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The effects of scale on granular mixing in a double screw pyrolyzer

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The effects of scale on granular mixing in a double screw pyrolyzer

by

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A thesis submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of
MASTER OF SCIENCE

Major: Mechanical Engineering

Program of Study Committee:
Theodore J. Heindel, Major Professor
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2015

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ABSTRACT

Granular mixing processes are important to many industries including the pharmaceutical, agricultural, and biotechnology industries. These processes often require both a high degree of homogeneity and a high degree of customizability. As granular mixing processes are so widely employed, a thorough understanding of the mixing dynamics is necessary to understand and control the resulting products. Research into granular mixing processes has been, thus far, largely focused on laboratory scale mixers with simple geometries, while actual industrial processes often require large mixers with complex geometries. Moreover, granular mixing processes are often very sensitive to changes in operating conditions and any solutions provided to deal with specific mixing problems are highly system-sensitive and do not readily carry over to other mixer types. These sensitivities mean it is necessary to study more complicated mixer geometries, more complicated operating condition, and industry scale mixers in order to apply experimental and theoretical knowledge of granular mixing to industrial processes.

One specific example of a complicated industrial mixer is a double screw pyrolyzer used in the bioenergy industry to produce bio-oil via fast pyrolysis. Bio-oil can be converted into synthetic gasoline, diesel, and other transportation fuels, or can be converted into bio-based chemicals for a wide range of purposes. Double screw pyrolyzers utilize a granular mixing process by mechanically conveying and mixing a biomass and heat carrier media together using two intermeshing screws. Fast pyrolysis is still a relatively new technology and much of the research that has been done with double screw pyrolyzers has focused on the products and not on the mixing dynamics within the mixer. However, understanding the
mixing dynamics is important because bio-oil yields depend on the heat transfer between the heat carrier and the biomass. Improving the mixing effectiveness within the pyrolyzer will improve the heat transfer rates, and thus, will improve the bio-oil yields.

The purpose of this study is to expand upon previous work done with double screw mixers, designed to geometrically replicate double screw pyrolyzers, by investigating the effects of various operating conditions and by developing, characterizing, and optimizing larger double screw mixers. Select operating conditions featuring changes in screw rotation speed, dimensionless screw pitch, and screw rotation orientation, previously investigated by Kingston and Heindel (2014b, c), were repeated while varying additional operating conditions such as material injection configuration, mass flow rate ratio, particle size and density, and mixer scale. Insights gained though this study aim to improve the viability of double screw pyrolyzers by increasing the mixing effectiveness through adjustments to operating conditions, and by bridging the gap between laboratory scale mixers and industry scale mixers.
CHAPTER 1. INTRODUCTION

1.1 Motivation

Granular mixing processes have applications in a wide range of industries including the pharmaceutical, energy, agricultural, and biotechnology industries (Bridgwater, 2012; Campbell, 2006; Mohan et al., 2006). As these processes often require both a high degree of homogeneity and a high degree of customizability (Ottino and Khakhar, 2001), a thorough understanding of the mixing dynamics is necessary to understand and control the resulting products (Paul et al., 2004).

Granular mixing has been widely studied in the past few decades, but most work thus far has focused on mixers with relatively simple geometries. While these studies have done much to improve the theoretical understanding of granular flows, these theoretical understandings are often difficult to apply to broader industrial uses. Moreover, granular mixing processes are often very sensitive to changes in operating conditions and any solutions provided to deal with specific mixing problems are highly system-sensitive. These sensitivities mean it is necessary to study more complicated mixer geometries and more complicated operating conditions in the hopes of applying knowledge of granular mixing to industrial processes (Hogg, 2009).

One example of a complicated industrial mixer is a double screw pyrolyzer used in the bioenergy industry to produce bio-oil via fast pyrolysis (Ingram et al., 2007; Mohan et al., 2006). In a double screw pyrolyzer, a biomass material is mixed with a heat carrier media at very high temperatures to produce vapors that are collected and condensed into bio-oil which then has the potential to be converted and upgraded into transport fuels (Bridgwater, 2003; Brown, 2011a). Fast pyrolysis is still, however, a relatively new technology (Bridgwater et
al., 1999) and much of the research that has been done with double screw pyrolyzers has focused on the products (Bahng et al., 2009; Brown, 2009; Ingram et al., 2007) and not on the mixing dynamics of the mixer. Recent work by Kingston and Heindel (2014b, c) has investigated the mixing dynamics within a double screw pyrolyzer to further understand both the mixing dynamics themselves, and the effects of operating conditions on the mixing effectiveness within the double screw mixer. As the mixing effectiveness of the two materials in a double screw pyrolyzer is directly correlated to the bio-oil yield—the better the mixing, the higher the yields—the insights gained through the study of the granular mixing process will hopefully improve the viability of double screw pyrolyzers and the production of bio-oil.

1.2 Goal and Objectives

The overall goal of this project is to assess scale effects on the mixing effectiveness of high density glass beads and low density biomass in a cold-flow double screw pyrolyzer. This goal will be realized through the following specific objectives:

Objective 1: Determine the effect of premixing the heat carrier and biomass using video and composition analysis in a D = 2.54 cm (1 in) double screw mixer, designed to geometrically replicate a double screw pyrolyzer.

Objective 2: Determine the effect of particle size, density, and concentration using video and composition analysis in a D = 2.54 cm (1 in) double screw mixer.

Objective 3: Determine the effect of mixer scale using video and composition analysis in a D = 3.81 cm (1.5 in) and a D = 5.08 cm (2 in) double screw mixer.
1.3 Practical Application

This study aims to provide an increased understanding of the effect of operating conditions on mixing effectiveness within a double screw mixer, and to begin to bridge the gap between laboratory scale mixers and industrial scale mixers. By relating conclusions from the double screw mixers to double screw pyrolyzers, recommendations can be made on how to best modify existing double screw pyrolyzers and how to design future pilot scale reactors to improve the viability of double screw pyrolyzers and bio-oil production. Furthermore, this project offers deeper insights into granular mixing processes in more complicated mixers that could be valuable to many industries beyond just the biochemical conversion industry.

1.4 Outline

This thesis is divided into six chapters which provide a complete description of this project. Chapter 2 provides a literature review of the main topics associated with this project. Chapter 3 describes all of the equipment, granular materials, and methods used. A reprint of a journal paper in preparation for submission to Powder Technology entitled “Effect of Particle Size, Density, and Concentration on Mixing in a Double Screw Pyrolyzer” is presented in Chapter 4. Chapter 5 also presents a reprint of a journal paper in preparation for submission to Powder Technology, entitled “Effect of Mixer Scale on Mixing in a Double Screw Pyrolyzer”. Chapter 6 summarizes the project conclusions and provides recommendations for future work. Comprehensive appendices are attached which include some additional results, engineering drawings, snapshots of the dynamic mixing videos for every operating
condition tested, the composition correlations for each material combination, and the mass flow rate ratios used for each operating condition.
CHAPTER 2. LITERATURE REVIEW

This chapter provides an overview of the background knowledge necessary to this project. The first section covers the fundamentals of granular mixing, while the second section covers a specific application of granular mixing that provides the motivation for this project.

2.1 Granular Mixing

2.1.1 Introduction

Granular materials, defined as those composed of individual granules, are found in everyday life, from sand and soil, to cereal and dog food. Granular materials are also important in many industrial applications, such as the pharmaceutical, mining, agricultural and energy industries, and in these cases, it is the flow and mixing of granular materials that is so important. For example, in the production of pharmaceuticals, it is vitally important that the mixing processes of the various chemicals and compounds be highly controlled and that the final compositions of the mixture be well known to ensure the safety and efficacy of the products. Unfortunately, granular flows are incredibly complex and not very well understood, leading to problems in the handling and processing of granular materials. Research interest into granular flows has grown in recent decades, but large holes in our fundamental knowledge of the mechanics and mixing mechanisms of granular flows remain. Additionally, much of the research that has been done so far has focused on simple granular flows and mixers with simple geometries, and not on the more complex flows and processes that are seen in actual industrial applications. Much more research is needed to help bridge
the gap between fundamental understanding and industrial practice. This chapter provides an introduction to granular mixing, the equipment involved, some industrial applications, and outlines the problems currently faced in the field.

2.1.2 History

Granular materials and mixing processes have been a vital part of daily human life for thousands of years, in a wide variety of forms from food stuffs to metal ores. However, unlike liquid flows and liquid mixing, granular mixing processes have not been well studied until quite recently (Jain et al., 2005). Bridgwater (2010) terms the period prior to the 1950’s as the age of intuition and mechanical design because until that point granular mixing processes and equipment were designed based on judgement and empiricism rather than on scientific justifications (Crowe, 1998; Seville, 1997). Few scientific studies on granular mixing existed before this period.

The period of 1950-1990 saw an increase in studies investigating granular flows, beginning with Lacey’s (1954) review on the theory of particle mixing in 1954. However, in this period, called the age of process design science by Bridgwater (2010), little fundamental progress was made. Experimental techniques, as well as theoretical understanding, remained poor and difficult to apply. Still, some quality books (Bourne, 1964) and reviews (Bridgwater, 1976; Hersey, 1975) did arise from the period.

The last decade of the 20th century and the beginnings of the 21st century have seen a dramatic increase in granular mixing research, both experimental and theoretical. Experimental methods and techniques have gained precision and scientific backing, instead of the purely intuitively derived methods used in previous decades. Advances in computer technologies have allowed for more complex simulations and modeling. Experimental
research has looked at a wide variety of mixers, including: chutes (Hajra et al., 2012; Holyoake and McElwaine, 2012; Wiederseiner et al., 2011), bladed mixers (Conway et al., 2005; Remy et al., 2010), vertical shakers (Hsiau and Chen, 2002; Yang, 2006), single screw mixers (Roberts, 1999; Tsai and Lin, 1994; Uchida and Okamoto, 2006, 2008), twin screw mixers (Amalia Kartika et al., 2006; Brown and Brown, 2012; Dhenge et al., 2013; Kingston and Heindel, 2014b) and rotating cylinders (Aït Aissa et al., 2010; Alexander et al., 2004b; Alexander et al., 2004c; Ingram et al., 2005). Many recent efforts have focused on simulations of granular mixers, often paired with supporting experimental evidence (Chand et al., 2012; Cleary and Sinnott, 2008; Gui et al., 2013; Huang and Kuo, 2012; Sarkar and Wassgren, 2009; Yang, 2006; Yijie et al., 2013). Several literature reviews also arose during this time period (Bridgwater, 2012; Bridgwater, 2010; Campbell, 1990; Campbell, 2006; Ottino and Khakhar, 2000, 2001). Though great advances came in this period, severe limitations still exist. The types of mixers most often investigated featured simple geometries with limited variables and unrealistic operating conditions. The simulations performed were largely of purely academic interest and consisted of overly simplistic models that limit their real world applicability (Campbell, 2006). Because real world applications of granular mixing require complicated mixers with complex operating conditions, much more research into granular mixing is needed.

2.1.3 Rheology

One factor that makes granular mixing processes so difficult to understand is their complex rheology. Granular materials have complex physical characteristics that do not completely fit into the behavior of any one phase. Depending on how the internal forces are transmitted, granular materials can pack and hold a shape like a solid, yet flow and take the
Gravity can encourage the packing and stabilization of granular materials, but at the same time granular flows are often gravity induced. While this dual nature of granular materials has been known since the time of Lucretius (ca 98-55 B.C.), it is a complicated phenomenon that remains poorly understood to this day (Campbell, 2006).

### 2.1.4 Classification

A flow can manifest in a myriad of ways. It can be single phase, consisting of only one phase of matter—solid, liquid, or gas—or it can be multiphase with two or more phases. Multiphase flows can be further broken up into four categories, outlined in Table 2.1. Any flow, whether single phase or multiphase, can also be single component or multicomponent, where a component is a chemical species such as nitrogen, or water (Crowe, 1998). Table 2.2 provides single component and multicomponent examples of both single and multiphase flows. The granular flows studied in this project are all multicomponent, multiphase flows.

<table>
<thead>
<tr>
<th>Gas-liquid flow</th>
<th>Bubbly flows, Separated flows, Gas-droplet flows</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas-solid flow</td>
<td>Gas-particle flows, Pneumatic transport, Fluidized beds</td>
</tr>
<tr>
<td>Liquid-solid flow</td>
<td>Slurry flows, Hydro-transport, Sediment transport</td>
</tr>
<tr>
<td>Gas-liquid-solid flow</td>
<td>Bubbles in a slurry flow, Droplets/particles in gaseous flows</td>
</tr>
</tbody>
</table>
Table 2.2: Examples of single and multiphase flows with single and multicomponent parts (from Crowe (1998)).

<table>
<thead>
<tr>
<th></th>
<th>Single Component</th>
<th>Multicomponent</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Single phase</strong></td>
<td>Water flow, Nitrogen Flow</td>
<td>Air Flow, Flow of Emulsions</td>
</tr>
<tr>
<td><strong>Multiphase</strong></td>
<td>Stream-water flow, Freon-Freon vapor flow</td>
<td>Air-water flow, Slurry flow</td>
</tr>
</tbody>
</table>

Granular flows can also be either a dispersed or a dense granular flow. In a dispersed flow, one phase of the flow is dispersed through another continuous phase. In the case of granular flows, the solid phase is always the dispersed phase, with the continuous phase being either gas or liquid. In a dispersed flow, the primary forces acting on the solid particles are fluid forces such as drag and lift (Crowe, 1998). As the solid particle concentration increases in a multiphase flow, the solid particles can begin to agglomerate and form plugs. At high solid phase flow rates, the flow is described as a dense granular flow. In dense granular flows, the kinetic energy of the particles is dissipated primarily through particle-particle collisions and particle-wall collisions. Thus, dense granular flows are dominated by collision and contact forces, as shown in Figure 2.1 (Campbell, 2006). The granular flows studied in this project are all contact dominated, dense granular flows.

Granular flows can also be classified by the speed of the flow—sludge flows or mudslides are very slow, while chute flows or avalanches are very fast. In slow granular flows, the particles are assumed to move collectively, while in a fast granular flow the particles move individually (Campbell, 2006). “Rapid granular flow” is the most commonly used term to describe fast granular flows and is short for “rapidly sheared granular flows”, originally termed by Campbell (Mehta, 2007). Rapid-Flow models have recently tried to explain the behavior of rapid granular flows by borrowing models from the kinetic theory of gases. These models treat the particles in a granular flow like molecules, assuming
instantaneous collisions and granular temperature, a quantity analogous to kinetic temperature (Campbell, 2006). However, even the best Rapid-Flow models fail to predict some of the behaviors of rapid granular flows, and further, they predict flow behavior contrary to the solid-like nature of granular flows. Variations on the Rapid-Flow model exist, such as the Elastic-Inertial model, but still, no current model fully explains the complex behavior and flow patterns of granular flows (Campbell, 2006). All the flows studied in this project are rapid granular flows.

![Dilute Flow and Dense Flow Diagram](image)

**Figure 2.1:** Dilute and dense granular flows (adapted from Crowe et al. (1998)).

### 2.1.5 Cohesive Forces

Dense granular flows, like the ones studied in this project, are dominated by inter-particle and particle-wall forces, also known as surface forces or cohesive forces. The most prominent cohesive forces in a granular flow include electrostatic forces, van der Waals forces, liquid-bridge forces, and steric forces, while gravitational attraction between particles play a minor role (Aït Aissa et al., 2010; Bridgwater, 2012). Any researcher investigating
granular mixing needs to have a thorough understanding of the cohesive forces at play within a granular flow in order to maximize or minimize their effects as needed to ensure the desired mixing, or other processes, is achieved. The dominance of the various cohesive forces at play in a granular flow changes with varying particle, mixer, and environmental properties. Particle size plays a huge part in determining the magnitude of cohesive forces. Smaller particles exhibit greater susceptibility to electrostatic and van der Waals forces, however, if liquid is present, the liquid-bridge force tends to dominate the cohesive forces. Particle surface roughness, wettability, container wall friction, environmental humidity, and temperature all play a role in determining the magnitude and behavior of the cohesive forces, which in turn all play a role in the efficiency of a mixing process (Seville, 1997; Uchida and Okamoto, 2006). Each of the major cohesive forces will be discussed further below with the exception of steric forces (forces due to adsorbed long chain molecules) and gravitational forces, which play only minor roles in this project.

2.1.5.1 Electrostatic forces

During the handling and processing of granular materials, electrostatic forces arise from friction between the particles themselves and between the particles and the container walls in which they are confined (Bridgwater, 2012). Electrostatic forces can also arise from charged particles in a gas component of the flow acting on the other particles in the flow (Crowe, 1998). The electrostatic attractive force $F_e$ is given by Coulomb’s Law:

$$F_e = \frac{q_1q_2}{r^2} \cdot \frac{1}{4\pi \varepsilon_0} \quad (2.1)$$

where $q_1$ is one charged particle, $q_2$ is the other charged particle, $r$ is the distance between the two particles, and $\varepsilon_0$ is the permittivity of free space, a constant. As the electrostatic attractive
force is proportional to the inverse square of the distance between the two particles, \( F \) drops off quickly as the distance between particles increases. However, as the size of the particle decreases, the effects of the electrostatic force on particles tends to increase significantly. This is because the mass of a particle decreases at a greater rate than the surface area (where the charge is distributed), resulting in a higher charge-to-mass ratio, giving \( F \) a larger influence. The combined effects of electrostatics on many particles can cause plugs and clogs within a granular flow.

2.1.5.2 Van der Waals forces

When solid surfaces interact, whether it is two solid particles colliding, or a solid particle colliding with the solid wall of its container, van der Waals forces act to adhere the two particles together. The van der Waals force is an attractive force between solid surfaces due to molecular interactions (Crowe, 1998). This adhesive force only dominates for very small particles however. When a large particle collides with another solid surface, its inertial forces cause it to rebound. While it loses some kinetic energy in the process, the initial inertial energy is great enough to overcome the molecular forces acting to adhere the two surfaces. When a particle has a very low mass and collides with another solid surface, however, the particle’s low inertial forces are not strong enough to overcome the molecular van der Waals forces and the particle adheres to the solid surface (Crowe, 1998; Mausda, 2006). The van der Waals force, \( F \), between two spheres is given by:

\[
F = \frac{Ad}{12(z+b)^2} \quad (2.2)
\]

\[
d = \left( \frac{D_1D_2}{D_1+D_2} \right) \quad (2.3)
\]
\[ b = \frac{(b_1 + b_2)}{2} \]  

(2.4)

where \( A \) is the Hamaker constant, generally very small for air, \( D_1 \) is the diameter of one sphere, \( D_2 \) is the diameter of the other sphere, \( z \) is the separation between spheres, and \( b_1 \) and \( b_2 \) are the roughness of the first and second sphere respectively. The van der Waals force, \( F \), between a sphere and a plane is given by:

\[ F = \frac{A d_p}{6z^2} \]  

(2.5)

where \( A \) is the Hamaker constant, \( d_p \) is the diameter of the sphere, and \( z \) is the distance between the sphere and the plane. While the electrostatic force, described in Section 2.1.5.1, also increases with decreasing particle size, the van der Waals force is typically the dominant force of the two for small particles (Aït Aissa et al., 2010).

2.1.5.3 Liquid bridge forces

When conditions are right, such as when humidity levels in the interstitial gas of a granular flow are high enough (>65%), molecules of mobile liquid can adhere to the surface of individual solid particles in a granular flow. At points of contact between solid surfaces, the presence of this liquid acts to form liquid bridges between solid particles, adhering them together (Seville, 1997). These bridges form between particles due to the curvature of the liquid-gas interface caused by the surface tension of the water (Crowe, 1998). The formation of these bridges depends not only on the environmental humidity, but also on the particle shape and surface wettability (Uchida and Okamoto, 2006). Figure 2.2 depicts a liquid bridge between two spherical particles.
Figure 2.2: Example of liquid bridge formation between two spherical particles (adapted from Seville et al. (1997)).

2.1.6 Mixing mechanisms

Many industrial processes involving granular materials involve mixing. These processes often desire an end product with specific properties. While the quality of the specified end product can be measured by any number of properties, from temperature to chemical reactivity, the most common mixing measure used to define the quality of the end product is composition, also known as concentration (Paul et al., 2004).

A mixture can be either perfect or random. In a perfect mixture, the particles in the mixture are alternated, one after the other, along a lattice (Paul et al., 2004). In a perfect mixture, the probability of finding a particle in a random sample of the mixture is identical to the probability of finding the particle in any other random sample of the mixture, or in the mixture as a whole. Figure 2.3a shows an example of a perfect mixture. A perfect mixture is never found in reality, unless created painstakingly, by placing each individual particle, one at a time (Rhodes and Rhodes, 2008).

In reality, the best a mixing process can aim for is a random mixture. In a random mixture, the probability of finding a certain type of particle is statistically independent of the
nature of its neighbors. In other words, the probability of finding a particle of any component is the same at all locations and equal to the proportion of that component in the mixture as a whole (Paul et al., 2004; Rhodes and Rhodes, 2008). Figure 2.3b shows an example of a random mixture. While a random mixture is theoretically achievable, in reality real mixtures tend to show some degree of heterogeneity due to overmixing, incomplete mixing, agglomerations, and/or segregation.

The “opposite” of a mixed system is a de-mixed system, one that is fully segregated by particle. Segregation can be due to any number of particle or mixer properties and is common in real world mixing processes. Figure 2.3c shows a fully segregated system in which the two unique particles are completely divided out of the other.

![Figure 2.3: Example of a a) perfect mixture, b) random mixture, and c) a de-mixed, or segregated mixture (adapted from Pernenkil et al. (2006)).](image)

2.1.6.1 Diffusive mixing

Diffusion is the completely random change of place of individual particles and, in fluids, is a process that has a governing equation (Weinekötter, 2000). However, in granular flows, this is not a physical process and, therefore, does not have a governing equation (Bridgwater, 2012). In granular flows, it is not a concentration gradient that causes diffusion, but mechanical agitation. The individual motion of the particles must be induced by an external energy source, such as from shaking, tumbling, or vibrating the mixture, and will not
occur spontaneously (Ortega-Rivas, 2012). While pure diffusion is essential for microscopic homogenization, and is highly effective when feasible, diffusion is a very slow process that is often of small influence when compared to the macroscopic mixing mechanisms of convection and shear forces (Mausda, 2006).

2.1.6.2 Shear mixing

In granular flows, velocity gradients give rise to narrow slip zones, in which particles are interchanged within layers in the zone (Bridgwater, 2012). These velocity gradients arise around the physical mixing device, be it the mixer wall, screw, or impeller, and is directly proportional to the surface contact area (Mausda, 2006). Shear mixing is sometimes considered to be related to convection.

2.1.6.3 Convective mixing

Convection is another mixing mechanism that arises from the action of the mixer (Bridgwater, 2012). Convection, caused by the agitation, pumping, vibrating, etc. of the mixer, is the transfer of masses or groups of particles from one location in the mixture to another. Convection improves the spatial homogeneity of the mixture (Lacey, 1954). This promotes diffusion, by breaking the mixture down into smaller chunks that diffusion can then act upon more readily (Weinekötter, 2000). Pure convection is a very rapid process, but less effective than diffusion, as it tends to leave more heterogeneity on the microscopic scale. A mixing process that combines the mechanisms of diffusion and convection results in a higher quality mixture than if only one mechanism was employed, because it is able to take advantage of both the speed of convection and the effectiveness of diffusion (Ortega-Rivas, 2012).
2.1.7 Segregation

While a fully segregated system, as shown in Figure 2.3c, is rarely seen in a real mixture, segregation of some scale and intensity commonly occurs in real mixtures. Even a mixture that has achieved a fully random state will likely segregate upon further handling, such as emptying out of the mixer, transport, or further processing (Rhodes and Rhodes, 2008). A fully segregated mixture has a high scale of segregation; we see only two homogeneous regions of particles adjacent to each other. Figure 2.4 illustrates how as the scale of segregation decreases, more clumps of homogeneous particles become adjacent to each other in smaller and smaller sized regions. As the intensity of segregation of a mixture decreases, the spread of the concentration of a component decreases (Pernenkil and Cooney, 2006). In order to determine the effectiveness of a mixing process, it is important to assess both the scale and intensity of segregation of the resulting mixture, as well as to understand the mechanisms that cause segregation to occur in the first place. Tang and Puri (2004) and McCarthy (2009) provide reviews of segregation and methods to minimize its effects, both in the mixers themselves, and in the handling of the resulting mixture.
The mixing dynamics of a granular flow can vary considerably based on particle characteristics (Campbell, 2006; Ottino and Khakhar, 2000), and on the mixing mechanisms used in the mixing process. Many studies have investigated the tendency for granular flows to segregate by varying particle characteristics, such as size (Alexander et al., 2004a; Arntz et al., 2014; Hogg, 2009; Jain et al., 2005), shape (Hilton and Cleary, 2011; Rao et al., 2011), and density (Hsiau and Chen, 2002; Nielsen et al., 2011; Yang, 2006). In addition to particle size, shape, and density, other particle characteristics like resilience, angle of repose, and cohesiveness have been shown to also affect the mixing dynamics and cause mixture segregation (Fayed, 1997; Paul et al., 2004). While all particle characteristics affect the mixing dynamics, particle size plays the biggest role in causing mixture segregation (Rhodes and Rhodes, 2008). Almost all studies found in the literature indicate that an increase in
particle size variation within a mixture increases the mixture segregation. However, a few studies have found that an increase in particle size ratio actually decreased mixture segregation (Arntz et al., 2014; Jain et al., 2005). Generally, density plays a relatively minor role in inducing particle segregation. However, in the case of gas fluidization, particle density can play the dominant role in causing segregation due to buoyancy forces (Rhodes and Rhodes, 2008). In a mixture with monosized particles of different densities, the particles having higher densities tend to settle to the bottom of the mixture, with the less dense particles rising to the top of the mixture (Tang and Puri, 2004). Particle shape is generally agreed to have the least effect on segregation. Five segregation mechanisms—segregation by agglomeration, by vibration, by percolation, trajectory segregation and elutriation segregation—are shown in Figure 2.5 and will be discussed below.

2.1.7.1 Segregation by agglomeration

The cohesive forces discussed in Section 2.1.5, electrostatic, van der Waals, and liquid-bridge forces, can sometimes lead to unwanted segregation of the particles within a mixture. Agglomerates of particles composed of the same species can form when the inter-particle forces are stronger than the shear forces within a mixture. Greater shear forces, from impellers, knives, blades, etc., are needed to break up these agglomerates (Tang and Puri, 2004). While typically negative, agglomeration can sometimes be positive and actually reduce mixture segregation. In the case of a very fine powder that is being mixed with larger particles, the fine powder can initially coat the larger particles, forming a mixture that is stabilized by van der Waals forces. This particle coating will, in turn, limit future segregation (Weinekötter, 2000). Hu et al. (2012) provides a brief review of granular agglomeration studies and anti-agglomeration methods.
Figure 2.5: Examples of various types of segregation mechanism (adapted from Weinekötter et al. (2000)).
2.1.7.2 Segregation by percolation

Differences in particle size leads to vertical segregation due to percolation. If a mixture of differently sized particles is disturbed in some way, whether from an outside force or via internal agitators, a rearrangement of the particles occurs in which the fine particles tend to percolate through the voids between the larger particles (Ortega-Rivas, 2012). This leads to a settling of the finer particles on the bottom of the mixture and a significant division between the differently sized particles. This phenomenon can occur even with closely sized particles; any size differences, even ones quite small, lead to segregation by percolation (Mausda, 2006). Percolation is the most important segregation mechanism (Weinekötter, 2000).

2.1.7.3 Segregation by vibration

Segregation by vibration is an offshoot of segregation by percolation, leading to vertical segregation of a bed of particles. When vertical vibration is induced in a bed of larger and small sized particles, the larger particles rise to the top of a bed, a phenomena termed the “Brazil Nut Effect” (Rhodes and Rhodes, 2008). When the entire bed is pushed upwards, the smaller sized particles readily fall into the gaps left under the larger particles, preventing the larger particles from falling back to their original position. The summation of many of these motion sequences results in the larger particles reaching the surface of the bed. Furthermore, because the larger particles are unlikely to be able to penetrate the small spaces between the smaller sized particles, it is unlikely that the large particles will ever move down into the bed again (Weinekötter, 2000). This phenomenon occurs even when the larger particles also have a greater density (Ortega-Rivas, 2012). Many studies have investigated vibration induced segregation (Hsiau and Chen, 2002; Metzger et al., 2011; Yang, 2006).
2.1.7.4 Trajectory segregation

Trajectory segregation results from a flow falling with a horizontal velocity (Weinekötter, 2000). As particles fall through a static gas atmosphere, differences in size, density, and air drag result in some particles traveling farther distances than others. This results in heaps that exhibit spatial segregation. The particle diameter once again plays the biggest role in determining the degree of segregation, with larger particles often traveling up to 4 times farther than particles half their diameter (Fayed, 1997).

2.1.7.5 Elutriation segregation

Elutriation segregation is closely related to trajectory segregation. In elutriation segregation, an upwards velocity of gas passing through a mixture results in vertical segregation of the mixture. If the upward velocity of the gas exceeds the terminal freefall velocity of the fine particles, these particles will rise with the air flow, while larger, and/or denser, particles will fall. If the mixture is falling through a rising gas stream, the fine particles may remain in suspension long after the large particles have settled at the bottom (Rhodes and Rhodes, 2008).

2.1.8 Mixing quantification

Being able to characterize the final end product from a mixing process is extremely important. In the pharmaceutical, it is vitally important that the composition of each pill or tablet is of a known and specified composition. In the agricultural industry, it is important that the final bags of fertilizer produced all have a uniform component makeup. However, in reality, in any real mixing application, even in a small scale laboratory mixer, testing the placement of every particle in a mixture, to assess composition and homogeneity, is
impossible. Instead, various sampling and visualization methods are employed to study smaller components of the whole mixture. Statistical analysis is then used to extrapolate the samples’ composition to the whole population, i.e., the mixture.

2.1.8.1 Statistical analysis

In order to assess the efficiency of a mixing process, a criterion for mixing needs to be established by which to judge the whole mixture. The most common way to judge mixedness is by composition, or, in other words, by testing the homogeneity of the mixture. A number of samples are taken from the mixture and the statistical parameter variance is typically used to then evaluate the sample compositions. If the mixture is not perfectly homogeneous, the composition will vary among the selected samples. The variance conveys the magnitude of these fluctuations. The smaller the fluctuations in composition from sample to sample, the higher quality the mixture, and the smaller the variance will be. A perfectly homogeneous mixture would have a variance of zero (Weinekötter, 2000). The variance is defined by

$$s^2 = \frac{\sum_{i=1}^{N} (x_i - \bar{x})^2}{N-1} \quad (2.6)$$

where $x_i$ is the composition of the $i^{th}$ sample, $\bar{x}$ is the mean composition of all the samples, and $N$ is the number of samples.

