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Further purification of food-grade alcohol to make a congener-free product

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Abstract

Most alcoholic beverages contain small amounts of chemicals other than ethanol, the congeners. These are byproducts of the fermentation process of the substrate. Congeners are implicated in contributing to hangover (veisalgia) symptoms and it is therefore considered expedient to remove these substances. This research compared 12 established vodka brands with a new product by GC-MS-olfactometry. A new vodka produced in Iowa from corn was found to be the purest while another corn-based vodka and a potato-based vodka contained eight and 12 impurities each. Eight other commercially available vodkas contained 15–19 impurities and three vodkas showed more than 30 impurities. Neither the raw material nor the country of origin made a difference to the level of the impurities. However, the treatment process was of great importance in terms of reaching lower impurity levels. Multiple distillations and filtration did not seem to benefit the quality, nor did charcoal and activated carbon alone. However, one vodka based on a multiple distilled neutral grain spirit process from corn contained zero measurable volatile impurities. The particular treatment process involved ozonation, followed by granular activated carbon and a nano-noble-metal catalysis and adsorption.

Keywords

Congeners, vodka, ozonation, SPME, GC-MS, impurities

Disciplines

Agriculture | Bioresource and Agricultural Engineering | Food Chemistry | Organic Chemistry

Comments

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Further Purification of Food-Grade Alcohol to Make a Congener-free Product

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Abstract

Most alcoholic beverages contain small amounts of chemicals other than ethanol, the congeners. These are byproducts of the fermentation process of the substrate. Congeners are implicated in contributing to hangover (veisalgia) symptoms and it is therefore considered expedient to remove these substances. The research compared twelve established vodka brands with a new product and involved comparing raw fermentation material, country of origin and purification process. IngeniOz, a new vodka produced in Iowa from corn, was by far the purest. Another corn-based vodka and a potato-based vodka contained 8 and 12 impurities each. Eight other commercially available vodkas contained 15 to 19 impurities and three vodkas had more than 30 impurities. The raw material nor the country of origin made no difference to the level of impurities. However, the treatment process was of great importance to reaching lower impurity levels. Multiple distillation and filtration did not seem to benefit the quality, nor did charcoal and activated carbon alone. However, IngeniOz vodka, based on treatment with a proprietary process contained zero measurable volatile impurities. The product is based on multiple-distilled neutral grain spirit and the treatment process involved ozonation, followed by granular activated carbon and nano-noble-metal catalysis and adsorption.

Keywords
Congeners, vodka, ozonation, SPME, GC-MS, impurities

Introduction

The purity of vodka has been of some interest to the consumer. It is well-known that single distillation and even double distillation can still produce a harsh, leathery taste. Discerning consumers are therefore willing to pay more for a purer vodka. However, there is an even more important reason to remove impurities from alcoholic beverages, i.e. their effect on post-consumption well-being.

Most alcoholic beverages contain small amounts of chemicals other than ethanol. These are by-products of the fermentation process of the substrate, e.g., grains, fruits, and tubers. Congeners are complex organic molecules with some toxic effects including acetone, acetaldehyde, furfural, higher or fusel alcohols, and tannins. The fusel alcohols (also called fusel oils) are mainly 2-methyl-1-butanol, isoamyl alcohol, isobutyl alcohol, and n-propyl alcohol.(*1*) While the main cause of hangover (veisalgia) symptoms is ethanol, congeners may increase symptom severity.(*2,3*) Congeners are implicated in contributing to hangover symptoms and it is therefore expedient to remove these substances.(*4,5*)

A novel process of purifying corn-based ethanol was developed.(*6,7*) The new process utilizes ozonation of ethanol followed by treatment with granular activated carbon and stripping with gas. Ten common congeners were tested, i.e., acetaldehyde, ethyl vinyl ether, 1,1-diethoxyethane, isoamyl alcohol, isoamyl acetate, styrene, 2-pentylfuran, ethyl hexanoate, ethyl octanoate, and ethyl decanoate. A 40 mg/L ozone treatment resulted in > 56 % and > 36 % removals of styrene and 2-pentylfuran, respectively, without significant generation of byproducts. A 55 g/L activated carbon and 270 min adsorption time, resulted in 84 %, >72 %, and >78 % removals of ethyl hexanoate, ethyl octanoate, and ethyl decanoate respectively. CO_2-based stripping, at 675 $L_{Stripping\ gas}/L_{Sample}$, removed 65 %, >82 %, and >83 % acetaldehyde, ethyl vinyl ether, and 1,1-diethoxyethane respectively. A combination of three approaches effectively removed 8 impurities and went a long way in purifying ethanol to achieve a higher quality product.(*7*)

