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Ethanol extraction of cottonseed

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ETHANOL EXTRACTION OF COTTONSEED

by

Rama Kanth Rao

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

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Approved:

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1957
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One of the reasons the solvent extraction industry has not developed in Asiatic countries like India, China and Japan, which produce enormous amounts of oilseeds, is the difficulty of obtaining a suitable solvent at a cheap price. The conventional petroleum and other solvents have to be imported and therefore become enormously costly due to high import duties and transit charges. The best way out appears to be the use of an indigenous solvent, which could be obtained cheaply.

Among the solvents produced in plenty in Asia is ethyl alcohol, which is available at about half the price of the imported solvents. It is known to be a good solvent for vegetable oils and is bound to become a very popular solvent in Asiatic countries if an economical process for its use is worked out.

Solubility data available in literature are quite favorable for the use of aqueous ethanol as a solvent for extracting all of the commonly used vegetable oils like corn, cottonseed, peanut, sesame and soybean oils. However, this project pertains to the study of cottonseed oil extraction only.

The main objectives of the project were to determine the
feasibility of extracting cottonseed directly using aqueous ethanol as a solvent; to test the adaptability of the Iowa State College extractor for the above purpose and to devise necessary modifications to it; to determine the optimum operating conditions for the alcoholic extraction of cottonseed; and to evaluate the economics of the process.

Laboratory extraction rate studies were carried out in a glass apparatus to determine the effect of temperature, moisture content of flakes, and the concentration of aqueous ethanol on the residual extractables for the extraction of cottonseed flakes by aqueous ethanol.

Pilot plant extractions of cottonseed flakes by aqueous ethanol were carried out in a small unit, similar to the commercial soybean oil extraction plant developed and patented by Iowa State College, to determine the effects of operating variables and the optimum operating conditions.

Both laboratory and pilot plant extraction studies have shown that direct extraction of cottonseed using aqueous ethanol as a solvent is a feasible process, and the optimum operating conditions for it have been established in the pilot plant unit.

Based on the results obtained in the pilot plant, it appears likely that the present Iowa State College extractor can be adopted, with some modifications, for the ethanol extraction of cottonseed. These modifications are discussed.
Pilot plant extractions have shown that in this process a prime quality crude oil and light colored meal of excellent quality with negligible free gossypol content are obtained.

Based on the data available in literature an economic cost study for a 50 tons per day commercial plant indicated an annual return on investment of 8.70 per cent.
INTRODUCTION

Solvent extraction is the most recent development of one of the oldest arts of man, that of obtaining oil from oilseeds and nuts. It has developed from the primitive hand or animal operated systems of pressing, which are wasteful and inefficient as the residual oil in the presscake is high. Even a modern screw press leaves about 5 to 8 per cent oil in the cake which could be almost completely recovered by solvent extraction methods. For low oil bearing materials like cottonseed and soybean, the pressing methods can be replaced by direct solvent extraction, which if properly done reduces the oil content of the meal to less than 1 per cent in almost every case.

While solvent extraction is a well established industry in the United States and European countries, it has not developed at all in India and other Asiatic countries which produce enormous amounts of oil bearing materials. India had its first solvent extraction plant of 50 tons a day capacity only about five years back which uses normal hexane as a solvent. However, there is an increasing need to set up more plants immediately for reasons to be discussed presently.
Paradoxical as it may seem, in spite of the enormous production of oilseeds in India, the production of vegetable oils is insufficient to meet the minimum requirements of the country. The annual per capita consumption in India is 8 pounds while the normal required is about 45 pounds (41). There are, however, many possible ways to increase the output. Increased agricultural production is no doubt one solution to the problem but that will be a comparatively long range project. Adoption of modern technological advances in the oil industry to obtain the maximum amount of oil from the oilseeds now being produced is quicker and more certain in its results.

The annual production of peanuts in India is about 3.5 million tons from which about 1.5 million tons of oilcakes are obtained. These oilcakes containing nearly 10 per cent oil are used as cattle feed and fertilizers and the oil in them is practically going to waste. By solvent extracting the cakes nearly 150,000 tons of oil could be recovered per year.

Another important way of increasing the oil production, which unfortunately has been badly neglected and needs immediate attention, is the utilization of cottonseed. India is one of the major cotton producing countries in the world, and the production of cottonseed is about 1.1 million tons per year from which nearly 150,000 tons of oil could be obtained. At present, the cottonseed is used only for cattle feed which is a very inefficient way of utilizing it.
The most appropriate way to recover the oil being wasted presently in the above two cases is by using solvent extraction procedures which if adopted will increase the total output of oil by 300,000 tons and go a long way in meeting the minimum fat requirements of the country.

One of the major factors hindering the establishment of the solvent extraction industry in India has been the difficulty of obtaining a suitable solvent at a cheap price.

The conventional solvents used in the U.S.A. are normal hexane and other low boiling petroleum fractions. In European countries benzene, trichloroethylene and cyclohexane have found much favor. In India there are resources neither to obtain petroleum solvents nor the other usual solvents like benzene, cyclohexane and trichloroethylene. It has, therefore, to depend entirely upon foreign countries for the supply of the conventional solvents for its extraction plants. These solvents become enormously costly due to very high import duties and the transit charges. Moreover, international events tend to make their supply uncertain and irregular, thus paralyzing the extraction industry. In order, therefore, to keep its solvent extraction industry absolutely free of foreign dependence and also because of the abnormal cost of the imported solvents, there is an urgent need to study the suitability of an indigenous solvent for extraction purposes.

Among the solvents available in plenty in India, the cheapest to compare with other imported solvents is ethanol.
The production of ethyl alcohol every year is about 10 million gallons in the form of absolute alcohol, 4 million gallons in the form of rectified spirit and 3 million gallons in the form of denatured spirit and is available at almost half the price of the imported conventional solvents. Alcohol is known to be a good solvent for oils at or above its boiling point. During the last war the Japanese were reported to have used ethanol as a solvent for a batch soybean extraction plant at Dairen, Manchuria (20, 28). Considerable work has been done in this country at the Northern Regional Research Laboratory, Peoria, Illinois (7, 8, 11), which has established alcohol to be a good solvent for soybean oil, producing a meal highly nutritious and of improved flavor. Some laboratory scale work done in India (39, 40) has also demonstrated the potentialities of ethyl alcohol for extracting cottonseed and peanut oils.

Thus for reasons mentioned earlier and in view of its plentiful supply at rather cheap price, ethyl alcohol is bound to become a very popular solvent in India if an economical method for its use is worked out. It may also become very popular in countries like China, Japan and Argentina where alcohol is more cheaply and abundantly available than the petroleum solvents.
Ethanol as a Solvent\textsuperscript{1}

Since the choice of the solvent plays a decisive part in the successful operation of the process, it is probably appropriate at this stage to discuss the advantages and disadvantages of alcoholic extraction and to examine closely as to how ethanol compares with the conventional solvents.

An ideal solvent must be stable, should have a uniform composition and a constant boiling range, which should not exceed 100°C., a low specific heat and a low freezing point. It should have a selective solvent power for oil, namely it should not extract pigments and mucilagenous matter with the oil. It should easily wet and penetrate the flakes, but should also be easily removable from the residual oil. It should preferably be noninflammable and its vapor nontoxic and non-explosive. It must have no corrosive action on the usual materials of construction. It should have a low specific gravity, should be easily available, store well, and not deteriorate in transport and above all it must be cheap (46).

Of course, there is no single solvent known that would completely satisfy all the above postulates, hence a compromise has to be made, depending upon the availability and the cost of the solvent.

Alcohol, in addition to having most of the characteristics

\textsuperscript{1}Based on the author's (34) and (35).
of an ideal solvent as postulated above such as low specific heat, and low specific gravity has been shown to possess certain unique advantages when compared to the conventional solvents. It has, however, a few disadvantages also. These disadvantages are its inflammability and its use at higher temperatures and pressures. It is also said that alcohol dilutes easily, thus decreasing its solvent power for oils and that it is very difficult to remove from the extracted oils.

As regards inflammability it is common to all the other conventional solvents and the usual precautions have to be taken.

Alcohol has to be used at temperatures near its boiling point, consequently the equipment has to be specially designed, which may involve extra initial cost of investment. Since the working temperatures in the case of alcohol will be about 80°C, unlike other solvents which are worked at low temperatures, it may be argued that more heat is to be supplied initially to the solvent. But this has an inherent advantage also. The oil dissolves completely in hot alcohol and the major portion of it separates out by cooling the miscella to about 20°C. (7). The usual method of obtaining the oil in the case of other solvents is by the distillation of the solvent. The use of hot alcohol in fact helps to do away with the "distillation step". This is a unique feature of the use of alcohol (9).

Use of the cooling step for the elimination of distillation, obviously substitutes a step that involves only a sensible heat
for one that requires the latent heat of vaporization. Furthermore, most of the sensible heat transfer may be affected in heat exchangers, since the cooled alcoholic solvent must be reheated for reuse in the extractor. Beckel, Belter and Smith (9) have developed a continuous non-distillation extraction process for soybean oil using ethyl alcohol as a solvent. They have investigated the economic aspects of the method and have found that alcoholic process required theoretically 7/10 as much energy as the hexane process.

One other advantage with ethanal is that although impurities are extracted by the alcohol together with the oil, the miscella when cooled to 25-30°C., separates into two layers. The bottom layer consists of pure oil of an excellent quality and little alcohol. The top layer contains alcohol and all the impurities, such as resins, pigments, proteins, and carbohydrates, and can be reused. After several recycles the contaminated alcohol has to be redistilled (7).

The meal resulting from the alcoholic extraction is shown to be of improved flavor (8, 15). It has also been shown that its nutritive value is higher than the meals obtained from other solvents and is bound to command a higher price than the other meals.

Alcohol has a great affinity for water. It easily absorbs moisture from the oleaginous material and the solubility of oil in alcohol is considerably reduced. This absorption can be prevented by simply predrying the material to reduce the
moisture content to less than 3 per cent, so that considerable
dilution does not take place. It has been shown by Beckel
that in the case of soybean flakes if the moisture content
is reduced to about 3 per cent, the moisture was not at all
lost to alcohol, but instead removed water from the latter,
leaving it a better solvent than when first used. He has
used the alcohol 85 times without distilling it and found it
a better solvent than it was at the beginning of the experi-
ment. He also found that the oil content of the flakes was
always reduced to less than 1 per cent in each case.