Often, a weighted composition variance is a better parameter to use, as it accounts for the variability in sample mass and therefore more accurately characterizes mixing. The weighted composition variance is defined as

$$s_w^2 = \frac{\sum_{i=1}^{n} m_i (x_i - \bar{x})^2}{(\sum_{i=1}^{N-1} m_i)\sum_{i=1}^{n} m_i} \quad (2.7)$$
where \( m_i \) is the mass of the \( i^{th} \) samples, \( x_i \) is the composition of the \( i^{th} \) sample, \( n \) is the number of the \( i^{th} \) sample, \( \bar{x}_w \) is the mass weighted composition mean of the samples, and \( N \) is the total number of samples being analyzed.

Another statistical parameter commonly used to evaluate mixing effectiveness is the coefficient of variation (CV), sometimes referred to as the relative standard deviation (RSD). The coefficient of variation normalizes the results by the initial mixture composition. This is useful when mixtures of widely differing component percentages are to be compared. Because the magnitude of the variance varies widely based on the percentage of each component in the mixture, the variances from different mixtures cannot be directly compared. The coefficients of variation, however, can be compared, as they are normalized by the initial component percentages (Paul et al., 2004). The coefficient of variation is defined as

\[
CV = \frac{s_w}{M}
\]

(2.8)

where \( s \) is the standard deviation, and \( M \) is the mean composition of the mixture if it were a perfect mixture.

2.1.8.2 Granular sampling

To determine variances and coefficients of variation, samples first need to be collected. It is very important to first figure out what the proper qualities of the sample(s) need to be. How many samples need to be drawn? From where should they be taken? How large should each sample be? How often do samples need to be collected? All of these questions, and many more, affect the ability of the statistical results to accurately portray the quality of the mixture as a whole.
Generally speaking, the larger the number of samples collected, the more reliable the results. Upwards of 50 samples could sometimes be needed for truly representative results, however, in practice this is often not economical or practical. It is more common for around 15 samples to be collected, although, even this can vary widely depending on the type of mixing process being investigated and the final purpose of the product (Ortega-Rivas, 2012).

Allen (1997) proposed two golden rules of sampling to ensure proper sample collection techniques:

1. Always collect the sample from a moving stream
2. The whole of the stream should be collected for many short periods of time instead of just part of the stream for a long period of time

Collecting a sample from only the surface of the mixture could potentially introduce errors from vertical segregation.

Some sample methods involve letting the mixture fall into a heap on a flat surface, smoothing the heap out flat and then collecting a portion of the flattened mixture. However, this method is particularly prone to selective sampling, due to trajectory segregation and vertical segregation (Allen, 1997). Many sampling methods involve scooping a portion of the sample from the internal structure of the mixture. These scoops are often called sampling thieves. The two most common sampling thieves are a side-sampling thief and an end-sampling thief. Both thieves rely on a tube inserted into the mixture that is then filled, either by particles falling into the side, as with a side-sampling thief, or by particles being pushed into the end, as with an end-sampling thief. While commonly used, these types of sampling scoops are not ideal because they depend on free-flowing particles to collect samples. Particles that are smaller and more free-flowing tend to fill the probes more readily than
larger, more cohesive particles. The probes are also invasive and disturb the mixture as they are being inserted. Both of these problems introduce potentially significant errors into the results (Allen, 1997; Bridgwater, 2012; Paul et al., 2004). Muzzio et al. (2003; 1997) studied the effects of many different, commonly used sampling probes to evaluate their efficacy in sample collection and determined that sampling probes impart a large degree of error due to their invasive nature. Researchers continue to look for new sampling probes that will enable sample collection without imparting errors on the results (Susana et al., 2011).

Because of the invasive nature of sampling probes, it is best to sample the granular mixture when it is in the form of a free flowing stream. This way, segregation effects are minimized, and no settling of the mixture is allowed to happen. Furthermore, by following Allen’s second golden rule of sampling, and collecting the whole outlet stream instead of only a portion, no effects of trajectory segregation or spatial segregation can skew the results.

2.1.8.3 Non-invasive characterization

There are also many advanced, non-invasive measurement techniques available to study granular flows and mixtures. Digital image analysis allows the surface of the flow to be analyzed without disturbing the flow itself (Aït Aissa et al., 2010; Busciglio et al., 2009; Chen and Yu, 2004; Daumann et al., 2009; Daumann and Nirschl, 2008). The use of clear casings further allow the sides and bottoms of the mixing region to be viewed (Kingston and Heindel, 2014c). However, due to the opaque nature of most granular materials, optical visualization of granular mixtures is limited. To study the internal structure of granular flows, newly developed methods such as particle image velocimetry (de Jong et al., 2012; Dhenge et al., 2013), magnetic resonance imaging (Hardy et al., 2007), positron emission particle tracking (Leadbeater et al., 2012; Portillo et al., 2009), X-ray radiography (Uchida and
Okamoto, 2006, 2008), X-ray stereography (Kingston et al., 2015; Kingston et al., 2014), and near-infrared spectroscopy (Koller et al., 2011) have been developed. Each of these techniques allows the internal structure and movements of the granular materials to be better understood and are invaluable to increasing the fundamental understanding of granular mechanics. However, each of these methods is highly expensive, typically require large facilities, and in some cases, can only handle mixers with simple geometries, limiting the feasibility of these measurement techniques.

2.1.9 Granular mixing equipment

For hundreds of years, the design of mixing equipment of any sort was based off of empiricism, experience, and judgement. Because of this, problems resulting from poorly designed mixers—poor quality of product, overmixed product (extraneous effort), and segregated end mixture—were common (Bridgwater, 2012). The quality of the final product is highly dependent on the efficiency of the mixing processes and equipment used. Three questions are often used when analyzing a mixing process (Weinekötter, 2000): 1) How good is the mixing? 2) How quickly is the final mixture achieved? 3) How high is the required energy input? While the answers to these questions do depend, in part, on the granular materials being mixed and the processes and equipment involved with the material handling before and after mixing, the mixing equipment itself plays one of the biggest roles in any effective mixing process.

2.1.9.1 Design considerations

When designing a mixer, there are many important things to consider. The size and shape of the mixer needs to be carefully considered so that the mixer will fit in its desired
location. The necessary supporting equipment, such as material feeders, chutes, and catch bins also need to fit. Future repositioning or transport of the mixer needs to be considered as well. The material the mixer will be made out of is especially important, especially if volatile or corrosive materials will be used.

It is also important to assure that all necessary requirements of the mixing process will be achieved. If there is heating or cooling of the mixture needed, how will this be accomplished? Will liquid need to be added before or during mixing? Does the mixing process need to be carried out under pressure or in a vacuum? Is the mixing process batch or continuous?

The shape, size, and nature of the particles is also important to consider when designing a piece of mixing equipment. Understanding how the granular materials will move through the mixer and what the dominant mixing mechanisms will be is important so that efficiency can be maximized and unwanted segregation, clogging and agglomeration can be avoided. Potential particle damage during mixing and potential mixer damage due to abrasive particles needs to be considered as well.

An engineer needs to consider more than the just mixing processes within the mixer itself. Mixing equipment often needs routine cleaning, and that process is much easier in certain types of mixers than in others. Ease of loading and discharging are important considerations. Can the mixer be hooked up to the next stage in the product’s processing sequence to avoid unnecessary handling? How will segregation upon discharge be limited? Power draw is also an important consideration, although there are unfortunately usually other concerns that are more pressing in design consideration, which often overrule the desire to reduce power consumption. A design engineer must take into account all of these questions
and concerns, and many more, when designing or modifying a piece of mixing equipment (Bridgwater, 2012).

2.1.9.2 Batch vs continuous mixing

There are two main ways a mixing process can be designed, either in a batch process or a continuous process. In a batch mixing process, the materials are loaded into the mixer all at once. The materials are mixed, and then emptied from the mixer after a set amount of time. The steps are completed separately, with the next step beginning only after the previous one has been completely finished (Weinekötter, 2000). Batch processes are the most common mixing process. Batch process mixers have the advantage of being easy to fill, as the materials can easily be weighed and added one at a time, and can handle small or large quantities of material at a time. Batch mixers, however, are susceptible to segregation and overmixing, are incompatible with cohesive materials, and result in the materials having to be emptied and transported to the next stage in the product’s processing sequence, resulting in increased processing times and additional chances for segregation (Pernenkil and Cooney, 2006).

In a continuous mixing process, the ingredients are continuously feed into the mixer. This results in the feeding, mixing, and discharge steps occurring continuously and simultaneously (Weinekötter, 2000). Continuous mixers are often more compact than batch mixers, because they can process new material continuously. They have the advantage of connecting directly to the next stage in the processing sequence, eliminating the need for mixture discharge and transport handling. Continuous mixers have less segregation than batch mixers and can handle both free-flowing and cohesive materials. However, continuous mixers have some significant disadvantages as well. They require a more complicated
material injection sequence, as the materials need to be continuously metered into the mixer simultaneously, instead of weighed and added separately as in batch mixing. This adds expense to the mixer and is often less accurate than batch weighing. Continuous mixers also have some loss of product during start up and shut down of the mixer, reducing the overall efficiency.

Continuous mixers have not been as well studied in the literature as batch systems. Pernenil and Cooney (2006) provide a review of continuous mixing, which not only covers the current state of the topic thoroughly, but also provides references to many additional reviews and studies in both batch and continuous mixing. Bridgwater’s (2012) review on the mixing of powders and granular materials also provides a good overview on continuous mixing.

2.1.9.3 Rotating shell mixers vs. stationary shell mixers

There are countless different mixer designs available, all with distinct advantages and disadvantages. Figure 2.6 shows various common mixer types, including (a) horizontal cylinder, (b) v-type, (c) double cone, (d) ribbon, (e) screw-in-cone, (f) high speed, (g) rotating disk, (h) fluidized bed, (i) motionless, and (j) vibrational. Mixers can be classified into two classes; Class I mixers with rotating shells, and Class II mixers with stationary shells (Pernenkil and Cooney, 2006).

Class I mixers feature closed compartments that rotate about an axis. These mixers are generally only suitable for free flow materials and the fill level of the compartments needs to be carefully controlled to prevent bulk motion of the internal granular flow. Class I mixers often feature cylindrical, cone or v-shaped containers. Asymmetry can help reduce the tendency towards bulk flow by increasing irregular flow patterns. Class I mixers are easy to
clean, but generally are only suitable for batch mixing (Bridgwater, 2012). The main mixing mechanism in rotating shell mixers is diffusion, meaning they generally require long mixing times, but result in high quality mixtures (Rhodes and Rhodes, 2008).

Figure 2.6: Examples of (a) horizontal cylinder (b) v-type (c) double cone (d) ribbon (e) screw-in-cone (f) high speed (g) rotating disk (h) fluidized bed (i) motionless (j) vibrational mixers (from Mausda et al. (2006)).
Class II mixers feature a stationary outer shell with some sort of internal mechanical agitator. These mixers are generally able to accommodate both free flowing and cohesive materials and can operate as both batch and continuous mixers. Class II mixers use paddles, blades, knives, and screws, among others, to achieve mixing (Bridgwater, 2012). Because of the internal agitation, convection cells form within the mixture, resulting in a fast mixing process with good macro homogeneity, but often with poor microscopic homogeneity. With their often complicated internal structure, Class II mixers can be difficult to clean (Rhodes and Rhodes, 2008).

Many studies have been done on both Class I and Class II mixers, both experimental and computational, as well as on many other types of mixers that may not fall directly into the Class I or Class II categories, such as motionless gravity mixers, or fluidized beds. Bridgwater (2012) provides a thorough review of various types of mixers as well as a brief summary of some of the current work. Khakhar (2011) and Cleary and Sinnott (2008) provide many examples of various modeling studies done with many different mixer types. However, even with the surge of studies in granular mixing in the past few decades, most of the work done has featured mixers with very simple geometries. While these studies have done much to improve the theoretical understanding of granular flows, these purely theoretical principles are often difficult to apply to broader industrial uses. Moreover, granular mixing processes are often very sensitive to changes in operating conditions and any specific solutions provided to deal with mixing problems are highly system-sensitive. These sensitivities mean it is necessary to study more complicated mixer geometries and more complicated operating condition interactions if knowledge of granular mixing is to be applied to industrial processes (Hogg, 2009).
This project investigates a complex double screw mixer under a wide variety of operating conditions and with a variety of particle characteristics, which more realistically represent actual industrial processes (see Section 2.2). Because the focus of this project is on double screw mixers, additional consideration will be given here to screw mixers.

2.1.9.3.1 Screw mixers

Archemedian screws have been employed since antiquity to transport liquids (Colijn, 1985). In modern times, screws have been adapted to transport granular materials as well. These devices are often referred to as screw conveyors and are very useful for the transport, feeding and discharging of free-flowing materials. Screw conveyors push the materials along while shearing the material in the radial direction, causing the material to tumble upon itself as it is conveyed (Colijn, 1985). This phenomenon is utilized in screw extruders, also referred to as screw mixers, to achieve a mixing of the materials inside.

Mixers employing the use of screws are most often Class II mixers with a stationary shell featuring one or more enclosed rotating screws that provide agitation and induce mixing. They can be designed for batch or continuous mixing and can feature the use of a single screw, twin screws, or sometimes ever three or more screws. Furthermore, the screws themselves can be mounted horizontally, at an incline, vertically, or be designed to sweep along the side of a mixer wall. Further increasing the incredible variability in screw mixers, there is a wide variety of screw flightings, screw rotation orientations, and screw speeds currently used in industry and research today.

2.1.9.3.1.1 Screw flighting geometry

A screw is typically composed of a continuous helix wrapped around a central shaft. Depending on how the helicoid, also called the screw flights or flightings, is wrapped around
the shaft, a screw is either left-hand threaded or right-hand threaded. A left-hand threaded screw, as viewed from the end, features a helicoid that wraps around the screw in a counterclockwise direction. A right-hand threaded screw features a clockwise wrapping of the helicoid (Colijn, 1985).

Some screws feature flights with a thicker inner edge and a thinner outer edge, while others feature flights of uniform thickness. Furthermore, the flightings themselves can have unique geometries and additional features. Notches can be cut out of the body of the flights. Paddles can be added to the screw, either attached to the flights at angles, or attached directly to the shaft itself, in between the flightings. These paddles can greatly improve the agitation within the material flow and counteract the bulk rotational flow of material, thus improving mixing (Colijn, 1985). Some examples of unique screw flightings are shown in Figure 2.7.

The spacing between the flightings themselves can be further altered to achieve different mixing mechanisms. A typical screw features a pitch equal to the screw diameter, where the pitch is defined as the distance from one flight to another (Martelli, 1983). The spacing between the flights can be increased for very free-flowing materials and liquids, or spaced closely, closer than 1 screw diameter, to improve the accuracy of conveying and feeding, especially for cohesive materials. However, this close spacing may limit mixing. Double flights, featuring two independent helicoids, can also be used to improve conveying accuracy. Tapered flights and stepped diameter flights are commonly used in feeders to ensure uniform material draw. Figure 2.8 shows many different screw flight arrangements (Colijn, 1985).
2.1.9.3.1.2 Screw rotation orientation

In screw mixers featuring more than one screw, the screws can either rotate together or in opposite directions. Two (or more) screws rotating in the same direction, either all clockwise, or all counterclockwise, are referred to as co-rotating. Screws that rotate opposite each other, such as in a two screw system where one screw rotates clockwise and the other rotates counterclockwise, are called counter-rotating. When two side-by-side screws, as
viewed from the end, feature the left screw rotating clockwise, and the right screw rotating counterclockwise, the system is referred to as having a counter-rotating down pumping screw rotation orientation. When the left screw has a counterclockwise rotation and the right a clockwise rotation, the system has a counter-rotating up pumping screw rotation orientation (Martelli, 1983). Figure 2.9 illustrates the three screw rotation orientations for a twin screw set-up.

Figure 2.8: Examples of various screw pitches (from Colijn (1985)).
2.1.9.3.1.3 Screw rotation speed

The rate at which the screw (or screws) rotates can vary widely and is one of the main determining factor, for the material residence time. Generally, as the screw rotation speed decreases, the material residence time increases. However, due to varying mixing and segregation mechanisms, it cannot be assumed that each component in a mixture has the same residence time (Bridgwater, 2012).

2.1.9.3.2 Single screw mixers

The first patent for a single screw extruder was filed in 1871. By the end of the 19th century, mixers and conveyors using single screws were commonplace in industrial and agricultural practices (White, 2010). Today, single screw devices are still commonly used.

Typically, single screw extruders maintain a pitch equal to the screw diameter (Martelli, 1983). Mixing paddles added to a single screw mixer can greatly improve mixing.
effectiveness. Various researchers have investigated the effect of changing operating conditions with single screw mixers (Roberts, 1999; Tsai and Lin, 1994) and Metcalf (1965) provides an overview of the mechanics of a screw feeder. While single screw mixers do achieve mixing, the primary mechanism driving mixing is friction, between the walls of the mixer, the screw flightings, and the material itself. Smooth surfaces, or smooth particles, can severely limit the mixing potential of a single screw mixer, as the material will tend to spin as a block within the barrel of the mixer (Martelli, 1983).

2.1.9.3.3 Double screw mixers

Twin screw mixing devices have been commercially available since the early 20th century, with only a co-rotating screw rotation orientation available at first. Counter-rotating options followed later, but to this day, the co-rotating orientation still remains the industry standard (Martelli, 1983). A twin screw extruder has various screw positioning options. If the two screws are positioned so that the flightings of one screw do not mesh or engage the flightings of the other screw, the screws are considered to be non-intermeshing. Non-intermeshing screws mixers essentially operate largely as two independent single screw mixers, and so have limited use as actual mixers (White, 2010).

Intermeshing screws, however, are positioned more closely together so that their flightings mesh and engage one another. Intermeshing screws encourage material mixing instead of simple conveyance, because one screw’s flights interfere with the material movement within the other screw’s flights. This acts to limit the rotational motion of the material around each screw, encouraging mixing. Furthermore, the flightings of the two screws wipe the spaces between the other screw’s flightings as they rotate, resulting in a cleaning of material accumulations or plugs that may form on an individual screw flight. An
An illustration of non-intermeshing and intermeshing screw is shown in Figure 2.10. Many studies, especially in recent decades, have focused on twin screw mixers (Brown and Brown, 2012; Dhenge et al., 2013; Kingston and Heindel, 2014a; Kingston and Heindel, 2014b, c), although more work still needs to be done on more complicated operating conditions, and on larger scale mixers.

Figure 2.10: Example of non-intermeshing and intermeshing screw placement for a twin screw system.

2.1.9.4 Mixer scale

Mixer size is a very important parameter that determines not only the quantity, but in many cases, the quality of the resulting product. In an industrial application, the end goal is most often a high quality product with economic viability (Mort, 2005). A small mixer that produces an excellent product may not have a high enough economy of production to validate its use. Scaling up from a laboratory sized mixer to one more suited to industrial outputs improves the economic viability of a mixing process, but it must be ensured that the larger mixer still produces a high quality product. A common axiom is to “commit your blunders on a small scale, and make your profits on a large scale” (Paul et al., 2004). It is much more cost effective to perform mixing studies on laboratory sized equipment, use
models or simulations to test larger industrial scale mixers, and then jump straight to pilot scale productions, but there is risk associated with not physically testing a larger scale mixer before production (Mort, 2005).

Complicating mixer scale-up, there are currently no clear scale-up laws concerning granular materials. For any given mixer type, we do not have sound scale-up laws, and only rudimentary assumptions of how granular mixers perform at different scales. There isn’t even a clear understanding of the important scaling parameters. The principle of similarity is a commonly used approach to modeling scale-ups, which states that

“the material flow patterns in two scales of mixing apparatuses are expected to be similar when the dimensions, velocity fields, and force distributions of corresponding points are proportional to each other” (Yijie et al., 2013).

However, the main similarity criterion required for proportionality seems to vary by mixer. While the principle of similarity is assumed to be valid, there is currently no theoretical validation for the principle. Furthermore, conventional scale-ups typically fail because the internal behavior of the mixture does not scale with the equipment size alone; particle properties and their proportions need to be considered in conjunction with equipment proportions (Bridgwater, 2012). Scale-up efforts need to consider how the macro-scale changes (such as mixer size) affect the micro-scale particle attributes (Mort, 2009).

Various researchers have studied scale-up laws using models and simulations for granular mixers (Mort, 2005, 2009; Rahmanian et al., 2008; Yijie et al., 2013) and Bridgwater (2012) and Paul (2004) provide good reviews of some general scaling rules for specific mixer geometries. However, as with all research concerning granular flows, studies to date have tended to only look at mixers with simple geometries and idealistic particle
properties. It is precisely the complicated mixer geometries and the non-uniform particle properties of real industrial processes that make scale-up to industrial mixers so uncertain. Furthermore, there is a fundamental lack in experimental validation to the models and theoretical rules concerning the scale-up of mixing equipment. This is due largely in part to increased costs associated with performing experimental studies on larger mixers. However, because it is nearly impossible to formulate generalized scale-up laws for granular mixers, extensive experimental studies are needed to provide usable criteria to aid in future scale-ups for industrial scale productions (Paul et al., 2004).

2.2 Biomass Thermochemical Conversion

The bioenergy industry uses a process known as fast pyrolysis to produce bio-oil (Ingram et al., 2007). Fast pyrolysis is the thermochemical conversion of biomass into bio-oil in the absence of oxygen (Mohan et al., 2006). In a double screw pyrolyzer, a biomass material is mixed with a heat carrier media at high temperatures to produce vapors, that are collected and condensed into bio-oil, and a solid that is collected as biochar (Bridgwater et al., 1999). Virtually any type of biomass can be pyrolyzed to produce bio-oil and biochar, although the biomass particles do generally need to be of a fine particle diameter, requiring processing of the biomass down to the necessary size. Various considerations, such as biomass growth times, ease of planting and harvesting, and media influences on bio-oil yields and composition can influence the availability and profitability of using different biomass (Mohan et al., 2006). Sand is the most common heat carrier media used in industrial pyrolysis applications (Brown, 2011a).

Bio-oil has the potential to be converted and upgraded into transport fuels, such as synthetic gasoline or diesel, to be used as a source of fuel via direct combustion, or to be
converted into a wide variety of biobased chemicals (Bridgwater, 2003; Brown, 2011a). Pyrolysis also produces ash and biochar along with the bio-oil vapors. This biochar can be burned for energy or used as a soil additive to greatly increase crop health and yields (Yoder et al., 2011).

Fast pyrolysis is still a relatively new technology (Bridgwater, 1999) and much of the research that has been done with double screw pyrolyzers has focused on the products (Bahng et al., 2009; Brown, 2009; Ingram et al., 2007) and not on the mixing dynamics of the mixer. As pyrolysis occurs due to the heat transfer from the heat carrier to the biomass material, the mixing between the two materials is vitally important. If the mixing between the two materials can be improved, the heat transfer rates will improve and the bio-oil yields will potentially increase (Mohan et al., 2006). Recent work by Kingston and Heindel (2015; 2014b, c) has begun to investigate the mixing dynamics within a double screw pyrolyzer in order to further understand both the mixing dynamics themselves, and the effects of operating conditions on the mixing effectiveness within the double screw mixer. Kingston and Heindel (2015; 2014b, c) found that higher screw rotation speeds, larger dimensionless screw pitches, and, most importantly, a counter-rotating down pumping screw rotation orientation improved the mixing effectiveness of a double screw pyrolyzer. Furthermore, they concluded that the industry standard screw rotation orientation, a co-rotating orientation, is a relatively poor mixing condition. More work investigating the mixing dynamics within a double screw pyrolyzer still needs to be done, looking both into the effects of various particle properties, as well as the effect of the mixer scale on the mixing dynamics.
2.3 Summary

Granular mixing processes are incredibly important to a wide range of industries. Unfortunately, granular materials exhibit a complex rheology that makes understanding their behavior difficult. Cohesive, diffusive, and shear forces dominate as the mixing mechanisms in most granular flows, but segregation mechanisms caused by differences in particle shapes, sizes and densities, often act to “de-mix” the flows.

There are dozens of different mixer designs available, all with their own advantages and disadvantages, but unfortunately, little research has been done into the mixing dynamics within actual industrial mixers. Most research so far has focused on laboratory scale mixers with simple geometries and uniform particles. More research needs to be done into larger scale, more complex mixers with more realistic non-uniform particles, to be applicable to real industrial applications.

One such industrial application of granular flows is found in the biomass thermochemical conversion industry. Bio-oil is produced from biomass via fast pyrolysis, a granular mixing process involving the mixing of a heat carrier media and a biomass material. Bio-oil yields are dependent on the heat transfer rates between the two materials; if the mixing effectiveness of the system can be improved, the heat transfer rates will increase and bio-oil yield may increase. This project aims to investigate the granular mixing dynamics within a double screw mixer, designed to geometrically replicate double screw pyrolyzers, in order to better understand how particle properties and mixer scale affect the mixing effectiveness. It is the hope that this project will help to improve our fundamental understanding of granular flows while also helping to improve the viability of bio-oil production.
CHAPTER 3. EQUIPMENT, MATERIALS, AND METHODS

This chapter outlines the three screw mixers, granular materials, material processing equipment, video and composition analysis equipment, and experimental methods used in this project. Detailed engineering drawings for all three screw mixers are provided in Appendix B.

3.1 Screw Mixers

3.1.1 Double screw pyrolyzer

The double screw mixers used in this study are designed to geometrically replicate double screw pyrolyzers used by some in the bioenergy industry to produce bio-oil (see section 2.2). The double screw pyrolyzer used by Iowa State University’s Biorenewables Research Laboratory, shown in Figure 3.1, features two horizontally mounted, intermeshing noncontact 2.54 cm (1 in) outside diameter screws, with a screw pitch of \( p/D = 3.175 \text{ cm} \) (1.25 in), co-rotating inside a stationary housing. The housing features an omega-shaped \((\omega)\) bottom plate profile which eliminates any dead space between the screw flights. The top of the housing is a flat, removable plate. There is a 0.159 cm (0.0625 in) clearance gap between the screw flights and the bottom of the housing. Measured from the centerline of the second injection port to the beginning of the outlet port, the effective mixing length is 25.4 cm (10 in), yielding a dimensionless mixing length of \( L/D = 10 \). Brown (2009) provides a detailed description of this double screw pyrolyzer.
Figure 3.1: The double screw pyrolyzer used at Iowa State University’s Biorenewables Research Laboratory (adapted from Brown (2009)).

3.1.2 Double screw mixer: baseline geometry (1x)

The clear, cold-flow laboratory scale double screw pyrolyzer used in these studies, shown in Figure 3.2, was originally designed by Kingston (2013). Hereafter, double screw mixer or just screw mixer will be used in place of its formal name. The double screw mixer was designed to geometrically replicate the double screw pyrolyzer used at Iowa State
University’s Biorenewables Research Laboratory, so that the results of this project may be applicable to the modification and future design of double screw pyrolyzers.

![Diagram of double screw pyrolyzer](image)

Figure 3.2: The cold flow, laboratory scale double screw pyrolyzer used in these studies. All parts shown are 3D printed.

The double screw mixer features two parallel, intermeshing noncontact screws with outside screw diameters of $D = 2.54$ cm (1 in). The housing features an overlapping “O” profile ($\infty$) eliminating any potential for dead space both above and below the screw flights. The clearance between the screws and the housing wall is $0.159$ cm (0.0625 in). The effective mixing length, measured from the center of the second inlet port to the beginning of the outlet ports measures 24.5 cm (10 in), giving the mixer a dimensionless mixing length of $L/D = 10$.

The biomass and heat carrier media are injected vertically into separate injection ports located two characteristic lengths apart, on top of the screw mixer. The granular materials exit from the bottom of the screw mixer under the force of gravity. The outlet port features a unique design that allows the exit stream to be divided into equal sections by four dividers.
(see Figure 3.3). These outlet ports allow the outlet flow of the pyrolyzer to be divided and sampled for non-invasive composition analysis.

![Cross-sectional view of the screw mixer housing’s outlet ports that allow the outlet stream to be divided and sampled for composition analysis.](image)

**Figure 3.3:** Cross-sectional view of the screw mixer housing’s outlet ports that allow the outlet stream to be divided and sampled for composition analysis.

A total of six screw designs are used in this project. Three different screw pitches are represented, $p = 1.901, 3.175, \text{ and } 4.445 \text{ cm (0.75, 1.25, 1.75 in)},$ yielding dimensionless screw pitches of $p/D = 0.75, 1.25, 1.75$ respectively. For a detailed explanation of why these screw pitches were initially chosen, see Kingston (Kingston, 2013). Both left-hand threaded and right-hand threaded screws were designed for each screw pitch. The ends of the screws fit into the screw gears and are tapered to a square shaft in order to transmit the torque from the screw gear to the screws. The six screw designs used in this study are shown in Figure 3.4.

Two supports fit to the end of the screw mixer housing to: (1) hold the screws in place, (2) support the screws’ radial and axial thrust loads, and (3) provide means for the screw mixer to be mounted in place. The screws fit into low friction sleeve bushings, placed...
within the supports, to prevent undue screw wear and are sealed by u-cup seals to prevent the granular material from escaping the housing enclosure.

![Diagram showing six unique screw designs](image)

Figure 3.4: The six unique screw designs used in this project (adapted from Kingston (2013)).

The spur gears used to turn the screws are designed specifically for this project. One spur gear attaches to the gearmotor, and two additional gears attach to the screws. In the case of a co-rotating screw rotation orientation, the screw gears are sized so as to be noncontact gears with the motor gear positioned between the two screw gears. In the case of a counter-rotating screw rotation, the screw gears are intermeshing, and only one screw gear is in contact with the motor gear. As the gearmotor has a maximum 35 rpm output, the gears were designed with a a 2:1 motor to screw gear ratio, to ensure the required max screw rotation speed of $\omega = 60$ rpm is achieved.

True double screw pyrolyzers must be manufactured out of a material that can withstand the $400 – 500 \degree C$ operating temperatures of fast pyrolysis (Mohan et al., 2006), and so are often made of steel. However, this project requires an optically transparent housing in order to perform $360^\circ$ optical visualization analysis of the internal flow dynamics. Furthermore, as all tests in this project are performed at room temperature, no extreme heat
or corrosion resistance is required of the screw mixer materials. Therefore, to ensure optical transparency and ease of manufacturing, the entire screw mixer, from the housing to the screws and gears, were 3D printed by Paradigm Development Group, Inc, in Elk Grove Village, Illinois. The main housing of the screw mixer was printed out of a clear, rigid, designer plastic, known as VeroClear, which required a post-process polishing step to produce the optically accessible housing. The support structures for the housing, the screws, and the screw and motor gears were printed from a grey designer plastic known as VeroGrey.

3.1.2.1 Characteristic Length

The characteristic length of a system is an important parameter that defines the scale of the system and provides a standard when normalizing the system. It is often difficult to define the characteristic length of a system as, in some cases, the characteristic length can be influenced by the operating conditions of the system and not just simply by the geometry.

