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

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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. Copyright © 2015 The Institute of Brewing & Distilling

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Keywords: congeners; vodka; ozonation; SPME; GC-MS; impurities

Introduction

The purity of vodka is 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 byproducts of the fermentation process of the substrate, for example, grains, fruits and tubers. Congeners are complex organic molecules with some toxic effects including acetone, acetaldehyde, furfural and higher or fusel alcohols. The fusel alcohols (or fusel oils) are mainly 2-methyl-1-butanol, isoamyl alcohol, isobutyl alcohol and *n*-propyl alcohol *(1)*. While the main cause of hangover symptoms is ethanol, congeners can increase symptom severity *(2,3)*. Congeners are implicated in contributing to hangover (veisalgia) symptoms and it is therefore considered 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 (GAC) and stripping with gas. Ten common congeners were tested (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 a $>56\%$ and a $>36\%$ removal 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 the three approaches effectively removed eight impurities and went a long way in purifying ethanol to achieve a higher quality product *(7)*.

A process similar to the one described in Onuki et al. *(7)* was developed to achieve a higher degree of purity and was used in developing a new brand of corn-based vodka (Fig. S1 in the Supporting Information). Certain adaptations were made, including multiple distillations 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

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filtration was developed to further aid in the removal of impurities. This study was aimed at establishing differences between different commercial vodkas, including the new purified brand, 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 the 13 commercially available vodkas that were studied.

Ozonation, activated carbon adsorption and gas stripping

This process is explained in detail by Onuki et al. *(7)*. Briefly, ozone was generated and passed through the 79% ethanol sample at a fixed flow rate, where the total ozone dose was time dependent. Post-ozonation ethanol was treated with specific amounts of GAC and agitated for a set amount of time. Air, N_2 or CO_2 was passed through the 79% ethanol sample at a fixed flow rate for a set amount of time. The resulting ethanol was diluted to 10% (v/v) ethanol concentration and analysed as follows.

Solid-phase microextraction

A 85 μm Carboxen/PDMS (57334-U, Supelco, Bellefonte, PA, USA) solid-phase microextraction (SPME) fibre was used for all samples to extract and pre-concentrate the 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 collected by 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 was 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 equilibration was followed by exposing the fibre, which was desorbed in the injection port for 2 min for cleaning the fibre prior to extraction, to the headspace of the vial for 5 min while agitating at 500 rpm. After the exposure period, the fibre was immediately inserted into the 260 °C GC injector for 2 min for desorption for further separation and analysis.

GC-MS-O

A multidimensional gas chromatography - mass spectrometry - olfactometry (MD GC-MS-O) (MOCON, Round Rock, TX, USA) was used for all analyses. The system integrates GC-O with conventional GC-MS (Agilent 6890 N GC/5973 MS, Wilmington, DE, USA) as the base platform with the addition of an olfactory port and flame ionization detector. The system was equipped with a non-polar precolumn (BP-5, 56 m × 530 μm inner diameter × 1.00 μm thickness, SGE, Austin, TX, USA) and polar analytical column (BP-20, 25 m × 530 μm inner diameter, 1.00 μm thickness, SGE, Austin, TX, USA) 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; flame ionization detector, 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/s and electron ionization energy was set at 70 eV. The MS detector was auto-tuned daily as a performance check of the MS.

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

Highly trained human panellists sniffed the GC separated compounds simultaneously with chemical analyses (Fig. S2). Odour evaluations consisted of qualitative comparisons of (a) the number of separated odour events and (b) the total odour defined here as sum of the product of odour intensity and odour event duration for all separated odour events and these were recorded in an aromagram. The aromagram was recorded by a panellist utilizing

Table 1. List of vodkas analysed

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

the human nose as a detector. Odour events resulting from separated compounds eluting from the column were characterized for odour descriptors with a 64-descriptor panel and odour intensity with Aromatrax software (Microanalytics, Round Rock, TX, USA). The olfactory responses of panellists were recorded using the Aromatrax software by applying an odour tag to a peak or a region of the chromatographic separation. The odour tag consisted of editable odour character descriptors, an odour event time span (odour duration) and perceived odour intensity.