As regards the argument that it is not possible to remove
the alcohol from the residual oil completely it has been shown
in a recent patent taken out by Beckel and Cowan (10) that
after removing the maximum quantity of oil the alcohol re-
main ing in the miscella is completely removed by distilling
out with benzene.

It is therefore evident from the above, that ethanol has
excellent oil-solvent properties. Its disadvantages are the
same as those of the conventional solvents, however, it has
certain unique properties due to which alcoholic extraction
process possesses the following advantages:

1. It is a simple direct process applicable to all
oilseeds and oil cakes, as well as to many types
of other extractable materials.

2. High grade edible oils can be obtained.

3. Efficiency of extraction is as high as with other
solvents.
4. Over-all processing costs are less by a third of the cost in other extraction methods. Steam costs are almost three quarters. Solvent loss is lower than with petroleum solvents (9, 40).

5. Alcohol is a nontoxic solvent so that even if traces of solvent remain in the extracted products, these are not rendered inedible.

6. Alcoholic extraction results in several valuable by-products which may be recovered such as fatty acids, sugars, lecithins, present in oilseeds and cakes (4, 5).

Thus ethyl alcohol with all the above inherent advantages is bound to become a very popular solvent if an economical process for its use is worked out, especially in Asiatic countries like India, China and Japan where ethyl alcohol is produced in plenty and available more cheaply than the conventional solvents.

Aim of Investigation

Solubility data available in literature and those determined recently by the author (36, 37, 38, 44, 45) are quite favorable for the use of aqueous ethanol as a solvent for extracting all of the commonly used vegetable oils like corn, cottonseed, peanut, sesame and soybean oils. However, this project will pertain to the study of cottonseed oil extraction only.
The main objectives of the project are:

1. To determine the feasibility of extracting cottonseed directly using aqueous ethanol as a solvent.
2. To test the adaptability of the Iowa State College extractor for the above purpose and to devise necessary modifications to it.
3. To determine the optimum operating conditions for the alcoholic extraction of cottonseed.
4. To evaluate the economics of the process.
REVIEW OF LITERATURE

Composition of Cottonseed

Cottonseed is obtained from the annual plant *Gossypium hirsutum* which is grown extensively throughout the world for its long cellulose base fiber. As obtained from the gin or fiber removing process, it is composed of the following physical parts: the kernel or meat which is the inner part of the seed; the hull which is a dark colored shell surrounding the kernel and the linters which are short fibers attached to the hulls.

Since the meats yield the oil and cake which are the most valuable products of cottonseed, processing developments have been focused on the completeness of their removal from the seed. The linters due to their high cellulose content are of value in production of nitrocellulose and viscose rayon. They are also used in coarse spun products such as twine, wicks or gauze. The hulls of cottonseed are the least developed of the major components of the seed. At the present time, they are used as additives to cottonseed meat to adjust the protein to one of the trade standards namely 41 or 36 per cent. The hulls are a good source of furfural yielding about 10 per cent of furfural by weight (5). They contain a considerable amount of
industrially suitable tannins (5). Numerous other uses have been found such as plastic fillers, source of activated carbon and as a base for sweeping compounds.

Cottonseed meal obtained after solvent extraction, on account of its high nitrogen content, is used as a fertilizer and as an animal feed (5, 6).

Pigments of Cottonseed

Cottonseed is peculiar in possessing a toxic pigment system which has introduced many complications in the solvent extraction industry. Considerable work has been done by Boatner and co-workers at the Southern Regional Research Laboratory, New Orleans, Louisiana, to determine the constituents of the pigment system and to evolve quantitative methods for determining their amounts. She has reviewed the entire work on this subject in an excellent chapter in Bailey's book on cottonseed (5).

The most important pigment peculiar to cottonseed is a bright yellow solid called gossypol. It is easily oxidized in the presence of air to a reddish black compound. Gossypol has a chemical formula of $C_{30}H_{30}O_8$ with a structure of 2,2' - bi - 1,6,7 - trihydroxy - 3-methyl - 5-isopropyl - 8-aldehydronaphthyl (5). Due to its toxic properties the removal or destruction of gossypol in processing cottonseed meal is of very great importance and consequently
is one of the major problems involved. The pigments are located throughout the tissue of the meat in glands which are strong semirigid, ovoid shaped cells about 100 microns long (5). If the gland wall holding the gossypol can be ruptured, the gossypol is readily converted to a nontoxic form called "bound gossypol" by oxidation. In contrast the "unbound" or toxic gossypol is called free gossypol.

Due to their small size the cells are generally not ruptured in the various mechanical operations carried out on the seed during the oil removal. Heating in the presence of moisture will effectively rupture the pigment glands but the degree of heating required to accomplish this also denaturates the proteins (5, 52).

Some work has been done on the removal of gossypol by selective solvents. Dechary and co-workers (18) studied the reduction of gossypol by various solvents and found that methanol, acetone and butanone were best for the purpose.

Olcott (29) reports that gossypol is almost completely extracted by chlorinated hydrocarbon solvents. Boatner found that pigment glands in sliced sections of cottonseeds were unaffected by exposure to moisture free trichloroethylene for 24 hours. Commercial solvent extraction with hexane removes the gossypol to some extent but the amount removed is variable. Wetting the flaked cottonseed with water causes immediate rupture of the pigment glands (13). Similar observation was also made by Bendler and McNeil (12) who extracted cottonseed
meats with hexane and water as cosolvents. By stirring violently for four hours at 4,100 r.p.m. a mixture of de-hulled and flaked cottonseed meal hexane and water, with respective hexane/meal and water/meal weight ratios of 6:1 and 1:5, all the pigment glands of the seeds were broken and the oil content of the meal was reduced to about 1 per cent.

Bollman (14) treated the cottonseed meats with 80 per cent ethanol to extract the coloring matter with relatively little oil, the meats were then extracted with a mixture of hydrocarbons to remove the oil.

Workers at the Southern Regional Laboratory, New Orleans, Louisiana (32, 49, 50, 55, 56) have developed a fractionating process, a modified solvent extraction method by means of which the cottonseed kernel is mechanically separated into pigment glands, gland free meal and lightly colored oil. By adjusting the density of the solvent at 1.378 gms./cc. the intact pigment glands rise to the surface, the gland-free flour sinks to the bottom and the oil and oil-soluble extra-glandular pigments are dissolved in the solvent. The pigment glands and gland-free flour can then be separated mechanically and the oil recovered from the solvent. However, this process could not be commercialized because its economics were not very favorable due to the poor quality of the meal produced in this process.

The amount of gossypol which causes cottonseed meal to be toxic is not definitely established. Lyman, Hale and Holland (26) found that the meal was not toxic to guinea pigs when the
free gossypol content was 0.07 per cent or lower. Reuther and co-workers (42) report that more than 0.03 per cent free gossypol in cottonseed meal will affect the growth of swine and poultry when included in their ration at a normal feeding level.

Commercial Processing Methods

Based on the method used for propelling the solids through the miscella, modern continuous extractors can be conveniently divided into six groups, each of which will be described briefly:

1. **Bucket type extractors**: This type of extractor marks the first real success in continuous extraction and was developed in Germany in the late twenties. It is usually referred to as the Bollman extractor and is characterized by small buckets or baskets with perforated bottoms fixed to endless chains which carry the solid particles through a vapor tight housing while solvent is sprayed into the baskets. Other extractors embodying the same principle have been produced by the French Oil Mill Machinery Company, Blaw Knox Company, and Bamag Ltd.

2. **Screw conveyor type extractors**: Probably the best known system in this category is the Hildebrandt system, which is made up of two vertical tubes connected with each other by a horizontal tube at
their bottom ends, thus forming a U tube. Each of these tubes is fitted internally with a power driven perforated screw conveyor. Oil bearing material is fed into the top of one leg, conveyed downward and picked up by the horizontal screw conveyor and then lifted up by the conveyor in the other vertical leg and discharged at the top of this leg. Solvent is fed in at the top of the leg just below the meal discharge and final miscella leaves the other vertical leg just below the initial feed point, thus ensuring complete countercurrent extraction. Modifications of Hildebrandt resulted in the Wilhelm, Bighouse, Detrex, Ford and Fauth extractors, which differ from the Hildebrandt in the relative positions and angles of the screw conveyors and solvents used.

3. Tower type extractors: In this type the extractor is a vertical tower of large diameter fitted internally with horizontal plates. The plates have segments cut in them through which the flaked oilseeds move from plate to plate downward through the tower. A rotating, centrally positioned shaft fitted with scraper arms acts as the motive force for this movement. The solvent enters at the bottom of the tower and the miscella discharged near the top from an overflow weir. The spent solids are discharged
at the bottom of the tower. These extractors are manufactured by the Allis Chalmers Manufacturing Company and the V. D. Anderson Company.

4. **Endless belt type extractors:** These operate on the percolation principle, utilizing an endless, perforated belt which moves horizontally in a vapor tight housing. The solvent and miscella of low oil content is sprayed on the bed of flaked oilseed as it is carried through the extractor on the belt, and the miscella is collected below the lower belt. The solids are scraped off the belt at the end of the forward path of the belt. The de Smet and Whitehead extractors are the most common of this group.

5. **Compartment type extractors:** These consist of a number of compartments in which the flakes are contacted with the solvent either by percolation or total immersion. The principle of operation is similar to multiple stage batch extractors, with the flakes and solvents moving in countercurrent flow. The stages or compartments are enclosed in one vapor tight housing and the contacting and separation of flakes and solvents are controlled automatically. The Blaw Knox "Rotocel" and the "Kennedy" are two typical extractors of this type.

The extractor developed and patented by the Engineering Experiment Station of Iowa State College
(51) can be considered in most respects similar to the compartment type of extractors. The flakes are totally immersed in this extractor and are held in a series of compact beds by a Redler type chain which moves the flake beds through the solvent. Each individual flake bed acts as a filtering media, and effectively removes most of the fine meal particles from the countercurrent flowing solvent.