In the case of the screw mixer, the screw flighting diameter is defined as the characteristic length of the system, \( D = 2.54 \text{ cm} \) (1 in). This definition is only dependent on the mixer geometry and not on operating parameters, thus providing a clear and straightforward definition.

3.1.3 Double screw mixer scale-up: 1.5x

Laboratory scale mixers, such as the screw mixer previously described, offer an economical way to study granular mixing processes due to their small size, and relative low cost of manufacturing and operation. However, a small mixer that produces a product of excellent quality may not produce at a rate that makes it economically viable. This is why larger mixers are used in industry, so that the products can be produced in quantities that
increase the economic viability of the mixing process (Mort, 2005). Unfortunately, because of the complicated nature of granular flows, there are currently no sound scaling laws for granular mixing equipment, and very few assumptions can be made about how granular flows change with changing mixer and particle scales. The quality of the product from a small scale, laboratory mixer may change, and even deteriorate, when the same process is used with a larger scale, mixer of the same geometry. Furthermore, because of the increased cost of larger scale mixers, very few studies have looked at how granular mixing changes with changes in mixer and particle scale. Extensive experimental studies are needed to provide usable criteria to aid in future scale-ups to industrial scale productions (Paul et al., 2004).

In order to study how mixer scale affects the mixing dynamics within a double screw pyrolyzer, two additional double screw mixers were designed, nearly geometrically identical to the original 1x scale double screw mixer described in Section 3.1.2. Detailed engineering drawings for the 1.5x scale double screw mixer are provided in Appendix B.2.

The characteristic length of the screw mixer, defined as the outside diameter of the screw flighting (D = 2.54 cm for the 1x scale mixer), was chosen as the scaling parameter. Therefore, the 1.5x scale mixer features an outside screw diameter of D = 3.81 cm (1.5 in). This parameter was then used to scale up all other components of the double screw mixer. The design of the screws was otherwise the same as that of the 1x scale screws.

It was desired that the larger screw mixers maintain a dimensionless mixing length of L/D = 10, and so, with the characteristic length defined as D = 3.81 cm (1.5 in), the mixing length of the 1.5x scale mixer is L = 38.1 cm (15 in). The 1.5x scale housing maintains the same design as the 1x scale housing, with an overlapping “O” (∞) profile, two inlet ports
located axially along the top of the housing, and four outlet ports on the bottom of the housing.

The three dimensionless screw pitches investigated with the 1x scale mixer, \( p/D = 0.75, 1.25, \) and 1.75, were maintained for the 1.5x scale mixer, yielding screw pitches of: \( p = 2.86, 4.77, \) and 6.67 cm (1.13, 1.88, and 2.62 in) for the three respective dimensionless screw pitches. Both left hand threaded and right hand threaded screws were designed for each dimensionless screw pitch. The clearance between the screw flights and the screw mixer housing was allowed to scale to 1.5x times that of the 1x scale mixer. Both the screw gears and the motor gears were scaled to maintain their 2:1 ratio, in order to ensure the three screw rotation speeds, \( \omega = 20, 40, \) and 60 rpm, were maintained.

The entire 1.5x screw mixer was 3D printed to ensure ease of manufacturing and optical transparency. The housing, supports, and gears were printed by 3D Systems of Rock Hill, South Carolina. The housing was printed from a clear designer plastic, Accura ClearVue, and the supports and gears were printed from a grey designer plastic, Accura Xtreme. The screws were printed by RedEye, Inc. of Minneapolis, Minnesota, out of white polycarbonate plastic.

### 3.1.4 Double screw mixer scale-up: 2x

A 2x scale double screw mixer was also designed, in order to more thoroughly investigate the effect of mixer scale on the granular mixing dynamics. Detailed engineering drawings for all parts associated with the 2x scale mixer are provided in Appendix B.3.

As with the 1.5x scale mixer, the characteristic length was chosen as the scaling parameter. The 2x scale mixer features an outside screw diameter of \( D = 5.08 \) cm (2 in). The 2x scale housing maintains the same design as the 1x scale housing, with an omega-shaped
top and bottom profile, two inlet ports located axially along the top of the housing, and four outlet ports on the bottom of the housing. To maintain the dimensionless mixing length of \( L/D = 10 \), the 2x scale mixer features a mixing length of \( L = 50.8 \text{ cm} \) (20 in).

To maintain the dimensionless screw pitches investigated with the 1x scale mixer, the 2x scale screws feature screw pitches of \( p = 3.81, 6.35, \) and \( 8.89 \text{ cm} \) (1.5, 2.5, 3.5 in), which yield the dimensionless screw pitches of \( p/D = 0.75, 1.25, \) and 1.75, respectively. The design of the screws was otherwise the same as that of the 1x scale screws. Both left hand threaded and right hand threaded screws were designed for each dimensionless screw pitch. The clearance between the screw flights and the screw mixer housing was allowed to scale to 2x times that of the 1x scale mixer. Both the screw gears and the motor gears were scaled to maintain their 2:1 ratio, in order to ensure the three screw rotation speeds, \( \omega = 20, 40, \) and 60 rpm, were maintained.

As with both the 1x and 1.5x scale mixers, the entire 2x screw mixer was 3D printed to ensure ease of manufacturing and optical transparency. The housing, supports, and gears were printed by 3D Systems of Rock Hill, South Carolina. The housing was printed from a clear designer plastic, Accura ClearVue, and the supports and gears were printed from a grey designer plastic, Accura Xtreme. The screws were printed by RedEye, Inc. of Minneapolis, Minnesota, out of white polycarbonate plastic. The three double screw mixers are shown in Figure 3.5.
Figure 3.5: The three double screw mixers with a selection of screws from each scale, shown against a standard 12 inch ruler.

3.2 Granular Materials

When it comes to the type of biomass used for the production of bio-oil, virtually any type of biomass can be pyrolyzed. Various considerations, such as biomass growth times, ease of planting and harvesting, and media influences on the yields and composition of bio-oil, influence the availability and profitability of using different biomass (Mohan et al., 2006). In terms of heat carrier media, while sand is the most commonly used material in industrial pyrolysis applications, other heat carriers, such as steel shot, can be used (Brown, 2011a). Because particle characteristics have a significant effect on the mixing and segregation mechanisms within a granular flow, changing the biomass and heat carrier particles could have dramatic effect on the mixing dynamics within a double screw pyrolyzer. Understanding how the particle characteristics will affect the mixing dynamics, and subsequently, the bio-oil yields, should be another important consideration when choosing biomass and heat carrier media for fast pyrolysis.

In order to investigate how changing the biomass and/or heat carrier media effects the mixing dynamics within a double screw mixer, four different biomass media and two different heat carrier media, in various combinations, were investigated in this project. The
individual media will be described below, and their specific combinations are listed in Section 3.5.2.1.

3.2.1 Biomass materials

3.2.1.1 Large red oak (LRO), 500 μm – 6350 μm size range

Large red oak chips (LRO) were the main biomass material used in this project in a size range of 500 – 6350 μm. The red oak chips were received in a general size range from dust sized particles to ~5 cm (~2 in). They were then repeatedly run through a 6350 μm (0.25 in) sieve, to remove all the very large pieces, and then through a 500 μm (0.020 in) sieve, to remove small particles and dust. As the red oak chips are needle-shaped, any particle with a diameter less than 6350 μm could pass through the first sieve, even if it were much larger than 6350 μm in length. To eliminate such particles, a two-step manual screening process was employed. First, the red oak chips that passed through the 6350 μm sieve, were spread out into a thin layer and any very large pieces (either longer than ~6350 μm, or overly square and knot-like) were manually discarded. A 1400 μm (0.0555 in) sieve was then used to eliminate all knot-like particles that would potentially catch between the 1600 μm (1/16 inch) clearance between the screw flightings and mixer housing. By choosing a sieve that had mesh opening just slightly less than this 1600 μm size all particles that had a minimum dimension greater than 1600 μm were eliminated, thus eliminating most all of the potential for red oak chip jams and screw breaks in the screw mixer. Due to the needle-like nature of the red oak chips, reducing the sieve size to 1400 μm did not eliminate the longer sized (up to 6350 μm) pieces as they were able to pass through the sieve in a vertical orientation. Thus we were left with a collection of red oak chips with a theoretical size range from 500 – 6350 μm
and no pieces with a minimum dimension greater than 1600 μm (Figure 2.3). The average true density of the red oak chips was 1330 kg/m³ as measured by a pycnometer. The final processed 500 – 6350 μm red oak chips are shown in Figure 3.6.

![Figure 3.6: LRO, captured by a) Nikon D50 camera and a b) Olympus Infinity microscope under 4x magnification.](image)

3.2.1.2 Small red oak, 300 μm – 710 μm size range

Small red oak particles (SRO) were also investigated in a smaller 300 – 710 μm size range. The red oak was obtained from the Biorenewables Research Laboratory at Iowa State. The red oak was milled through a 0.32 cm (0.125 in) screen using a hammer mill to reduce the particle size to the desired range. This red oak had a true density, as measured by a pycnometer, of 142 kg/m³. The particles, shown in Figure 3.7, are still needle-like in shape, like their larger counterparts, but are much more uniform in shape and size.
3.2.1.3 Corn stover, 300 μm – 710 μm size range

Corn stover (CS) was also used in this project, in the 300 – 710 μm size range, shown in Figure 3.8. The corn stover was obtained from the BioCentury Research Farm at Iowa State University. The material was single pass corn stover and already processed to a 0.64 cm (0.25 in) size. To achieve the required 300 – 710 μm range, the corn stover was first run through a hammer mill with a screen opening of 710 μm to reduce the particles to the upper limit of the size range and then through a 300 μm sieve to eliminate all particles less than 300 μm. The corn stover had a true density of 1370 kg/m³ as measured by a pycnometer. The corn stover, though in the same size range as the 300 – 710 μm red oak, was more stringy and heterogeneous than the red oak. Although the two materials had similar true densities the corn stover was much more voluminous and needle-like than the 300 – 710 μm red oak (i.e. Figures 3.8 and 3.7, respectively).
3.2.1.4 Cork, 300 μm – 710 μm size range

Fast pyrolysis forms a solid ash/char along with the liquid bio-oil. The true density of the ash/char formed from fast pyrolysis has a true density of 600 kg/m³ and a size range of approximately 300 – 710 μm (Brewer et al., 2014). Because ash/char is black and dusty, it is not feasible to run through the screw mixer, as it would coat the mixer housing and make optical visualization impossible. Ground cork (CO) was identified as a moderate substitute to match the size range and true density of the ash/char, with the measured true density of the ground cork being 900 kg/m³ as measured by a pycnometer. The cork was purchased from Hobby Lobby in the form of craft cork board. It was then cut up, and run through a hammer mill with a screen opening of 710 μm, and then through a 300 μm sieve to eliminate the particles below the lower limit of the size range. Once ground, the cork was homogeneous with a particle shape more square than needle-like, shown in Figure 3.9. The ground cork is highly susceptible to electrostatic forces, a trait that made it very difficult to work with.
3.2.2 Heat Carrier Media

Sand is the most commonly used heat carrier media in fast pyrolysis. One specific type that is used in laboratory experiments, Quikrete commercial grade (fine No: 1961) sand, works well in a screw pyrolyzer made from stainless steel. However, the nature of the abrasive sand granules, would quickly scratch and damage the clear plastic screw mixer housing used in this project. This would quickly render optical visualization impossible. A suitable replacement was needed which would closely resemble the particle size and density characteristics of sand without causing damage to the screw mixer. The sand’s average true density was determined to be 2.68 g/cc as measured by a pycnometer.

3.2.2.1 Small glass beads, 300 μm – 500 μm size range

Small glass beads (SGB) in a 300 – 500 μm size range were chosen to simulate the traditional sand as the heat carrier media in this project. The spherical shape of the glass beads is well characterized and minimizes abrasive damage to the screw mixer. The glass beads were obtained from McMaster-Carr and have a true density of 2500 kg/m³ as measured
by a pycnometer (just 5.6 % less than the 2.68 g/cc sand) and are 95% spherical, as shown in Figure 3.10.

Figure 3.10: SGB, captured by a a) Nikon D50 camera and a b) Olympus Infinity microscope under 4x magnification.

3.2.2.2 Large glass beads, 800 μm – 1000 μm size range

Large glass beads (LGB), as heat carrier, were also investigated in a larger particle size. The glass beads were obtained from CeroGlass Industries in a specified size range of 595 – 2000 μm. However, once the materials were received and the size range was tested, it was determined that 95% of the glass beads fell within the 800 – 1000 μm range. The entire supply of glass beads was then repeatedly run through a 1000 μm sieve and a 800 μm sieve and the extraneous 5% discarded for a final size range of 800-1000 μm, shown in Figure 3.11. The glass beads were found to have a true density of 500 kg/m³ as measured by a pycnometer. These glass beads were only guaranteed 65% spherical, as opposed to the 95% spherical guarantee of the 300 – 500 μm glass beads, however these characteristics were acceptable because the beads were spherical enough as to not damage the clear screw mixer housing and the minor irregularities in particle shape were assumed to have little effect on flow conditions (See Section 2.1.7).
Figure 3.1: LGB, captured by a a) Nikon D50 camera and a b) Olympus Infinity microscope under 4x magnification.

3.3 Major Equipment

3.3.1 Volumetric auger feeders

Two stainless steel Tecweigh CR5 volumetric auger feeders were used to independently feed the biomass and heat carrier media into the screw mixers. A similar volumetric feeder as the two used in this work is shown in Figure 3.12. Each feeder features a 0.014 m³ (0.5 ft³) hopper and a 15.24 cm (6.0 in) long feed tube. The auger’s rotation speed was adjusted using a potentiometer which controlled a variable-speed 1/8 horsepower, 167 rpm maximum gearmotor. A Tecweigh 14A - 20M sprocket kit was swapped with the standard 16A - 16M sprocket kit to upgrade the drive system on the biomass volumetric feeder, and was used to increase the biomass volumetric output. The 14A - 20M sprocket kit features 14 teeth on the auger sprocket (i.e., 14A) and 20 teeth on the motor sprocket (i.e., 20M). The gearmotor to auger gear ratio was increased by using a larger motor sprocket and a smaller auger sprocket. A clear plastic lid was placed over the top of each feeder to ensure the granular materials were not contaminated.
Figure 3.12: A Techweigh CR5 volumetric auger feeder similar to the two used in these studies to feed the biomass and heat carrier media into the screw mixer.

3.3.1.1 1x scale mixer

When running the 1x scale screw mixer, the biomass feeder features a 2.54 cm (1 in) outside diameter feed tube with an 1.27 cm (0.5 in) open flight auger, designed for the largest of the LRO (6350 μm) particles. The same feed tube size was used for the heat carrier feeder, but with a larger open flight auger, 1.905 cm (0.75 in), designed to work for the SGB (300 – 500 μm) and LGB (800 – 1000 μm). Each feeder conveys their respective material horizontally along the feed tube into a clear polyvinyl chloride (PVC) 90° elbow, and then falls down a vertical semi-clear polyethylene (PE) tube into the screw mixer inlet ports. Clear PVC elbows were employed after a red oak chip jam in the biomass feeder feed tube resulted in a catastrophic feeder auger failure. With a clear PVC elbow, the material ejection from the feed tube can be continually monitored to minimize the possibility of a feeder jam going unnoticed and leading to complete auger failure.

3.3.1.2 1.5x and 2x scale mixers

To provide enough volumetric flow rate to fill the 1.5x and 2x scale mixers to the desired 65% fill level, the auger and feed tubes for both the biomass and the heat carrier feeders required upgrades. When running the 1.5x and the 2x scale mixers, the biomass
feeder features a 3.81 cm (1.5 in) outside diameter feed tube with a 2.54 cm (1 in) open flight auger. The heat carrier feeder features the same 3.81 cm (1.5 in) outside diameter feed tube, but with a larger 3.17 cm (1.25 in) open flight auger. Although larger PVC elbows and PE tubes were needed to convey the increased material volumes, the elbow and tube set-ups were the same as for the 1x scale mixer.

Unfortunately, even with the upgraded augers and feed tubes, the heat carrier feeder was unable to output the needed flow rate to reach a 65% fill of the 2x scale mixer for the operating condition featuring the highest screw rotation speeds paired with the largest dimensionless screw pitch. Because of this, this one particular condition is underfilled for all tests with the 2.0 scale reactor.

### 3.3.2 Gearmotor

The screws of all three screw mixer scales are driven by a Leeson 985-627H gearmotor mounted on a fully adjustable x-y-z axis plate. The gearmotor requires a 90 V direct current, and is powered by a Mastech HY10010EX digital DC power supply, to achieve a 35 rpm rotation speed.

An emergency shut-off switch is used to simultaneously deactivate both the volumetric feeders and the gearmotor. The power supply is allowed to remain energized, while the motor is not, using an alternating current control relay to break the circuit between the power supply and gearmotor. This allows both the feeders and the gearmotor to be simultaneously reactivated with no time delay, allowing the screw mixer to be started and stopped with no interruption to the steady state flow.
3.3.3 Pycnometer

The composition analysis used in this project requires the true density of a sample to be calculated. The true density of a material is different from the bulk density because, while the bulk density takes into count the void space in between individual particles, the true density of a material counts only the particles themselves and neglects the void spaces. Hence, the true density of a material is often much larger than the bulk density. While the bulk density of a material is generally easy to measure, the true density of a material requires special equipment that can measure, and then factor out, the void spaces when computing the density. This is accomplished, with a Pentapyc 5200e gas pycnometer from Quantachrome Instruments, shown in Figure 3.13.

Figure 3.13: A Quantachrome Instruments Pentapyc 5200e Gas Pycnometer used to calculate the true density of a sample.
The pycnometer features five sample chambers, used to simultaneously calculate the true density of five separate samples. The pycnometer first measures the true volume of a sample using Archimedes’ principle of fluid displacement and gas expansion, using helium gas as a displacing fluid. The ideal gas equation governs the process:

\[ PV = nRT \]  

where \( p \) is the pressure, \( v \) is the volume, \( n \) is the number of moles of gas, \( T \) is the temperature, and \( R \) is the molar gas constant. Helium is used as the displacing gas because it is inert and, due to its small atomic dimension, is able to penetrate even the finest pores.

The sample material is loaded into a sample chamber \((V_C)\), and the sample’s predetermined mass (as measured by a Cole-Parmer Symmetry PA-Analytical Balance) is manually input into the pycnometer. The sample chamber is then repeatedly flushed, and then filled with helium gas to a target pressure and sealed. The equilibrium pressure \((P_A)\) is measured, the sample chamber is opened into vent to another chamber of known volume \((V_A)\), and the new equilibrium pressure measured \((P_B)\). The change in pressure is related to the overall change in volume and can be used to calculate the true volume of the sample \((V_S)\) by:

\[ P_A(V_C - V_S) = P_B(V_C - V_S + V_A) \]

The accuracy of the pycnometer is \(<\pm 0.02\%\), and the repeatability is \(<\pm 0.01\%\).

### 3.3.4 Material separation

After mixing the biomass and heat carrier materials in the screw mixer, a separation process is needed to separate out each of the granular materials for reuse. The mixture, as received straight from the screw mixer, is first manually run through sieves of the relevant
sizes to separate the two materials. The size of these sieves vary according to the specific materials being run through the screw mixer at the time, and are chosen in order to separate out the non-overlapping size ranges (see Section 3.5.2.2, Table 3.2 for specific material combinations). For example, when the LRO (500 – 6350 µm) is mixed with the SGB (300 – 500 µm) a 500 µm sieve is used to separate out the majority of the biomass (some of the smallest, needle-like pieces of LRO still pass through the sieve in a vertical orientation, to be removed later). However, when the LRO is mixed with the LGB (800 – 1000 µm), a 1000 µm sieve is first used to remove the largest of the red oak particles, and then a 800 µm sieve is used to remove the smallest of the red oak. What remains is a mixture of all the glass beads with a portion of the red oak chips that pass through a 1000 µm sieve but not through an 800 µm sieve.

After one or more hand sieves are employed to separate out as much of the mixture as possible, the remaining mixture is placed inside a 15.24 cm (6 in) diameter cold-flow fluidized bed attached to a shaker, shown in Figure 3.14. The bed is lightly fluidized, at an air flow rate just above the minimum fluidization velocity needed to fluidize the granular material, and rocks back-and-forth to allow the lighter particles to percolate to the surface. The less dense biomass material accumulates on the top layer of the material where it is vacuumed off with a custom-made Venturi vacuum. With a few repeated runs, the heat carrier media regains almost 99.99% purity.
3.3.5 Optical visualization equipment

To capture the dynamic mixing process within the screw mixer, four Panasonic HC-V700M high definition cameras are used, following the procedures devolved by Kingston (2013). The cameras each have a $1920 \times 1080$ resolution and a frame rate of 60 FPS.

Combining the four independent video projections into one comprehensive dynamic mixing video requires the use of Adobe Premiere Pro CS6 video editing software. Premiere is a powerful video editing software, providing numerous options and a wide range of flexibility in editing and compiling videos. With the aid of Premiere, one dynamic mixing video, comprising 360º of the screw mixer’s granular flow, is composed.
3.4 Methods

3.4.1 Optical visualization

To capture the dynamic mixing process, the four Panasonic HC-V700M high definition cameras are positioned to the top, left, right and bottom of the screw mixer, in order to simultaneously capture four independent projections. The experimental setup is shown in Figure 3.15.

![Experimental setup used to capture qualitative optical visualization of the dynamic mixing process inside the screw mixer (adapted from Kingston (2013)).](image)

To eliminate glare on the curved reflective surface of the screw mixer, all metal components of the surrounding support structure and volumetric feeders are covered in black fabric. Furthermore, the entire system is enclosed in a black fabric canopy to eliminate ambient light from the room falling on the surface of the screw mixer. To provide lighting on the inside of the black fabric canopy, six 85 W compact florescent lamps (providing 5700
lumens each) are positioned, three on either end. Care is taken to ensure the lamps are positioned level with the screw mixer and that the light rays, reflected off a photography umbrella, are parallel to the surface of the screw mixer housing. This helps to further reduce glare off the curved surface of the screw mixer housing.

For the 1.5x and 2x scale mixers, the optical visualization setup is the same as described, but eight florescent lamps are used. Because the larger mixers feature longer mixing regions, the distance from the middle of the mixer to the lamps on either end is increased. The intensity of light falls off as the inverse square of the distance, and so, additional lamps are needed to compensate for the loss in light intensity due to distance.

For the 1x scale mixer the four cameras are each placed 25.4 cm (10 in) from the surface of the screw mixer. For the 1.5x and 2x mixers, this distance is increased, to 38.1 cm (15 in) and 50.8 cm (20 in), respectively.

In Adobe Premiere Pro CS6, each captured video is cropped so that only the defined mixing region of each projection is shown, and inverted (in the case of the left and bottom projections) so that the granular flow travels from left to right in all projections. An audio spike is used to temporally sync all four video projections. Figure 3.16 shows how each projection fits into the final dynamic mixing video. With the aid of Premiere, one dynamic mixing video, comprising 360° of the screw mixers granular flow, is composed, an example of a video snapshot is shown in Figure 3.17.
Figure 3.16: a) The original four projections of the screw mixer. b) The cropped and aligned images in the final dynamic mixing video (adapted from Kingston (2013)).

Figure 3.17: An example of a dynamic mixing video single frame, combining four independent projections into one 360° view of the screw mixer.

3.4.2 Composition analysis

Non-invasive composition analysis is made possible by the unique outlet port design of the screw mixer. The mixing effectiveness of the screw mixer is evaluated by determining
the degree of heterogeneity of the outlet flow. Samples from the four separate outlet ports are collected, and the mixture composition of each is determined. The individual outlet port mixture compositions are then compared to each other to determine the mixing effectiveness of the screw mixer.

3.4.2.1 Sample Collection

With the unique outlet port design of the screw mixers, no sampling probes are needed to collect samples from the granular flow. This is advantageous because sampling probes, though there are dozens of different designs available, all impart some degree of disturbance on the granular flow, inducing errors in the resulting analysis (Muzzio et al., 1997). Furthermore, according to Allen’s golden rules of sampling, it is best to sample a stream while it is in motion, and to sample the entirety of the stream, not just a portion (Allen, 1997). The outlet port design of the screw mixers used in this study allow samples to be collected while the mixer is running, with no disturbance to the internal structure of the flow, and allows the entire stream to be collected simultaneously, in the form of four separate samples.

To collect samples for each operating condition, the screw mixer is set up and allowed to reach steady state operating conditions. A quick shut-off switch is activated which stops the volumetric feeders and the screw motor simultaneously, freezing the screw mixer at steady state. Collection bags are placed over plastic tubes attached to each of the four outlet ports. The screw motor and volumetric feeders are then simultaneously reactivated and the system run for ~30 seconds, to ensure a representative sample is collected. The system is then stopped, and the bags removed and sealed. This process was repeated three times for each operating condition, to ensure repeatability.
3.4.2.2 Composition Analysis

Once the samples are collected, the composition of each sample, defined as the glass bead mass fraction, is determined in order to gauge the mixing effectiveness of the screw mixer. The glass bead mass fraction ranges from zero to one, with a mass fraction of one representing a mixture composed entirely of glass beads and a mass fraction of zero representing a mixture composed entirely of biomass material. To determine the glass bead mass fraction, an empirical correlation between mixture composition and mixture density was first determined. This was done by manually creating 11 mixtures featuring varying glass bead mass fractions, from a mixture composed entirely of biomass, to a mixture composed of 90% biomass, 10% glass beads (a glass bead mass fraction of 0.1), and so on, all the way to a mixture composed of entirely glass beads. These manually created mixtures were analyzed by a pycnometer to determine their true densities. The densities were then plotted against the glass bead mass fractions, shown in Figure 3.18, to reveal an empirically derived equation for converting from mixture density to mixture composition:

\[
\rho_{\text{mix}} = 915.86x^2 + 232.09x + 1358.9 \\
(3.3)
\]

where \( \rho_{\text{mix}} \) is the mixture density and \( x \) is the mixture composition in terms of glass bead mass fraction ranging from zero to one. The mixture density in nonlinear with respect to the mixture composition; the correlation corresponds to a harmonic mean and not an arithmetic mean. The theoretical harmonic mean equation consisting of a binary mixture is:

\[
\rho_{\text{mix}} = \left( \frac{x}{\rho_1} + \frac{1-x}{\rho_2} \right)^{-1} \\
(3.4)
\]

where \( \rho_{\text{mix}} \) is the mixture density, \( x \) is the mixture composition, and \( \rho_1 \) and \( \rho_2 \) are the individual material densities.
Figure 3.18: Empirical correlation used to relate the mixture density to the mixture composition for 500 – 6350 μm large red oak chips and 300 – 500 μm small glass beads.

The uncertainty in the mixture density is measured using a Taylor series expansion propagation of error (Devore, 2014; Ku, 1966) with the error in the mixture mass and volume combined using a root sum of squares (RSS). The uncertainty in the mixture true density is less than 1% with the correlation’s least squares regression equation having a coefficient of determination, $R^2$, greater than 0.999.

Each unique pairing of biomass and heat carrier media tested necessitated its own empirical composition correlation, as the mixture density of each unique combination has different densities depending on the true densities of the constituent particles. The empirical correlations for each material combination presented in this study are provided in Appendix E.
3.4.2.3 Statistical Analysis

The degree of homogeneity of the four samples collected from the outlet stream of the screw mixer indicates the mixing effectiveness of the screw mixer. The more homogeneous the samples are, the better the mixing within the screw mixer. Ideally, an even distribution of biomass and heat carrier would be found in all four samples, indicating a homogenous distribution of the two granular materials throughout the screw mixer. However, if the mixture is not perfectly homogeneous, the composition will vary among the selected samples. The variance conveys the magnitude of these fluctuations. The smaller the fluctuations in composition from sample to sample, the higher quality the mixture, and the smaller the variance will be. In this study, the mass weighted variance is used, to normalize against varying sample masses. A perfectly homogeneous mixture has a weighted variance of zero (Weinekötter, 2000). The mass weighted variance, $s_w^2$, is defined by

$$s_w^2 = \frac{\sum_{i=1}^{n} m_i (x_i - \bar{x}_w)^2}{(N-1) \sum_{i=1}^{n} m_i}$$

(3.5)

where $m_i$ is the mass of the $i^{th}$ sample, $x_i$ is the composition of the $i^{th}$ sample, $n$ is the number of the $i^{th}$ sample, $\bar{x}_w$ is the mass weighted composition mean of the samples, and $N$ is the total number of samples being analyzed.

The coefficient of variation, CV, is then calculated to normalize the results by the initial mixture composition. This is useful when mixtures of widely differing component percentages are compared. Because the magnitude of the variance varies based on the percentage of each component in the mixture, the variances from mixtures of different particle concentrations cannot be directly compared. The coefficients of variation, however,
can be compared, as they are normalized by the initial component percentages (Paul et al., 2004). The coefficient of variation is defined as

\[ CV = \frac{s_w}{M} \]  

(3.6)

where \( s_w \) is the standard deviation (square root of Equation (3.5)), and \( M \) is the mean composition of the mixture if it were a perfect mixture. A coefficient of variation of zero is the desired result because it corresponds to a perfectly homogeneous granular flow. A higher coefficient of variation indicates increased segregation of the granular materials and decreased mixing effectiveness. In this way, the coefficient of variation is used to evaluate the mixing effectiveness within the screw mixer.

### 3.4.2.4 1.5x and 2x scale modifications

The sample chamber of the pycnometer used to determine the true density of the samples has a fixed volume of 135 cm\(^3\). This volume perfectly matches the sample volumes collected from the 1x scale mixer. However, due to the increased volume of the 1.5x and 2x scale mixers, the sample volumes far exceeded the 135 cm\(^3\) pycnometer sample chamber. In order to calculate the true density of the samples collected from the two larger screw mixers, the collected samples have to first be carefully divided into subsamples of less than 135 cm\(^3\). For the 1.5x scale mixer, the number of subsamples range from two to five, while the 2x scale mixer requires from three to nine subsamples (the volume of the samples varies by operating condition—higher screw speeds yield samples of greater volume). The subsamples are then individually run through the pycnometer, and the collective masses and true densities summed together in Microsoft Excel. The sample true density is then calculated by:
\[
\rho = \frac{\sum_{i=1}^{n} m_i}{\sum_{i=1}^{n} V_i}
\]  

(3.7)

where \(\rho\) is the sample true density, \(m_i\) is the subsample mass, \(V_i\) is the subsample true volume, and \(n\) is the number of subsamples.

The mass weighted composition variance and coefficient of variation are calculated by the same equations as for the 1x scale mixer, and the determination of mixing effectiveness for the larger scale mixers is as described above in Section 3.4.2.3.