Results

Thirteen commercially available vodkas (Table 1) were analysed for chemical impurities in headspace and associated aromas. Since chemical and sensory analysis was performed simulta-

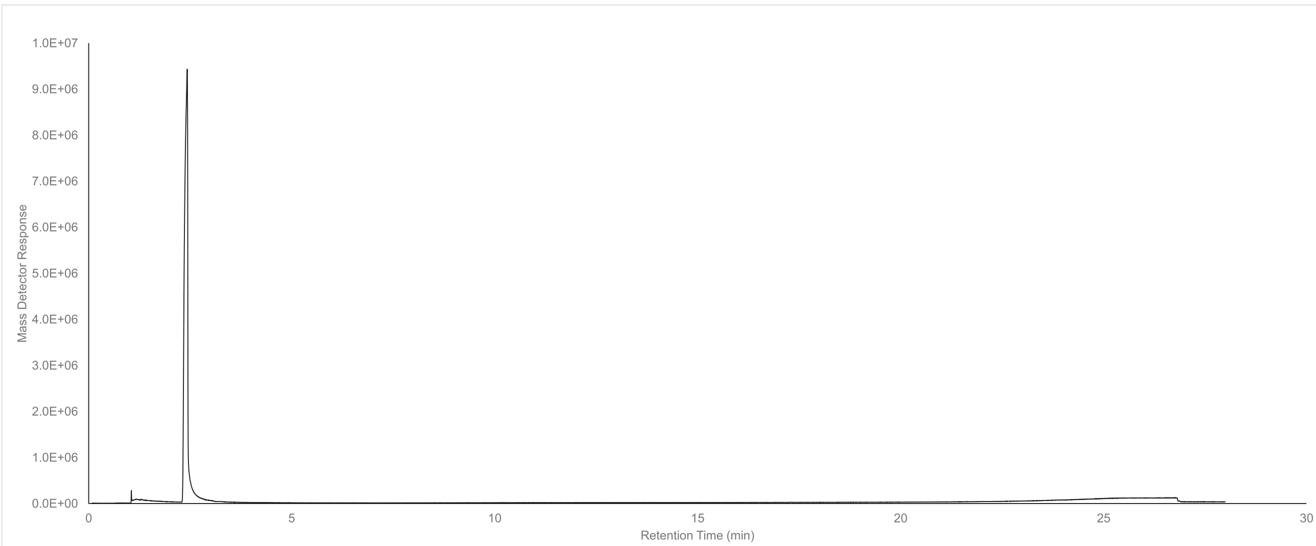

Figure 1. Total ion chromatogram of volatile organic compounds (VOCs) from headspace of new purified vodka by solid-phase microextraction (SPME)-MDGC-MS-O.