6. Filtration extraction system: One of the new processes is the filtration-extraction system (17) developed by the Southern Regional Research Laboratory, New Orleans, Louisiana. It is designed particularly for cottonseed but claims are made for its versatility in processing other oilseeds like peanut and soybean. The process has been licensed to several companies. Commercial plants have been designed and are offered under the names of "Rotofil" by the Blaw Knox Company, "Filterex" by Wurster and Sanger, Inc. and "Filtration-Extraction" by the Lukenwald division of Luken Steel Company.

In this process delinted and dehulled cotton-seed meats are rolled, cooked and then crisped. Continuous countercurrent extraction is accomplished in two units, a horizontal immersion type of mixing
or soaking extractor and a horizontal rotary oliver
vacuum filter of fully enclosed vapor tight con-
struction. The prepared meats are slowly conveyed
through the miscella filled, immersion extractor
and discharged as a slurry on to the filter screen.
From this point miscella of high oil concentration
is drained from the meal by vacuum for oil recovery
in the evaporators. Then as the filter screen
rotates it carries a bed of meal under solvent
sprays of successively decreasing oil concentration
with vacuum drainage between each wash. The miscella
drained from the first wash is pumped into the im-
mersion extractor for slurrying more meal. The last
wash through which the meal bed is rotated is fresh
solvent. This is drained off from the preceding
wash. The fully extracted meal is removed from the
filter screen by a revolving screw for conveying to
the meal desolventizer.

Theoretical Considerations in Solvent Extraction

The process of extracting oil bearing materials is complex
and up to this time there is no generally accepted, well defined
theory of vegetable oil extraction. Several unknown factors
like the physical structure of the solids, the adsorption
effect of the solute by the carrier solid, the distribution
Investigations of the types of solutions and found that the
structure of the solid before extraction. These authors
expanded the theory of diffusion to include consideration of
subsequent experimental work by Cahn and Kastr (30) has

suggested.

efficiency as a function of temperature of the solution and
were used. Further, they correlated the diffusion co-

of the solid was proved that it could permit measurement
of the temperature and independent of the extraction solution.

found that the extraction was one of pure molecular
were implemented with some extract to improve quality of the
mechanical batch extractions of thin porous permeable plates that
refractance, bounded and co-workers (15) carried out experi-
ments of extraction and the control of a short delay of their
several investigators have tried to explain the mechanism

equipment.

measurement and generally of the process taking place in the
temperature. It is still based upon experience rather than the

operations conditions. The design of extraction apparatus,

possible to devise a simple theory for design purposes.

solutions suitable to one specific solution have made it

30
shape of the extraction curve and the rate of extraction were greatly influenced by the structure of the material. They have recommended that the extraction data for different materials be compared only when details of the structures of the solids are known.

This expanded theory has been confirmed by King, Brier and Katz (24), who have presented data on the extraction of porous clay plates as well as soybean flakes of varying thickness. This work indicates close adherence to molecular diffusion when the clay plates are solvent extracted but variation from simple diffusional operation in the case of soybean flakes points out clearly that the cell structure of the flakes is a variable in the extraction process. The failure of the diffusion theory to be applicable to the extraction of the flakes was believed by the authors to be due to the lack of knowledge of the structure of the material and the distribution of the extractables.

Ruth (43) obtained an approximate correlation of the King, Brier and Katz data (24) on the extraction of soybean flakes by trichloroethylene. He noted that the rate of extraction was approximately proportional to the square of the fraction of oil unextracted. He has demonstrated the use of such an equation for calculating the time of extraction in a conveyor type extractor.

Fan and co-workers (19) studied extraction of peanut slices cut with a microtome, using Skellysolve "B" and
Skellysolve "F" and found that the general diffusion equation would correlate their data with some success. The calculated diffusion coefficient was independent of time of extraction but dependent on the initial moisture content of the slices and to some extent on the thickness of the slices.

Wingard and Phillips (54) report that the amount of oil extractable from any given type of oilseed is variable, dependent upon the temperature at which the extraction is carried out and the solvent used. They found that the time required to reduce the residual oil content of cottonseed flakes to 1 per cent by extraction with commercial hexane varied with the \(-1.9\) power of the extraction temperature. They also found that no theoretical basis for the correlation existed and that the relation was purely empirical.

According to Karnofsky (23) there are two possible extraction mechanisms for vegetable oilseeds. These are molecular diffusion and rate of solution of the oil into the solvent. During the flaking operation the cell walls are crushed and the oil which is contained in the cells is released, and this portion is easily removable. However, at this stage the bulk of the cells are intact and the oil in them is not in direct contact with the solvent and therefore is removable with greater difficulty. According to Karnofsky, vegetable oilseeds fail to conform to the diffusion theory completely. Adherence to the theory occurs during the extraction of the easily removable oil. Non-adherence to the
diffusion theory occurs during the extraction of the
difficult to remove oil. In a series of presoaking tests
he found that both mechanisms, diffusion and undissolved
oil, are in operation, with diffusion being most important
initially and the undissolved oil theory applying in the
later period of extraction. Dialysis is also felt to con-
tribute to the theory of extraction. Thus, according to this
author the mechanism seems to be a combination of diffusion,
dialysis, and the solution of slowly soluble materials.

The presence of slowly soluble materials has been fur-
ther investigated by Coats and Karnofsky (16), who report
that the rate of the oil present is relatively independent
of the concentration of the extraction solvent and depends
largely upon soaking time.

Smith (47) feels that the rate equations and rate
constants will be most useful for correlation of the effects
of the variables. The empirical rate equation which he found
applicable was:

\[-\frac{dx}{dt} = K(x + b)^2\]

where \(x\) is the residual oil, \(K\) is the rate constant and \(b\) is
a constant.

The rate constant was found to vary with reciprocal of
temperature, directly with the product of oil and solvent
viscosities and approximately with the reciprocal of the
square of the material thickness. This author concludes
that diffusion controls the entire process in the solvent extraction of the commercial oil bearing materials.

Smutz (46) obtained rate extraction data by extracting soybean particles in a batch extractor with several different solvents under variety of process conditions. He found that the data could be divided into two distinct phases, a falling rate period and a constant rate period. During the falling rate period the rate of oil removal was found independent of the agitation, the ratio of the weight of particles to the volume of the solvent, the nature of the solvent and the original moisture content of the particles. The rate of oil removal and the amount of oil that was removed were a function of the particle size. He correlated data during this period by equation:

\[-dw/d\theta = K_1 \left( \frac{W_o - W_c}{\theta} \right)\]

where

- \( w \) is the weight of oil unextracted at time \( \theta \) divided by the inert solid.
- \( W_o \) is the original value of \( W \) prior to extraction.
- \( W_c \) is the value of \( W \) at the end of falling rate period.
- \( K_1 \) is a constant independent of \( \theta, W, \) agitation, the ratio of the weight of particles to the volume of solvent, the nature of the fat solvent and the original moisture content of the particles.
During the constant rate period the rate of extraction was a function of the original moisture content of the particles, the nature of the fat solvent and the particle size. He correlated the rate data during this period by equation:

\[ \frac{dW}{d\theta} = K_2 \]

where

- \( K_2 \) is a constant independent of \( W, \theta, \) agitation and the ratio of the weight of particles to the volume of the solvent.

Further he was able to correlate rate data of King, Katz and Brier (24) on soybean flakes using trichloroethylene as a solvent, and those of Fan, Morris and Wakeham (19) on peanut slices using petroleum solvents by equations of the same form as above.

Arnold and Patel (2) made rate extraction studies of soybean and cottonseed flakes and found that a moisture content below 10 per cent does not influence the residual oil content appreciably. Juhl (22) in a study of the solvent extraction of cottonseed using trichloroethylene as a solvent found that this material processed satisfactorily and that the residual oil content of the meal could be calculated by empirical equations involving the cottonseed meal diameter, extraction time, the kinematic viscosity, the average flake thickness and the temperature.

Recently Othmer and Agarwal (31) working on the extraction
of soybean flakes using Skellysolve "B" as a solvent have come out with entirely new ideas about the mechanism of solvent extraction of vegetable oils. Neither the ordinary process of molecular diffusion through cell walls, the concentration of the oil in the extracting solvent or miscella, nor the countercurrent use of solvent has been found by authors to affect the extraction of oil from soybean flakes. According to the authors, extraction is a problem of fluid dynamics and extraction rate is physically and mathematically defined by the Hagen-Poiseuille's law governing viscous fluid flow in capillaries.

They found that the rate of extraction is proportional to the 3.5 power of the oil remaining in the flakes at any time of the extraction and decreases approximately as flake thickness raised the fourth power. Further, they found that the retention of oil in the flakes was independent of the solvent composition.

It should be noted, however, that the solvent composition used by them was limited to a maximum of 20 per cent oil. When the solvent composition becomes higher, it reduces the extraction rate. Juhl (22) has proved this in his work cited earlier. Higher residual content was obtained at high miscella concentration when a low solvent to feed ratio was employed. With high miscella concentration at the exit the solvent composition at any section along the extractor was correspondingly higher, hence a lower extraction rate resulted.
Very recently Kulkarni et al. (25) have studied the extractability of raw and cooked cottonseed using Skelly-solve "B" and miscella concentration up to 50 per cent oil as solvents. They also found that the effect of increasing miscella concentration for both the raw and cooked flakes of medium and thick sizes was to slow down the initial extraction rate, which is again in contradiction to Othmer's claim and confirms Juhl's finding. They further observed that the cooked flakes prepared from the medium and thick flakes extracted at a more rapid rate and to a greater degree in all miscella concentrations, than the raw flakes of comparative thicknesses, but the rate and degree of extraction were about equal for the very thin flakes. No explanation has been given by the authors for the above observations.
LABORATORY EXTRACTION RATE INVESTIGATION

Materials

Cottonseeds

The cottonseeds used were prime, delinted seed supplied by Swift and Company, Cairo, Illinois in October 1955.

Solvents

The solvents used were aqueous ethanol of 99.9, 98.0, 95.4, and 91.5 per cent concentrations (weight percentage). The concentrations of the alcoholic solutions were determined by determining the densities by the pycnometer method.