3.5 Parameter Selection and Design of Experiments

A double screw mixer is a complex system with a large number of interconnected parameters that can vary widely. These parameters can have a large influence on the mixing effectiveness of a screw mixer. Parameters such as housing orientation, volumetric fill level, screw rotation orientation, screw rotation speed, screw pitch, screw flighting geometry, material injection configuration, mixing length, mass flow rate ratio, particle properties such as size and density, and mixer scale can all affect the mixing dynamics within the mixer. It is important to understand not only the individual effects of each parameter, but also the interplay of each, in order to better understand the operation and design of double screw mixers.

3.5.1 Reference study

A previous study, conducted by Kingston (2013) and Kingston and Heindel (2015; 2014b, c), investigated a number of parameters and their interactions in a double screw mixer. They investigated: (i) screw rotation speed (\(\omega = 20, 40, \text{ and } 60 \text{ rpm}\)), (ii) screw
rotation orientation (co-rotating, counter-rotating up pumping, and counter-rotating down pumping), (iii) dimensionless screw pitch (p/D = 0.75, 1.25, and 1.75), and (iv) material injection configuration (biomass inlet port 1 and biomass inlet port 2). A full-factorial design of experiments was chosen so that all possible combinations of the parameter levels would be taken into account, yielding 54 distinct operating conditions, detailed in Table 3.1. A detailed description of why each parameter was chosen, and why the particular parameter levels were chosen, can be found in Kingston (2013).

Kingston and Heindel (2014b) concluded that, of the four parameters they investigated, screw rotation orientation had the biggest effect on mixing effectiveness within a laboratory scale double screw mixer. The counter-rotating down pumping screw rotation orientation was shown to offer the greatest mixing effectiveness of the three screw rotation orientations investigated. It was further concluded that higher screw rotation speeds and larger dimensionless screw pitches also increased the mixing effectiveness. The counter-rotating up pumping screw rotation condition resulted in the worst mixing dynamics of the three screw rotation orientations, and the co-rotating condition, which is currently the standard screw rotation orientation used in industry, was found to be a poor mixing condition as well. Material injection configuration was found to have little effect on the mixing effectiveness, although a slight preference towards the biomass being injected first was chosen.
Table 3.1: The 54 operating conditions investigated by Kingston and Heindel (2014b, c) with their respective parameters. The operating condition numbering system is arbitrary and used only for identification.

<table>
<thead>
<tr>
<th>Operating Condition</th>
<th>Screw Speed [rpm]</th>
<th>Dimensionless Screw Pitch [-]</th>
<th>Screw Rotation Orientation</th>
<th>Biomass Injection Port</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>20</td>
<td>0.75</td>
<td>Co-rotating</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>0.75</td>
<td>Co-rotating</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>1.25</td>
<td>Counter, Up Pumping</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>4</td>
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<td>Counter, Down Pumping</td>
<td>1</td>
<td></td>
</tr>
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<td>5</td>
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<td></td>
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</tr>
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</tr>
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<td></td>
</tr>
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<td></td>
</tr>
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<td></td>
</tr>
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<td>Counter, Up Pumping</td>
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<td></td>
</tr>
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<td></td>
</tr>
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<td></td>
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<td>2</td>
<td></td>
</tr>
<tr>
<td>29</td>
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<td></td>
</tr>
<tr>
<td>30</td>
<td>1.75</td>
<td>Counter, Up Pumping</td>
<td>1</td>
<td></td>
</tr>
<tr>
<td>31</td>
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<td></td>
</tr>
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<td></td>
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<tr>
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<td>2</td>
<td></td>
</tr>
<tr>
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<td></td>
</tr>
<tr>
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<td></td>
</tr>
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<td>43</td>
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<td>2</td>
<td></td>
</tr>
<tr>
<td>44</td>
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<td></td>
</tr>
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<td>1.75</td>
<td>Counter, Up Pumping</td>
<td>2</td>
<td></td>
</tr>
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<td>46</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td>2</td>
<td></td>
</tr>
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<td>1.75</td>
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<td></td>
</tr>
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<td>48</td>
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<td>Counter, Up Pumping</td>
<td>2</td>
<td></td>
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<td>49</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
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<td></td>
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<td></td>
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<td>51</td>
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<td>52</td>
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<td>Counter, Down Pumping</td>
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<td>53</td>
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</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Counter, Down Pumping</td>
<td>2</td>
</tr>
</tbody>
</table>
3.5.2 Parameter selection

This project builds upon the work done by Kingston and Heindel (2014b, c) by investigating additional parameters of the double screw mixer. Kingston and Heindel laid the foundation by investigating four key parameters. Extensive review of the literature suggests that particle characteristics and mixer scale play significant roles the mixing dynamics within a screw mixer. Therefore, specific operating conditions tested by Kingston and Heindel are repeated while selectively varying additional parameters. This way, the further effect of parameters on mixing effectiveness can be investigated, while still analyzing how parameters interact and affect each other. Specifically, this project further investigates the parameters: (i) material injection (premixing), (ii) particle size, (iii) particle density, (iv) particle concentration, (v) and mixer scale.

3.5.2.1 Premixing

Double screw pyrolyzers are much more complex in design than screw mixers. Their designs must feature not only the screws and housing, but multiple vapor outlet ports, temperature measurement ports, solids outlet ports, supporting parts, and heat shielding. Figure 3.19 shows the schematic of a laboratory-scale double auger reactor used for biomass fast pyrolysis at Iowa State University, illustrating the complicated design of actual pyrolyzers (Brown, 2009). In order to reduce the complexity of the system, even in a minor way, it would be advantageous if the two material injection ports could be combined into a single inlet port. While this would simplify the external aspects of the system, it is unknown what effects this combined material injection would have on the mixing dynamics within the system.
This project briefly investigates the effects of premixing the heat carrier and biomass media (by injecting the two granular materials through one injection port) on the mixing effectiveness within the screw mixer. The original biomass particles and heat carrier media used by Kingston and Heindel (2014b) were used so that the effects of the premixing would be isolated. The results of the premixing investigation are presented in Appendix A.

3.5.2.2 Particle size and density

Numerous studies found in the literature indicate that particle size and particle density play a key role in determining the mixing effectiveness of a granular mixing process.
(Alexander et al., 2004a; Arntz et al., 2014; Hogg, 2009; Hsiau and Chen, 2002; Jain et al., 2005; Nielsen et al., 2011; Yang, 2006). When the particles in a granular flow have differing sizes and/or densities, segregation mechanisms act to limit the mixing potential of the process. Understanding how certain particles interact and segregate due to particle differences is crucial in order to design mixers that mitigate these segregation effects.

Kingston and Heindel (2014b, c) only investigated one biomass particle (red oak chips, 500 – 6350 µm, 1.47 g/cc, referred to as LRO in this study) and one heat carrier particle (glass beads, 300 – 500 µm, 2.50 g/cc, referred to as SGB in this study) in their study. Because of the important influence particle properties can have on the mixing dynamics within a screw mixer, this project investigates various particles with differing sizes and densities. One additional biomass particle (red oak chips, 300 – 710 µm, 1.53 g/cc, referred to as SRO) is used to investigate the effect of changing biomass particle size. One new heat carrier particle (glass beads, 800 – 1000 µm, 2.50 g/cc, referred to as LGB) is investigated to determine the effect of heat carrier particle size. To determine the effect of biomass particle density, three biomass particles are compared: SRO, corn stover (300 – 710 µm, 1.37 g/cc, CS) and cork particles (300 – 710 µm, 0.87 g/cc, CO). The specific material combinations used in all studies of this project are outlined in Table 3.2. Note that in each case, only one particle was changed from the conditions investigated by Kingston and Heindel (2014b, c) so that the effects of changing particle size and density are isolated.
Table 3.2: All granular material combinations used in this project, outlined by specific study.  
*The reference study was conducted by Kingston and Heindel (2014b, c).

<table>
<thead>
<tr>
<th>Study</th>
<th>Biomass Material</th>
<th>Heat Carrier Media</th>
<th>Mass Flow Rate Ratio, Heat Carrier to Biomass</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Particle Abbrv.</td>
<td>Size [µm]</td>
<td>Density [kg/m³]</td>
</tr>
<tr>
<td>Reference study**, Premixing</td>
<td>Red Oak LRO</td>
<td>500–6350</td>
<td>1470</td>
</tr>
<tr>
<td>Particle Size</td>
<td>Red Oak LRO</td>
<td>500–6350</td>
<td>1470</td>
</tr>
<tr>
<td>Particle Size and Density</td>
<td>Red Oak SRO</td>
<td>300–710</td>
<td>1530</td>
</tr>
<tr>
<td>Particle Density</td>
<td>Corn Stover CS</td>
<td>300–710</td>
<td>1370</td>
</tr>
<tr>
<td>Particle Density</td>
<td>Cork CO</td>
<td>300–710</td>
<td>900</td>
</tr>
<tr>
<td>Mass Flow Rate Ratio</td>
<td>Red Oak LRO</td>
<td>500–6350</td>
<td>1470</td>
</tr>
</tbody>
</table>

**3.5.2.3 Particle concentration**

Particle concentration is an important parameter in fast pyrolysis. As pyrolysis occurs due to the heat transfer from the heat carrier to the biomass material, the heat transfer potential between the two materials is vitally important (Mohan et al., 2006). Changing the biomass mass particle concentrations, or in other words, the mass flow rate ratio between the two granular materials, can influence the rates of heat transfer, potentially increasing the bio-oil yields. However, changing the particle concentration can influence the mixing dynamics, which also have an influence on bio-oil yields. Thus, it is important to understand how changing the mass flow rate ratios influence the mixing dynamics, so that in the future, when the effect of heat transfer rates on pyrolysis is investigated, the mixing dynamics will be better understood and able to be considered.
Kingston and Heindel (2014b, c) held the mass flow rate ratio between the biomass particle and the heat carrier constant for all tests. This project investigates the effect of changing mass flow rate ratio by testing four additional biomass particle concentrations. In total, including the results from Kingston and Heindel (2014b, c), five mass flow rate ratios are compared: 10:1, 20:1, 30:1, 40:1, and 50:1 mass flow rate ratios between the heat carrier and biomass materials. In all cases, the LRO and SGB are used.

3.5.2.4 Mixer scale

Mixer scale is perhaps one of the most important parameters concerning granular mixers today. While granular flow research has gained momentum in the past few decades, the majority of the research that has been done has been on small, laboratory scale mixers, which are not directly applicable to industrial uses (Bridgwater, 2012). Due to the complicated nature of granular flows, it is vitally important that large scale mixers be experimentally tested in order to develop scale up laws to aid in the future design of industrial scale mixers (Paul et al., 2004). It may even be that, given the complicated rheology of granular materials, no sound scale up laws will be able to be developed, meaning that extensive experimental research with larger scale mixers will be even more vitally important.

In this project, a 1.5x and 2x scale screw mixer was designed and tested, based on the original screw mixer designed by Kingston and Heindel (2014b, c). Select operating conditions, based on the conclusions of Kingston and Heindel, were then exactly replicated (LRO:SGB, 10:1 mass flow rate ratio) with the larger screw mixers to determine the effect of mixer scale on the mixing dynamics.
3.5.3 Design of experiments

3.5.3.1 Premixing Study

Operating conditions investigated in the premixing task were determined based on the conclusions of Kingston and Heindel (2014b, c). The optimal mixing condition, with a screw rotation speed of $\omega = 60$ rpm, a dimensionless screw pitch of $p/D = 1.75$, and a counter-rotating down pumping screw rotation orientation was the starting point for the study. As a counter-rotating down pumping screw rotation orientation was determined to be the most advantageous screw rotation condition, regardless of screw rotation speed and dimensionless screw pitch, all other operating conditions involving the counter-rotating down pumping screw rotation orientation were also investigated. These consisted of screw rotation speeds of $\omega = 20, 40$ and $60$ rpm, each with a dimensionless screw pitch of $p/D = 0.75, 1.25$, and $1.75$.

For the sake of completeness, two additional operating conditions were chosen for testing: (1) Kingston and Heindel’s (2014b, c) original standard condition with a screw speed of $\omega = 40$ rpm, dimensionless screw pitch of $p/D = 1.25$ and a co-rotating screw rotation orientation; and (2) a condition deemed to be a poor mixing condition, with a screw speed of $\omega = 20$ rpm, dimensionless screw pitch of $p/D = 0.75$ and a counter-rotating up pumping screw rotation orientation.

The goal of this small study was to investigate the effect of premixing so the two materials, LRO and SGB, were metered out of their respective volumetric feeders into one single inlet tube which fed into the second inlet port of the screw mixer. Thus, only one inlet port was used. The experimental setup for the premixing study is shown in Figure 3.20. All premixing tests were conducted at a 10:1 mass flow rate ratio between the SGB and the LRO.
with a 65% fill of the screw mixer, outlined in Table 3.2. In total, 11 testing conditions were investigated in the premixing task, outlined in Table 3.3.

Figure 3.20: The experimental setup for the premixing study. The two volumetric feeders (biomass on the left, heat carrier on the right) feed into the same inlet tube, which injects the premixed materials into the second inlet port of the screw mixer.
Table 3.3: The 11 operating conditions with their respective parameters tested in the premixing study. Operating condition numbers correlate to the arbitrary numbering system of the shown in Table 3.1.

<table>
<thead>
<tr>
<th>Operating Condition</th>
<th>Screw Speed [rpm]</th>
<th>Dimensionless Screw Pitch [-]</th>
<th>Screw Rotation Orientation [-]</th>
<th>Injection Orientation [-]</th>
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</thead>
<tbody>
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<td>RO 2, GB 2</td>
</tr>
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<td>RO 2, GB 2</td>
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</tr>
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</tr>
<tr>
<td>53</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td>RO 2, GB 2</td>
<td></td>
</tr>
</tbody>
</table>

3.5.3.2 Particle Size and Density Study

The operating conditions investigated in the particle size tests were determined based on the conclusions of Kingston and Heindel’s (2014b, c). The same 11 operation conditions described in Section 3.5.2.1 and outlined in Table 3.3 were originally chosen to be replicated with each new particle combination, outlined in Table 3.2. However, unexpected detrimental mixing behavior observed in the initial particle size tests of the counter-rotating up pumping screw rotation orientation led to the abandonment of later tests for this condition. As a result, only the dynamic mixing video and composition results of the SRO:SGB was captured for the counter-rotating up pumping screw rotation orientation, all other particle composition tests (CS:SGB, CO:SGB, and LRO:LGB) were abandoned for this screw orientation for reasons more thoroughly explained in Chapter 4.

All conditions tested had a material injection configuration of biomass injected into port 1, heat carrier injected into port 2, a 10:1 mass flow rate ratio, and a 65% volumetric fill
of the screw mixer. In total, 44 operating conditions were attempted (11 operating conditions x 4 particle combinations), of which only 41 were able to be fully realized.

Table 3.4: The 11 operating conditions with their respective parameters tested in the particle size and density study. Operating condition numbers correlate to the arbitrary numbering system of Table 3.1. The highlighted operating condition was later abandoned for the CS:SGB, CO:SGB, and LRO:LGB conditions) due to an increased potential for screw breaks.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Screw Speed [rpm]</th>
<th>Dimensionless Screw Pitch [-]</th>
<th>Screw Rotation Orientation</th>
<th>Injection Orientation [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>20</td>
<td>0.75</td>
<td>Counter, Up Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>5</td>
<td>20</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>11</td>
<td>20</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>17</td>
<td>20</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>23</td>
<td>40</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>25</td>
<td>40</td>
<td>1.25</td>
<td>Co, CW</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>29</td>
<td>40</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>35</td>
<td>40</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>41</td>
<td>60</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>47</td>
<td>60</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
<tr>
<td>53</td>
<td>60</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td>BM 1, GB 2</td>
</tr>
</tbody>
</table>

3.5.3.3 Mass Flow Rate Ratio Study

Operating conditions investigated in the mass flow rate ratio study were the same as described in Section 3.5.2.1, and outlined in Table 3.3. Interesting behavior seen in initial tests of the counter-rotating up pumping screw rotation orientation led to the addition of three more counter-rotating up pumping screw rotation orientations at screw rotation speeds of \( \omega = 20, 40 \) and 60 rpm, all with a dimensionless screw pitch of \( p/D = 1.25 \). In total, 56 operating conditions were investigated (14 operating conditions x 4 mass flow rate ratios) for the mass flow rate ratio study and are outlined in Table 3.5.

Four mass flow rate ratios were investigated for all operating conditions: 20:1, 30:1, 40:1, and 50:1 GB:RO. It was decided to investigate decreasing biomass particle concentrations (i.e., increasing mass flow rate ratios) because the goal in fast pyrolysis is to
increase the potential for heat transfer, accomplished by increasing the ratios of heat transfer media to biomass material. All conditions tested in the mass flow rate ratio study of this project used LRO and SGB, had a material injection configuration of LRO 1, SGB 2, and maintained a 65% fill of the screw mixer. Details of the granular material combinations used in the study are outlined in Table 3.2.

Table 3.5: The 56 operating conditions with their respective parameters tested in the mass flow rate ratio tests. Operating condition numbers correlate to the arbitrary numbering system of Table 3.1

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Screw Speed [rpm]</th>
<th>Dimensionless Screw Pitch [-]</th>
<th>Screw Rotation Orientation</th>
<th>Injection Orientation [-]</th>
<th>Mass Flow Rate Ratio [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>20</td>
<td>0.75</td>
<td>Counter, Up Pumping</td>
<td></td>
<td>20:1, 30:1, 40:1, 50:1</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>11</td>
<td>1.25</td>
<td>Counter, Up Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>17</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>23</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>23</td>
<td>25</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>40</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>29</td>
<td>1.25</td>
<td>Co-rotating</td>
<td></td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>35</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>41</td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>41</td>
<td>45</td>
<td>0.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>45</td>
<td>47</td>
<td>1.25</td>
<td>Counter, Up Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>47</td>
<td>53</td>
<td>1.25</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
<tr>
<td>53</td>
<td></td>
<td>1.75</td>
<td>Counter, Down Pumping</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.5.3.4 Mixer Scale Study

The exact parameters for selected conditions, from Kingston and Heindel (2014b, c), were replicated for the larger screw mixer tests. The 11 conditions outlined in Section 3.5.2.1 were chosen as the conditions for the mixer scale study and are outlined in Table 3.3. The LRO:SGB material combination, 10:1 mass flow rate ratio, material injection configuration of LRO inlet port 1, and a 65% fill of the screw mixer was maintained (except for one operating condition which was under filled, as explained in Section 3.3.1), for all tests with the 1.5x and 2x scale screw mixers.
CHAPTER 4. EFFECT OF PARTICLE SIZE, DENSITY, AND CONCENTRATION ON MIXING IN A DOUBLE SCREW PYROLYZER¹

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Keywords:
- Granular materials
- Mixing
- Multiphase flow
- Particulate process
- Powder technology
- Renewable energy

Highlights

- Decreasing biomass particle size increases mixing effectiveness
- Increasing heat carrier particle size decreases mixing effectiveness
- Changing biomass particle density has little effect on mixing effectiveness
- Decreasing biomass particle concentration decreases mixing effectiveness.

4.1 Abstract

Double screw pyrolyzers can be used to convert cellulosic biomass into bio-oil. Bio-oil can then be converted into synthetic gasoline, diesel, and other transportation fuels, or can be converted into bio-based chemicals for a wide range of purposes. One method of industrial bio-oil production is called fast pyrolysis, the fast thermal decomposition of organic material in the absence of oxygen. One type of pyrolyzer, a double screw pyrolyzer, features two intermeshing screws encased in a reactor which mechanically conveys and mixes the biomass and heat carrier media. The mixing effectiveness of the two materials in

¹ Manuscript to be submitted to Powder Technology.
the pyrolyzer is directly correlated to the bio-oil yield—the better the mixing, the higher the yields. This study investigates the effects of particle size, density, and concentration on mixing effectiveness in a double screw pyrolyzer. Using glass beads as simulated heat carrier media and various organic particles as biomass, a cold-flow double screw mixer with 360° of optical access and full sampling capabilities was used to collect mixing data. Unique optical visualization and composition analysis paired with statistical methods were used to evaluate the effects of varying the biomass particle size and density, the heat carrier particle size, and the biomass particle concentration. The particle combinations used in this study include: (1) red oak chips (300 – 710 μm, 1.53 g/cc) and glass beads (300 – 500 μm, 2.50 g/cc); (2) corn stover (300 – 710 μm, 1.37 g/cc) and glass beads (300 – 500 μm, 2.50 g/cc); (3) cork particles (300 – 710 μm, 0.87 g/cc) and glass beads (300 – 500 μm, 2.50 g/cc); (4) red oak chips (500 – 6350 μm, 1.47 g/cc) and glass beads (800 – 1000 μm, 2.50 g/cc); and (5) red oak chips (500 – 6350 μm, 1.47 g/cc) and glass beads (300 – 500 μm, 2.50 g/cc). Condition (5) was used for the varying biomass concentration tests. Both qualitative and quantitative analysis indicated that reducing the biomass particle size, for counter-rotating down pumping screw rotation orientations, noticeably increased mixing effectiveness. Increasing the heat carrier media particle size showed both increases and decreases in mixing effectiveness depending on operating condition. For all screw rotation orientations, a change in biomass particle density resulted in little change in mixing effectiveness, while reducing the biomass particle concentration reduced the overall mixing effectiveness.

4.2 Introduction

Granular mixing processes are very important across many industries including, but not limited to, the pharmaceutical, agricultural, and biotechnology industries (Bridgwater,
2012; Campbell, 2006; Mohan et al., 2006). These processes often require both a high degree of homogeneity and a high degree of customizability (Ottino and Khakhar, 2001). As granular mixing processes are so widely employed, a thorough understanding of the mixing dynamics is necessary to understand and control the resulting products (Paul et al., 2004).

Granular mixing has been widely studied since the 1950’s when the science began to gain attention (Bridgwater, 2010). Considerable experimental research has since been done on a wide variety of mixers, including: bladed mixers (Conway et al., 2005; Remy et al., 2010), vertical shakers (Hsiau and Chen, 2002; Yang, 2006), single screw mixers (Tsai and Lin, 1994; Uchida and Okamoto, 2006, 2008), and rotating cylinders (Aït Aissa et al., 2010). Granular mixing processes have also been studied using simulations (Cleary and Sinnott, 2008; Sarkar and Wassgren, 2009; Yang, 2006). Several literature reviews are available on the subject (Bridgwater, 2012; Campbell, 1990; Campbell, 2006; Ottino and Khakhar, 2000).

Although many studies have investigated granular mixing, most work has focused on mixers with relatively simple geometries. While these studies have done much to improve the theoretical understanding of granular flows, these theoretical understandings are often difficult to apply to broader industrial uses. Moreover, granular mixing processes are often very sensitive to changes in operating conditions and any solutions provided to deal with specific mixing problems are highly system-sensitive. These sensitivities mean it is necessary to study more complicated mixer geometries and more complicated operating condition interactions if we hope to apply knowledge of granular mixing to industrial processes (Hogg, 2009).

One example of a complicated industrial mixer is a double screw pyrolyzer used in the bioenergy industry to produce bio-oil via fast pyrolysis (Ingram et al., 2007; Mohan et al.,
Fast pyrolysis is the thermochemical conversion of biomass into bio-oil in the absence of oxygen (Mohan et al., 2006). In a double screw pyrolyzer, a biomass material is mixed with a heat carrier media at high temperatures to produce vapors that are collected and condensed into bio-oil (Bridgwater, 1999). Bio-oil has the potential to be converted and upgraded into transport fuels, such as synthetic gasoline or diesel, to be used as a source of fuel via direct combustion, or to be converted into a wide variety of bio-based chemicals (Bridgwater, 2003; Brown, 2011b). Fast pyrolysis is still, however, a relatively new technology (Bridgwater, 1999) and much of the research that has been done with double screw pyrolyzers has focused on the products (Bahng et al., 2009; Brown, 2009; Ingram et al., 2007) and not on the mixing dynamics of the mixer. Recent work by Kingston and Heindel (2015; 2014b, c) began to correct this by investigating the mixing dynamics within a double screw pyrolyzer to further understand both the mixing dynamics themselves, and the effects of operating conditions on the mixing effectiveness within the double screw mixer.

Kingston and Heindel (2015; 2014b, c) developed measurement techniques to determine the mixing effectiveness of the continuous mixing process inside a double screw mixer. Using both quantitative and qualitative methods, they investigated the interactive effects of screw rotation speed, screw rotation orientation, material injection configuration, dimensionless screw pitch, and dimensionless mixing length on the mixing dynamics and mixing effectiveness. Their results provided a greatly improved understanding of the mixing dynamics within a double screw mixer and offered valuable insights into the optimization of industrial double screw pyrolyzers.

However, the studies performed by Kingston and Heindel featured only one size, type, and concentration of biomass and heat carrier media, whereas in actual fast pyrolysis
there is often a wide variability in particle characteristics. Various considerations, such as biomass growth times, ease of planting and harvesting, and media influences on bio-oil yields and composition can influence the availability and profitability of using different biomass and heat carrier media, which may have different particle sizes and densities (Mohan et al., 2006). Investigating different particles in a double screw pyrolyzer is important because the mixing dynamics of a granular flow can vary considerably based on particle characteristics (Campbell, 1990; Campbell, 2006; Ottino and Khakhar, 2001). Additionally, many studies have investigated the tendency for granular flows to segregate by varying particle characteristics, such as size (Hogg, 2009; Rao et al., 2011), shape (Hilton and Cleary, 2011; Rao et al., 2011), and density (Hsiau and Chen, 2002; Nielsen et al., 2011; Yang, 2006). All studies have shown that changes in these particle characteristics can have a dramatic effect on the mixing dynamics within a granular flow.

Furthermore, as fast pyrolysis is the thermochemical conversion of biomass into bio-oil, it is highly dependent on the heat transfer from the heat carrier media to the biomass media. Increasing the ratio of heat carrier to biomass media, by reducing the biomass concentration in the system, may increase the rate of heat transfer into the biomass material, thus increasing potential bio-oil yields (Bridgwater, 2003; Ingram et al., 2007). Thus, it is important to also investigate the effect of biomass particle concentration on the mixing effectiveness within a double screw pyrolyzer.

The purpose of this study is to expand upon the work completed by Kingston and Heindel, and to investigate the effect of changing particle size, density, and concentration on mixing effectiveness in a double screw pyrolyzer. Selected operating conditions investigated by Kingston and Heindel (2013; 2014b), featuring changes in screw rotation speed,
dimensionless screw pitch, and screw rotation orientation, were repeated using different combinations of biomass and heat carrier media. In this study, particle size in the form of a new biomass particle size and a new heat carrier particle size, particle density in the form of three different biomass particle densities, and particle concentration in the form of changing biomass inlet concentrations, were investigated. The specific biomass/heat carrier combinations used in this study are listed in Table 1. Qualitative optical visualization methods and quantitative composition analysis, as developed by Kingston and Heindel (2014c), were used to assess the mixing effectiveness of the double screw mixer. The operating conditions, based on these parameters, which led to the highest degree of granular homogeneity—and thus the optimized mixing effectiveness—were determined.

4.3 Experimental Procedures

The experimental setup and procedures used in this study are very similar to those developed by Kingston and Heindel (2013; 2014c). Only a summary is provided here.

4.3.1 Experimental setup

The clear, laboratory scale cold-flow double screw pyrolyzer used in these studies is shown in Figure 4.1 and will hereafter be referred to as a double screw mixer. The double screw mixer was manufactured using 3D printing technology using a rigid, clear, designer plastic material. This unique manufacturing method allows complete 360° optical access to the internal structure of the screw mixer. The double screw mixer features two intermeshing, noncontact screws with screw diameter \( D = 2.54 \) cm. The characteristic length of the mixer is defined as the screw diameter. The effective mixing length, \( L \), is defined from the center of inlet port 2 in Figure 4.1 to the beginning of the outlet ports, where \( L/D = 10 \).
Figure 4.1: The cold flow, laboratory scale pyrolyzer used in these studies (referred to as a double screw mixer) with a cross-sectional view of the unique outlet ports that allows the outlet stream to be divided for sample composition analysis (Kingston and Heindel, 2014c). The double screw mixer can operate at three screw rotation orientations: co-rotating (CoR), counter-rotating up pumping (CtrR UP), and counter-rotating down pumping (CtrR DP).

Two material injection ports are positioned axially two characteristic lengths apart and laterally halfway between the two screws. The biomass and heat carrier media are injected vertically into ports 1 and 2, respectively, which was the preferred injection configuration determined by Kingston and Heindel (2014b). The two granular materials are fed into their respective inlet ports by two Tecweigh CR5 volumetric auger feeders. The exit stream of the double screw mixer is divided into four equal sections by a unique outlet port design, located on the bottom of the double screw mixer. These outlet ports, shown in Figure 4.1, spatially divide the outlet flow of the pyrolyzer into four separate channels from which four unique samples are collected for composition analysis.

This study varied dimensionless screw pitches ($p/D = 0.75, 1.25, \text{ and } 1.75$) and screw rotation speeds ($\omega = 20, 40, \text{ and } 60 \text{ rpm}$). Screw rotation orientation was also varied and
included a co-rotating (CoR), counter-rotating up pumping (CrtR UP), and counter-rotating down pumping (CrtR DP), as schematically shown in Figure 4.1.

4.3.2 Granular materials

The biomass materials were red oak, corn stover, or cork (to simulate pyrolysis char), while the heat carrier was simulated with glass beads. Six specific material combinations were investigated and are listed in Table 4.1. All granular materials are shown in Figure 4.2.