A process similar to the one described in (*7*) was developed to achieve a higher degree of purity to make a vodka called IngeniOz from corn-based ethanol. See Figure S 1. Certain adaptations were made, including multiple distillation before treatment to lower the level of further treatment required. Gas stripping was combined with ozonation, a suitable GAC was developed to remove the oxidized impurities and a new proprietary unit process of nano-noble-metal filtration was developed to further aid the removal of oxidized impurities. Oz Spirits, LLC in Iowa was licensed to produce and sell IngeniOz vodka based on this new process. This study was aimed at establishing differences between different commercial vodkas, including IngeniOz and to establish the effect of raw materials and type of treatment on the number of impurities in these popular alcoholic beverages.

Materials and methods

Commercial vodka samples. Table 1 summarizes the raw material, known preparation information, the country of origin, and packaging material of 13 commercially available vodkas as studied.

Solid Phase Microextraction (SPME). 85 µm Carboxen/PDMS (57334-U, Supelco, Bellefonte, PA) solid phase microextraction (SPME) fiber was used for all samples to extract and pre-concentrate volatile organic compounds (VOCs) from the headspace of vodka samples.

All samples were diluted to 10% ethanol content by diluting 2.5 mL 80 proof vodka to 7.5 mL pure water in a 20 mL amber vial. All diluted samples were subjected to headspace extraction with SPME. The SPME procedure was performed automatically using a CTC Combi PAL™ LEAP GC autosampler (LEAP Technologies, Inc., Carrboro, NC, USA) equipped with a heated agitator. For each sample, the automated sequence started by transferring the glass vial prefilled with diluted vodka to the agitator, set to 40 ˚C, and the vial was equilibrated at this temperature for 5 min with 500 rpm agitation. The fiber was desorbed in the injection port for 2 min for cleaning prior to extraction. The equilibration was followed by exposing the fiber to the headspace of the vial for 5 min while agitating at 500 rpm. After the exposure period, the fiber was immediately inserted into the 260 ˚C GC injector for 2 min for desorption for further separation and analysis.

GC-MS-O. Multidimensional GC-MS-O (MOCON, Round Rock, TX) was used for all analyses. The system integrates GC-O with conventional GC-MS (Agilent 6890N GC / 5973 MS, Wilmington, DE, USA) as the base platform with the addition of an olfactory port and flame ionization detector (FID). The system was equipped with a non-polar precolumn and polar analytical column in series as well as system automation and data acquisition software (MultiTrax™ V. 6.00 and AromaTrax™ V. 6.61, Microanalytics and ChemStation™, Agilent). The general run parameters used were as follows: injector, 260 °C; FID, 280 °C, column, 40 °C initial, 6 min hold, 10 °C /min, 220 °C final, 4 min hold; carrier gas, He. Mass to charge ratio (m/z) range was set between 29 and 280. Spectra were collected at 6 scans/sec and electron multiplier voltage was set to 2100 V. The MS detector was auto-tuned daily.

The identity of compounds was verified using (a) reference standards (Sigma-Aldrich, Fisher, Fluka) and matching their retention times on multidimensional GC capillary column and mass spectrums; (b) matching mass spectrums of unknown compounds with BenchTop/PBM (Palisade Mass Spectrometry, Ithaca, NY, USA) MS library search system and spectrums of pure compounds, and (c) by matching the description of odor character.

A highly trained analyst sniffed GC separated compounds simultaneously with chemical analyses (Figure S 2). Odor evaluations consisted of qualitative comparisons of (a) the number of separated odor events and (b) the total odor defined here as sum of the product of odor intensity and odor event duration for all separated odor events were recorded in an aromagram. An aromagram was recorded by the analyst utilizing the human nose as a detector. Odor events resulting from separated compounds eluting from the column were characterized for odor descriptor with a 64-descriptor panel and odor intensity with Aromatrax software (Microanalytics, Round Rock, TX). The olfactory responses of a panelist were recorded using Aromatrax software by applying an odor tag to a peak or a region of the chromatographic separation. The odor tag consisted of editable odor character descriptors, an odor event time span (odor duration) and perceived odor intensity.

Results

Thirteen commercially available vodkas (Table 1) were analysed for chemical impurities in headspace and associated aromas. Please see Supplemental Material for full details of the results (Figures S 3 to S 22, Tables S 1 to S 20). Only selected examples of one grain-based and two corn-based vodkas are discussed in the following sub-sections.