Equipment

Extraction rate apparatus

The extraction rate apparatus used is shown in Figure 1. It consisted of three major parts: a 3-liter round bottomed flask serving as the solvent tank; a straight tube condenser 16 inches in length serving as a solvent preheater; and the extractor. The latter was a glass tube 1 inch in diameter, 6 inches in height, equipped with a jacketed section through which water is circulated from a constant temperature bath.
Figure 1. Extraction rate apparatus.
SOLVENT TANK

THERMOMETER

SOLVENT PREHEATER

EXTRACTION CHAMBER

SOLUTION OVER-FLOW

WATER
The solvent is also heated by the same water similarly. The temperature of the solvent could be controlled very accurately.

**Flake preparation equipment**

This is the same equipment as described and used by Juhl (22). The flaking rolls were constructed from a set of 17 1/2 inch diameter gasoline engine flywheels with 1 5/8 inch faces. The faces of the rolls were machined to give an even flaking surface. The rolls were mounted between parallel I-beams on stud shafts free to rotate in pillow bearings. The bearings of one shaft were fixed to the I-beams and the bearings on the other shaft were free to slide in guides, in a horizontal plane. An adjustable, spring loaded, tie rod was connected to each of the sliding bearings, which enabled variation of the pressure exerted by the rolls and consequently the thickness of the flakes produced. The rolls were driven at 206 r.p.m. by a 1-horsepower, 1160 r.p.m. electric motor and roller chain speed reduction arrangement.

**Procedure**

**Preparation of flakes**

The cottonseeds were dehulled in the Allis Chalmers' type cracking rolls and the hulls were separated from the meats in a vibrating screen shaker. The moisture content of the meats was then determined to know the amount of water to be added to the meats to produce good flakes. It is known that meats
containing about 10 per cent moisture produce flakes of good quality with minimum amount of fines. The necessary amount of water was added to weighed amounts of meats (about 2 pounds for each batch) in a wide mouth jar provided with an air tight cover. The mixture was shaken thoroughly and set aside for overnight to allow the water to soak into the meats. The wet meats were next flaked in the flaking equipment described earlier, after setting the distance between the rolls for the desired thickness of flakes. A composite flake sample was carefully screened over an 8-mesh screen and the flakes remaining on the screen were used for making the extraction runs. The thickness of 20 individual flakes was determined by the use of a micrometer and the average thickness of the batch was determined.

**Extraction rate determination**

An accurately weighed sample of cottonseed flakes (about 15 grams) was added to the extractor, heated to the desired temperature by circulating water from the heated water bath. At the same time solvent heated similarly was allowed to flow at a controlled rate of 10 milliliters per minute. The starting time was taken as the first drop of the miscella flowed from the extraction tube. The miscella was collected in 100 milliliter graduated jars at the rate of 10 milliliters per minute, transferred into tared flasks and evaporated free of solvent on a water bath. At the end of the extraction experiment, the remaining liquid was drained from the extractor and
the flake sample transferred to a soxhlet extractor, where
the residual oil was removed using the drained liquid as
part of the extracting solvent.

The rate of extraction was determined from the total
extractables and the amounts extracted at each time interval.

Results

The objective of this investigation was to study the
effects of the following extraction variables on the residual
extractables:

1. Concentration of aqueous ethanol.
2. Temperature.
3. Moisture content of flakes.

The effect of flake thickness was not studied in this
project, since it is fairly well known as discussed in the
literature review. A uniform flake thickness of 0.013 inch
was used.

The data obtained in this study are plotted in Figures
2 to 12 and tabulated in Table 1.

Figures 2, 3, and 4 are extraction rate curves for flakes
of 6.97 per cent moisture content at 65°, 70°, and 78.3°C.
Figures 5 to 7 and 8 to 10 are similar curves for flakes of
moisture content 12.78 and 16.55 per cent, respectively.

It is seen that all the rate extraction curves follow
the same general pattern. For the first 15 to 20 minutes
Figure 2. Extraction rate curves for cottonseed flakes with 6.97 per cent moisture at 65°C.
Figure 3. Extraction rate curves for cottonseed flakes with 6.97 per cent moisture at 70°C.
Figure 4. Extraction rate curves for cottonseed flakes with 6.97 per cent moisture at 78.3°C.
Figure 5. Extraction rate curves for cottonseed flakes with 12.78 per cent moisture at 65°C.
Figure 6. Extraction rate curves for cottonseed flakes with 12.78 per cent moisture at 70°C.
Figure 7. Extraction rate curves for cottonseed flakes with 12.78 per cent moisture at 78.3°C.
Figure 8. Extraction rate curves for cottonseed flakes with 16.55 per cent moisture at 65°C.
Figure 9. Extraction rate curves for cottonseed flakes with 16.55 per cent moisture at 70°C.
Figure 10. Extraction rate curves for cottonseed flakes with 16.55 per cent moisture at 78.3°C.
Figure 11. Extraction rate curves for cottonseed flakes showing the effect of temperature on residual extractables.
Figure 12. Extraction rate curves for cottonseed flakes showing the effect of moisture on residual extractables.
Table 1. Summary of laboratory extraction rate data for cottonseed flakes with aqueous ethanol.

<table>
<thead>
<tr>
<th>Moisture content %</th>
<th>Temperature °F.</th>
<th>Ethanol Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>99.9%</td>
</tr>
<tr>
<td>16.55</td>
<td>149 (65°C.)</td>
<td>4.28</td>
</tr>
<tr>
<td></td>
<td>158 (70°C.)</td>
<td>3.28</td>
</tr>
<tr>
<td></td>
<td>173 (78.3°C.)</td>
<td>2.88</td>
</tr>
<tr>
<td>12.78</td>
<td>149 (65°C.)</td>
<td>2.36</td>
</tr>
<tr>
<td></td>
<td>158 (70°C.)</td>
<td>2.31</td>
</tr>
<tr>
<td></td>
<td>173 (78.3°C.)</td>
<td>2.19</td>
</tr>
<tr>
<td>6.97</td>
<td>149 (65°C.)</td>
<td>1.45</td>
</tr>
<tr>
<td></td>
<td>158 (70°C.)</td>
<td>1.41</td>
</tr>
<tr>
<td></td>
<td>173 (78.3°C.)</td>
<td>0.78</td>
</tr>
</tbody>
</table>
there is a rapid extraction rate period, followed by a slow extraction period in the last 80 minutes. Similar curves have appeared in the literature for cottonseed and other oils with other solvents. In each one of the above cases, it is seen that the extraction rate decreases with the increase in the water content of ethanol. In other words, better extraction is obtained by increasing the concentration of ethanol.

As is to be expected, and proved for other solvents in the literature, in the case of ethanol extraction of cottonseed higher temperature has a definite effect in the residual oil content of the flakes. This effect is noticeable in all the curves presented. Figure 11 shows the effect of the temperature on the residual extractables.

Figure 12 shows the effect of moisture content on the extraction rate. The moisture in the flakes reduces the extraction rate and consequently higher values of residual extractables are obtained with flakes of high moisture contents. Thus, the residual oil increases with the increase in the moisture content of the flakes.

Table 1 summarizes the entire data. It can be readily seen that the residual extractables decrease with the increase in the temperature and concentration of ethanol but increase with the moisture content of the flakes.

In these experiments residual extractable value was reduced to less than 1 per cent, which is the commercially accepted value for modern solvent extraction equipment.
These laboratory experiments have thus shown that it should be feasible to use ethanol as a solvent for extracting cotton-seed oil on a commercial scale.
PILOT PLANT EXTRACTION INVESTIGATION

The results of the laboratory extraction rate studies presented in the previous section have shown that ethanol can and does extract the oil from cottonseed flakes to about 1 per cent residual extractables, which is the standard practice in the solvent extraction industry.

Due to the inability to duplicate plant operating conditions in rate extraction experiments, it becomes necessary to test the validity of the results in a pilot plant extractor similar to the one to be used commercially. Since one of the aims of this project was to test the feasibility of the use of the Iowa State College extractor for the ethanol extraction of cottonseed, a pilot plant similar to it built and used by Juhl (22), was used in this investigation.

Materials

Cottonseeds

The cottonseeds used were prime, delinted seeds supplied by the Southern Cottonseed Oil Company, Memphis, Tennessee in February 1956.

Solvents

The solvents used were aqueous ethanol of 99.90, 95.40,
and 91.50 per cent (by weight) concentrations.

Equipment

flake preparation equipment

The flake preparation equipment used in this study was an Allis Chalmers' experimental unit. It consisted of two sections, a cracking section and the flaking section. Each section consisted of a pair of rolls 9 1/2 inch in diameter and 14 1/2 inch in length. The unit is so designed that either one or both the sections could be used at the same time.

pilot plant countercurrent extractor

The pilot plant investigation was carried in the pilot plant unit, details of which are shown in Figures 13, 14 and 15.

Flakes to be extracted were carried countercurrent to the solvent through the outer loop by a Redler type conveyor, 25 feet in length and consisting of modified circular flights, 2 inches in diameter, attached to standard No. 35 roller chain at intervals of 3 inches along the chain. A section of the chain is shown in Figure 14. The loop casing enclosing the chain conveyor was made of standard 2 inch pipe. This casing was surrounded by a steam jacket of standard 4 inch pipe, along its upper length to form the first of three driers, or meal desolventizer sections. Extracted meal was discharged
Figure 13. A view of the pilot plant extractor.
Figure 14. Conveyor chain and flights.
Figure 15. Flow diagram of the pilot plant extractor.
from the chain at the left end (Figure 15) of this first drier through a connecting chute into the second drier section. The second and third driers consisted of 2 1/2 inch pipe surrounded by 4 1/2 inch steam jackets, and connected through another vertical chute at the discharge end of the second drier. The meal finally was discharged at the left end of the last drier. All drier sections were insulated with a 1 inch layer of magnesia.

The lower horizontal part of the loop was heated by a steam coil. Ten equally spaced copper-constantan thermocouples, connected to a 20-point selector switch and a Leeds and Northrup portable potentiometer, were used to measure the temperatures at different points. The average of these points was taken as the average extraction temperature.