The biomass materials were processed down to the specified size ranges using a series of hammer mills and hand sieves. The heat carrier media were purchased in bulk quantities of broad size ranges and refined, using hand sieves, down to their final, specified size ranges. The true density of each material was measured using a Pentapyc 5200e gas pycnometer (Quantachrome instruments). In the biomass thermochemical conversion industry, refractory sand is the typical heat carrier media used, because of its ready availability and high heat transfer rates (Brown and Brown, 2012). However, due to the abrasive nature of sand particles, sand was not a viable choice for this study, as the rough, irregular sand particles would scratch the clear 3D printed double screw mixer. Instead, highly spherical glass beads were chosen as the heat carrier media in this study because they were much less abrasive than sand and will cause less damage to the surface of the plastic double screw mixer. The glass beads have a measured true density of 2.50 g/cc, comparable to sand’s (Quikrete No: 1961) true density of 2.68 g/cc (Kingston, 2013), making them an adequate substitute for refractory sand.
Table 4.1: The granular material combinations investigated. *Reference study conducted by Kingston and Heindel (Kingston and Heindel, 2014b)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Red Oak*</td>
<td>LRO</td>
<td>500–6350</td>
<td>1.47</td>
<td>Glass Beads*</td>
<td>SGB</td>
<td>300–500</td>
<td>2.50</td>
<td>10:1</td>
</tr>
<tr>
<td>Red Oak</td>
<td>LRO</td>
<td>500–6350</td>
<td>1.47</td>
<td>Glass Beads</td>
<td>LGB</td>
<td>800–1000</td>
<td>2.50</td>
<td>10:1</td>
</tr>
<tr>
<td>Red Oak</td>
<td>SRO</td>
<td>300–710</td>
<td>1.53</td>
<td>Glass Beads</td>
<td>SGB</td>
<td>300–500</td>
<td>2.50</td>
<td>10:1</td>
</tr>
<tr>
<td>Corn Stover</td>
<td>CS</td>
<td>300–710</td>
<td>1.37</td>
<td>Glass Beads</td>
<td>SGB</td>
<td>300–500</td>
<td>2.50</td>
<td>10:1</td>
</tr>
<tr>
<td>Cork</td>
<td>CO</td>
<td>300–710</td>
<td>0.87</td>
<td>Glass Beads</td>
<td>SGB</td>
<td>300–500</td>
<td>2.50</td>
<td>10:1</td>
</tr>
<tr>
<td>Red Oak</td>
<td>LRO</td>
<td>500–6350</td>
<td>1.47</td>
<td>Glass Beads</td>
<td>SGB</td>
<td>300–500</td>
<td>2.50</td>
<td>20:1, 30:1, 40:1, 50:1</td>
</tr>
</tbody>
</table>

Figure 4.2: Images of the granular materials used in the study at 4x magnification.
4.3.3 Operating conditions

Table 4.1 summarizes the original granular material combination investigated by Kingston and Heindel (2013; 2014b) as well as the 5 additional biomass and heat carrier media combinations investigated in this study. Table 4.2 summarizes the operating conditions investigated for each new combination of biomass and heat carrier media. 54 original operating conditions were tested by Kingston and Heindel; the 14 in Table 4.2 were selected based on the conclusions made by Kingston and Heindel (2013; 2014b).

Table 4.2: Select operating conditions investigated in this study. *Counter-rotating up pumping was only successfully investigated for the biomass particle size and biomass particle concentration tests. **Counter-rotating up pumping at 40 rpm was only investigated for the biomass particle concentration tests.

<table>
<thead>
<tr>
<th>Operating Conditions</th>
<th>Screw Rotation Orientation</th>
<th>Screw Rotation Speed ω [rpm]</th>
<th>Dimensionless Screw Pitch p/D [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Counter, Up Pumping*</td>
<td>20</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td></td>
<td>40**</td>
<td>0.75**</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.25**</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>1.75**</td>
</tr>
<tr>
<td></td>
<td>Co-rotating</td>
<td>40</td>
<td>1.25</td>
</tr>
<tr>
<td></td>
<td>Counter, Down Pumping</td>
<td>20</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.25</td>
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<td></td>
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<td>1.75</td>
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<td></td>
<td></td>
<td>0.75</td>
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<td></td>
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<td></td>
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<td>0.75</td>
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<td></td>
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<td></td>
<td></td>
<td>1.75</td>
<td></td>
</tr>
</tbody>
</table>
The counter-rotating down pumping screw rotation orientation was determined by Kingston and Heindel (2013; 2014b) to be the most advantageous screw rotation orientation, regardless of screw rotation speed and dimensionless screw pitch. Based on this conclusion, all operating conditions involving the counter-rotating down pumping screw rotation orientation were investigated in this study. One co-rotating screw rotation condition, determined by Kingston and Heindel to represent a standard operating condition, with a screw speed of $\omega = 40$ rpm and dimensionless screw pitch of $p/D = 1.25$, was chosen. Additionally, for all material combinations, one counter-rotating up pumping screw rotation orientation was chosen with a screw rotation speed of $\omega = 20$ rpm and a dimensionless screw pitch of $p/D = 0.75$. This up pumping condition was chosen to represent a “poor” operating condition determined by Kingston and Heindel (2014b). The biomass particle concentration tests showed unexpected mixing dynamics for the one counter-rotating up pumping condition, and so additional up pumping operating conditions were investigated for this particular material combination. Note that the LRO and SGB 10:1 mass flow rate ratio data (referred to in the results section as the reference study) was originally obtained by Kingston and Heindel (2013; 2014b) and is used in our analysis as a comparison. A 10:1 mass flow rate ratio between the heat carrier media and the biomass was maintained for all particle size and density tests, as this was the original mass flow rate ratio investigated by Kingston and Heindel (2013; 2014b). For the biomass particle concentration tests mass flow rate ratios of 10:1, 20:1, 30:1, 40:1 and 50:1, corresponding to biomass inlet mass concentrations of 9%, 4.7%, 3.2%, 2.4%, and 1.9% respectively, were investigated. In all tests, a 65% fill of the double screw mixer was maintained, as this fill level is optimal to avoid problematic operating conditions and achieve optimal mixing (Colijn, 1985).
4.3.4 Optical visualization

To capture the dynamic mixing process within the double screw mixer, the methods developed by Kingston and Heindel (2014c) were used.

Four Panasonic HC-V700M high definition cameras, one each positioned to the top, left, right, and bottom of the double screw mixer, were used to simultaneously capture the dynamic mixing process from four independent views. The video cameras each feature a 1920 × 1080 resolution and a frame rate of 60 frames per second. To provide lighting, six 85 W compact florescent lamps were used to illuminate the mixing region, positioned three on either end, level with the screw mixer. To minimize glare from ambient light, the entire experimental setup was encased in a canopy of black fabric.

Adobe Premiere Pro CS6 video editing software was then used to combine the four separate video projections into one temporally and spatially synced dynamic mixing video. An audio spike was used to temporally synchronize the four videos files. Figure 4.3 shows how the four independent projections (Figure 4.3a) are combined into one dynamic mixing video (Figure 4.3b). The top and right projections were captured in the shown orientation, but the left and bottom projections necessitated inverting using Premiere Pro to spatially align all projections into one common material flow direction (i.e., from left to right in Figure 4.3b). Figure 3b shows the typical pathline of a particle injected into port 2. This technique of optical visualization allows simultaneous 360° visualization of the entire mixing region within the double screw mixer.
4.3.5 Composition analysis

The mixing effectiveness within the double screw mixer was determined by the homogeneity of the mixture. An ideally mixed system features a high degree of homogeneity throughout. In the double screw mixer, an even distribution of the biomass and glass beads everywhere within the screw mixer would indicate a high degree of mixing effectiveness. Mixture homogeneity was determined by the method developed by Kingston and Heindel (2014b).

The outlet port of the double screw mixer features a unique dividing system that spatially partitions the outlet stream into four equal sections (see Figure 4.1). Samples were collected, simultaneously, from each of the four outlet ports. To ensure repeatability, three sets of samples were collected for each operating condition. To gauge the mixture homogeneity, the mixture composition of each sample was determined by calculating the true
density of each sample and using an empirical correlation between mixture density and composition to determine the sample composition. Using the method developed by Kingston and Heindel (2014c), unique empirical correlations between mixture density and composition were developed for each material combination investigated in this study.

The mixture composition is defined as the glass bead mass fraction and ranges from one to zero. The sample compositions, from each of the four outlet ports, were then compared to each other, as well as across operating conditions, by calculating the mass weighted composition variance, $s^2$, of the four outlet ports:

$$s^2 = \frac{\sum_{i=1}^{n} m_i (x_i - \bar{x}_w)^2}{(N - 1) \sum_{i=1}^{n} m_i} \quad (4.1)$$

where $n$ is the number of the $i^{th}$ sample, $m_i$ is the mass of the $i^{th}$ sample, $x_i$ is the measured composition of the $i^{th}$ sample, $\bar{x}_w$ is the mass weighted mean composition of the samples, and $N$ is the total number of samples. The mass weighted composition variance was used instead of a standard composition variance because the mass flow rates through each of the four outlet ports were not equal, and thus the standard composition variance, if not weighted by mass, would have been skewed.

The mass weighted composition variance was then used to calculate the coefficient of variation, CV, a statistical parameter used to gauge mixture homogeneity:

$$CV = \frac{s}{\mu} \quad (4.2)$$

where $s$ is the standard deviation determined by the square root of Eq. (4.1), and $\mu$ is the theoretical mean of the outlet composition, which, in an ideally mixed system equals the inlet mixture composition. The coefficient of variation is a dimensionless parameter that allows
the variability in a series of numbers to be independently compared, regardless of the units or magnitude of the individual measurements. When using the CV to determine the mixing effectiveness of the double screw mixer, a smaller CV indicates a more homogeneous mixture and greater mixing effectiveness. Therefore, a CV of zero is the desired result.

4.3.6 Uncertainty analysis

An uncertainty analysis was performed to calculate the amount of uncertainty associated with computing the coefficient of variation. The standard error of the coefficient of variation was used, an empirically calculated error that represents the maximum possible error in the data set (Devore, 2014). The calculated standard errors could be large and ranged from 0.004 to 0.26. Thus, the error bars, when plotted on the three-way interaction plot, often overlapped each other. This indicates that our ability to differentiate one set of results from another may be limited. However, close analysis of the individual data points indicate that, in most cases, the data points are clustered together and remain distinct from other sets of data points, indicating that the trends observed in the averaging lines in the following figures are consistent and represent actual mixing behavior.

4.4 Results and Discussion

In all figures, tables, and results that follow, the reference study results (LRO-SGB, 10:1) were originally obtained by Kingston and Heindel (2013; 2014b), and will henceforth be identified simply as the reference study. Their composition analysis results were presented only as a mass weighted composition variance, $s^2$, not as a coefficient of variation, CV. Their original results have been converted, with permission, to the coefficient of variation used in this study, in order to allow for direct comparison to the results present here.
4.4.1 Particle size

The reference study featured only one combination of biomass material and heat carrier media. This study replicates the operating conditions tested in the reference study for two new particle sizes, one smaller biomass material and one larger heat carrier material. In each case, only one particle size was changed from the reference study at a time. The new, smaller biomass material, SRO, was paired with the original reference study’s heat carrier media, SGB. Likewise, the new, larger heat carrier media, LGB, was paired with the original reference study’s biomass material, LRO.

4.4.1.1 Biomass particle size

To understand the effect individual parameters have on the mixing effectiveness in the double screw mixer, and to optimize the mixing effectiveness, the coefficients of variation of each operating condition are plotted against each other in a single plot. Figure 4.4 shows the three-way interaction between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the new biomass particle size (plotted in black) compared to the reference study results (Kingston and Heindel, 2014b) (plotted in gray). Recall that a lower coefficient of variation indicates better mixing—a coefficient of variation of zero is a perfectly homogeneous mixture and is the desired state. For each operating condition, three data points are plotted, representing the set of three samples collected for each operating condition to ensure repeatability. To aid the visualization of trends, an averaging line had been applied to each set of three samples and across operating conditions. The error bars represent the standard error in the average measurement (Section 4.3.6), and in some cases are concealed by the black data symbols.
Figure 4.4: The coefficient of variation as a function of the three-way interaction between screw rotation orientation, dimensionless screw pitch, and screw rotation speed for the SRO-SGB (black) and LRO-SGB (reference, gray) systems. The reference data are from Kingston and Heindel (2014b).

The counter-rotating down pumping screw rotation orientation (CtrR DP), across all screw rotation speeds and dimensionless screw pitches, featured very low coefficients of variation for the SRO-SGB tests. Higher screw rotation speeds showed decreasing coefficients of variation as the dimensionless screw pitch increased. However, at the slowest screw rotation speed tested, $\omega = 20$ rpm, the mid-size dimensionless screw pitch ($p/D = 1.25$) showed the lowest coefficient of variation of the three pitches, with an increase in the coefficient of variation as the pitch was increased further to $p/D = 1.75$. Overall, the SRO-SGB tests appear to have a higher sensitivity to changes in dimensionless screw pitch than
the reference study’s LRO-SGB results. All counter-rotating down pumping coefficients of variation for the SRO-SGB tests were equal to or lower than the LRO-SGB results. In fact, all but three of the operating conditions tested showed coefficients of variation significantly lower than the reference study results. This indicates that reducing the biomass particle size significantly increased the mixing effectiveness of the double screw mixer for most of the counter-rotating down pumping conditions. The one co-rotating condition tested also showed improved mixing, but the improvements (reductions in the coefficients of variation) are less extreme.

Of all the conditions tested, only one condition, the counter-rotating up pumping screw rotation orientation condition, displayed an increase in the coefficient of variation for the SRO-SGB test, and thus a decrease in the mixing effectiveness, when compared to the reference study results.

Overall, reducing the biomass particle size results in a lower coefficient of variation for nearly all the conditions tested, indicating that, quantitatively, reducing the biomass particle size increases the mixing effectiveness of the double screw mixer.

The trends observed in the quantitative three-way interaction plot were also observed in the qualitative dynamic mixing videos, of which select snapshots are provided in Figure 4.5. The reference study snapshots are from Kingston (2013) and are provided for comparison. Dynamic mixing videos for all 11 operating conditions were collected, and while results presented here are based on conclusions from all 11 videos, only selected snapshots are presented for reference. In all snapshots, the granular materials are injected from the top of the screw mixer at the left, travel left to right, and are ejected from the bottom of the screw mixer at the far right. The biomass particles appear brown and the glass beads
appear gray. Three operation conditions are represented in Figure 4.5 and represent a range of outcomes from poor mixing conditions (a and d), standard operating conditions (b and e), and good mixing conditions (c and f).

![Image of Figure 4.5](image)

**Figure 4.5:** Snapshots of the dynamic mixing process for select operating conditions of both the LRO-SGB (reference) study and the SRO-SGB particle condition. The reference study images are from Kingston (2013).

The counter-rotating down pumping screw rotation orientation videos (Figure 4.5f) for the SRO-SGB size exhibit very good mixing dynamics. The red oak chips are evenly mixed throughout the system with no agglomerations present anywhere in the mixing region. Though it is difficult to see in the static images provided in Figure 4.5, the dynamic mixing videos showed that the red oak particles are in constant motion—axially, transversely, and vertically—with no trapped or static particles. This indicates that the two granular materials are mixing together while they are conveyed through the double screw mixer.

When compared to the counter-rotating down pumping screw rotation orientation videos of the reference study, with the LRO-SGB (Figure 4.5c), the SRO-SGB videos show greatly improved mixing dynamics. The reference study’s videos feature higher concentrations of red oak chips in some areas and lower concentrations in others, indicating
granular segregation, whereas the SRO-SGB condition features a near uniform concentration of red oak chips throughout. In fact, due to the even distribution of the SRO throughout the system, it is difficult to distinguish the red oak chips from the glass beads in the dynamic mixing videos. Qualitatively, the mixing dynamics of the counter-rotating down pumping screw rotation are greatly improved with the SRO-SGB combination. These qualitative conclusions agree with the quantitative conclusions represented in Figure 4.4.

The co-rotating screw rotation orientation condition tested (Figure 4.5e) with the SRO-SGB shows adequate mixing. Due to the co-rotation of the screws, the bulk of the granular flow is pushed into the left screw, essentially turning the double screw mixer into a single screw mixer. Some of the flow is visible in the right screw, though it appears to consist predominantly of red oak chips. The differences from the reference study’s co-rotating condition, however, are profound (Figure 4.5b). While the reference study’s granular flow was similarly pushed into the left screw, and also saw a high concentration of red oak in the right screw, the significant difference between the two conditions is in the concentration of biomass in the left screw. In the reference study, the LRO remained in the right screw with little red oak visible in the left projection. In the SRO-SGB study, however, there is a very high concentration of red oak remaining in the left screw. Furthermore, the red oak in the SRO-SGB tests is in constant movement, appearing to move back and forth between the two screws. The LRO of the reference study became more vertically segregated from the much smaller SGB and traveled along the top of the glass beads. In contrast, the SRO, while still separating out, were continually mixed back into the bulk of the glass beads. The smaller biomass particle size appears to, qualitatively, greatly improve the mixing dynamics of the
double screw mixer for the co-rotating screw rotation orientation, again in agreement with the quantitative results seen in the composition analysis.

The dynamic mixing video for the counter-rotating up pumping screw rotation orientation condition (Figure 4.5d), unlike the co-rotating and counter-rotating down pumping conditions, displays very poor mixing dynamics for the SRO-SGB combination. The system took a significantly longer time to reach a quasi-steady state, whereas all other conditions reached a steady state condition quickly. In the counter-rotating up pumping condition, the SRO particles back up in the double screw mixer, building up and eventually jamming the screws, leading to breaks of the plastic 3D printed screws. The comparative reference study’s condition (Figure 4.5a), with the larger biomass particles, did not display any of these phenomena. The mixing dynamics were poor in the reference study’s up pumping screw rotation condition, but the system did not back up and jam the way it did with the SRO. As the amount of red oak in the system initially increased, the dynamic mixing video of the SRO-SGB shows how the close spacing of the screw flightings (p/D = 0.75) essentially “cut” through the red oak/glass bead mixture, no longer acting as a mixer or even a conveyer, thus causing the material backup. Qualitatively, it appears that decreasing the biomass particle size greatly decreases the mixing effectiveness in an up-pumping double screw mixer to the point of screw failure. This is in agreement with the quantitative composition results, though the composition results do not convey the dramatic change in mixing dynamics, like the mixing videos do. This is because the coefficient of variation only conveys how much each outlet port varies from one another, it does not tell anything about what happens in the double screw mixer up until the point of collection.
Overall, taking into account both the quantitative composition results and the qualitative optical visualization results, it appears that reducing the biomass particle size greatly increases the mixing effectiveness for both the counter-rotating down pumping and the co-rotating screw rotation orientations tested. This is further supported by previous granular mixing studies looking at segregation and particle size (Hogg, 2009; Ottino and Khakhar, 2000; Rao et al., 2011). In the reference study, the density difference between the light red oak chips and the heavy glass beads leads to buoyancy forces pushing the red oak chips to the top of the flow, where, the large size difference between the two granular materials then prevents the red oak chips from being able to penetrate back into the glass beads. This results in some vertical segregation between the two granular materials that is visually observed but not captured in the composition analysis. With the smaller biomass particle sizes however, the SRO are more readily able to penetrate the spaces between the similarly sized glass beads. The buoyancy forces remain comparable as the true densities of the SRO and LRO are nearly the same, but the SRO are much more able to percolate through the glass beads than their large counterparts, thus increasing the mixing effectiveness of the double screw mixer. A study by Hogg (2009) explains that changes in the ratio of the particle size from one granular particle to another in a granular mixer has a profound effect on the mixing dynamics within a granular flow, with more similarly sized particles exhibiting the best mixing. In the reference study the ratio between the midpoint biomass particle size to the midpoint heat carrier particle size was 8.6:1. In the SRO-SGB study, the particle size ratio reduced to 1.3:1, effectively eliminating the issue of different particle size. This explains the dramatic increase in the mixing effectiveness seen in the SRO-SGB study.
4.4.1.2 Heat carrier particle size

Figure 4.6 shows the three-way interaction between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the new heat carrier particle size, LRO-LGB (plotted in black), against the reference study results, LRO-SGB (Kingston and Heindel, 2014b) (plotted in gray). The counter-rotating down pumping screw rotation orientation conditions for the LGB features coefficients of variation that tend to increase as the dimensionless screw pitch increases. The smallest dimensionless screw pitches (p/D = 0.75) have low coefficients of variation, lower than the reference study in some cases, but they increase rapidly as the pitch is increased, with the largest pitch (p/D = 1.75) having the largest coefficients of variations. The one exception to this trend occurs at a screw rotation speed of \( \omega = 20 \text{ rpm} \), where the mid-size dimensionless screw pitch has the lowest coefficient of variation when compared to the other two dimensionless screw pitches at that screw speed, though the p/D = 1.75 pitch still has the largest coefficient of variation. The effect of screw rotation speed seems to be limited as the coefficients of variation across screw rotation speeds are relatively unchanged, except the \( \omega = 20 \text{ rpm}, \text{p/D = 1.25} \) condition mentioned above.

The coefficient of variation for the co-rotating condition investigated with the LRO-LGB is significantly larger than the coefficient of variation for the same reference study condition. This indicates that the mixing effectiveness of the double screw mixer for this co-rotating condition decreases as the heat carrier particle size is increased.
Figure 4.6: The coefficient of variation as a function of the three-way interaction between screw rotation orientation, dimensionless screw pitch, and screw rotation speed, for the LRO-LGB (black) and LRO-SGB (reference, gray) systems. The reference data are from Kingston and Heindel (2014b).

The one counter-rotating up pumping screw rotation condition tested for the biomass particle size study was not completed for the heat carrier particle size study because of an increased risk of screw jams and subsequent screw breaks. The combined particle size of the LRO-LGB strained the clearance between the screw flightings and housing for all operating conditions tested with the larger glass beads. As a result, the counter-rotating up pumping condition for the LRO-LGB was abandoned, due to excessive screw failures and damage to the double screw mixer in attempts to collect that data.
The trends observed in the quantitative three-way interaction plot were also observed in the dynamic mixing videos, of which select snapshots are provided in Figure 4.7. The reference study snapshots are from Kingston (2013) and are provided for comparison.

Dynamic mixing videos for 10 of the 11 operating conditions were collected (counter-rotating up pumping screw rotation orientation condition abandoned), and results presented here are based off of conclusions from all 10 videos, though only 3 select snapshots are presented here for the LRO-LGB conditions.

![Particle Size: Heat Carrier Particle](Image)

Figure 4.7: Snapshots of the dynamic mixing process for select operating conditions of both the LRO-SGB (reference) study and the LRO-LGB study. The reference study images are from Kingston (2013).

The dynamic mixing videos for the counter-rotating down pumping screw rotation orientation are consistent with the composition results. The smallest dimensionless screw pitch, $p/D = 0.75$, features very good mixing (Figure 4.7f). The red oak appears evenly distributed throughout the system with large, even concentrations of red oak visible on the top and bottom as well as in both the left and right projections. However, in the left and the right projections, the red oak particles tend to remain stationary, as individual red oak chip
particles are visible for long periods of time, while the internal structure of the flow continues to be conveyed. This is apparent only in the dynamic mixing videos and not the static pictures. It appears as if the LGB are creating a sort of film between the screw flightings and the mixer wall, creating a region of particles that are not mixed into the rest of the granular flow. However, from the top and bottom projections, it is obvious that the rest of the granular flow continues to be mixed and conveyed because movement of the red oak is visible in these regions.

As the dimensionless screw pitch increases, the mixing effectiveness appears to decrease for the counter-rotating down pumping conditions (Figure 4.7d). Less red oak is visible on the sides and bottom of the double screw mixer, and the same tendency for the particles on the sides to remain stationary is observed. The larger dimensionless screw pitch conditions, when compared to the reference study (Figure 4.7a), also feature a higher concentration of red oak on the top surface of the granular flow. This indicates that the red oak is not being as readily mixed into the system with the LGB than as with the SGB for these conditions. However, at the smallest dimensionless screw pitch (Figure 4.7f), the concentrations of red oak seen on the sides and bottom of the LGB tests, compared to the reference study’s SGB tests (Figure 4.7c), is much higher. This indicates that as the heat carrier particle size is increased, the mixing effectiveness for the double screw mixer is increased for the smallest dimensionless screw pitch, but then decreased as the dimensionless screw pitch is increased.

The one co-rotating screw rotation orientation condition tested shows very poor mixing dynamics (Figure 4.7e). The bulk of the granular flow is pushed into the left screw, as expected by the rotation orientation of the two screws. However, the amount of red oak that
accumulated in the right screw was greatly increased when compared to the reference study’s co-rotating condition (Figure 4.7b). The LGB condition had a significantly higher degree of segregation between the red oak and glass beads, as much more red oak was trapped in the right screw. This qualitative observation was consistent with the quantitative composition results that showed a much higher coefficient of variation for the LGB than the SGB.

Overall, it appears that increasing the heat carrier particle size increases the double screw mixer’s sensitivity to screw rotation orientation and, especially, to dimensionless screw pitch. The mixing effectiveness in the double screw mixer increased for the counter-rotating down pumping screw rotation orientation at the smallest of the dimensionless screw pitches tested, but then decreased rapidly as the dimensionless screw pitch was increased. Furthermore, the co-rotating condition continued to be a poor mixing condition, as in the reference study. The overall decrease in the mixing effectiveness within the double screw mixer is most likely due to the increase in inertia forces associated with the increased heat carrier particle size. For the most part, as no particle densities were changed from the reference study, the inertial forces were increased for only the heat carrier, and not the biomass. With the higher inertial forces of the LGB compared to the SGB, the LGB have a much higher tendency to resist changes to their state of motion, i.e., to resist being displaced by the LRO. The minor improvement in mixing found in the conditions with the smallest dimensionless screw pitch can most likely be attributed to the closely spaced screw flighting helping the biomass to overcome the inertia of the LGB. As the spacing between screw flightings becomes larger, there are less shear forces from the screw flightings to break up the LGB and allow for LRO penetration. The implications of changing particle size, and thus inertial forces, has been recognized by previous research (Alexander et al., 2004a; Arntz et
al., 2014; Jain et al., 2005), though in the case of the rotating drum mixers investigated by Arntz et al (2014), and Jain et al (2005), the increase in the inertial forces of one particle resulted in an increase in the mixing and a decrease in the particle segregation. This indicates that the mixer geometry is incredibly important, and that mixing effectiveness for similar particle conditions can vary widely depending on mixer geometry.

### 4.4.2 Particle density

The effect of particle density on mixing effectiveness was also investigated in this study. Biomass particles of three different densities were investigated for select operating conditions. In each case, all three biomass particles had the same size range, and the heat carrier media paired with all three biomass particles remained the same. The three biomass-heat carrier combinations include SRO-SGB, CS-SGB, and CO-SGB (Table 4.1).

#### 4.4.2.1 Red oak trends

See Section 4.4.1.1 for observations of the SRO-SGB system. Figure 4.8 again presents select snapshots of the dynamic mixing videos for the SRO-SGB condition. Figure 4.8a and 4.8c, are repeated from Figure 4.5b and 4.5c, for completeness. Figure 4.8b is a snapshot of another dynamic mixing video, representing a counter-rotating down pumping screw rotation condition at \( \omega = 20 \text{ rpm} \) with a dimensionless screw pitch of \( p/D = 1.75 \). Figure 4.9 is a three-way interaction plot between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the three biomass particles investigated in the particle density investigation. The SRO-SGB results are identical to those presented in Figure 4.4.
Figure 4.8: Snapshots of the dynamic mixing process for select operating conditions of all three biomass particles tested in the particle density tests.

4.4.2.2 Corn stover trends

In Figure 4.9, the counter-rotating down pumping screw rotation orientation conditions for the CS-SGB system features low coefficients of variation that remain relatively constant as the dimensionless screw pitch changes, especially at higher screw rotation speeds. However, the slowest screw rotation speed tested ($\omega = 20$ rpm) features some changes in the coefficient of variation as the dimensionless screw pitch changes with the mid-size dimensionless screw pitch having a noticeably lower coefficient of variation. Overall, the coefficients of variations for the counter-rotating down pumping CS-SGB conditions appear to have little sensitivity to changes in dimensionless screw pitch and screw rotation speed are generally low and stable, and show good mixing conditions.
Figure 4.9: The coefficient of variation as a function of the three-way interaction between screw rotation orientation, dimensionless screw pitch, and screw rotation speed for 3 different density systems (Table 4.1).

The one co-rotating screw rotation orientation condition tested shows a fairly high coefficient of variation, indicating poor mixing.

The one counter-rotating up pumping screw rotation orientation condition in Table 4.2 was abandoned after the same detrimental mixing dynamics observed for the SRO-SGB (i.e., material backup, screw failures) were observed for the CS-SGB condition. No composition results or dynamic mixing videos were captured for this condition.

The CS particles are fluffy and stringy, making them susceptible to electrostatic effects. Due to this, the dynamic mixing videos of the CS-SGB condition suffer a slightly
reduced visibility as the smallest CS particles stuck to the inside surface of the double screw mixer. However, it is still readily apparent, especially in the counter-rotating down pumping screw rotation orientation conditions, that the CS-SGB condition offers good mixing (Figure 8e and 8f). The even distribution of CS throughout the system is noticeably higher for counter-rotating down pumping screw rotation conditions featuring larger dimensionless screw pitches than small dimensionless screw pitches. At the smallest dimensionless screw pitch \( p/D = 0.75 \), a clear vertical segregation of the CS and SGB is visible, as the CS particles rise to the top of the granular flow and are never mixed back in. As the dimensionless screw pitch is increased, this effect lessens and disappears entirely by the largest dimensionless screw pitch, \( p/D = 1.75 \) (i.e., Figure 4.8e and 4.8f), where the CS is evenly distributed throughout the system and is in constant motion.

The discrepancy between the visually poor mixing conditions observed in the smallest dimensionless screw pitch conditions and the quantitatively good coefficients of variation shown in the composition analysis for the same conditions can be explained by the vertical segregation of the granular flow. Visually it is apparent that the two granular materials are segregated and that the mixture is not homogenous. However, the dividing system of the double screw mixer’s outlet ports divides the outlet stream horizontally, and is unable to capture vertical slices of the flow. If the granular segregation of the two materials is evenly distributed horizontally, it will be evenly distributed across all four outlet ports, and the coefficient of variation will not reflect the vertical granular segregation. This most likely explains the seemingly good mixing as indicated by the composition analysis, and emphasizes the importance of paired optical visualization with composition analysis to thoroughly understand the actual mixing dynamics within the double screw mixer. Overall,
the counter-rotating down pumping screw rotation is a good mixing condition only at the two larger dimensionless screw pitches, as the smallest dimensionless screw pitch results in a vertically segregated mixture.

The one co-rotating screw rotation condition tested for the CS-SGB (Figure 4.8d) showed similar behavior to all other co-rotating conditions tested; the granular materials pushed into the left screw, where the SGB predominantly remained, while the lighter CS fell into the right screw, the two materials remaining segregated. As the granular flow progresses down the length of the double screw mixer the concentration of CS in the right screw increases significantly. Qualitatively, the co-rotating screw rotation orientation is a poor mixing condition for the CS-SGB, consistent with the quantitative composition results.

4.4.2.3 Cork trends

In Figure 4.9, the counter-rotating down pumping screw rotation orientation for the CO-SGB condition features a high sensitivity to dimensionless screw pitch. The lowest and highest dimensionless screw pitches, at all screw rotation speeds, show higher coefficients of variation than the mid-size dimensionless screw pitch, with higher screw rotation speeds resulting in progressively higher coefficients of variations. Note that this is the opposite of the usual trends with screw rotation speeds; typically a higher screw rotation speed yields a lower coefficient of variation. Overall, the CO condition features a high degree of sensitivity to both dimensionless screw pitch and screw rotation speed, and exhibits trends opposite those observed for other particles. Of the counter-rotating down pumping screw rotation conditions, the dimensionless screw pitch of \( p/D = 1.25 \) is the best mixing condition across all screw rotation speeds.
The one co-rotating screw rotation condition tested for the CO-SGB systems shows very low coefficients of variation, indicating it may be a good mixing condition.

The one counter-rotating up pumping screw rotation orientation condition was abandoned after the same detrimental mixing dynamics observed for the SRO-SGB (i.e., material backup, screw failures) were observed for the CO condition. No composition results or dynamic mixing videos were captured for this condition.