IngeniOz vodka. Tests were performed to demonstrate the purification effect of the two main stages of treatment, i.e. the effect of ozonation and subsequent granular activated carbon adsorption (GAC). No volatile impurities were detected chemically by mass spectrometer (Figure 1). Only ethanol was detected by human olfaction (Figure 2).

A Swedish vodka from grain. Similar tests were performed on a commercial Swedish vodka from grain. Chemical analysis of this sample resulted in 19 volatile impurities in headspace, detected by mass spectrometer (Figure 3). Identifications of these impurities are given in Table 2. Sensory analysis of this sample resulted in 10 aroma notes in headspace, as detected by human olfaction (Figure 4). Details of these 10 aromas are given in Table 3.

An American vodka from corn. Similar tests were performed on a commercial American vodka from corn. Chemical analysis of this sample resulted in 49 volatile impurities in headspace and identified in Table 4. Sensory analysis of this sample resulted in 11 aroma notes in headspace, as detected by human olfaction (Figure 5). Details of these 11 aromas are given in Table 5.

Summary of 13 vodka analyses. A summary of the analyses of all thirteen vodkas is presented in Table 6.

Discussion

Distillation will not remove high volatile compounds that would be at the base of odor events. Multiple distillation alone cannot get rid of all impurities. One of the vodkas, 5x column distilled, had a much higher number of odor events than two other vodkas with a similar number of impurities. However, the general trend was that higher impurity levels resulted in more odor events.

The source of raw material for fermentation did not seem to play a significant role in the quality of the vodka, certainly not as quantified in the number of impurities, nor in the amount of odor events. This is illustrated by the fact that the five vodkas with the lowest impurity levels were based on four different raw materials. Likewise, it would not seem that the country of origin was unimportant. Both the purest and impurest vodkas originated from the same country and based on the same raw material, corn. The packaging, glass or plastic, made no difference, although there was no direct comparison between different packaging of the same product.

Charcoal or "loose" activated carbon treatment alone does not do much to remove impurities. Neither does multiple filtration.

The 13 vodkas can be ranked according to impurities and odor events as in Table 6. The only treatment able to remove all the impurities was a combination of selective oxidation with ozone, granular activated carbon and nano-noble-metal filtration, as was demonstrated with the new brand IngeniOz vodka.

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Author Contributions

All authors contributed to this manuscript. HvL, LC, JAK developed experimental design. LC performed research, analyzed data, and wrote the original commercial report. SR performed some supplemental analyses. JAK, HvL, SR, WSJ co-wrote the manuscript.

Notes

Dr. Hans van Leeuwen is the founder of Oz Spirits LLC and developed IngeniOz vodka. The IngeniOz brand belongs to Oz Spirits, LLC in Iowa, USA and the proprietary treatment technology belongs to co-author J. (Hans) van Leeuwen. The other co-authors declare no competing financial interest. The research was sponsored by various small grants from the State of Iowa through Iowa State University and contributions by private entities, but other than producing a report, there was no commercial commitment nor any obligation.

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Figure Captions

Figure 1. Total ion chromatogram of VOCs from headspace of IngeniOz vodka by SPME-MDGC-MS-O.

Figure 2. Comparison of aromagram and chromatogram of IngeniOz vodka from headspace SPME-MDGC-MS-O. Only ethanol was detected by human olfaction, and characterized as "alcoholic" with a "Neutral 0" hedonic tone.

Figure 3. Total ion chromatogram of VOCs from headspace of a Swedish vodka from grain (distilled five times, filtered through activated carbon) by SPME-MDGC-MS-O.

Figure 4. Comparison of aromagram and chromatogram of a Swedish vodka from grain (distilled five times, filtered through activated carbon) from headspace SPME-MDGC-MS-O.

Figure 5. Total ion chromatogram of VOCs from headspace of an American vodka from corn (distilled six times, filtered through activated carbon) by SPME-MDGC-MS-O.

Figure 6. Comparison of aromagram and chromatogram of an American vodka from corn (distilled six times, filtered through activated carbon) from headspace SPME-MDGC-MS-O.