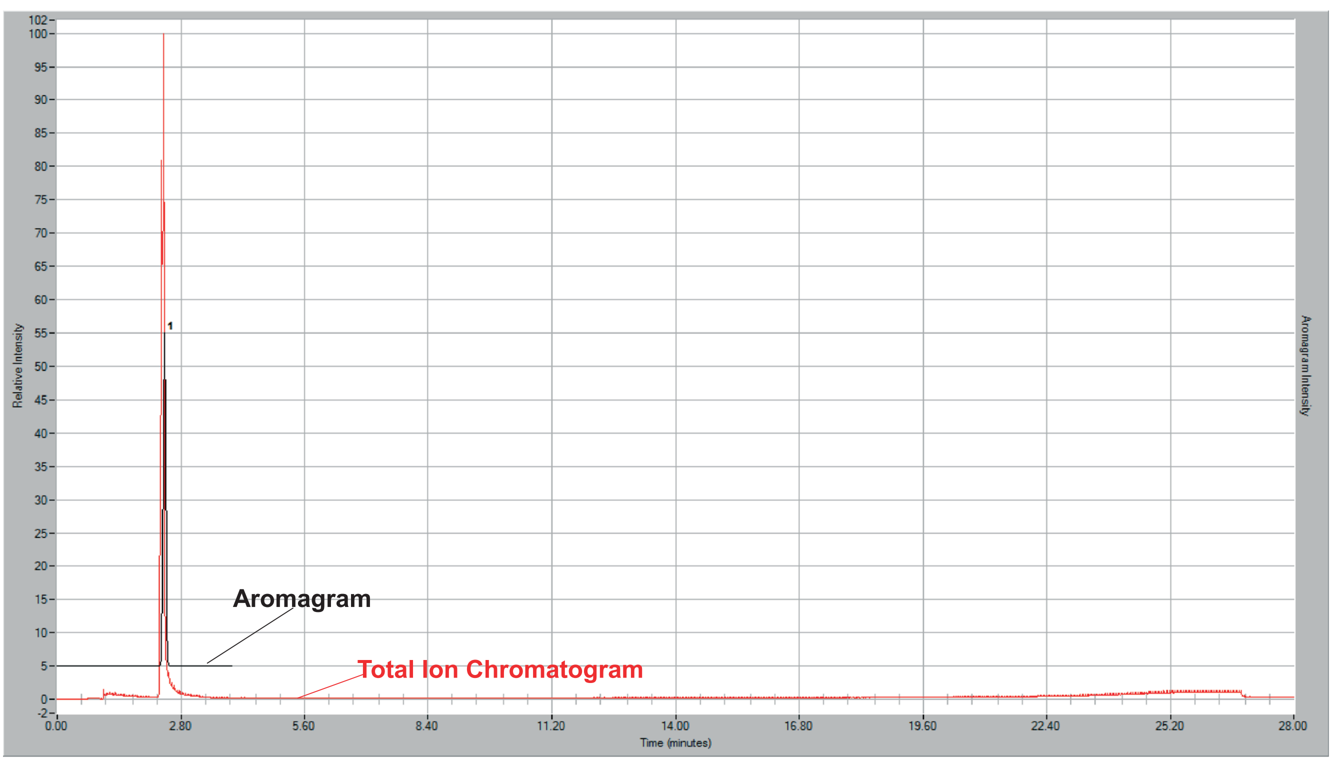

Figure 2. Comparison of aromagram and chromatogram of the new purified vodka headspace by SPME-MDGC-MS-O. Only ethanol was detected by human olfaction, and characterized as 'alcoholic' with a 'neutral 0' hedonic tone.

neously, the odour events can be tentatively identified by matching retention time to the GC-MS compound identification from probability matched spectra. The Supporting Information contains full details of the results (Figs S3–S22, Tables S1–S20). SPME of headspace of water used for dilution was analysed as a control sample and showed no interfering odours or volatile compounds. Only selected examples of one grain-based and one corn-based vodka are discussed in the following subsections.

The 13 vodkas were ranked according to impurities and odour events. One of the vodkas, the 5× column distilled, had a much higher number of odour events than two other vodkas with a similar number of impurities. This illustrates that the distillation did not remove the high volatile compounds that

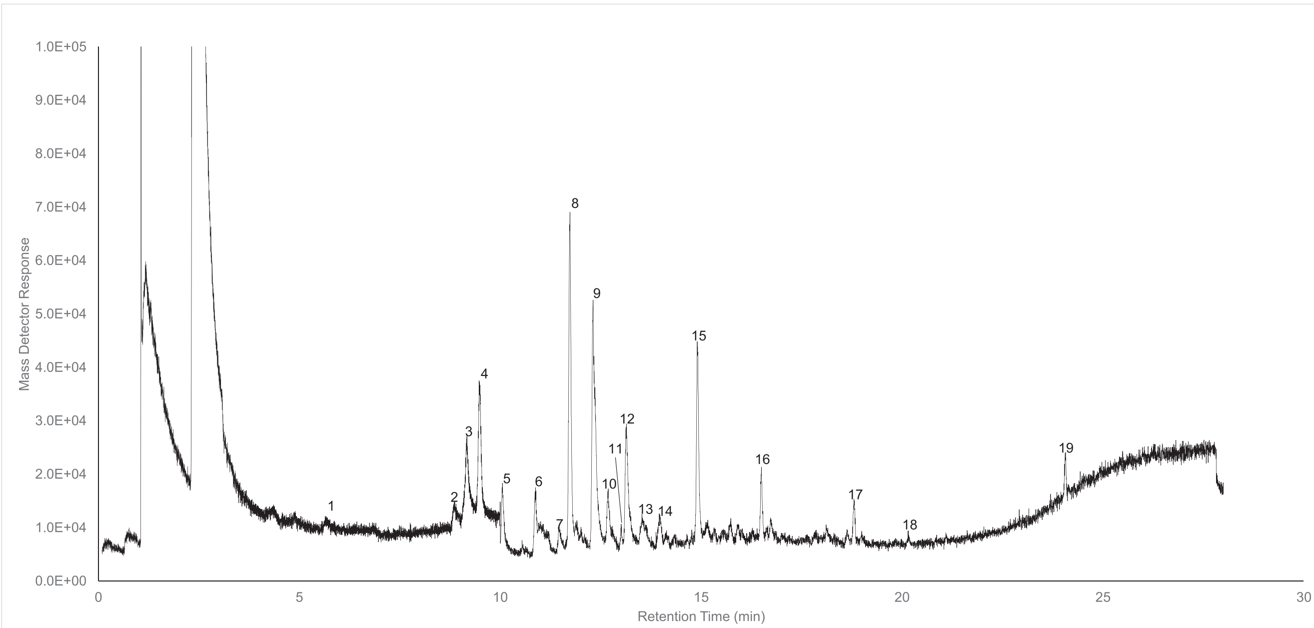

Figure 3. Total ion chromatogram of VOCs from headspace of a Swedish vodka from grain (distilled five times, filtered through activated carbon) by SPME-MDGC-MS-O.