Similar to the CS particles, the CO particles are very fluffy, and extremely susceptible to electrostatic effects. Due to this, the composition results may have a significant amount of induced error as the CO particles had a high tendency to coat anything they contacted, including the inside of the outlet tubes and sample collection bags. As the samples were collected and handled, more and more CO was successively lost as it clung to surfaces, possibly skewing the final composition results towards higher percentages of glass beads than may have originally been in the outlet sample. Furthermore, the dynamic mixing videos of the CO conditions suffer a significantly reduced visibility as the CO particles coated the inside surface of the double screw mixer, sometimes obscuring up to 80% of the inside surface. Because of this, observations from dynamic mixing videos of the CO conditions are limited.

For the CO-SGB conditions, the counter-rotating down pumping screw rotation orientation appears to be a very good mixing condition (Figure 4.8h and 4.8i). The CO particles are so evenly distributed throughout the system that the mixture appears almost one solid color, with little visual differentiation between the SGB and CO particles. This observation is present for all dimensionless screw pitches and all screw rotation speeds. The CO is very thoroughly mixed into the glass beads with no segregation or CO agglomerations
visible in any of the dynamic mixing videos. Even the one co-rotating condition tested with the CO particles (Figure 4.8g) visually displays good mixing, with the CO particle concentrations very evenly distributed between the left and right screws. More of the granular flow is present in the left screw, due to the screw rotation orientation, but the concentration of CO in the left and right screws visually appears to be similar.

While the co-rotating screw rotation orientation condition’s dynamic video supports the composition analysis results—that the condition is a good mixing condition—the counter-rotating down pumping screw rotation orientations’ dynamic mixing videos do not show any indication of the dimensionless screw pitch or screw rotation speed sensitivity that the composition results indicate. This may be due to the difficulty in collecting and handling the cork samples, and the resulting possible skewing of the composition results. However, the consistency of the trends seen in the counter-rotating down pumping screw rotation orientation composition results, and the close clustering of the three data points for most operating conditions, indicate that the results are indicative of actual mixing dynamics. It is most likely that the reduced visibility of the dynamic mixing videos is limiting the ability to detect these parameter sensitivities.

4.4.2.4 Particle density comparison

Figure 9 allows for direct comparison between the three different particle densities investigated. The SRO, with the highest density of the three particles tested, consistently has the lowest coefficients of variation for the counter-rotating down pumping screw rotation orientation. Only a few conditions of the CS, namely the \( p/D = 0.75 \) dimensionless screw pitch at screw rotation speeds of \( \omega = 40 \) and 60 rpm, have lower coefficients of variation. Furthermore, the SRO coefficients of variation are all lower than the CO coefficients of
variation. That is, the particle with the lowest density consistently had the highest coefficient of variation. The CO particle is also the only one to display a high degree of sensitivity to changes in dimensionless screw pitch and especially to changes in screw rotation speed, while the SRO and CS particle results are stable across all dimensionless screw pitches and screw rotation speeds. For the one co-rotating condition tested, the trends from the counter-rotating down pumping appear to be reversed, as now the CO shows the lowest coefficient of variation and the CS and SRO feature higher coefficients of variation.

Qualitatively, the three particles behaved quiet similarly. Overall, all three exhibited good mixing conditions for the counter-rotating down pumping screw rotation condition, however the CS did show unwanted vertical segregation of the two granular materials at the smallest dimensionless screw pitch. The CO displayed the best mixing for the co-rotating screw rotation condition with an even distribution of biomass in both the left and the right projections, something not seen in either the SRO or the CS tests. Unfortunately, optical visualization limitations due to electrostatic effects of the biomass materials limited some observations of the CS and, especially, the CO particle tests.

Overall, the conclusions from the density study are consistent with conclusions from previous studies (Hsiau and Chen, 2002; Yang, 2006) that found that an increase in the density ratio between particles increased mixture segregation. Even though there were small qualitative changes in mixing effectiveness observed with changing biomass particle densities, the changes in mixing effectiveness due to density changes were much less significant that the changes in mixing effectives due to changes in biomass particle size. This can be understood by looking at the density ratio changes compared to the particle size ratio changes. In the SRO condition, the particle size ratio was reduced from the 8.6:1 ratio of the
reference study to a 1.3:1 ratio, a sizable decrease. However, the magnitude of the changes in the particle density ratios between the three biomass particles tested was much less. The SRO-SGB condition had a density ratio of 0.60:1; the CS-SGB condition a 0.54:1 ratio; and the CO-SGB condition a 0.35:1 ratio. The changes in the density ratios were small, each being close to a 1:1 ratio to begin with. This explains why the changing biomass particle size, with its resulting large change in particle size ratio, had the greater effect on mixing than the changing biomass particle density, with its resulting small change in particle density ratios.

### 4.4.3 Particle Concentration

The reference study featured only one mass flow rate ratio between the heat carrier media and the biomass media, a 10:1 ratio. This study replicates the operating conditions tested in the reference study for four new mass flow rate ratios, 20:1, 30:1, 40:1 and 50:1. In each case, only the mass flow rate ratio was changed, the original LRO and SGB from the reference study were used in all cases.

Figure 4.10 shows the three-way interaction between screw rotation speed, dimensionless screw pitch and screw rotation orientation for all investigated conditions. All five mass flow rate ratios have been plotted together in Figure 4.10 to allow a direct comparison. The results of the 10:1 mass flow rate ratio are those of Kingston and Heindel (2014b). To keep the graph concise and readable, only averaging lines of the data points are provided in Fig. 4.10.
Figure 4.10: The coefficient of variation as a function of the three way interaction plot between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the LRO-SGB mass flow rate ratio tests. To aid visualization, only the averaging line of each test condition is shown. The reference data (10:1 condition) are from Kingston and Heindel (2014b).

The counter-rotating down pumping screw rotation conditions for the 10:1 mass flow rate ratio featured the greatest mixing effectiveness, regardless of other parameters, as evident by the black lines for the counter-rotating down pumping conditions having generally lower coefficients of variation than the black lines (or dots) of the other screw rotation orientations in Figure 4.10. This trend was observed in each higher mass flow rate ratio tested; quantitatively, the counter-rotating down pumping screw rotation orientations...
continued to provide the best mixing of the three screw rotation orientations tested, confirming the results of Kingston and Heindel (2014b).

However, increasing the mass flow rate ratio increased the double screw mixer’s sensitivity to changes in screw rotation speed and dimensionless screw pitch. In the 10:1 mass flow rate ratio, the counter-rotating down pumping screw rotation condition showed little change in the coefficients of variation across changes in screw rotation speed and dimensionless screw pitch, indicating that mixing effectiveness was not affected by such changes. However, the higher mass flow rate ratios show a much greater variability in the counter-rotating down pumping screw rotation conditions across changes in screw rotation speed and dimensionless screw pitch. This indicates that as the mass flow rate ratio is increased, the mixing effectiveness of the double screw mixer is more dependent on screw rotation speed as well as dimensionless screw pitch.

The 10:1 mass flow rate ratio is the best mixing condition as it generally offers the overall lowest coefficients of variation. However, select counter-rotating up pumping conditions, specifically at 20:1 and 30:1 ratios, exhibit slightly lower coefficients of variation, and thus better mixing, for this orientation. A slight advantage for the higher mass flow rate ratios was also observed in the counter-rotating down pumping screw rotation orientations at dimensionless screw pitches of p/D=1.25 and screw rotation speeds of ω=40 and 60 rpm. Especially at ω=60 rpm, the higher ratios consistently offer lower coefficients of variation than the 10:1 ratio, with the 50:1 ratio offering the lowest coefficient of variation and thus the greatest mixing effectiveness. However, for these operating conditions the actual difference between mass flow rate ratios is within the error bars (discussed in Section 4.3.6).
The trends observed in the quantitative three-way interaction plot were also qualitatively observed in the dynamic mixing videos, of which snapshots are provided in Figure 4.11. Again, the 10:1 mass flow rate ratio condition is from Kingston and Heindel (2014b). In Figure 4.11, the materials travel from left to right, the LRO appears brown and the SGB appear grey. However, due to the reduction of LRO in the system at higher mass flow rate ratios, the LRO is difficult to visualize in some of the snapshots. Only one operating condition is represented in Figure 4.11, shown across all mass flow rate ratios. Even though only one set of snapshots is provided, conclusions presented here are based on thorough analysis of all recorded dynamic mixing videos.

Operating Condition
- Screw rotation orientation: Counter-rotating down pumping
- Screw rotating speed: \( \omega = 60 \, \text{rpm} \)
- Dimensionless screw pitch: \( p/D = 1.75 \)

Figure 4.11: Snapshots of the dynamic mixing process across all five mass flow rate ratios for one operating condition. The 10:1 mass flow rate ratio, a), is from Kingston and Heindel (2014b).

At each mass flow rate ratio, the counter-rotating down pumping screw rotation orientations showed better mixing than the other two screw rotation conditions tested. The counter-rotating down pumping condition videos, of which one condition is shown in Figure 4.11, showed an even distribution of LRO throughout the system with no agglomeration accumulation. The counter-rotating up pumping and co-rotating screw rotation condition
videos, however, showed large agglomerations of LRO on the tops and sides of the flow. These agglomerations of LRO are due to the natural tendency of the two materials to segregate and demonstrate poor mixing.

Due to the nature of the higher mass flow rate ratios, very little LRO was present in the system at the higher ratios. This made optical visualization, especially at the higher mass flow rate ratios, limited because of the preponderance of SGB in the system. Because of this, the conclusions of the composition results—that increasing the mass flow rate ratio increases the systems sensitivity to screw rotation speed and dimensionless screw pitch, and overall, decreases mixing effectiveness—can be neither supported nor unsupported by the dynamic mixing videos, although the dynamic mixing videos do support that the counter-rotating down pumping condition is the best screw rotation condition for all mass flow rate ratios.

4.5 Conclusions

This study investigated the effect of particle size, density and concentration on the mixing effectiveness in a double screw mixer. Using a study performed by Kingston and Heindel (2013; 2014b) as a reference study, SRO and LGB were investigated to determine the effects of biomass and heat carrier particle size on mixing dynamics. To determine the effects of particle density, three biomass particles, SRO, CS, and CO, were investigated, each of the same particle size range, but with different true densities. Lastly, to determine the effects of particle concentration five biomass inlet concentrations of 9%, 4.7%, 3.2%, 2.4% and 1.9%, with the LRO and SGB, were investigated.

Decreasing the biomass particle size greatly increased the mixing effectiveness in the double screw mixer, in almost all conditions tested, especially the counter-rotating down pumping screw rotation orientations at larger dimensionless screw pitches. However, the
counter-rotating up pumping screw rotation condition with the SRO-SGB exhibited very detrimental mixing dynamics that resulted in screw jams and system failures. Overall, decreasing the biomass particle size greatly increased the mixing effectiveness within the double screw mixer because it essentially removed the issue of differing granular material size, bringing the particle size ratios between the biomass material and the heat carrier media from the 8.6:1 ratio of the reference study to a 1.3:1 ratio with the SRO.

Increasing the heat carrier particle size reduced the mixing effectiveness within the double screw mixer, especially for counter-rotating down pumping screw rotation orientations with large dimensionless screw pitches. While the larger heat carrier media, paired with the LRO did reduce the particle size ratio from 8.6:1 to 3.8:1, the reduction in particle size ratio was not as significant as the SRO-SGB condition and most importantly, the increased LGB particle size increased the inertial forces of the glass beads, resisting the low density LRO’s ability to penetrate the bulk of the glass beads leading to vertical segregation of the mixture.

The particle density tests revealed that a biomass particle density closer to that of the heat carrier particle yields a better mixing condition. The biomass particle/heat carrier combination that resulted in the greatest density difference between the two particles exhibited the worst mixing conditions, at least quantitatively. Qualitatively, this was not apparent in the optical visualization as all three particles showed generally good mixing dynamics. Overall, the changes observed in mixing effectiveness for changes in particle density were much less than the changes observed for changes in particle size. This is likely due to the fact that changes in size ratios achieved in the particle size tests were much greater than the changes in density ratios achieved in the particle density tests. In the biomass
particle size tests, a reduction in the particle size ratio from 8.6:1 to 1.3:1 was achieved; but with the density tests, the density ratios only varied from 0.60:1 for the red oak tests, to 0.35:1 for the cork tests.

Quantitative composition results from the biomass inlet concentration tests indicate that the mixing effectiveness decreased as the biomass inlet concentration decreased, although qualitative visual analysis was neither able to confirm nor deny this due to limited visual capabilities. Composition analysis further indicates that decreasing the biomass inlet concentrations increases the system’s sensitivity to changes in dimensionless screw pitch, and, that the counter-rotating down pumping remains the best screw rotation condition for all mass flow rates tested. Even though it was concluded that higher mass flow rate ratios result in a generally decreased mixing effectiveness, if higher mass flow rate ratios are required, select operating conditions, specifically the counter-rotating down pumping screw rotation condition at a screw speed of $\omega=60$ rpm and a dimensionless screw pitch of $p/D=1.75$, do offer good mixing effectiveness at higher mass flow rate ratios.

Overall, when optimizing a double screw mixer, particle size and concentration effects should be given greater consideration than particle density effects as greater increases in mixing effectiveness can be achieved with changes in particle size and mass flow rate ratios, for the conditions of this study.

4.6 Acknowledgments

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CHAPTER 5. EFFECT OF MIXER SCALE ON MIXING IN A DOUBLE SCREW PYROLYZER

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5.1 Abstract

Granular mixing processes are common throughout the industrial world, seen in the agricultural, pharmaceutical, mineralogical, and bioenergy industries, among many others. These processes seek a high degree of control and customization, while maintaining the quality and homogeneity of the final product. However, granular flows have a very complex rheology, a high tendency to segregate, and the mixing mechanisms that govern their behavior are not very well understood. Extensive research into granular flows and mixers is vitally important to understanding the mixing process, improving the design of mixers, and ensuring the quality of the products. Most granular mixing studies thus far conducted have focused on laboratory scale mixers, which are not applicable to true industrial applications that need large scale mixers to improve the economic viability of the processes.

Double screw pyrolyzers, featuring twin rotating screws, can be used to convert biomass into bio-oil via fast pyrolysis, a granular mixing process. Understanding the mixing dynamics within a double screw pyrolyzer is crucial to maximizing bio-oil yields. Increasing the size of the pyrolyzer may be another way to increase the volume of bio-oil produced.

This study aims to investigate the effect of mixer scale on mixing effectiveness in a cold-flow double screw pyrolyzer. Select operating conditions featuring changes in screw...
rotation speed, dimensionless screw pitch, and screw rotation orientation, are compared for three double screw mixers of differing scales. Conclusions from this study provide additional insights into pyrolyzer scale-up and the mixing dynamics in larger scale granular mixers. Advanced 360° optical visualization techniques, paired with non-invasive composition analysis, indicates that for counter-rotating down pumping screw rotation conditions, increasing the scale of the mixer decreases the mixing effectiveness of the system. The up-pumping screw rotation orientation remains a poor mixing condition at all mixer scales. And, for a co-rotating screw rotation condition, increasing the mixer scale from 1x to 1.5x greatly increased the mixing effectiveness, but further increasing the mixer scale to 2x decreased the mixing effectiveness back down to that of the 1x scale mixer.

5.2 Introduction

Granular mixing processes are common throughout the industrial world, seen in the agricultural, pharmaceutical, mineralogical, and bioenergy industries, among many others (Bridgwater, 2012; Mohan et al., 2006). These processes often seek a high degree of control and customization, while maintaining the quality and homogeneity of the final product (Ottino and Khakhar, 2001). However, granular flows have a very complex rheology, behaving like both a solid and a liquid. Therefore, the mixing mechanisms that govern their behavior are complicated and, at this point, not very well understood (Bridgwater, 2012; Campbell, 2006; Weinekötter, 2000). Granular flows also have a high tendency to segregate due to differences in particle characteristics (Hogg, 2009; McCarthy, 2009; Pernenkil and Cooney, 2006). All of these factors complicate the design of granular mixing processes. Extensive research into granular flows and mixers is vitally important to understanding the mixing process, improving the design of mixers, and ensuring the quality of the products.
While granular mixing has been studied since the 1950’s, intensive research only truly began in the last decade of the 20th century (Bridgwater, 2010). Since that time, considerable research has been done into granular mixers, including bladed mixers (Conway et al., 2005; Remy et al., 2010), vertical shakers (Hsiau and Chen, 2002; Yang, 2006), single screw mixers (Tsai and Lin, 1994; Uchida and Okamoto, 2006, 2008), and rotating cylinders (Aït Aissa et al., 2010). Several literature reviews are available on the subject (Bridgwater, 2012; Campbell, 1990; Campbell, 2006; Ottino and Khakhar, 2000). However, these studies often focused on laboratory scale mixers with relatively simple geometries and idealized operating conditions. In true industrial applications, the mixers are often much larger, much more complex, and feature a wider range of interacting operating parameters. Further complicating matters, granular mixing processes are highly system sensitive, and changes in any one parameter can have dramatic effects on the quality of the resulting products. Because of this, it is vitally important that research is conducted with more intricate granular mixers featuring complex geometries and operating conditions (Hogg, 2009).

One additional matter still complicating granular mixing design and process implementation is that most granular mixing studies thus far conducted have focused on laboratory scale mixers which are not applicable to true industrial applications. Even though various researchers have studied scale-up laws using models and simulations of granular mixers (Mort, 2005, 2009; Rahmanian et al., 2008; Yijie et al., 2013) and Bridgwater (2012) and Paul (2004) provide good reviews of some general scaling rules for specific mixer geometries, there are currently no sound scale-up laws concerning granular mixers. Those that do exist have not been experimentally tested (Bridgwater, 2012). Industrial applications need large scale mixers to improve the economic viability of the processes, but the quality of
the product must still be assured (Mort, 2005). This is why extensive experimental research is still needed regarding the mixing dynamics of larger scale mixers, in order to provide usable criteria to aid in the future design of industrial scale operations (Paul et al., 2004).

A double screw pyrolyzer, featuring twin rotating screws, is one example of a complex industrial mixer in which biomass is converted into bio-oil, via fast pyrolysis (Ingram et al., 2007). Fast pyrolysis is a relatively new technology, but bio-oil has the potential to be a source of fuel via direct combustion, to be upgraded into transport fuels, or to be converted into a wide variety of bio-based chemicals (Bridgwater, 2003; Brown, 2011b; Mohan et al., 2006). Fast pyrolysis is a granular mixing process in which a heat carrier media is mixed with a biomass material in order to heat and pyrolyze the biomass to produce bio-oil (Brown and Brown, 2012). Most recent work with double screw pyrolyzers has focused on the products and not the mixing dynamics within the mixers (Bahng et al., 2009; Ingram et al., 2007). However, as the rate of heat transfer between the two granular materials is directly correlated to bio-oil yields, and increasing the heat transfer rates increases the bio-oil yields, understanding the mixing dynamics within a double screw pyrolyzer is crucial to maximizing bio-oil yields. Kingston and Heindel (2015; 2014b, c) began to fill this gap by investigating the effect of select operating conditions on the mixing effectiveness with a cold-flow double screw pyrolyzer. Kingston and Heindel developed measurement techniques to determine the mixing effectiveness of various operating conditions, featuring varying screw rotation speeds, dimensionless screw pitches, screw rotation orientations, and material injection configurations. Results from Kingston and Heindel (2014b) have improved the understanding of the mixing dynamics within a double screw pyrolyzer and have provided key insights into the important parameters which determine the mixing effectiveness.
One major factor limiting the viability of fast pyrolysis is the rate at which bio-oil is produced. Most of the existing research into fast pyrolysis has focused on laboratory scale mixers which do not produce great enough volumes of bio-oil to be economically viable (Bridgwater, 2007). Increasing the size of the pyrolyzer may be one way to increase the volume of bio-oil produced. However, because the process of fast pyrolysis is a granular mixing process, the mixing effectiveness of larger double screw pyrolyzers is unknown. Therefore, experimental studies are needed to investigate the mixing dynamics in larger scale double screw mixers, so that the improved understanding of the mixing dynamics can be used in future scale-up studies of fast pyrolysis to improve the viability of bio-oil production.

This study aims to expand on work completed by Kingston and Heindel (2014b, c), by investigating the effect of mixer scale on mixing effectiveness in a cold-flow double screw pyrolyzer. Select operating conditions investigated by Kingston and Heindel (2013; 2014b), featuring changes in screw rotation speed, dimensionless screw pitch, and screw rotation orientation, are repeated for two larger scale double screw mixers, one 1.5x times the original mixer scale, as designed by Kingston and Heindel (2014c), and one 2x scale. In this study, red oak chips are used as the biomass material, and glass beads are used as the heat carrier media. Advanced 360° optical visualization techniques, paired with non-invasive composition analysis, are used to determine the mixing effectiveness of the larger scale mixers. Conclusions from this study provide additional insights into pyrolyzer scale-up and the mixing dynamics in larger scale granular mixers.
5.3 Experimental Procedures

The screw mixers and experimental procedures used in this study are very similar to those developed by Kingston and Heindel (2013; 2014c). Only a summary is provided here.

5.3.1 Experimental setup

The cold-flow double screw pyrolyzer design used in these studies features two horizontally mounted, intermeshing, noncontact screws, and is shown in Figure 5.1. The system will hereafter be referred to as a double screw mixer. The original reference scale mixer was designed by Kingston and Heindel (2013; 2014c), and featured screws of diameter $D = 2.54$ cm (1 in), defined as the characteristic length of the system, and an effective mixing length of $L = 25.4$ cm (10 in), with $L/D = 10$.

Figure 5.1: The cold flow, laboratory scale pyrolyzer design used in these studies (referred to as a double screw mixer) with a cross-sectional view of the unique outlet ports that allows the outlet stream to be divided for sample composition analysis (Kingston and Heindel, 2014c). The double screw mixer can operate at three screw rotation orientations: co-rotating (CoR), counter-rotating up pumping (CtrR UP), and counter-rotating down pumping (CtrR DP).
To study the effects of mixer scale on mixing dynamics, this study designed and analyzed two larger scale double screw mixers based on the reference design by Kingston and Heindel (2013; 2014c). The characteristic length of the system, defined as the outside screw diameter, was chosen as the scaling parameter and was used to scale up all other components of the double screw mixer. Therefore, the 1.5x scale mixer features a characteristic length of $D = 3.81$ cm (1.5 in), and the 2x scale mixer features a characteristic length of $D = 5.08$ cm (2 in). The original 1x scale mixer featured a mixing length of $L = 25.4$ cm (10 in), ten characteristic lengths, and thus the 1.5x scale mixer features a mixing length of $L = 38.1$ cm (15 in) and the 2x scale mixer features a $L = 50.8$ cm (20 in).

Both the 1.5x and 2x scale mixers feature two material injection ports, axially located two characteristic lengths apart on the top of the mixer, positioned laterally halfway between the two screws. The two granular materials are fed into the injection ports separately from two Tecweigh CR5 volumetric auger feeders. The outlet port of the screw mixers feature a unique division system that allows the entire outlet stream to be spatially divided into four sections, thus enabling non-invasive composition analysis.

Both the 1.5x and 2x double screw mixers were manufactured using 3D printing technology. The housing of the mixers was printed from a rigid, clear designer plastic material which allows complete $360^\circ$ optical access to the internal structure of the screw mixer. The supporting structures and screws were printed out of a strong, opaque plastic. All three double screw mixers, with a selection of screws for each scale, are shown in Figure 5.2.
Figure 5.2: The three double screw mixers with a selection of screws from each scale, shown against a standard 12 inch ruler.

This study investigated three dimensionless screw pitches (p/D = 0.75, 1.25, and 1.75), three screw rotation speeds (ω = 20, 40, and 60 rpm), and three screw rotation orientations (co-rotating (CoR), counter-rotating up pumping (CrtR UP), and counter-rotating down pumping (CrtR DP)). The screw rotation orientations are schematically shown in Figure 5.1.

5.3.2 Granular materials

Two granular materials were used in this study, red oak chips as the model biomass (1.53 g/cc, 500 – 6350 μm) and glass beads as the model heat carrier (2.50 g/cc, 300 – 500 μm). The red oak chips were refined down to their specified size range using a combination of hammer mills and hand sieves. The glass beads were used as the heat carrier, instead of the more traditionally used sand, because the abrasiveness of sand would scratch the clear, plastic housing and render optical visualization impossible. The glass beads used in this study have a true density within 5% of a typical sand and are 95% spherical, making them a good substitute for sand in this study. The glass beads were purchased in their specified size range. The true density of each material was measured using a Quantachrome Pentapyc 5200e gas pycnometer. The two materials are shown in Figure 5.3.
5.3.3 Operating conditions

Kingston and Heindel (2013; 2014b) investigated 54 operation conditions, featuring all possible parametric combinations of three screw rotation speeds, three screw rotation orientations, three dimensionless screw pitches and two material injection configurations. Based on conclusions presented by Kingston and Heindel (2013; 2014b), 11 select operating conditions were chosen to be replicated in this study, using the larger scale mixers. Table 1 outlines the selected operating conditions and their specific parameters. As Kingston and Heindel (2013; 2014b) concluded that the counter-rotating down pumping screw rotation
orientation is the most effective of the three screw rotation conditions investigated, this study focuses primarily on investigating all possible operating conditions with this screw rotation orientation.

Table 5.1: Select operating conditions investigated in this study.

<table>
<thead>
<tr>
<th>Operating Conditions</th>
<th>Screw Rotation Orientation</th>
<th>Screw Rotation Speed $\omega$ [rpm]</th>
<th>Dimensionless Screw Pitch $p/D$ [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Counter, Up Pumping</td>
<td>Counter, Up Pumping</td>
<td>20</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td>Co-rotating</td>
<td>40</td>
<td>1.25</td>
</tr>
<tr>
<td>Counter, Down Pumping</td>
<td>20</td>
<td>0.75</td>
<td></td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>1.25</td>
<td></td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0.75</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.25</td>
<td></td>
</tr>
</tbody>
</table>

Additionally, this study investigates one co-rotating screw rotation orientation, with a screw rotation speed of $\omega = 40$ rpm, and a dimensionless screw pitch of $p/D = 1.25$. This was chosen as it represents the industry reference used by Kingston and Heindel (2013; 2014b). One counter-rotating up pumping screw rotation orientation with a $\omega = 20$ rpm screw speed and $p/D = 0.75$ dimensionless screw pitch was also investigated, determined by Kingston and Heindel (2013; 2014b) to be a poor mixing condition.

As material injection configuration was previously determined to have little effect on the mixing dynamics, only one material injection configuration was used in this study—biomass injection port 1, heat carrier injection port 2. For all tests, the mass flow rate ratio between the heat carrier and the biomass was maintained at a 10:1 ratio. To achieve optimal mixing conditions and avoid problematic effects, a 65% fill level was maintained for all tests.
(Colijn, 1985), except where limitation from the volumetric feeders resulted in an under
filled system (see Section 5.4.2.2).

### 5.3.4 Optical visualization

To capture the dynamic mixing process within the double screw mixer, the methods
developed by Kingston and Heindel (2014c) are used.

To simultaneously capture the dynamic mixing process with the double screw mixer
from four independent views, four Panasonic HC-V700M high definition cameras are placed
above, below, and to each side of the screw mixer. The video cameras each feature a 1920 ×
1080 resolution and a frame rate of 60 frames per second. The cameras are placed 38.1 cm
(15 in) from the surface of the 1.5x scale screw mixer, and 50.8 cm (20 in) from the surface
of the 2x scale screw mixer. Eight compact 85 watt florescent lamps (providing 5300
lumens) are placed on the ends of the screw mixers, four at each end, to illuminate the
mixing region. To minimize glare from ambient light, the entire experimental setup is
encased in a canopy of black fabric, and all metal components of the feeders and supporting
structure are similarly covered in black fabric.

The four independent video projections are then combined into one comprehensive
dynamic mixing video using Adobe Premiere Pro CS6 video editing software, comprising
360° of the screw mixers granular flow. In Adobe Premiere Pro CS6, each captured video is
cropped so that only the defined mixing region of each projection is shown, and inverted (in
the case of the left and bottom projections), so that the granular flow travels from left to right
in all projections. An audio spike is used to temporally synchronize all four video
projections. Figure 5.4 shows how each projection fits into the final dynamic mixing video.
5.3.5 Composition analysis

One of the most common ways to measure the mixing effectiveness of a granular flow is by determining the homogeneity of the mixture. A double screw mixer with a high degree of mixing effectiveness would have an even distribution of biomass and heat carrier throughout the entire system, giving the mixture a high degree of homogeneity. Mixture homogeneity was determined by the method developed by Kingston and Heindel (2014b).

The unique outlet port design of the screw mixers allows the entire outlet stream to be collected, whilst in motion, thus satisfying Allen’s two golden rules of sampling (Allen, 1997). Samples are collected from the screw mixer once the system has achieved steady state operating conditions. The samples from the four outlet ports are collected simultaneously, and to ensure repeatability, three sets of samples are collected for each operating condition.
The composition of each sample is then determined and compared in order to gauge the mixture homogeneity. To determine the samples’ composition, the true density of the samples are first calculated using pycnometer and mass measurements. Due to the large volume capacity of the large mixers and the small fixed volume of the pycnometer sample chamber, each sample collected from the outlet ports needs to be first separated into subsamples to fit into the pycnometer sample chamber. Each subsample is then individually run through the pycnometer. The pycnometer calculates the true volume of each subsample. The resulting true volumes are then summed together, along with the subsample masses, and used to calculate the original sample’s true density. An empirical correlation between mixture density and composition, a method used by Kingston and Heindel (2013; 2014c), is then used to determine the sample composition from the true density. The mixture composition is defined as the glass bead mass fraction and ranges from one to zero.

Statistical parameters are then used to compare the four outlet port samples to each other, as well as to the samples from other operating conditions. The mass weighted composition variance, $s^2$, of the four outlet ports:

$$s^2 = \frac{\sum_{i=1}^{n} m_i (x_i - \bar{x_w})^2}{(N - 1) \frac{1}{N} \sum_{i=1}^{n} m_i}$$

(5.1)

where $n$ is the number of the $i^{th}$ sample, $m_i$ is the total mass of the $i^{th}$ sample, $x_i$ is the measured composition of the $i^{th}$ sample, $\bar{x_w}$, is the mass weighted mean composition of the samples, and $N$ is the total number of samples, is first calculated. The mass weighted composition variance is used instead of a standard composition variance because the mass flow rates through each of the four outlet ports may not be equal, and thus the standard composition variance, if not weighted by mass, would be skewed.
To gauge mixture homogeneity, the mass weighted composition variance was then used to calculate the coefficient of variation, CV:

\[
CV = \frac{s}{\mu} \tag{5.2}
\]

where \( s \) is the standard deviation determined by the square root of Equation (5.1), and \( \mu \) is the theoretical mean of the outlet composition, which, in an ideally mixed system, equals the inlet mixture composition. The coefficient of variation is a dimensionless parameter that allows the variability in a series of numbers to be independently compared, regardless of the units or magnitude of the individual measurements. A smaller CV indicates a more homogeneous mixture and greater mixing effectiveness. A CV of zero is, thus, the desired result in a mixing process.