Table 1. List of vodkas analyzed

No	Raw material	Purification technique	Country	Bottle material
1	Corn	Neutral grain spirits involving multiple distillation, ozonation, GAC adsorption and nano-noble-metal filtration	USA	Glass
2	Corn	Four column distillation + triple filtration	USA	Plastic
3	Corn	Triple distilled and charcoal filtered	USA	Plastic
4	Corn	Distilled six times, filtered through activated carbon	USA	Plastic
5	Grain	Distilled	Finland	Glass
6	Grain	Distilled five times with five columns	Sweden	Plastic
7	Grain	Distilled	Sweden	Glass
8	Grain	Distilled	Poland	Glass
9	Potato	Distilled four times	Poland	Glass
10	Wheat	Distilled, filtered through loose charcoal	Netherlands	Glass
11	Wheat	Distilled	France	Glass
12	Wheat	Distilled	Russia	Glass
13	Grape	Distilled five times	France	Glass

Table 2. Preliminary identification of VOCs from headspace of a Swedish vodka from grain (distilled five times, filtered through activated carbon)

No	GC column retention time (min)	Chemical name	CAS	Significant ion	MS Spectral Identification Match%
1	5.58	Toluene	108-88-3	91, 92	68
2	8.80	Ethylbenzene	100-41-4	91, 106	94
3	9.08	Xylene(s)		91, 106	93
4	9.43	α-Pinene	80-56-8	93, 77	93
5	10.00	Xylene(s)		91, 106	91
6	10.83	β-pinene	18172-67-3	93, 41	93
7	11.43	*o*-Ethyltoluene	611-14-3	105, 120	75
8	11.65	Δ-3-Carene	13466-78-9	93, 77	95
9	12.25	*dl*-Limonene	138-86-3	68, 93	96
10	12.63	*o*-Cymene	527-84-4	119, 134	81
11	12.98	γ-Terpinene	99-85-4	93, 91	88
12	13.06	Undecane	1120-21-4	57, 43	88
13	13.50	9-Methyl-3-undecene	74630-54-9	70, 41, 55	58
14	13.90	Unknown			
15	14.85	Dodecane	112-40-3	57, 43, 71	93
16	16.45	Tridecane	629-50-5	57, 71, 85	95
17	18.61	Ethyl tridecanoate	28267-29-0	88, 101	33
18	18.78	Viridiflorol	552-02-3	109, 69	50
19	24.03	1,1,3-Trimethyl-3-phenylindane	3910-35-8	221, 143	95

Table 3. Aromas detected by human olfaction from headspace of a Swedish vodka from grain (distilled five times, filtered through activated carbon).

Event#	Aroma Descriptor	Aroma Intensity (%)	Start Time (min)	Width (min)	Event Area (Aroma Intensity × Width × 100)
1	Alcoholic Solvent Neutral 0	50	2.45	0.19	948
2	Solvent Unpleasant -1	40	3.3	0.06	239
3	Solvent Unpleasant -1	30	6.1	0.05	149
4	Plastic Unpleasant -1	30	9.36	0.05	149
5	Mint Neutral 0	40	9.56	0.08	319
6	Plastic Solvent Unpleasant -1	40	11.93	0.25	998
7	Solvent Unpleasant -1	30	12.62	0.07	209
8	Moldy Neutral 0	30	13.27	0.09	269
9	Cardboard Neutral 0	40	15.15	0.14	559
10	Moldy Milky Neutral 0	30	15.81	0.09	269

Table 4. Preliminary identification of VOCs from headspace of an American vodka from corn (distilled six times, filtered through activated carbon).