The main drive section consisted of a 3/4 horsepower motor, which was connected through a speed reducer and a series of chains and sprockets to the conveyor drive sprocket, which was housed as shown in Figure 15 at the upper left hand corner of the conveyor loop casing.

A series of sprockets were used on the speed reducer shaft in order to provide changes in the speed of conveyor chain and hence a change in feed rate and extraction time.

The solvent entered the extractor loop between the two glass sections on the right hand leg of the loop (Figure 15) through a rotameter.
Distillation still

Since in this investigation large amounts of ethanol were used, it was considered desirable to recover the ethanol from the alcoholic miscellas. For this purpose a distillation still was set up.

The still consisted of a vertical glass column 4 inches in diameter and 6 feet high packed with Berl Saddles. A steam heated boiler was provided at the bottom to heat the miscella and a condenser at the top to condense the alcohol vapors. The miscella was fed to the boiler from the miscella drum, set on the second floor, by gravity. The still could be operated continuously.

Flake drier

After a dozen pilot plant runs were made, it was felt necessary to dry the flakes. For this purpose a galvanized sheet 2 1/2 feet in width and 6 1/2 feet in length was used. This sheet was heated on two steam heated steel plates of about the same total dimensions. This drier provided enough flakes for two to three runs.

Procedure

Experimental countercurrent extractions were carried out in the extractor described above, to study the effect of extraction variables on the residual oil and free gossypol content of the meal and the oil quality in terms of color,
refining loss and free gossypol content.

The experiments were divided into "series" in each of which the purpose was to study the effect of one of the extraction variables. Each series was subdivided into a number of runs in which the effects were studied over a wide range. The variables considered were:

1. Extraction temperature
2. Extraction time
3. Moisture content of flakes
4. Concentration of aqueous ethanol
5. Solvent to feed ratio

As in the case of laboratory extraction rate studies, the effect of flake thickness was not studied in this project, since it is fairly well established. Instead a uniform flake thickness of 0.013 inch was maintained in all the runs in the pilot plant work.

The general operational procedure for all runs consisted essentially of three steps:

1. Preparation of flakes
2. Extraction
3. Analysis of products

Preparation of flakes

Cottonseeds were dehulled in the double rolled cracker described earlier. The hulls were separated from the meats in a vibrating screen shaker. The moisture content of the
meats was then determined.

Fifty pounds of cottonseed meats were weighed out into a barrel provided with an air tight lid. The necessary amount of water (to provide about 10 per cent moisture content in the flakes) was added to the meats. The barrel was closed well and rolled for about an hour to ensure thorough mixing. The wet meats were then left overnight to ensure thorough penetration of water.

After setting the distance between the flaking rolls for the desired flake thickness, the necessary amounts of meats were flaked.

**Extraction**

Prior to making any actual extraction runs, preliminary experiments were made to determine the relation between sprocket size, chain speed, extraction time and the feed rate. The results are tabulated in Table 2. This table was used subsequently in setting some of the desired operating conditions.

Before making any extraction run, the following operating variables had to be set: concentration of ethanol, moisture content of flakes, feed rate, extraction temperature, extraction time, solvent to feed ratio and hence the solvent rate.

Before the start of actual extraction, a warm-up period was required during which the desolventizers and extractor loop were brought up to the desired operating temperature.
Table 2. Operating characteristics of the pilot plant for cottonseed extraction.

<table>
<thead>
<tr>
<th>Number of teeth in sprocket</th>
<th>Chain speed (inches per minute)</th>
<th>Extraction time (minutes)</th>
<th>Feed rate(^a) lbs. per hour (dry basis)</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>8.10</td>
<td>12.50</td>
<td>24</td>
</tr>
<tr>
<td>30</td>
<td>4.10</td>
<td>25.00</td>
<td>12</td>
</tr>
<tr>
<td>15</td>
<td>2.10</td>
<td>50.00</td>
<td>6</td>
</tr>
<tr>
<td>10</td>
<td>1.40</td>
<td>75.00</td>
<td>4</td>
</tr>
</tbody>
</table>

\(^a\)Feed consisted of cottonseed flakes 0.013 inch thickness and known moisture content.

The extractor conveyor was then started followed by the feed and solvent. The feed rate of the flakes was measured by recording the weight of flakes fed as determined by the loss in weight of the containing vessel in 20 minutes. The solvent rate was controlled by the rotameter reading. Approximately two hours after the feeding of the flakes began, the unit approached the steady state conditions and at this time two gallons of miscella sample and two samples of meal were collected. The length of each run varied depending upon the extraction time used. After the run was completed, the unit
was emptied and made ready for the next run. The miscella, other than the samples withdrawn for analysis, was collected in a 45 gallon drum and when the drum was full, it was fed to the distilling column, described earlier, for the recovery of alcohol.

Analysis of products

The products obtained in a particular run were analyzed to determine the efficiency of extraction.

Meal analysis. The samples of meal obtained were analyzed for moisture content and residual oil using normal hexane as solvent by the official A.O.C.S. methods (1). The residual oil was determined on a moisture free basis.

Miscella analysis. The miscella analysis was done for only those eight runs shown in Table 6, for which the operating conditions were optimum and extraction efficiency quite high. The miscella collected in the gallon jug was allowed to cool at the room temperature (28°C.) The solvent rich upper layer was decanted and a small portion of it analyzed for oil content. A small portion of the lower oil rich layer was analyzed for ethanol content.

Oil analysis. Ethanol was boiled off from the oil rich layer obtained in each of the eight runs analyzed above, and the oil obtained was dried in an air oven. The oil in each case was analyzed for color, and refining loss using the standard A.O.C.S. methods (1).
Gossypol determination. Free gossypol was determined in the extracted oil and meal samples obtained in the eight runs listed in Table 6 using the official A.O.C.S. methods (1).

Results

The flakes as fed to the extraction unit contained 35.20 per cent oil and a free gossypol content of 1.16 per cent on a moisture free basis. The results of the pilot plant investigation carried on to determine the adaptability of the process and the effects of the variables on the extraction efficiency and the quality of the resulting products are tabulated in Tables 3 to 9. The effect of each of the extraction variables will be discussed.

Extraction temperature

The effect of extraction temperature can be easily seen from the data for runs 1 to 6 in Table 3. As in the case of laboratory extraction rate studies, it is observed that increasing the extraction temperature results in better extraction which could be expected since the extracting solution is less viscous, which results in higher diffusion rates in the flakes and the solvent. It is also observed that best extraction is obtained with ethanol at or near its boiling point (173°F.). For that reason, subsequent experiments with other concentrations of ethanol were performed at the boiling point of the solvent.
**Table 3. Pilot plant extraction of cottonseed with 91.50 per cent ethanol.**

<table>
<thead>
<tr>
<th>Run</th>
<th>Moisture content of flakes (per cent)</th>
<th>Average extraction temperature (°F.)</th>
<th>Solvent to feed ratio</th>
<th>Extraction time (minutes)</th>
<th>Residual oil (per cent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10.60</td>
<td>150</td>
<td>2.0</td>
<td>50</td>
<td>20.10</td>
</tr>
<tr>
<td>2</td>
<td>10.60</td>
<td>160</td>
<td>2.0</td>
<td>50</td>
<td>17.89</td>
</tr>
<tr>
<td>3</td>
<td>10.60</td>
<td>172</td>
<td>2.0</td>
<td>50</td>
<td>15.07</td>
</tr>
<tr>
<td>4</td>
<td>2.32</td>
<td>150</td>
<td>3.0</td>
<td>75</td>
<td>14.71</td>
</tr>
<tr>
<td>5</td>
<td>2.32</td>
<td>160</td>
<td>3.0</td>
<td>75</td>
<td>12.18</td>
</tr>
<tr>
<td>6</td>
<td>2.32</td>
<td>172</td>
<td>3.0</td>
<td>75</td>
<td>10.78</td>
</tr>
<tr>
<td>7</td>
<td>1.80</td>
<td>172</td>
<td>3.0</td>
<td>75</td>
<td>9.61</td>
</tr>
<tr>
<td>8</td>
<td>1.80</td>
<td>172</td>
<td>4.0</td>
<td>75</td>
<td>9.12</td>
</tr>
<tr>
<td>9</td>
<td>1.80</td>
<td>172</td>
<td>5.0</td>
<td>75</td>
<td>8.32</td>
</tr>
</tbody>
</table>

aData common to all runs:
- average flake thickness = 0.013 inch

bResidual oil calculated on moisture free basis.
Extraction time

Extraction time was the next variable studied. The capacity of an extractor is determined by the extraction time. Table 2 shows the relation of the capacity of the extractor unit at different extraction times, and the former is in inverse proportion to the latter. While longer extraction times result in better extraction, they reduce the capacity of the extractor. Therefore, the extraction plants have to operate at optimum conditions so as to get reasonably good extraction, and also maintain the economical capacity of the plant. The results of the variation in this variable are evident from the data in runs 10 to 13 in Table 4 and runs 22 to 25 in Table 6. It is seen from these data that longer extraction time results in better extraction.