5.3.6 Uncertainty analysis

To determine the amount of uncertainty associated with computing the coefficient of variation, the standard error of the coefficient of variation is used. This empirically calculated error represents the maximum possible error in the data set (Devore, 2014). The calculated standard errors range from 0.04 to 0.18. Therefore, the error bars, when plotted on the three-way interaction plot, often overlapped each other. This indicates that our ability to differentiate one set of results from another may be limited. However, close analysis of the individual data points indicate that, in most cases, the data points are clustered together and remain distinct from other sets of data points, indicating that the trends observed in the averaging lines in the following figures are consistent and are a good representation of the actual mixing behavior.
5.4 Results and Discussion

In all figures, tables, and results that follow, the 1.5x and 2x scale screw mixer results are compared to results originally presented by Kingston and Heindel (2013; 2014b), featuring a D = 2.54 cm (1 in) double screw mixer. This study will henceforth be identified as the 1x scale mixer study. Kingston and Heindel’s composition analysis results were presented only as a mass weighted composition variance, $s^2$, not as a coefficient of variation, CV. Their original results have been converted, with permission, to the coefficient of variation used in this study, in order to allow for direct comparison to the results present here.

5.4.1 Screw mixer: 1.5x

5.4.1.1 Composition Analysis

Figure 5.5 shows the three-way interaction between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the three double screw mixers. The 1x scale mixer results, from Kingston and Heindel (2014b), are plotted in black, the 1.5x scale results in blue and the 2x scale results in red. Recall that a lower coefficient of variation indicates better mixing—a coefficient of variation of zero is a perfectly homogeneous mixture and is the desired state. For each operating condition three data points are plotted, representing the set of three samples collected for each operating condition to ensure repeatability. To aid the visualization of trends, an averaging line had been applied to each set of three samples and across operating conditions. The error bars represent the standard error in the average measurement (Section 5.3.6).
Figure 5.5: The coefficient of variation as a function of the three-way interaction between screw rotation orientation, dimensionless screw pitch, and screw rotation speed for the three screw mixer scales, 1x black, 1.5x blue, 2x red. The 1x scale reference data are from Kingston and Heindel (2014b).

For the counter-rotating down pumping screw rotation conditions, the 1.5x double screw mixer’s composition results consistently reveal low coefficients of variation for the largest dimensionless screw pitch, p/D = 1.75, compared to the other two dimensionless screw pitches. However, the 1.5x system shows sensitivity to changes in the dimensionless screw pitch, as the p/D = 0.75 and 1.25 pitches, especially at lower screw rotation speeds, show fluctuating, often high, coefficients of variation.
The one counter-rotating up pumping condition and the one co-rotating condition tested both display very low coefficients of variation for the 1.5x system. This indicates these conditions may be good mixing conditions, as the low coefficients of variation of the outlet stream indicate the system is fairly homogeneous, at least horizontally.

5.4.1.2 Optical Visualization

Dynamic mixing videos of the 1.5x scale screw mixer, for all 11 operating conditions shown in Table 5.1, were collected, and conclusions presented here are derived from all 11 dynamics mixing videos. However, snapshots of only three select videos are shown in Figure 5.6 b, e, and h.

The counter-rotating down pumping screw rotation orientation videos show differing mixing dynamics depending on the dimensionless screw pitch, indicating that the 1.5x scale mixer is sensitive to changes in dimensionless screw pitch. This is in agreement with the composition results. At the smallest dimensionless screw pitch, an example of which is shown in Figure 5.6b, a layer of red oak particles forms between the wall of the screw mixer housing and the screw flightings, essentially eliminating these particles from the mixing potential of the granular flow. However, this particle layer forms during system start up, and once the screw mixer has reached a steady state, the particle layer does not changeover with new particles. Thus, while the particle layer formation initially disrupts the granular flow, once its formation is complete, the layer no longer has an active effect on the mixing dynamics. Elsewhere in the system, the red oak particles are in constant motion, and are visible in all four projections. However, the red oak particles do tend to ride on top of the granular flow for prolonged periods of time before eventually being pulled back into the mixture. This vertical segregation is likely due to the difference in biomass and heat carrier
particle size and density. The particle layer effect and prolonged red oak presence on top of the flow was seen in all counter-rotating down pumping conditions involving the p/D = 0.75 dimensionless screw pitch, at screw rotation speeds of \( \omega = 20, 40, \) and 60 rpm.

Counter-rotating down pumping, p/D = 0.75, \( \omega = 40 \) rpm

[Images of dynamic mixing process for select operating conditions from all mixer scales. The 1x scale reference study images are from Kingston (2013).]

Counter-rotating down pumping, p/D = 1.75, \( \omega = 20 \) rpm

The p/D = 1.25 dimensionless screw pitch conditions, for the counter-rotating down pumping screw rotation orientation, show different mixing behaviors than the p/D = 0.75 dimensionless pitch conditions. The particle layer effect seen with the smallest pitch is no longer present for the p/D = 1.25 pitch, at any screw rotation speed. The agglomerations of red oak on the top of the granular flow are still present and tend to clump together before and
after the screw flightings. The specific particles of red oak that make up these agglomerations are continuously changing over; however, they remain static on the surface of the flow for longer periods of time than other particles in the system.

The counter-rotating down pumping screw rotation conditions featuring the largest dimensionless screw pitch, $p/D = 1.75$, show similar mixing behavior to the $p/D = 1.25$ conditions, with a slightly better distribution of red oak particles throughout the system. An example is shown in Figure 5.6e. There is no particle layer formation along the mixer walls at any screw rotation speed, and while the red oak particles do form agglomerations on the top of the flow around the screw flightings, they are quickly pulled back into bulk of the flow.

The 1.5x screw mixer, for the counter-rotating down pumping screw rotation orientation, exhibits better mixing effectiveness as the dimensionless screw pitch is increased. This supports the results seen in the coefficients of variation for these operating conditions.

The one up-pumping screw rotation condition tested, which seems to be a good mixing condition according to the low coefficients of variation the composition results provided, is actually a visually poor mixing condition. The system has a moderate amount of vertical motion, with the biomass particles sifting down through the glass beads and then rising back up, indicating that some mixing is occurring. However, the process is very slow and the granular materials are being conveyed along more than they are being mixed together. The composition results only indicate that the distribution of particles is even across the horizontal plane, the results provide no information about the possible vertical segregation or the conveyor-like nature of the system. This is why it is important to combine
optical visualization with composition analysis, in order to fully understand the mixing dynamics within the system.

On the other hand, the dynamic mixing video from the one co-rotating condition tested, shown in Figure 5.6h, fully supports the conclusions from the composition results. The dynamic mixing video shows a very even distribution of red oak throughout the system. This alone is significant because the co-rotating screw rotation orientation, in any double screw mixer, causes the granular flow to be pushed into the left screw, leaving the right screw nearly empty. This usually results in significant horizontal segregation between the two granular materials—manifesting as a disproportionally large amount of red oak in the right screw and very little red oak in the left screw. However, with 1.5x scale screw mixer, while the bulk of the granular flow is still pushed into the left screw, there is very little particle segregation occurring. There is an even amount of red oak visible in the both projections, and very few agglomerations in the right screw. The dynamic mixing video of the co-rotating screw rotation supports the composition results, and shows that this condition has good mixing effectiveness.

5.4.2 Screw mixer: 2x

5.4.2.1 Composition Analysis

The composition results for the 2x scale mixer, shown in red in Figure 5.4, generally feature high coefficients of variation, especially at the lower screw rotations speeds for the counter-rotating down pumping screw rotation conditions. The coefficients of variation for the $\omega = 20$ and 40 rpm screw rotation speed conditions show a high sensitivity to changes in dimensionless screw pitch. The lowest screw rotation speed features high coefficients of
variation at the smallest dimensionless pitch, and then decreasing coefficients of variations as the dimensionless pitch increases. However, the middle screw rotation speed sees the lowest coefficients of variation at the smallest dimensionless screw pitch with increasing coefficients of variation as the dimensionless screw pitch increases. The highest screw rotation speed features coefficients of variation that are mostly unaffected by changes in dimensionless screw pitch.

The 2x scale mixer displays low coefficients of variation for the one counter-rotating up pumping condition tested indicating a potentially well mixed system. The one co-rotating condition tested, however, has high coefficients of variation, and likely remains a poor mixing condition.

5.4.2.2 Optical Visualization

Observations from all 11 dynamic mixing videos of the 2x scale mixer are used to form the conclusions presented here, but only snapshots from three select videos are shown in Figure 5c, f, and i.

The operating conditions featuring counter-rotating down pumping screw rotation orientations display differing mixing dynamics depending on the dimensionless screw pitch. For the smallest dimensionless screw pitch, p/D = 0.75, an example of which is shown in Figure 5.5c, the red oak particles form a stationary particle layer between the screw mixer housing and the screw flightings. This layer forms during system start up, and once the screw mixer has reached a steady state, it likely no longer has an effect on the mixing dynamics. Elsewhere in the system, the red oak particles are in constant motion and are visible in all four projections, although the red oak does tend to remain on the surface of the flow for long periods of time.
The middle dimensionless screw pitch, p/D = 1.25, for the down pumping screw rotation conditions, does not show any signs of a red oak layer forming between the screw flightings but the red oak does form agglomerations on the surface of the granular flow, especially before and after the screw flightings. At the higher screw rotation speeds, the size of the agglomerations does not change, but they begin to consist of red oak particles that remain stationary for longer periods of time, relative to the number of screw rotations.

For counter-rotating down pumping operating conditions featuring the largest of the three dimensionless screw pitches, p/D = 1.75, the \( \omega = 20 \) screw speed condition yields good mixing dynamics. There are still some red oak agglomerations present on the surface of the flow, but these are minimal and the individual particles quickly cycle back into the flow. The rest of the system shows an even distribution and constant motion of the red oak. The condition featuring a screw rotation speed of \( \omega = 20 \) rpm is shown in Figure 5.5f. The \( \omega = 40 \) and 60 rpm screw speed conditions, however, show a high concentration of red oak forming over the entire top surface of the flow, not just around the screw flightings. This leads to a high degree of vertical segregation in the flow and poor mixing. However, it is important to note that for these specific operating conditions, the 2x screw mixer is under filled due to limitations of the volumetric feeders. A fill level of 65% was intended for all conditions, but for these particular conditions only an approximate fill of 50% was achieved. Fill level is crucial to optimal mixing dynamics and an under filled system can be detrimental to the mixing effectiveness in a mixer (Colijn, 1985). Therefore, the poor mixing seen in the counter-rotating down pumping conditions at a screw speeds of \( \omega = 40 \) and 60 rpm, and dimensionless screw pitch of p/D = 1.75 for the 2x scale mixer is most likely the result of the under filled system, and not the stated operating conditions.
The dynamic mixing video for the one counter-rotating up pumping screw rotation orientation condition tested with the 2x scale mixer reveals fairly poor mixing, contrary to the results of the composition analysis for the same condition. While the composition results yield low coefficients of variation for this operating condition, the dynamic mixing video reveals that the seemingly even distribution of the two materials in the flow is only in the horizontal direction and not the vertical. The dynamic video reveals heavy vertical segregation between the two granular materials, with the red oak chips on top. There is some vertical changeover of particles, as the red oak is drawn down into the glass beads, but by and large, the majority of the red oak in the system resides on the top of the flow and the two granular materials are simply conveyed rather than mixed. This condition is revealed to be a poor mixing condition, and stresses the importance of combining composition analysis with optical visualization.

The one dynamic mixing video of the co-rotating screw rotation orientation condition tested with the 2x scale mixer, shown in Figure 5.5i, reveals poor mixing, in agreement with the composition results. The granular flow is pushed into the left screw, as is typical of the co-rotating screw rotation orientation. The red oak particles fall into the right screw and are unable to penetrate back into the left screw, resulting in the right screw having a high percentage of red oak particles, and thus, horizontal segregation of the two granular materials. Therefore, this operating condition for the 2x scale screw mixer is a poor mixing condition.
5.4.3 Mixer scale comparison

5.4.3.1 Composition Analysis

Direct comparison of the composition results from the three screw mixer scales reveals significant changes in the mixing dynamics as the screw mixer dimensions change. Kingston and Heindel (2013; 2014b) observed that the operating conditions involving the counter-rotating down pumping screw rotation orientation, for the 1x scale screw mixer, provided nearly universally good mixing conditions, shown by the low coefficients of variation. The counter-rotating down pumping conditions were largely unaffected by changes in dimensionless screw pitch and screw speed, with only the slowest screw rotation speed conditions, $\omega = 20$ rpm, showing any noticeable change in the coefficients of variations as the dimensionless screw pitch changed. The $\omega = 40$ and 60 rpm conditions’ coefficients of variation were very steady across changes in dimensionless screw pitch with the 1x scale mixer.

With the 1.5x and 2x scale screw mixers, however, the counter-rotating down pumping screw rotation conditions show some higher, fluctuating coefficients of variation, indicating unwanted flow dynamics negatively affecting the mixing effectiveness. The larger scale mixers show an increase in sensitivity to changes in dimensionless screw pitch, with the 1.5x scale slightly more sensitive than the 1x, and the 2x successively more sensitive than the 1.5x.

The nature of the sensitivity to changes in dimensionless screw pitch for the larger mixers, in terms of the resulting coefficients of variation, is not identical however. The two behave similarly for the $\omega = 20$ rpm screw rotation speed conditions, where the largest dimensionless screw pitch has the lowest coefficients of variation. However, for the $\omega = 40$
rpm conditions, the 1.5x scale screw mixer features decreasing coefficients of variation as the
dimensionless screw pitch increased, whereas the 2x scale mixer saw increasing coefficients
of variation as the dimensionless screw pitch increased.

In terms of magnitude, the 2x scale mixer consistently has some of the highest
coefficients of variation at each counter-rotating down pumping screw rotation condition,
with the 1.5x screw mixer regularly falling between the 1x and 2x scale mixers. The 1.5x
scale screw mixer has a few select operating conditions where it features lower coefficients
of variation, below that even of the 1x scale mixer, but at other conditions, it has the highest
coefficients of variation of the three scales. Overall, the composition analysis of the counter-
rotating down pumping screw rotation conditions, for the three scales, generally indicate that
increasing the scale of the screw mixer decreases the mixing effectiveness of the system and
increases the variability in mixing dynamics across changes in operating conditions.

The composition results for the one counter-rotating up pumping condition tested
with the two larger scale mixers show results compared to the 1x scale mixer, with slightly
lower coefficients of variation for the 2x scale mixer. The co-rotating system however, shows
changes in the coefficients of variation for both the 1.5x and 2x scale mixer when compared
to the 1x scale mixer results. The 2x scale mixer revealed slightly higher coefficients of
variation than the 1x scale mixer, indicating a decreased mixing effectiveness. However, the
1.5x scale mixer features lower coefficients of variation for this one condition, much lower
than the 1x scale results. This indicates that the mixing effectiveness of the system, from a
composition analysis standpoint, is increased at the 1.5x scale.
5.4.3.2 Optical visualization

Just as the composition results indicate, the 1.5x and 2x scale mixers display differing dynamic mixing behaviors than their 1x scale counterpart. The increased sensitivity to changes in dimensionless screw pitch is very apparent in the dynamic mixing videos of the larger scale mixers and some of the mixing phenomena seen in the larger mixers is unseen in the 1x scale mixer. At the smallest dimensionless screw pitch, the formation of the red oak particle layer that occurs between the sides of the housing and the screw flightings does not occur in any of the dynamic mixing videos for the 1x scale screw mixer. This film formation is most likely due to the increased clearance between the screw housing and the flightings, which was allowed to scale with the screw mixer scale. Therefore, the 1.5x scale mixer has 1.5 times the clearance as the 1x scale mixer and similarly with the 2x scale mixer. With the 1x scale mixer, the clearance (1590 μm) was smaller than the bulk of the red oak particles (500 – 6350 μm) and so only the smallest of the red oak particles could pass between the housing wall and the screw flightings. With the larger scale mixers the clearance increases (2388 μm and 3180 μm for the 1.5x and 2x scale mixer, respectively), allowing a much larger proportion of the red oak particles to pass between the housing and the screw flightings. Red oak particles that end up in this space remain there, likely due to electrostatic forces from the plastic housing holding them in place. As the screw flightings never come in contact with these particles, the electrostatic bonds remain in place.

Other than the film formation, the larger screw mixers see moderate red oak movement throughout the rest of the system for the p/D = 0.75 counter-rotating down pumping conditions, compared to the 1x scale mixer. However, the 1.5x and 2x scale mixers see an increase in the proportion of red oak that rides along the surface of the granular flow,
indicating that the larger scale mixers exhibit increased vertical segregation compared to the 1x scale mixer.

For the operating conditions with a p/D = 1.25 dimensionless screw pitch and a counter-rotating down pumping screw rotation orientation, the 1.5x and 2x scale screw mixers show large agglomerations of red oak particles forming in the surface of the granular flow around the screw flightings. This is a significant development, as there is very few red oak agglomerations visible on the surface of the 1x scale screw mixer. Only at the highest screw rotation speed, ω = 60 rpm, does the 1x scale mixer start to see small agglomerations forming on the surface of the flow. The larger mixers, however, see large formations of red oak clusters on the surface, with the 2x scale mixer seeing larger formations and a longer residence time of the individual particles on the surface of the flow than the 1.5x scale mixer. The two larger scale mixers for the p/D = 1.25 conditions show increased segregation and therefore decreased mixing effectiveness when compared to the original 1x scale mixer.

For the counter-rotating down pumping condition with a screw pitch of p/D = 1.75 and a screw rotation speed of ω = 20 rpm, the 1.5x and 2x scale mixer display good mixing dynamics similar to the 1x scale screw mixer. The red oak is well distributed throughout the system in both the 1.5x and 2x scale mixers, and there are minimal red oak agglomerations on the surface of either mixer. The ω = 40 and 60 rpm conditions for the 1.5x scale mixer are also similar to the 1x scale mixer, in that the system shows a high degree of mixing effectiveness with good red oak movement, and no vertical or horizontal segregation. However, the ω = 40 and 60 rpm conditions for the 2x show increased vertical segregation and red oak agglomerations on the surface of the flow. This however, is most likely due to
the under filled nature of the 2x scale mixer at these conditions and should not be attributed entirely to the scale of the mixer.

The dynamic mixing videos with the counter-rotating up pumping screw rotation condition showed similar behavior for all the scale mixers in that the system is heavily vertically segregated, and contrary to what the composition results suggests, is poorly mixed. It is concluded that, for the larger scale mixers, this condition exhibits poor mixing effectiveness, compared to the 1x scale mixer.

The one co-rotating condition tested with the larger mixers provides the most unexpected results of the tests conducted with the larger scale mixers. The mixing effectiveness of the system is significantly increased as the mixer scale is increased from the 1x scale to the 1.5x scale, but then significantly decreased as the mixer scale is further increased from 1.5x to 2x. The particle scale to mixer scale ratio for the 1.5x system seems to be optimized for this co-rotating condition to minimize to segregation effects that are typical of this screw rotation orientation, yielding a very well mixed granular flow. The 1x scale mixer and 2x scale mixer feature similarly poor mixing dynamics.

5.5 Conclusions and Discussion

This study investigated the effect of mixer scale on the mixing effectiveness in a double screw mixer. Using a study performed by Kingston and Heindel (2013; 2014b) with a D = 2.54 cm (1 in) screw diameter double screw mixer as a reference study, select operating conditions tested by Kingston and Heindel were replicated with a D = 3.81 cm (1.5 in) and 5.08 cm (2 in) screw diameter double screw mixer in order to investigate the effect of mixer scale on mixing dynamics.
The counter-rotating down pumping screw rotation conditions were the focus of this study based on the conclusions presented by Kingston and Heindel (2013; 2014b). The larger scale mixers displayed both an increased sensitivity to changes in dimensionless screw pitch, a phenomenon evident in both the composition results and the dynamics mixing videos, and an increased tendency towards vertical segregation of the two granular particles. These phenomena are unique to the larger scale mixers, as the 1x scale mixer displayed consistently good mixing dynamics across all dimensionless screw pitches of the counter-rotating down pumping screw rotation orientation conditions. The 1.5x and 2x scale mixers only feature comparatively good mixing at the largest dimensionless screw pitch. Overall conclusions from the counter-rotating down pumping screw rotation conditions, with the larger scale mixers, indicates that the mixing effectiveness of the system is decreased as the mixer scale is increased.

The one counter-rotating up pumping screw rotation condition tested with the larger scale mixers showed very similar composition results and dynamic mixing behavior to that of the 1x scale mixer. Conclusions from this operating conditions indicate that the up-pumping screw rotation orientation is still a poor mixing condition at larger mixer scales.

Unique mixing behavior was seen for the one co-rotating screw rotation condition investigated with the larger screw mixers. The 1x scale mixer saw poor mixing dynamics with high coefficients of variation and horizontal and vertical segregation. While the 2x scale saw these same mixing behaviors, the 1.5x scale mixer featured good mixing dynamics for this condition, with an even distribution of red oak throughout the system and little segregation. For this one co-rotating condition, increasing the mixer scale from 1x to 1.5x
greatly increased the mixing effectiveness, but further increasing the mixer scale to 2x decreased the mixing effectiveness back down to that seen with the 1x scale mixer.

Results from this study provide insights into how granular flows behave as the mixer scale is increased. However, granular flows are incredibly complex, and as the results here suggest, while some general trends can be recognized, scale-up of granular flows are very difficult to predict and understand. Granular mixers can be incredibly sensitive to changes in operating parameters at a laboratory scale, and the results from this study suggest that, as the mixer scale is increased, sensitivity to changes in operating parameters is only increased.

Understanding the key parameters affecting the mixing dynamics as a granular mixing process is scaled-up is also a challenge. This study aimed to isolate the effect of mixer scale by exactly replicating the operating conditions of a D = 2.54 cm (1 in) screw diameter double screw mixer with a 1.5x and 2x scale double screw mixer. However, the particles used in this study were identical in size to that of the 1x scale mixer study. Therefore, the mixer geometry was not the only parameter scaled; the particle size to mixer size ratio was also scaled. Many researchers have looked into the significant effect particle size has to play on the mixing dynamics within a granular flow (Alexander et al., 2004a; Arntz et al., 2014; Hogg, 2009; Jain et al., 2005), and by changing this ratio, the mixing dynamics of the screw mixer were likely affected. Furthermore, the shear force exerted on the granular particles in a mixer is directly proportional to the surface area of the mixer (Mausda, 2006). By changing the particle surface area to mixer surface area ratio, the proportion of shear forces imparted on the particles to other forces in the system, likely changed from the 1x scale mixer to the 1.5x and 2x scale mixers, further affecting the mixing dynamics.
This all demonstrates how difficult it is to isolate parameters when researching mixer scale-up. Many different parameters affect the mixing dynamics in a granular mixer, and the proportional role each parameter plays in the mixing dynamics is not only unknown, but most likely changes with varying mixer scale. For this reason, extensive research, both experimentally and with simulations, is needed on larger granular mixers in order to fully understand the complex, ever changing mixing dynamics in industrial scale granular flows.

5.6 Acknowledgments

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CHAPTER 6. PROJECT CONCLUSIONS

6.1 Conclusions

This project provides valuable insights into the effects of scale on the mixing effectiveness in a double screw mixer. Particle characteristic play a crucial role in influencing granular mixing dynamics and there is a pressing need for a better understanding of larger scale industrial mixers. Granular flows are complex and not very well understood, and larger mixers are needed to make industrial mixing applications more economically viable. This project focused on the important issues of particle scale effects and mixer scale effects on granular mixing in the hope of improving the working knowledge of granular flows and the viability of bio-oil production via double screw pyrolyzers. This project accomplished this goal via three main objectives described in Section 1.2.

6.1.1 Objective 1

**Objective 1:** Determine the effect of premixing the heat carrier and biomass using video and composition analysis in a D = 2.54 cm (1 in) double screw mixer, designed to geometrically replicate a double screw pyrolyzer.

**Conclusion 1:** The experimental procedures and results from Objective 1 are provided in detail in Appendix A. In fast pyrolysis, premixing the biomass and heat carrier media allows the design of double screw pyrolyzers to be simplified by requiring only one inlet port instead of two. This study investigated premixing by injecting the heat carrier and biomass simultaneously into inlet port 2. Results, obtained from composition analysis and optical visualization, indicate that premixing the heat carrier media and the biomass has little to no
effect on the mixing dynamics within the double screw mixer. While premixing in actual fast pyrolysis introduces the risk of pyrolysis occurring within the inlet tube and not the reactor itself, from a mixing standpoint, conclusions from Objective 1 indicate that premixing is an adequate solution to the issue of complicated mixer design.

6.1.2 Objective 2

Objective 2: Determine the effect of particle size, density, and concentration using video and composition analysis in a D = 2.54 cm (1 in) double screw mixer.

Conclusion 2: Numerous research studies have shown that particle characteristics have a very large influence in the mixing and segregation mechanisms within granular flows. Because industrial applications of granular mixing often involve complicated operating conditions with irregular particles, it is important to study granular mixing processes with a wide range of particle combinations. This study used composition analysis and optical visualization techniques to investigate the effect of changing particle size, density, and concentration.

The particles used in this study were: (1) large red oak, LRO, 500 – 6350 μm, 1.47 g/cc; (2) small red oak, SRO, 300 – 710 μm, 1.53 g/cc; (3) corn stover, CS, 300 – 710 μm, 1.37 g/cc; (4) cork particles, CO, 300 – 710 μm, 0.87 g/cc; (5) small glass beads, SGB, 300 – 500 μm, 2.50 g/cc; and (6) large glass beads, LGB, 800 – 1000 μm, 2.50 g/cc. LRO:SGB was used as the reference condition for all tests in this study (Kingston, 2013; Kingston and Heindel, 2014b). To investigate the effect of particle size, SRO:SGB and LRO:LGB were compared to the reference study. For particle density effects, SRO:SGB, CS:SGB, and CO:SGB were compared to each other. To determine the effects of changing particle...
concentration, LRO:SGB were tested at mass flow rate ratios of 20:1, 30:1, 40:1, 50:1 (heat carrier to biomass) and the results were compared to the 10:1 ratio of the reference study.

The SRO:SGB results, when compared to the reference study’s LRO:SGB, indicate that reducing the biomass particle size greatly increasing the mixing effectiveness of the system, especially for the counter-rotating down pumping screw rotation conditions. The LRO:LGB results however, when compared to the LRO:SGB reference study, indicate that increasing the heat carrier particle size decreases the overall mixing effectiveness.

The changing particle density results, from comparing the SRO:SGB, CS:SGB, and CO:SGB, indicate that there is little change in mixing effectiveness with change in biomass particle density. Composition results indicate some change in the mixing effectiveness with changing dimensionless screw pitch as biomass particle density changes, however, the dynamic mixing videos did not indicate this, instead showing similarly good mixing dynamics for all the particle density combinations.

Results from the four particle concentration tests, when compared to the reference study, indicate that reducing the biomass particle concentration (by increasing the mass flow rate ratio between the heat carrier and biomass) decreases the mixing effectiveness of a double screw mixer. There are some select conditions that exhibit improved mixing dynamics at the 20:1 and 30:1 mass flow rate ratios, but overall smaller biomass concentrations decrease the mixing effectiveness of the system.

Overall, conclusions from Objective 2 indicate that when designing granular mixers and double screw pyrolyzers, the greatest considerations should be given to managing particle characteristics and concentrations, with the effects of changing particle density being the least important.
6.1.3 Objective 3

**Objective 3:** Determine the effect of mixer scale using video and composition analysis in a \( D = 3.81 \) cm (1.5 in) and a \( D = 5.08 \) cm (2 in) double screw mixer.

**Conclusion 3:** Industrial granular mixing processes require large scale mixers that produce a high quality product with an accurately known composition. However, because granular flows are very complex and little understood, especially on larger scales, mixer design is still largely based on trial and error and intuition rather than analytically derived knowledge. Experimental studies with larger scale mixers are vitally important to improve the understanding and efficiency of industrial scale mixers.

This project investigates how the mixing dynamics within a double screw mixer change as the mixer is geometrically scaled to 1.5x and 2x times the original scale. A double screw mixer designed and tested by Kingston and Heindel (2013; 2014b, c) was used as the original 1x scale mixer.

Composition analysis paired with optical visualization, conducted on both the 1.5x and the 2x scale double screw mixers, generally indicate that increasing the size of the screw mixer decreases the overall mixing effectiveness of the system. One exception to this conclusion occurs for the operating condition involving a co-rotating screw rotation orientation, in which the mixing effectiveness of the system was actually increased as the mixer scale was increased to 1.5x. However, as the scale was increased further to 2x, the mixing effectiveness decreased to the levels seen in the 1x scale mixer. Most all other conditions saw an increase in either vertical segregation, sensitivity to changes in dimensionless screw pitch, or both, as the mixer scale was increased, leading to a conclusion of decreased mixing effectiveness with increasing mixer scale.
6.2 Future Work

This project investigated the effect of premixing the heat carrier and biomass, changing particle size, density, concentration, and mixer scale on the mixing effectiveness within a double screw mixer. Non-invasive composition analysis was paired with advanced 360° optical visualization to provide a thorough understanding of the mixing dynamics within the system. However, there are still certain particle and mixer scale characteristics that require further study.

One area that could benefit from further investigation is the role that particle size plays on granular mixing. This study showed that the particle size ratio between the heat carrier media and the biomass has a large effect on the mixing dynamics within a double screw pyrolyzer. Many previous research studies, using different types of mixers, have likewise confirmed the significance of particle size on the mixing mechanisms within granular flows. Additional research needs to be done to formalize rules about the effects of particle characteristics, so that suggestions for the mitigation of the negative effects caused by non-uniform particles, such as segregation, can be better made.

A better understanding of the local three-dimensional mixing within the double screw mixer with the smaller biomass particles, SRO, would be valuable as well. Kingston et al. (2015) used X-ray particle tracking velocimetry to characterize the three-dimensional granular flow structures in a double screw mixer, using the LRO:SGB indicated as the reference study in this project. As the SRO:SGB resulted in greatly improved mixing as compared to the LRO:SGB, it would be valuable to better understand how the smaller biomass particles move throughout the system by characterizing the internal flow structure of the SRO:SGB.
There still remains much work to be done with the 1.5x and 2x scale double screw mixers used in this study. The LRO:SGB used in the larger mixers resulted in decreased mixing effectiveness when compared to the same operating conditions of the 1x scale mixer. It would be interesting to investigate how SRO:SGB would behave in the larger systems, given how significantly the smaller biomass particles improved the mixing effectiveness of the 1x scale mixer. In the 1.5x and 2x scale studies conducted in this project, the mixer geometry was scaled, but so was the particle size to mixer size ratio. Testing another combination of different size particles with the larger scale mixers, especially one that has already been tested with the 1x scale mixer, would provide a better understanding of how this ratio affects mixer scale up.