No	GC column retention time (min)	Chemical name	CAS	Significant ion	MS Spectral Identification Match%
1	3.23	Acetal	105-57-7	45, 73,103	8
2	4.65	2,4-Dimethylhexane	589-43-5	43, 57,85	54
3	5.61	5-Methyl-1-heptene	13151-04-7	70,55,43	35
4	6.43	4-Methyl-octane	2216-34-4	43, 85, 71	88
5	10.93	Styrene	100-42-5	104, 78,51	24
6	11.15	3,3-Dimethyloctane	4110-44-5	43, 71, 57	54
7	11.25	4-Methyldecane	2847-72-5	43, 71, 57	68
8	11.41	2,5,6-Trimethyl-octane	62016-14-2	57, 43	74
9	11.58	2,2,5,5-Tetramethyl-hexane	1071-81-4	57, 71	20
10	11.68	3,7-Dimethyldecane	17312-54-8	43, 57, 71	63
11	11.78	5-Ethyl-2,2,3-trimethylheptane	62199-06-8	57, 56,43	53
12	12.10	2,7,10-Trimethyldodecane	74645-98-0	57, 71, 43	39
13	12.28	*dl*-Limonene	138-86-3	68, 93	95
14	12.58	*o*-Cymene	527-84-4	119, 134	94
15	12.75	1-Dodecanol	112-53-8	70, 56	39
16	12.83	4-Methyl-5-propylnonane	62185-55-1	57, 71	50
17	12.98	α-Terpinyl propionate	80-27-3	93, 121	24
18	13.1	5-Methylundecane	1632-70-8	43, 57,	74
19	13.21	Pentadecane	629-62-9	57, 71	54
20	13.38	2,5,6-Trimethyloctane	62016-14-2	57, 43	63
21	13.46	2,2,4-Trimethylheptane	14720-74-2	57, 56	59
22	13.75	3,3,8-Trimethyldecane	62338-16-3	71, 57	72
23	14.18	3,6-Dimethyloctane	15869-94-0	57, 71	50
24	14.53	3,3,8-Trimethyldecane	62338-16-3	71, 43	69
25	14.66	Benzaldehyde	100-52-7	77, 105	93
26	15.53	6-Ethylundecane	17312-60-6	57, 43,71	63
27	15.65	Ethyl caprylate	106-32-1	88, 101	85
28	15.83	*o*-Vinylphenylacetic acid	81598-12-1	117, 162	39
29	16.08	7,9-Dimethylhexadecane	21164-95-4	57, 71, 85	58
30	16.56	2-Methylundecyl-2-thiol	10059-13-9	41, 55	50
31	16.68	7-Methyl-1-undecene	74630-42-5	43, 69	63
32	16.81	Didecyl sebacate	2432-89-5	57, 71	58
33	17.16	Ethyl nonanoate	123-29-5	88, 101	95
34	17.83	Cuminic aldehyde	122-03-2	133, 148	54
35	18.00	β-Cadinene	523-47-7	161, 204	72
36	18.55	β -Elemene	515-13-9	81, 93,68	86
37	18.65	β -Guaiene	88-84-6	161, 105	93
38	18.75	Epizonarene	41702-63-0	161, 204	93
39	18.81	Cedr-8-ene	469-61-4	119,93	93
40	19.13	Alloaromadendrene	25246-27-9	105, 91	72
41	19.63	Dehydroaromadendrene		159, 105	92
42	19.80	α-Amorphene	23515-88-0	161,105	95

43	20.11	α-Muurolene	31983-22-9	105, 161	94
44	20.25	Aromadendrene	489-39-4	91, 105	94
45	20.43	Δ-Cadinene	483-76-1	161, 204	93
46	20.80	Calamene	483-77-2	159	93
47	20.91	Cinnamaldehyde	104-55-2	131,130	93
48	21.23	Ethyl dodecanoate	106-33-2	88, 101	85
49	23.86	Cadalene	483-78-3	183, 198	91

Table 5. Aromas detected by human olfaction from headspace of an American vodka from corn (distilled six times, filtered through activated carbon).

Event#	Descriptor	Aroma Intensity (%)	Start Time (min)	Width (min)	Event Area (Aroma Intensity × Width × 100)
1	Alcoholic Solvent Neutral 0	50	2.36	0.22	1098
2	Sweet Pleasant +2	40	3.28	0.09	359
3	Mint Pleasant +1	40	12.42	0.06	239
4	Moldy Unpleasant -1	40	12.8	0.06	239
5	Smoky Burnt Unpleasant -2	40	13.59	0.13	519
6	Burnt plastic Skunky Unpleasant -2	50	15.09	0.18	898
7	Moldy Mushroom Resiny Unpleasant -1	60	15.76	0.13	778
8	Mushroom Moldy Neutral 0	40	17.34	0.23	918
9	Smoky Unpleasant -1	30	17.97	0.06	179
10	Sweet Fruity Pleasant +1	50	20.41	0.38	1896
11	Sweet Fruity Pleasant +1	60	20.82	0.25	1497

Table 6. Ranking of 13 vodkas according to the number of impurities and aroma events and total odor present in the headspace of each vodka sample.

Rank	Brand	Country	Number of impurities	Number of odor events	Total odor*
1	IngeniOz	USA	0	1	798
2	Corn-based, 3x distilled, charcoal filtered	USA	8	1	1,048
3	Potato-based, 4x distilled	Poland	12	4	3,313
4	Grain-based	Poland	15	3	1,855
5	Wheat-based	Russia	17	3	2,155
6	Wheat-based	France	14	4	3,196
7	Grain-based	Sweden	16	2	1,846
8	Charcoal filtered	Netherlands	18	3	1,646
9	Corn-based, 4x distilled 3x filtered	USA	19	4	2,284
10	5x column distilled	Sweden	19	10	4,108
11	Grain-based	Finland	31	2	1,896
12	Grape-based, 5x distilled	France	39	7	4,000
13	6x distilled, activated carbon filtered	USA	49	11	8,620

*note: Total Odor = sum of Event Areas; Event Area = Aroma Intensity × Width × 100