Moisture content of flakes

This was the most significant variable studied. In most of the extraction processes, solvents nonmiscible with water are used, and the moisture content of the flakes is generally maintained at about 10 per cent. However, in the case of ethanol, the moisture in the flakes is absorbed thus diluting the ethanol and decreasing its solvent properties. The results of the variation of this variable are shown in Tables 3, 4 and 5 and will be discussed in detail. Flakes containing about 10 per cent flakes are generally used in the case of extraction with other solvents, and therefore the initial runs in the study of this variable were made with
Table 4. Pilot plant extraction of cottonseed with 95.40 per cent ethanol\(^a\)

<table>
<thead>
<tr>
<th>Run</th>
<th>Moisture content of flakes (per cent)</th>
<th>Average extraction temperature (°F.)</th>
<th>Solvent to feed ratio</th>
<th>Extraction time (minutes)</th>
<th>Residual oil (per cent)(^b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>6.80</td>
<td>172</td>
<td>4</td>
<td>50</td>
<td>5.95</td>
</tr>
<tr>
<td>11</td>
<td>6.80</td>
<td>171</td>
<td>4</td>
<td>75</td>
<td>4.50</td>
</tr>
<tr>
<td>12</td>
<td>3.70</td>
<td>172</td>
<td>4</td>
<td>50</td>
<td>4.85</td>
</tr>
<tr>
<td>13</td>
<td>3.70</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>3.65</td>
</tr>
<tr>
<td>14</td>
<td>3.70</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>3.46</td>
</tr>
<tr>
<td>15</td>
<td>3.70</td>
<td>172</td>
<td>3</td>
<td>75</td>
<td>2.90</td>
</tr>
<tr>
<td>16</td>
<td>2.32</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>2.53</td>
</tr>
<tr>
<td>17</td>
<td>2.32</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>2.38</td>
</tr>
<tr>
<td>18</td>
<td>1.80</td>
<td>172</td>
<td>2</td>
<td>75</td>
<td>1.98</td>
</tr>
<tr>
<td>19</td>
<td>1.80</td>
<td>172</td>
<td>3</td>
<td>75</td>
<td>1.54</td>
</tr>
<tr>
<td>20</td>
<td>1.80</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>1.48</td>
</tr>
<tr>
<td>21</td>
<td>1.80</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>1.42</td>
</tr>
</tbody>
</table>

\(^a\)Data common to all runs:
average flake thickness = 0.013 inch

\(^b\)Residual oil calculated on a moisture free basis.
Table 5. Pilot plant extraction of cottonseed with 99.90 per cent ethanol

<table>
<thead>
<tr>
<th>Run</th>
<th>Moisture content of flakes (per cent)</th>
<th>Average extraction temperature (°F.)</th>
<th>Solvent to feed ratio</th>
<th>Extraction time (minutes)</th>
<th>Residual oil (per cent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>22</td>
<td>10.42</td>
<td>171</td>
<td>2</td>
<td>25</td>
<td>14.10</td>
</tr>
<tr>
<td>23</td>
<td>10.42</td>
<td>171</td>
<td>2</td>
<td>50</td>
<td>7.75</td>
</tr>
<tr>
<td>24</td>
<td>6.80</td>
<td>172</td>
<td>2</td>
<td>50</td>
<td>6.64</td>
</tr>
<tr>
<td>25</td>
<td>6.80</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>5.57</td>
</tr>
<tr>
<td>26</td>
<td>6.80</td>
<td>171</td>
<td>3</td>
<td>75</td>
<td>3.95</td>
</tr>
<tr>
<td>27</td>
<td>3.70</td>
<td>171</td>
<td>4</td>
<td>75</td>
<td>5.10</td>
</tr>
<tr>
<td>28</td>
<td>3.70</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>3.02</td>
</tr>
<tr>
<td>29</td>
<td>3.70</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>2.64</td>
</tr>
<tr>
<td>30</td>
<td>2.32</td>
<td>171</td>
<td>3</td>
<td>75</td>
<td>2.35</td>
</tr>
<tr>
<td>31</td>
<td>2.32</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>2.35</td>
</tr>
<tr>
<td>32</td>
<td>2.32</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>2.16</td>
</tr>
<tr>
<td>33</td>
<td>1.80</td>
<td>172</td>
<td>2</td>
<td>75</td>
<td>1.76</td>
</tr>
<tr>
<td>34</td>
<td>1.80</td>
<td>172</td>
<td>3</td>
<td>75</td>
<td>1.36</td>
</tr>
<tr>
<td>35</td>
<td>1.80</td>
<td>172</td>
<td>4</td>
<td>75</td>
<td>1.26</td>
</tr>
<tr>
<td>36</td>
<td>1.80</td>
<td>172</td>
<td>5</td>
<td>75</td>
<td>1.25</td>
</tr>
</tbody>
</table>

aData common to all runs:
average flake thickness = 0.013 inch

bResidual oil calculated on a moisture free basis.
such flakes. Runs 1 to 3 in Table 3 and 22 to 24 in Table 5 show the results of extraction with 91.50 per cent and 99.90 per cent ethanol. It is seen that although the extraction was greater with 99.90 per cent ethanol, as is to be expected, yet the residual oil content of only 7.75 per cent was reached. Hence, it was necessary to dry the flakes.

Table 3 shows the effect of reducing the moisture content of the flakes in extraction runs with 91.50 per cent ethanol. It is seen that even with a low moisture content of 1.80 per cent the residual oil is 8.32 per cent, which is very high. The results of extraction with 95.40 per cent ethanol are shown in Table 4. It is seen in this case, that reducing the moisture content improves the efficiency of extraction and that with flakes of 1.80 per cent moisture content in runs 19 to 21 approximately 1.50 per cent residual oil was obtained. Thus by reducing the moisture in this case to 1.80 per cent, the commercially accepted standard of about 1.0 per cent residual oil can be reached.

Table 5 shows the results of extraction with absolute alcohol. The reduction of moisture in the flakes definitely improves the extraction efficiency and in run 36, a residual oil content of 1.25 per cent was reached, which is quite a low figure considering the fact, that ethanol is a very miscible solvent, and loses its solvent property considerably by dilution.
Concentration of ethanol

The next variable studied was the concentration of ethanol. The laboratory extraction runs had shown that better extraction was obtained with ethanol of higher concentration and the pilot plant runs also have shown similar effect. This can be easily seen by comparing results shown in Tables 3, 4 and 5 for comparable runs; for example, runs 8, 20 and 35. However, one noticeable point in the study of this variable is that after the flakes are dried to below 3 per cent, 95.40 per cent and 99.90 per cent ethanol appear to be almost equally effective as solvents for the extraction of cottonseed oil, while 91.50 per cent ethanol appears to be definitely less effective. In Table 6 are shown the results obtained for extraction runs with flake moisture content of 1.80 per cent. It is seen that the maximum difference in the amount extracted in comparable runs is of the order of 0.20 per cent. It is therefore possible to use 95.40 per cent ethanol, which is more easily obtainable by distilling the ethanol layer of the miscella, with a sacrifice of 0.20 per cent oil, than using absolute alcohol, which is expensive and not obtainable from the miscella without going to the complicated process of azeotropic distillation.

The reason for the behaviour of 95.40 per cent and 99.90 per cent ethanol as solvents of about the same effective solvent power when the moisture content of the flakes is quite low can be easily explained. At such low moisture content
Table 6. Variation of residual oil with ethanol concentration and solvent to feed ratio\textsuperscript{a}

<table>
<thead>
<tr>
<th>Run</th>
<th>Ethanol concentration (weight per cent)</th>
<th>Solvent to feed ratio</th>
<th>Residual oil\textsuperscript{b} (per cent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>95.40</td>
<td>2</td>
<td>1.98</td>
</tr>
<tr>
<td>19</td>
<td>95.40</td>
<td>3</td>
<td>1.54</td>
</tr>
<tr>
<td>20</td>
<td>95.40</td>
<td>4</td>
<td>1.48</td>
</tr>
<tr>
<td>21</td>
<td>95.40</td>
<td>5</td>
<td>1.42</td>
</tr>
<tr>
<td>33</td>
<td>99.90</td>
<td>2</td>
<td>1.76</td>
</tr>
<tr>
<td>34</td>
<td>99.90</td>
<td>3</td>
<td>1.36</td>
</tr>
<tr>
<td>35</td>
<td>99.90</td>
<td>4</td>
<td>1.28</td>
</tr>
<tr>
<td>36</td>
<td>99.90</td>
<td>5</td>
<td>1.25</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Data common to all runs:
average flake thickness = 0.013 inch
moisture content of flakes = 1.80 per cent
average temperature of extraction = 172\textdegree F.
extraction time = 75 minutes

\textsuperscript{b}Residual oil calculated on moisture free basis.
the flakes do not lose any moisture to the solvent and consequently the alcohol is not diluted. On the contrary, it is possible that the flakes might absorb some water from 95.40 per cent ethanol, thus increasing its concentration and solvent power.

Similar results were obtained by the workers at the Northern Regional Research Laboratory, who worked on the extraction of soybean oil with aqueous ethanol. This work has been discussed in the literature review.

**Solvent to feed ratio**

The last variable studied was solvent to feed ratio. In general, an increase in the solvent to feed ratio increases the extraction, however, there is an optimum point above which the additional yield of the product does not justify the increased cost of the additional solvent. It is sometimes possible to avoid excess solvent use by reducing the feed rate, however, the reduction in residual oil may be more than offset by the decreased production. Thus, an economically feasible solvent to feed ratio has to be arrived at by considering all the aspects.

Table 6 shows the effect of solvent to feed ratio on the residual oil for flakes of 1.80 per cent moisture. With 95.40 per cent ethanol, it is seen that, if the solvent to feed ratio is increased from two to three, the yield increases by 0.44 per cent, but with a further increase in the ratio to four and
to five, the increase in yield is only 0.06 per cent in each case. In the case of 99.90 per cent ethanol also similar observation is observed. An increase in the solvent to feed ratio from two to three increases the extraction by 0.40 per cent, while further increase of the ratio to four and to five results in an increase of only 0.08 and 0.03 per cent, respectively.

Thus, a solvent to feed ratio of three appears to be optimum for the ethanol extraction of cottonseed.

Optimum conditions

The best results obtained in the pilot plant extraction investigation, which come close to the commercially accepted standard of 1 per cent residual oil are tabulated in Table 6. Since as already mentioned, it is quite convenient to obtain 95.40 per cent ethanol, the optimum operating conditions for ethanol extraction of cottonseed appear to be those set in run 19, although the residual oil in this case is not the lowest of all the runs listed. However, the convenience and economy in cost effected by the use of 95.40 per cent ethanol more than offsets the increased yield obtained by using 99.90 per cent ethanol.

Quality of products

Miscella. The results of miscella analysis for the eight runs listed in Table 6 are tabulated in Table 7. It is
Table 7. Results of the miscella analysis.