X-ray particle tracking velocimetry with the larger double screw mixers would also provide valuable information about how the individual particle flow dynamics change with changing mixer scale. Knowledge of the three-dimensional flow structures within the larger scale mixers would prove to be even more valuable than with the 1x scale mixer because, with the larger scale mixers, more of the flow is hidden inside the opaque flow and the information that can be gained by surface optical visualization alone is more limited. X-ray particle tracking velocimetry would reveal the internal flow structures.

Additional work also needs to be done with even larger scale mixers. The 1.5x and 2x double screw mixers used in this study, while excellent starting places for scale-up studies, are still not approaching industrial scales. Given the results of this study, which indicate the unpredictability of the larger scale systems with their increased sensitivity to parameter changes, more experimental work will be needed with even larger double screw mixers before industrial scale systems can be manufactured and utilized efficiently.
Valuable insights were gained from this project, which provide a better understanding of granular mixing processes in general, and specifically, the mixing dynamics within double screw pyrolyzers used for fast pyrolysis. However, because granular mixing is so intricate and important to such a wide variety of industries, additional research is still needed to fully understand the complex and intriguing nature of granular flows.
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APPENDIX A. PREMIXING STUDY

A.1 Introduction

Double screw pyrolyzers are much more complex in design than screw mixers. Their designs must feature not only the screws and housing, but multiple vapor outlet ports, temperature measurement ports, solids outlet ports, supporting parts, and heat shielding. Figure 3.18 shows the schematic of a laboratory-scale double auger reactor used for biomass fast pyrolysis at Iowa State University, illustrating the complicated design of actual pyrolyzers (Brown, 2009). In order to reduce the complexity of the system, even in a minor way, it would be advantageous if the two material injection ports could be combined into one single inlet port. While this would simplify the external aspects of the system, it is unknown what the effects of this combined material injection would be on the mixing dynamics within the system.

A.2 Parameter Selection

Operating conditions investigated in the premixing task were determined based on the conclusions of Kingston and Heindel (2014b, c). The optimal mixing condition, with a screw rotation speed of \( \omega = 60 \) rpm, a dimensionless screw pitch of \( p/D = 1.75 \), and a counter-rotating down pumping screw rotation orientation was the starting point for the study. As a counter-rotating down pumping screw rotation orientation was determined to be the most advantageous screw rotation condition, regardless of screw rotation speed and dimensionless screw pitch, all other operating conditions involving the counter-rotating down pumping
screw rotation orientation were also investigated. These consisted of screw rotation speeds of $\omega = 20, 40$ and 60 rpm, each with a dimensionless screw pitch of $p/D = 0.75, 1.25,$ and 1.75.

For the sake of completeness, two additional operating conditions were chosen for testing: (1) Kingston and Heindel’s (2014b, c) original standard condition with a screw speed of $\omega = 40$ rpm, dimensionless screw pitch of $p/D = 1.25$ and a co-rotating screw rotation orientation; and (2) a condition deemed to be a poor mixing condition, with a screw speed of $\omega = 20$ rpm, dimensionless screw pitch of $p/D = 0.75$ and a counter-rotating up pumping screw rotation orientation.

As the goal of this small study was to investigate the effect of premixing, the two materials, LRO and SGB, were metered out of their respective volumetric feeders into one single inlet tube which fed into the second inlet port of the screw mixer. Thus, only one inlet port was used. The experimental setup for the premixing study is shown in Figure 3.19. All premixing tests were conducted at a 10:1 mass flow rate ratio between the SGB and the LRO with a 65% fill of the screw mixer, outlined in Table 3.2. In total, 11 testing conditions were investigated in the premixing study, outlined in Table 3.3.

**A.3 Experimental Set-Up**

Two Tecweigh CR5 volumetric auger feeders were used to meter the granular materials into the screw mixer. To achieve premixing, the two granular materials, biomass and heat carrier media, were conveyed horizontally out of their respective feeders into a single vertical inlet tube that fed into the second of two inlet ports (port 2) on top of the screw mixer. To prevent material jamming in the feeder outlet tubes, the heat carrier feeder was raised an arbitrary height above the biomass feeder so that the more dense glass beads
were injected into the vertical inlet tube first and fell on top of the less dense red oak chips as the red oak chips were injected second into the vertical inlet tube. The second inlet port was chosen as the point of material injection as this is the start of the screw mixer’s dedicated mixing region, measured from the center of the second inlet port to the beginning of the outlet ports.

**A.4 Results and Discussion**

The results from the premixing study are compared to the results presented by Kingston and Heindel (2013; 2014c) which feature a separate biomass injection configuration. More specifically, the results of this premixing study will be compared to the optimal material injection configuration as determined by Kingston and Heindel, in which the biomass is injected into port 1 and the heat carrier into port 2.

**A.4.1 Composition analysis**

Figure A.1 shows the three-way interaction between screw rotation speed, dimensionless screw pitch, and screw rotation orientation for the premixing tests (plotted in black) compared to the reference study results (Kingston and Heindel, 2014b) (plotted in green). Recall that a lower coefficient of variation indicates better mixing—a coefficient of variation of zero is a perfectly homogeneous mixture and is the desired state. For each operating condition, three data points are plotted, representing the set of three samples collected for each operating condition to ensure repeatability. To aid the visualization of trends, an averaging line had been applied to each set of three samples and across operating conditions. The error bars represent the standard error in the average measurement (Section 3.4.2).
The premixing coefficients of variation are very similar to the reference study results, except for a few select conditions which have slightly higher coefficients of variation. The counter-rotating down pumping screw rotation orientation conditions display a slight increase in sensitivity to changes in dimensionless screw pitch, which manifest as higher coefficients of variation. The higher screw rotation speeds, $\omega = 40$ and $60$ rpm, feature the largest deviations in coefficient of variation as compared to the reference study. The composition results for this screw rotation orientation indicate that premixing may decrease the mixing effectiveness, but only slightly.
The one counter-rotating up pumping condition tested with the premixed material injection configuration resulted in coefficients of variation very similar to those of the reference study, indicating the mixing dynamics are likely similar.

Premixing the two granular materials resulted in higher coefficients of variation for the one co-rotating screw rotation condition tested, indicated the mixing effectiveness for this condition decreased.

A.4.2 Optical visualization

Dynamic mixing videos of the premixing condition were captured for all 11 operating conditions specified in Table 3.3. Snapshots of three select dynamic mixing videos are shown in Figure A.2. Conclusions presented here are based on all 11 dynamic videos.

![Figure A.2: Snapshots of the dynamic mixing process for select operating conditions from the premixing study.](image)

The counter-rotating down pumping screw rotation orientation mixing videos, an example of which is shown in Figure A.2a, display good mixing dynamics with the biomass evenly distributed throughout the system and in constant motion. The conditions featuring the largest dimensionless pitch, \( p/D = 1.75 \), tend to have more red oak riding along the surface of the flow than the smaller dimensionless screw pitch conditions, however, the red oak particles are all eventually mixed back into the system. In all other counter-rotating down pumping conditions very few red oak agglomerations are visible and, for the most part, the
premixing dynamic mixing videos display very similar mixing dynamics to the reference study dynamic mixing videos (Kingston, 2013).

The counter-rotating up pumping screw rotation condition with the premixing material injection configuration, shown in Figure A.2b, features very poor mixing. The biomass material rises to the surface of the flow where it remains segregated from the heat carrier. The granular flow as a whole also tends to be simply conveyed along and little mixing occurs.

Premixing the two granular materials results in a flow that tends to segregate more than the reference study’s separate material injection configuration. Figure A.2c shows the co-rotating premixing condition. The bulk of the flow is pushed into the left screw, a result of the screw rotation orientation, and large agglomerations of biomass form on the surface of the flow in the right screw. While the reference study did see similar agglomerations of red oak particles forming on the top of the flow in the right screw, these agglomerations were more significant and appeared earlier for the premixing condition. This indicates that the premixing conditions is a worse mixing condition than the reference study for this co-rotating operating condition.

The biggest observational difference between the premixing study and the reference study appears in the material injection region of the double screw mixer. When the biomass is injected first, as in the reference study, the biomass particles form a large initial agglomeration which rides, as a bulk, to the surface of the flow once the glass beads are injected. This large agglomeration takes a few turns of the screws to be fully broken up and worked into the flow, meaning that mixing of the two granular materials is delayed. With the premixing condition, there is no biomass build up at the front of the screw mixer, meaning no
additional time is needed to break up the agglomerated biomass materials. Mixing
commences immediately upon the material injection into the screw mixer. The phenomenon
is shown in Figure A.3.

Counter-rotating down pumping, $\omega = 20$, $p/D = 1.25$

Figure A.3: Snapshots of one operating condition for both the (a) reference study, and (b) the
premixing study. The inlet region of each mixer is highlighted to show how
premixing eliminates the biomass material backup that otherwise occurs in the
separate material injection configuration of the reference study. The reference
study snapshot is from Kingston (2013).

A.5 Conclusions

Overall, it appears that premixing has little effect on the mixing dynamics within a
double screw mixer. The composition results indicate a slight increase in sensitivity to
changes in dimensionless screw pitch, manifesting as higher coefficients of variation. While
this indicates a decrease in mixing effectiveness, the increases in the coefficients of variation
are minor. The dynamic mixing videos for the premixing condition reveal mixing behavior
very similar to the reference study’s separated material injection configuration; further
indicating that premixing has little effect on the flow dynamics. One positive effect of the
premixing was seen at the material injection region of the screw mixer, where the premixing
condition eliminated the biomass build up that delayed the onset of mixing in the reference
study.
While premixing in actual fast pyrolysis introduces the risk of pyrolysis occurring within the inlet tube and not the reactor itself, from a granular mixing standpoint, conclusions from the premixing study indicate that premixing has little negative effect on mixing effectiveness and is therefore an adequate solution to the issue of complicated mixer design.
APPENDIX B. ENGINEERING DRAWINGS

Engineering drawings for all the 3D printed parts associated with both the 1.5x and 2x scale double screw mixers are provided here. All dimensions are in inches.

**B.1 Screw Mixer: 1.5x**
B.2 Screw Mixer: 2x
Appendix C provides the mass flow rates for the biomass and heat carrier media needed to achieve a 65% fill of the screw mixer, for each material combination used in this project.

### C.1 Premixing, LRO:SGB

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<th>Screw Rotation Speed [rpm]</th>
<th>Dimensionless Screw Pitch (p/D) [-]</th>
<th>Biomass Mass Flow Rate [kg/hr]</th>
<th>Glass Beads Mass Flow Rate [kg/hr]</th>
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### C.2 Particle Size, Density, and Concentration

#### C.2.1 Particle Size

#### C.2.1.1 SRO:SGB

Data file corrupted
### C.2.1.2 LRO:LGB

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### C.2.2 Biomass Particle Density

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C.2.3 Particle Concentration

C.2.3.1 Mass Flow Rate Ratio 20:1

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### C.3 Mixer Scale

#### C.3.1 LRO:SGB, 1.5x Scale

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C.3.2 LRO:SGB, 2x Scale

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*Maximum volumetric feeder setting. Resulted in an under filled (~50% fill) system.
APPENDIX D. VIDEO SNAPSHOTS

Snapshots of the dynamic mixing process for each material combination used in this project are presented in Figures D.1 through D.130. The operating condition numbers refer to the arbitrarily chosen number system presented in Table 3.1. The parameters of each operating condition can also be found in Table 3.1. The snapshots of the dynamic mixing videos from the reference study can be found in Kingston (2013).

D.1 Premixing

Figure D.1: Premixing, Operating Condition 3
Figure D.2: Premixing, Operating Condition 5

Figure D.3: Premixing, Operating Condition 11
Figure D.4: Premixing, Operating Condition 17

Figure D.5: Premixing, Operating Condition 23
Figure D.6: Premixing, Operating Condition 25

Figure D.7: Premixing, Operating Condition 29
Figure D.8: Premixing, Operating Condition 35

Figure D.9: Premixing, Operating Condition 41
Figure D.10: Premixing, Operating Condition 47

Figure D.11: Premixing, Operating Condition 53
D.2 Particle Size

D.2.1 SRO:SGB

Figure D.12: SRO:SGB, Operating Condition 3
Figure D.13: SRO:SGB, Operating Condition 5

Figure D.14: SRO:SGB, Operating Condition 11
Figure D.15: SRO:SGB, Operating Condition 17

Figure D.16: SRO:SGB, Operating Condition 23
Figure D.17: SRO:SGB, Operating Condition 25

Figure D.18: SRO:SGB, Operating Condition 29
Figure D.19: SRO:SGB, Operating Condition 35

Figure D.20: SRO:SGB, Operating Condition 41
Figure D.21: SRO:SGB, Operating Condition 47

Figure D.22: SRO:SGB, Operating Condition 53
D.2.2 LRO:LGB

Figure D.23: LRO:LGB, Operating Condition 5

Figure D.24: LRO:LGB, Operating Condition 11
Figure D.25: LRO:LGB, Operating Condition 17

Figure D.26: LRO:LGB, Operating Condition 23
Figure D.27: LRO:LGB, Operating Condition 25

Figure D.28: LRO:LGB, Operating Condition 29
Figure D.29: LRO:LGB, Operating Condition 35

Figure D.30: LRO:LGB, Operating Condition 41
Figure D.31: LRO: LGB, Operating Condition 47

Figure D.32: LRO: LGB, Operating Condition 53
D.3 Particle Density

D.3.1 SRO:SGB

See Section D.2.1

D.3.2 CS:SGB

Figure D.33: CS:SGB, Operating Condition 5
Figure D.34: CS:SGB, Operating Condition 11

Figure D.35: CS:SGB, Operating Condition 17
Figure D.36: CS:SGB, Operating Condition 23

Figure D.37: CS:SGB, Operating Condition 25
Figure D.38: CS:SGB, Operating Condition 29

Figure D.39: CS:SGB, Operating Condition 35
Figure D.40:  CS:SGB, Operating Condition 41

Figure D.41:  CS:SGB, Operating Condition 47
Figure D.42: CS:SGB, Operating Condition 53

D.3.3 CO:SGB

Figure D.43: CO:SGB, Operating Condition 5
Figure D.44: CO:SGB, Operating Condition 11

Figure D.45: CO:SGB, Operating Condition 17
Figure D.46: CO:SGB, Operating Condition 23

Figure D.47: CO:SGB, Operating Condition 25
Figure D.48: CO:SGB, Operating Condition 29

Figure D.49: CO:SGB, Operating Condition 35
Figure D.50: CO:SGB, Operating Condition 41

Figure D.51: CO:SGB, Operating Condition 47
Figure D.52: CO:SGB, Operating Condition 53
D.4 Particle Concentration

Figure D.53: Operating Condition 3, 20:1
Figure D.54: Operating Condition 3, 30:1

Figure D.55: Operating Condition 3, 40:1
Figure D.56: Operating Condition 3, 50:1

Figure D.57: Operating Condition 5, 20:1
Figure D.58: Operating Condition 5, 30:1

Figure D.59: Operating Condition 5, 40:1
Figure D.60: Operating Condition 5, 50:1

Figure D.61: Operating Condition 9, 20:1
Figure D.62: Operating Condition 9, 30:1
Figure D.63: Operating Condition 9, 40:1

Figure D.64: Operating Condition 9, 50:1
Figure D.65: Operating Condition 11, 20:1

Figure D.66: Operating Condition 11: 30:1
Figure D.67: Operating Condition 11, 40:1

Figure D.68: Operating Condition 11, 50:1
Figure D.69: Operating Condition 17, 20:1

Figure D.70: Operating Condition 17, 30:1
Figure D.71: Operating Condition 17, 40:1

Figure D.72: Operating Condition 17, 50:1
Figure D.73: Operating Condition 23, 20:1

Figure D.74: Operating Condition 23, 30:1
Figure D.75: Operating Condition 23, 40:1

Figure D.76: Operating Condition 23, 50:1
Figure D.77: Operating Condition 25, 20:1

Figure D.78: Operating Condition 25, 30:1
Figure D.79: Operating Condition 25, 40:1

Figure D.80: Operating Condition 25, 50:1
Figure D.81: Operating Condition 27, 20:1

Figure D.82: Operating Condition 27, 30:1
Figure D.83:  Operating Condition 27, 40:1

Figure D.84:  Operating Condition 27, 50:1
Figure D.85: Operating Condition 29, 20:1

Figure D.86: Operating Condition 29, 30:1
Figure D.87: Operating Condition 29, 40:1

Figure D.88: Operating Condition 29, 50:1
Figure D.89: Operating Condition 35, 20:1

Figure D.90: Operating Condition 35, 30:1
Figure D.91: Operating Condition 35, 40:1

Figure D.92: Operating Condition 35, 50:1
Figure D.93: Operating Condition 41, 20:1

Figure D.94: Operating Condition 41, 30:1
Figure D.95: Operating Condition 41, 40:1

Figure D.96: Operating Condition 41, 50:1
Figure D.97: Operating Condition 45, 20:1

Figure D.98: Operating Condition 45, 30:1
Figure D.99: Operating Condition 45, 40:1

Figure D.100: Operating Condition 45, 50:1
Figure D.101: Operating Condition 47, 20:1

Figure D.102: Operating Condition 47, 30:1
Figure D.103: Operating Condition 47, 40:1

Figure D.104: Operating Condition 47, 50:1
Figure D.105: Operating Condition 53, 20:1

Figure D.106: Operating Condition 53, 30:1
Figure D.107: Operating Condition 53, 40:1

Figure D.108: Operating Condition 53, 50:1
D.5 Mixer Scale

D.5.1 Mixer Scale: 1.5x

Figure D.109: Operating Condition 3
Figure D.110: Operating Condition 5

Figure D.111: Operating Condition 11
Figure D.112: Operating Condition 17

Figure D.113: Operating Condition 23
Figure D.114: Operating Condition 25

Figure D.115: Operating Condition 29
Figure D.116: Operating Condition 35

Figure D.117: Operating Condition 41
Figure D.118: Operating Condition 47

Figure D.119: Operating Condition 53
D.5.2 Mixer Scale: 2x

Figure D.120: Operating Condition 3
Figure D.121: Operating Condition 5

Figure D.122: Operating Condition 11
Figure D.123: Operating Condition 17

Figure D.124: Operating Condition 23
Figure D.125: Operating Condition 25

Figure D.126: Operating Condition 29
Figure D.127: Operating Condition 35

Figure D.128: Operating Condition 41
Figure D.129: Operating Condition 47

Figure D.130: Operating Condition 53
APPENDIX E. COMPOSITION CORRELATION DATA

Appendix E provides the composition correlation for each material combination which allows the composition of a sample to be calculated from its true density. The uncertainty in the true density, as measured by a pentapycnometer, was calculated using the root sum of squares (RSS) procedure.
E.1 LRO:SGB

Table E.1: Composition correlation calibration data for SRO:LGB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Mass [g]</th>
<th>SGB Mass Fraction [-]</th>
<th>LRO Mass Fraction [-]</th>
<th>Average True Density [kg/m³]</th>
<th>Average True Density Uncertainty [kg/m³]</th>
<th>Average True Density Uncertainty Percentage [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>35.000</td>
<td>1.0</td>
<td>0.0</td>
<td>2534.6</td>
<td>3.1</td>
<td>0.1%</td>
</tr>
<tr>
<td>2</td>
<td>35.000</td>
<td>0.9</td>
<td>0.1</td>
<td>2309.8</td>
<td>3.4</td>
<td>0.1%</td>
</tr>
<tr>
<td>3</td>
<td>35.000</td>
<td>0.8</td>
<td>0.2</td>
<td>2106.0</td>
<td>3.7</td>
<td>0.2%</td>
</tr>
<tr>
<td>4</td>
<td>35.000</td>
<td>0.7</td>
<td>0.3</td>
<td>1953.4</td>
<td>4.0</td>
<td>0.2%</td>
</tr>
<tr>
<td>5</td>
<td>35.000</td>
<td>0.6</td>
<td>0.4</td>
<td>1818.6</td>
<td>4.3</td>
<td>0.2%</td>
</tr>
<tr>
<td>6</td>
<td>35.000</td>
<td>0.5</td>
<td>0.5</td>
<td>1697.4</td>
<td>4.6</td>
<td>0.3%</td>
</tr>
<tr>
<td>7</td>
<td>35.000</td>
<td>0.4</td>
<td>0.6</td>
<td>1613.7</td>
<td>4.9</td>
<td>0.3%</td>
</tr>
<tr>
<td>8</td>
<td>35.000</td>
<td>0.3</td>
<td>0.7</td>
<td>1520.2</td>
<td>5.2</td>
<td>0.3%</td>
</tr>
<tr>
<td>9</td>
<td>35.000</td>
<td>0.2</td>
<td>0.8</td>
<td>1469.0</td>
<td>5.3</td>
<td>0.4%</td>
</tr>
<tr>
<td>10</td>
<td>35.000</td>
<td>0.1</td>
<td>0.9</td>
<td>1394.5</td>
<td>5.6</td>
<td>0.4%</td>
</tr>
<tr>
<td>11</td>
<td>35.000</td>
<td>0.0</td>
<td>1.0</td>
<td>1355.8</td>
<td>5.8</td>
<td>0.4%</td>
</tr>
</tbody>
</table>

Figure E.1: Empirical correlation between the mixture composition and the mixture density for the LRO:SGB.
Table E.2: Composition correlation calibration data for SRO:SGB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Mass [g]</th>
<th>SGB Mass Fraction [-]</th>
<th>SRO Mass Fraction [-]</th>
<th>Average True Density [kg/m³]</th>
<th>Average True Density Uncertainty [kg/m³]</th>
<th>Average True Density Uncertainty Percentage [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150.6</td>
<td>1.0</td>
<td>0.0</td>
<td>2505.7</td>
<td>13.4</td>
<td>0.5%</td>
</tr>
<tr>
<td>2</td>
<td>100.0</td>
<td>0.9</td>
<td>0.1</td>
<td>2333.9</td>
<td>9.6</td>
<td>0.4%</td>
</tr>
<tr>
<td>3</td>
<td>100.0</td>
<td>0.8</td>
<td>0.2</td>
<td>2192.2</td>
<td>10.2</td>
<td>0.5%</td>
</tr>
<tr>
<td>4</td>
<td>83.3</td>
<td>0.7</td>
<td>0.3</td>
<td>2068.7</td>
<td>9.0</td>
<td>0.4%</td>
</tr>
<tr>
<td>5</td>
<td>62.5</td>
<td>0.6</td>
<td>0.4</td>
<td>1929.6</td>
<td>7.2</td>
<td>0.4%</td>
</tr>
<tr>
<td>6</td>
<td>60.0</td>
<td>0.5</td>
<td>0.5</td>
<td>1862.3</td>
<td>7.2</td>
<td>0.4%</td>
</tr>
<tr>
<td>7</td>
<td>50.0</td>
<td>0.4</td>
<td>0.6</td>
<td>1783.4</td>
<td>6.3</td>
<td>0.4%</td>
</tr>
<tr>
<td>8</td>
<td>45.7</td>
<td>0.3</td>
<td>0.7</td>
<td>1732.9</td>
<td>5.9</td>
<td>0.3%</td>
</tr>
<tr>
<td>9</td>
<td>40.0</td>
<td>0.2</td>
<td>0.8</td>
<td>1640.8</td>
<td>5.5</td>
<td>0.3%</td>
</tr>
<tr>
<td>10</td>
<td>35.6</td>
<td>0.1</td>
<td>0.9</td>
<td>1560.3</td>
<td>5.1</td>
<td>0.3%</td>
</tr>
<tr>
<td>11</td>
<td>18.9</td>
<td>0.0</td>
<td>1.0</td>
<td>1544.4</td>
<td>2.7</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

Figure E.2: Empirical correlation between the mixture composition and the mixture density for the SRO:SGB.
Table E.3: Composition correlation calibration data for LRO:LGB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Mass [g]</th>
<th>LGB Mass Fraction [-]</th>
<th>LRO Mass Fraction [-]</th>
<th>Average True Density [kg/m³]</th>
<th>Average True Density Uncertainty [kg/m³]</th>
<th>Average True Density Percentage [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>161.1</td>
<td>1.0</td>
<td>0.0</td>
<td>2532.0</td>
<td>14.2</td>
<td>0.6%</td>
</tr>
<tr>
<td>2</td>
<td>100.0</td>
<td>0.9</td>
<td>0.1</td>
<td>2311.8</td>
<td>9.7</td>
<td>0.4%</td>
</tr>
<tr>
<td>3</td>
<td>90.0</td>
<td>0.8</td>
<td>0.2</td>
<td>2123.6</td>
<td>9.5</td>
<td>0.4%</td>
</tr>
<tr>
<td>4</td>
<td>90.0</td>
<td>0.7</td>
<td>0.3</td>
<td>2034.5</td>
<td>9.9</td>
<td>0.5%</td>
</tr>
<tr>
<td>5</td>
<td>70.0</td>
<td>0.6</td>
<td>0.4</td>
<td>1828.7</td>
<td>8.6</td>
<td>0.5%</td>
</tr>
<tr>
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<td>50.0</td>
<td>0.5</td>
<td>0.5</td>
<td>1716.3</td>
<td>6.5</td>
<td>0.4%</td>
</tr>
<tr>
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<td>50.0</td>
<td>0.4</td>
<td>0.6</td>
<td>1606.3</td>
<td>7.0</td>
<td>0.4%</td>
</tr>
<tr>
<td>8</td>
<td>42.9</td>
<td>0.3</td>
<td>0.7</td>
<td>1512.6</td>
<td>6.3</td>
<td>0.4%</td>
</tr>
<tr>
<td>9</td>
<td>37.5</td>
<td>0.2</td>
<td>0.8</td>
<td>1431.8</td>
<td>5.9</td>
<td>0.4%</td>
</tr>
<tr>
<td>10</td>
<td>33.3</td>
<td>0.1</td>
<td>0.9</td>
<td>1358.2</td>
<td>5.5</td>
<td>0.4%</td>
</tr>
<tr>
<td>11</td>
<td>35.0</td>
<td>0.0</td>
<td>1.0</td>
<td>1355.8</td>
<td>5.8</td>
<td>0.4%</td>
</tr>
</tbody>
</table>

Figure E.3: Empirical correlation between the mixture composition and the mixture density for the LRO:LGB.
Table E.4: Composition correlation calibration data for CS:SGB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Mass [g]</th>
<th>SGB Mass Fraction [-]</th>
<th>CS Mass Fraction [-]</th>
<th>Average True Density [kg/m$^3$]</th>
<th>Average True Density Uncertainty [kg/m$^3$]</th>
<th>Average True Density Percentage [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>1.0</td>
<td>0.0</td>
<td>2497.5</td>
<td>13.4</td>
<td>0.5%</td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>0.9</td>
<td>0.1</td>
<td>2304.6</td>
<td>8.7</td>
<td>0.4%</td>
</tr>
<tr>
<td>3</td>
<td>60</td>
<td>0.8</td>
<td>0.2</td>
<td>2145.3</td>
<td>6.3</td>
<td>0.3%</td>
</tr>
<tr>
<td>4</td>
<td>45</td>
<td>0.7</td>
<td>0.3</td>
<td>1998.4</td>
<td>5.0</td>
<td>0.3%</td>
</tr>
<tr>
<td>5</td>
<td>35</td>
<td>0.6</td>
<td>0.4</td>
<td>1860.1</td>
<td>4.2</td>
<td>0.2%</td>
</tr>
<tr>
<td>6</td>
<td>25</td>
<td>0.5</td>
<td>0.5</td>
<td>1730.3</td>
<td>3.2</td>
<td>0.2%</td>
</tr>
<tr>
<td>7</td>
<td>25</td>
<td>0.4</td>
<td>0.6</td>
<td>1625.4</td>
<td>3.4</td>
<td>0.2%</td>
</tr>
<tr>
<td>8</td>
<td>15</td>
<td>0.3</td>
<td>0.7</td>
<td>1545.0</td>
<td>2.2</td>
<td>0.1%</td>
</tr>
<tr>
<td>9</td>
<td>15</td>
<td>0.2</td>
<td>0.8</td>
<td>1459.9</td>
<td>2.3</td>
<td>0.2%</td>
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<tr>
<td>10</td>
<td>15</td>
<td>0.1</td>
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<td>1401.0</td>
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<td>0.2%</td>
</tr>
<tr>
<td>11</td>
<td>18.9</td>
<td>0.0</td>
<td>1.0</td>
<td>1370.0</td>
<td>3.1</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

Figure E.4: Empirical correlation between the mixture composition and the mixture density for the CS:SGB.
E.5 CO:SGB

Table E.5: Composition correlation calibration data for CO:SGB.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Total Mass [g]</th>
<th>SGB Mass Fraction [-]</th>
<th>CO Mass Fraction [-]</th>
<th>Average True Density [kg/m³]</th>
<th>Average True Density Uncertainty [kg/m³]</th>
<th>Average True Density Percentage [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>150</td>
<td>1.0</td>
<td>0.0</td>
<td>2497.5</td>
<td>13.4</td>
<td>0.5%</td>
</tr>
<tr>
<td>2</td>
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<td>0.1</td>
<td>2151.0</td>
<td>8.5</td>
<td>0.4%</td>
</tr>
<tr>
<td>3</td>
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<td>0.2</td>
<td>1887.2</td>
<td>4.8</td>
<td>0.3%</td>
</tr>
<tr>
<td>4</td>
<td>27.02</td>
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<td>0.3</td>
<td>1692.8</td>
<td>3.6</td>
<td>0.2%</td>
</tr>
<tr>
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<td>22.5</td>
<td>0.6</td>
<td>0.4</td>
<td>1544.3</td>
<td>3.3</td>
<td>0.2%</td>
</tr>
<tr>
<td>6</td>
<td>18.04</td>
<td>0.5</td>
<td>0.5</td>
<td>1444.8</td>
<td>2.8</td>
<td>0.2%</td>
</tr>
<tr>
<td>7</td>
<td>15.02</td>
<td>0.4</td>
<td>0.6</td>
<td>1314.8</td>
<td>2.6</td>
<td>0.2%</td>
</tr>
<tr>
<td>8</td>
<td>14.28</td>
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<td>0.7</td>
<td>1241.6</td>
<td>2.6</td>
<td>0.2%</td>
</tr>
<tr>
<td>9</td>
<td>12.5</td>
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<td>0.8</td>
<td>1162.1</td>
<td>2.4</td>
<td>0.2%</td>
</tr>
<tr>
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<td>0.9</td>
<td>1062.7</td>
<td>2.4</td>
<td>0.2%</td>
</tr>
<tr>
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<td>967.2</td>
<td>2.3</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

Figure E.5: Empirical correlation between the mixture composition and the mixture density for the CO:SGB.