<table>
<thead>
<tr>
<th>Run</th>
<th>Per cent oil in solvent-rich layer</th>
<th>Per cent oil in oil-rich layer</th>
<th>Per cent solvent in oil-rich layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>2.74</td>
<td>91.11</td>
<td>8.89</td>
</tr>
<tr>
<td>19</td>
<td>2.81</td>
<td>91.26</td>
<td>8.74</td>
</tr>
<tr>
<td>20</td>
<td>2.85</td>
<td>91.32</td>
<td>8.68</td>
</tr>
<tr>
<td>21</td>
<td>2.89</td>
<td>91.44</td>
<td>8.56</td>
</tr>
<tr>
<td>33</td>
<td>3.03</td>
<td>92.42</td>
<td>7.58</td>
</tr>
<tr>
<td>34</td>
<td>3.16</td>
<td>92.70</td>
<td>7.30</td>
</tr>
<tr>
<td>35</td>
<td>3.24</td>
<td>92.83</td>
<td>7.17</td>
</tr>
<tr>
<td>36</td>
<td>3.30</td>
<td>92.92</td>
<td>7.08</td>
</tr>
</tbody>
</table>

*a Obtained by difference

seen that approximately 3 per cent oil is left in the solvent rich layer of the miscella in these runs. It has been shown by Juhl (22) and also Othmer and Agarwal (31) that miscellas containing up to 20 per cent oil have the same solvent effect as the pure solvent. Thus, it is safe to assume that the alcohol-rich layer obtained by cooling the miscella could be recycled effectively as was shown by Beckel for soybean oil (7).
It is seen that the oil-rich layer contains about 91.0 per cent and 92 per cent oil respectively in the case of 95.40 per cent and 99.90 per cent ethanol runs. The solvent from this layer can easily be removed in an oil stripper, as was demonstrated in the case of alcoholic extraction of soybean oil at the Northern Regional Research Laboratory (9, 10).

**Oil.** The free fatty acids, refining loss and refined oil color obtained for the runs in Table 6 are tabulated in Table 8. It is noticed that the free fatty acids of the resulting oil are very low in all cases. These results are similar to those obtained by Harris, in his extensive work on the extraction of cottonseed by isopropanol (21). Thus, alcohols appear to produce oils of low free fatty acids. The refining loss of the oils obtained in all those runs is around 7.30 per cent, which is quite a low figure and meets the standards for the prime quality oil set by National Cottonseed Products Association (27).

The refined color oil for all the runs are about 5.30, which also satisfies the requirements of the National Cottonseed Products Association for prime quality oil (27).

**Gossypol in oil and meal.** The free gossypol content of the extracted oil and meal are shown in Table 9.

It is seen that the oil samples obtained by extraction with 95.40 per cent ethanol show a free gossypol content of about 0.11 per cent and those extracted with 99.90 per cent
Table 8. Results of oil analysis.

<table>
<thead>
<tr>
<th>Run</th>
<th>Free fatty acids(^a) of oil (per cent)</th>
<th>Refining loss (per cent)</th>
<th>Refined oil(^b) color</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>0.71</td>
<td>7.28</td>
<td>5.23</td>
</tr>
<tr>
<td>19</td>
<td>0.73</td>
<td>7.31</td>
<td>5.32</td>
</tr>
<tr>
<td>20</td>
<td>0.73</td>
<td>7.33</td>
<td>5.31</td>
</tr>
<tr>
<td>21</td>
<td>0.74</td>
<td>7.42</td>
<td>5.33</td>
</tr>
<tr>
<td>33</td>
<td>0.71</td>
<td>7.30</td>
<td>5.42</td>
</tr>
<tr>
<td>34</td>
<td>0.72</td>
<td>7.38</td>
<td>5.36</td>
</tr>
<tr>
<td>35</td>
<td>0.73</td>
<td>7.34</td>
<td>5.52</td>
</tr>
<tr>
<td>36</td>
<td>0.72</td>
<td>7.36</td>
<td>5.40</td>
</tr>
</tbody>
</table>

\(^a\) Free fatty acids determined by A.O.C.S. method (1).

\(^b\) Photometric color by A.O.C.S. method (1).

Ethanol show a free gossypol content of about 0.06 per cent which are, of course, very low when compared to the cottonseed oils extracted with petroleum solvents and trichloroethylene (21, 22).

The reason for such a low free gossypol content of the extracted oil, in spite of the fact that alcohol extracts almost all of the gossypol present in the flakes (as shown
Table 9. Results of gossypol determination in extracted oil and meal.

<table>
<thead>
<tr>
<th>Run</th>
<th>Per cent free gossypol in oil</th>
<th>Per cent free gossypol in meal</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>0.108</td>
<td>0.0002</td>
</tr>
<tr>
<td>19</td>
<td>0.108</td>
<td>0.0002</td>
</tr>
<tr>
<td>20</td>
<td>0.112</td>
<td>0.0002</td>
</tr>
<tr>
<td>21</td>
<td>0.118</td>
<td>0.0002</td>
</tr>
<tr>
<td>33</td>
<td>0.064</td>
<td>0.0002</td>
</tr>
<tr>
<td>34</td>
<td>0.065</td>
<td>0.0002</td>
</tr>
<tr>
<td>35</td>
<td>0.068</td>
<td>0.0002</td>
</tr>
<tr>
<td>36</td>
<td>0.067</td>
<td>0.0002</td>
</tr>
</tbody>
</table>

from the free gossypol content of the extracted cake in the same table), is quite easy to explain. Hot alcohol does extract all the free gossypol along with the cottonseed oil, however, when the miscella is cooled, pure oil separates out and the gossypol remains in the alcoholic layer in which it is more soluble. The small amounts of gossypol in the cooled and separated oil is explained by the fact that gossypol is soluble in cottonseed oil itself, although its solubility in the oil is much lower than in alcohol.
The free gossypol in the extracted meal is almost negligible, and this is easily explained by the fact that all the gossypol in the flakes is extracted by the hot alcohol, in the extraction process.

The free gossypol content of the oil and meal as shown in Table 9 are considerably lower than the maximum limits on those set by the National Cottonseed Products Association (27).

Thus the quality of the oil and the meal obtained by the ethanol extraction of cottonseed are of quite acceptable quality and meet all the standards and specifications of the National Cottonseed Products Association (27).
DISCUSSION

In this section the results obtained in the investigation and their significance for the development of the process will be discussed.

In the laboratory rate extraction study several factors which affect the rate of extraction were studied. The final result showed that with a flake thickness of 0.013 inch, moisture content of 6.97 per cent, extraction time of 100 minutes, residual extractable values of 0.78 and 1.04 per cent are reached with 99.90 per cent and 95.40 per cent ethanol respectively. Under the same conditions for an extraction time of 75 minutes, the residual extractable figures from Figure 4 are 1.50 and 1.90 per cent.

In the pilot plant investigation with a flake thickness of 0.013 inch, moisture content of 1.80 per cent, solvent to feed ratio of 5, extraction time of 75 minutes, the residual oil figures were 1.25 and 1.42 with 95.40 per cent and 99.90 per cent ethanol respectively.

The results of the two studies can not be compared on any theoretical basis because of the different principles involved in the studies. In the laboratory rate extraction study, the flakes are stationary and the solvent moves
continuously, thus the solvent to feed ratio is infinite. In the pilot plant unit, the flakes and solvent move counter-currently at a controlled solvent to feed ratio and extraction time.

Since the final results obtained, based on the residual oil content of the extracted meal are in the same range of values, it is evident that the laboratory stage results do compare fairly well with those obtained in the pilot plant in a general way. Similar observation was made by other work done in this laboratory (3, 22).

One of the significant results of the pilot plant investigation is that 95.40 per cent ethanol acts as good a solvent as absolute ethanol at the boiling point when the moisture content is below 3 per cent. This is explained by the fact that no moisture is lost to the alcoholic solvent at such a low moisture content. Similar results were obtained in the case of soybeans using alcohol as a solvent (7).

Cottonseed flakes contain around 35 per cent oil on a dry basis, and the fact that by direct extraction a residual oil content of 1.50 per cent can be achieved using 95.40 per cent ethanol as solvent is a very significant thing. This study did not involve any work on the heating of the meats, which is known to improve the efficiency of extraction, as was shown by Juhl, as well as Olcott (22, 29). It is probably possible to reach the commercially accepted practice of less than 1.00 per cent residual oil by preheating the meats before
flaking. This phase will have to be studied further before adopting the process.

One important phase of this project, which could not be investigated, is the reuse of the alcoholic phase of the miscella obtained in the process. As previously mentioned, the cottonseed oil along with the other non-glyceride constituents are dissolved in the hot alcohol and when the miscella is cooled to the room temperature, pure oil separates as the lower heavy layer and the alcohol along with other dissolved solids as the upper light layer. It was shown in the pilot plant runs that the oil content of this alcohol phase is only 3 per cent. It should be possible to use this alcoholic phase for further extraction, since it has been shown that such a low amount of oil will not affect the solvent property of the solvent. In fact, Juhl as well as Othmer found that miscellas up to 20 per cent concentration behaved as effective solvents as the pure solvent itself (22, 31). In the case of soybean extraction with alcohol, it was possible to use the alcoholic phase several times for extraction (7), and it is reasonable to presume that the same should hold for cottonseed also. This phase has to be studied in detail before adopting the process on a commercial scale.

It was not possible in the time spent on this project to make any investigations on the desolventizing of the oil rich phase of the miscella. Table 7 shows that this phase contains only about 8 per cent of ethanol and it will be possible to
remove that amount by passing through a stripping column. However, more work has to be done on this phase of this problem.

Harris and co-workers had shown that isopropanol was a good solvent for extracting cottonseed oil, since in addition to good quality oil, meal of very low gossypol content is obtained (21). Present work has shown that alcohol is definitely a better solvent than isopropanol, as regards the quality of the oil and meal is concerned. Table 8 shows that the oil obtained is of very low free fatty acids and refining loss. Table 9 shows that the free gossypol content of the oil is of the order of 0.10 per cent, which could be completely removed during refining, and the free gossypol content of the meal is almost negligible. Thus this type of meal will be definitely better priced than the other meals.

Although all the free gossypol is extracted from the flakes, it does not show either in the oil or the meal. What probably happens is that it gets detoxified during extraction with hot solvent. Some of the gossypol which is dissolved in the oil, shows up in the extracted oil, as shown in Table 9. The alcoholic phase of the miscella might contain some free gossypol also, but this would be easily detoxified during the reuse of this layer for further extraction, or will appear as "rejects" of the distilling still.

No work was done on this project on the protein quality
of the extracted meal, however, laboratory stage work done earlier in India has established that the protein quality of the meal is excellent in ethanol extraction of cottonseed (39). To meet the trade requirements the protein quantity has to be adjusted to 41.0 per cent by adding hulls to the extracted meal, which is the usual practice in this industry.

Since one of the purposes of the project was to devise necessary modifications to the existing design of the present plant, the following suggestions are made to improve the suitability of the plant for the use of aqueous ethanol.

Since the alcoholic extraction of cottonseed involves the use of alcohol at its boiling point, there is a liability of heavy solvent loss by evaporation, when compared to the usual solvents, which are not used at their boiling points. Therefore, necessary steps have to be taken to make the equipment fully leak-proof.

The feeding arrangement in the present set up could be replaced by a better arrangement so that the height of the feed column of flakes, should be effective in cutting down the solvent loss from this end.

The use of suitable heat exchanger to heat the incoming solvent by outgoing hot miscella is suggested to effect considerable economy in the heat requirements of the process.

A settling tank has to be provided for the cooling and subsequent separation of the hot miscella into two layers.

This process will need an arrangement to dry the flakes
and a distilling still to recover the ethanol. These have to be provided.
PROCESS EVALUATION

The direct extraction of cottonseed using aqueous ethanol as a solvent was shown feasible in the pilot plant equipment, resulting in a prime quality crude oil and a light colored, good quality meal with negligible free gossypol content. P'Pool (33) found that the extraction data duplicated commercial plant results for extraction of soybean oil, which enables direct application of the pilot plant results on cottonseed to commercial plant scale.

Thus it is reasonable to assume that the optimum operating conditions established for the pilot plant runs will produce a meal of approximately 1.50 per cent residual oil.

The process as envisaged consists essentially in delinting, and dehulling raw cottonseeds, and conditioning the meats to about 10 per cent moisture. The moist meats are rolled to flakes of about 0.013 inch thickness and dried to about 2.0 per cent moisture. They are extracted in the extractor countercurrent to 95.40 per cent ethanol, the temperature of extraction being maintained at the boiling point of the solvent. The miscella is allowed to cool in a settling vessel which separates into two layers. The upper alcoholic layer is separated and used for further extraction. The lower oil
rich layer is stripped of the solvent and the oil is recovered. The extracted meal is collected and sold as such. A proposed flow sheet for the ethanol extraction of cottonseed is shown in Figure 16.

The final products of this process are prime quality oil, good quality meal, linters and hulls. The economic analysis of this process is worked out based on the available equipment cost, and process data in the literature for a 50 ton per day plant.

Since the complete analysis of the cottonseeds used in this study were not available, typical data for cottonseed analysis has been taken from the extensive data of Harris and Hayward on the extraction of cottonseed by isopropanol (21).

The fixed capital cost and processing data used are the same as in Juhl's analysis (22), which were obtained from A. J. Kaiser of the Crown Iron Works, Minneapolis, Minnesota which is licensed to manufacture the commercial scale plant of the type used in the pilot plant studies patented by Iowa State College.

The prices of the cottonseed and its products were obtained from the most recent information of the U. S. Department of Agriculture (53). The cost of the solvent was based on January 1957 prices.

The estimated installed cost of a complete plant of 50 ton per day capacity plant is $336,000 (22). Although the alcoholic extraction process will involve some modifications,
Figure 16. Flow sheet for the ethanol extraction of cottonseed.
alterations, additions and deletions of some of the equipment, it is assumed that the net price of the modified plant will not exceed the above amount, in order to be competitive. For comparison purposes, the basis of estimating the working capital used is the same as used by Juhl (22). The calculations based on the above assumptions and data are presented in Tables 10 to 12.

It is seen from the data in Table 12, that according to the present prices, the process yields a return on investment of 8.70 per cent. This figure is considerably lower than 14.3 per cent reported by Juhl (22) for trichloroethylene extraction of cottonseed in 1953. However, the difference is due to the tremendous decline in the cost of cottonseed products, when compared to that for cottonseed itself in the last four years.

The figure of 8.70 per cent, which is a low return, is however approximately in the same range as most of the other oil extraction processes are yielding presently, due to adverse market conditions. If the ethanol extracted meal, due to its excellent quality and negligible free gossypol content, yields higher prices than those used in the calculations, the process will become more attractive.

The markets of vegetable oils, protein meal supplements, and other by-products are very unstable and have shown considerable decline in the last five years, resulting in poor returns in this industry (53). However if the trend changes,
<table>
<thead>
<tr>
<th>Product</th>
<th>Base price</th>
<th>Product yield</th>
<th>Product value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Meal</td>
<td>$58.00</td>
<td>917</td>
<td>26.60</td>
</tr>
<tr>
<td>Oil</td>
<td>$0.1145</td>
<td>346</td>
<td>50.20</td>
</tr>
<tr>
<td>Lint</td>
<td>$0.038</td>
<td>190</td>
<td>7.23</td>
</tr>
<tr>
<td>Hull</td>
<td>$5.60</td>
<td>408</td>
<td>1.14</td>
</tr>
<tr>
<td>Gross value</td>
<td></td>
<td></td>
<td>85.16</td>
</tr>
</tbody>
</table>

aRecent prices from The fats and oils situation published by United States Department of Agriculture (53).

bData from Harris, W.D. and Hayward, J.W. (21).

cDifference between total and 2,000 pounds is moisture lost during processing.

dMeal adjusted to 41 per cent protein and 12 per cent moisture by addition of hulls and water.
Table 11. Production cost estimate for a 50 tons per day cottonseed oil extraction plant

<table>
<thead>
<tr>
<th>Costs per ton of cottonseed processed (dollars)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw materials and storage&lt;sup&gt;a&lt;/sup&gt;</td>
<td>52.00</td>
</tr>
<tr>
<td>Delinting and dehulling</td>
<td>10.00</td>
</tr>
<tr>
<td>Direct extraction expense&lt;sup&gt;b&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>power</td>
<td>0.72</td>
</tr>
<tr>
<td>light</td>
<td>0.10</td>
</tr>
<tr>
<td>maintenance</td>
<td>0.38</td>
</tr>
<tr>
<td>fuel</td>
<td>1.64</td>
</tr>
<tr>
<td>solvent</td>
<td>1.20</td>
</tr>
<tr>
<td>water</td>
<td>0.32</td>
</tr>
<tr>
<td>labor</td>
<td>3.60</td>
</tr>
<tr>
<td></td>
<td>7.96</td>
</tr>
<tr>
<td>Indirect extraction expense&lt;sup&gt;b&lt;/sup&gt;</td>
<td></td>
</tr>
<tr>
<td>depreciation</td>
<td>3.36</td>
</tr>
<tr>
<td>insurance and taxes</td>
<td>0.64</td>
</tr>
<tr>
<td>laboratory expense</td>
<td>0.10</td>
</tr>
<tr>
<td></td>
<td>4.10</td>
</tr>
<tr>
<td>Total production cost</td>
<td>74.06</td>
</tr>
</tbody>
</table>

<sup>a</sup>Price of cottonseed from (53) and storage charge estimated.

<sup>b</sup>Data from Kaiser, A. J. of Crown Iron Works Company, Minneapolis, Minnesota from Juhl's thesis (22).
Table 12. Estimated return on investment for a 50 tons per day cottonseed oil extraction plant\textsuperscript{a}

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
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</tr>
</thead>
<tbody>
<tr>
<td>Annual tons processed</td>
<td>15,000</td>
</tr>
<tr>
<td>Annual sales, dollars</td>
<td>1,277,400</td>
</tr>
<tr>
<td>Annual processing cost</td>
<td>1,110,900</td>
</tr>
<tr>
<td>Gross profit</td>
<td>166,500</td>
</tr>
<tr>
<td>Sales and administration expense (5 per cent of annual sales)</td>
<td>63,870</td>
</tr>
<tr>
<td>Net profit</td>
<td>102,630</td>
</tr>
<tr>
<td>Income tax (50 per cent of net profit)</td>
<td>51,315</td>
</tr>
<tr>
<td>Net earnings</td>
<td>51,315</td>
</tr>
<tr>
<td>Return on fixed capital (per cent)</td>
<td>15.30</td>
</tr>
<tr>
<td>Return on fixed plus working capital\textsuperscript{b} (per cent)</td>
<td>8.70</td>
</tr>
</tbody>
</table>

\textsuperscript{a} 300 operating days per year.

\textsuperscript{b} Working capital estimated as 20 per cent of annual sales.
which is quite likely, then this process along with other
extraction processes will be fairly lucrative in this country.

As stated in the early part of this thesis, this process
was considered more likely for commercial exploitation in
Asiatic countries like India, China and Japan. Preliminary
estimates made by the author and other workers in India
(40, 41) have shown that this process, if fully developed
for successful commercial exploitation, will be fairly
attractive for the conditions in India. Lack of information
on the present market conditions and other necessary data
about India make it impossible for the author to evaluate
the economics of the process for the present conditions in
India.
CONCLUSIONS

1. Laboratory extraction rate studies were carried out in a glass apparatus to determine the effects of temperature, moisture content of flakes, and the concentration of aqueous ethanol on the residual extractables for the extraction of cottonseed flakes by aqueous ethanol.

2. Pilot plant extractions of cottonseed flakes by aqueous ethanol were carried out in a small unit, similar to the commercial soybean oil extraction plant developed and patented by Iowa State College, to determine the effects of operating variables and the optimum operating conditions.

3. Both laboratory and pilot plant extraction studies have shown that direct extraction of cottonseed using aqueous ethanol as a solvent is a feasible process, and the optimum operating conditions for it have been established in the pilot plant unit.

4. Based on the results obtained on the pilot plant, it appears likely that the present Iowa State College extractor can be adopted, with some modifications, for the ethanol extraction of cottonseed.

5. Pilot plant extractions have shown that in this process a prime quality crude oil and light colored meal of
excellent quality, with negligible free gossypol content, are obtained.

6. Based on the data available in literature, an economic cost study for a 50 tons per day commercial plant indicated an annual return on investment of 8.70 per cent.
LITERATURE CITED


52. Thornton, M. K. Press room operation as it affects the quality of cottonseed oil. Oil and Soap. 14: 151-152. 1937.


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