Table 2. Preliminary identification of volatile organic compounds (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

would be at the base of the odour events. However, the general trend was that vodkas with higher impurity levels resulted in more odour events. The number of times distilled is really just a 'commercial expression'. Larger commercial alcohol distillation plants use multistage distillation columns, where each stage could be considered a distillation, and thus a much

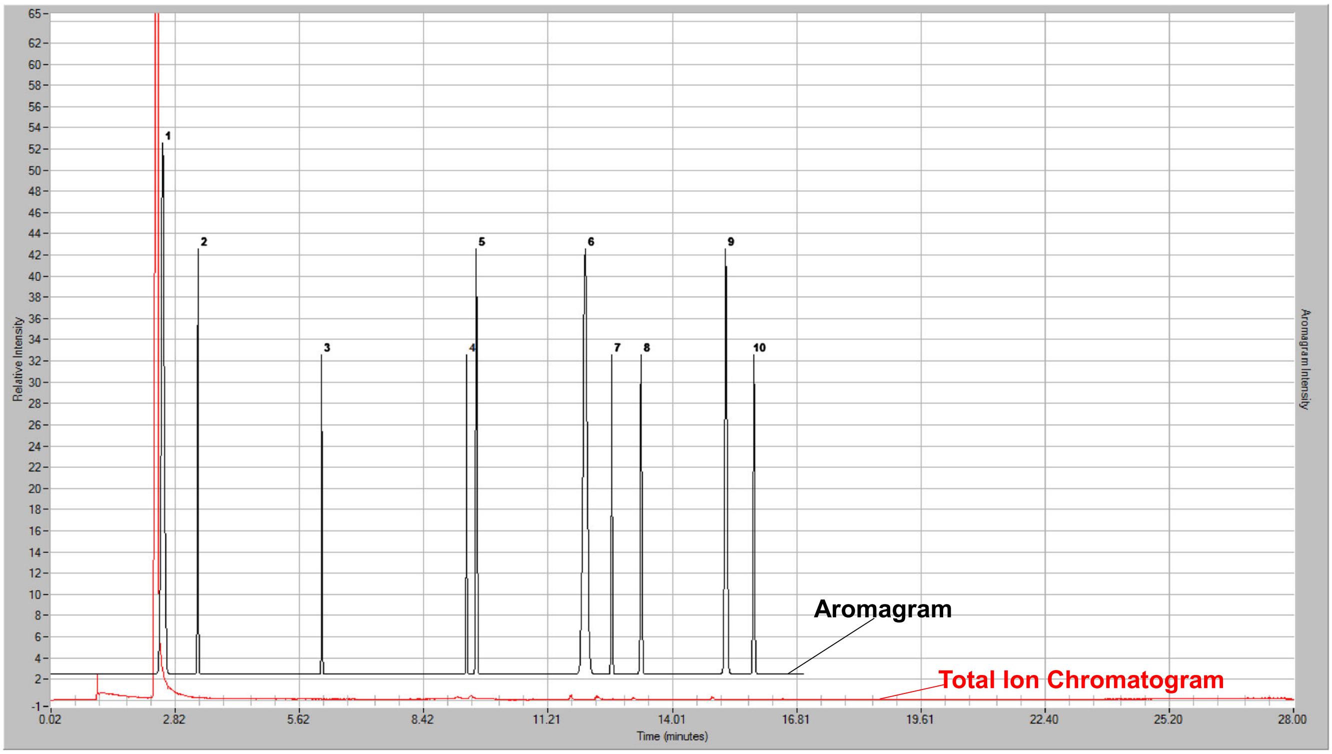

Figure 4. Comparison of aromagram and chromatogram of a Swedish vodka from grain (distilled five times, filtered through activated carbon) by SPME-MDGC-MS-O.

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

Event no.	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	Mouldy Neutral 0	30	13.27	0.09	269
9	Cardboard Neutral 0	40	15.15	0.14	559
10	Mouldy 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

higher number of distillation stages could be claimed if desirable for marketing purposes.

While detailed tests were performed on all 13 vodkas tested, only the results of three the vodkas are presented in the main part of this paper. The Supporting Information to this paper shows the other results. The vodkas are only described in general terms in order not to interfere with any sensitive commercial information.

New vodka from corn using physical-chemical purification

Tests were performed to demonstrate the purification effect of the two main stages of treatment, i.e. the effect of ozonation and the subsequent granular activated carbon adsorption (GAC) process. No volatile impurities were detected chemically by mass spectrometer (Fig. 1). Only ethanol was detected by human olfaction (Fig. 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 the headspace as detected by mass spectrometry (Fig. 3). Identifications of these impurities are given in Table 2. Sensory analysis of this sample resulted in 10 aroma notes in headspace,

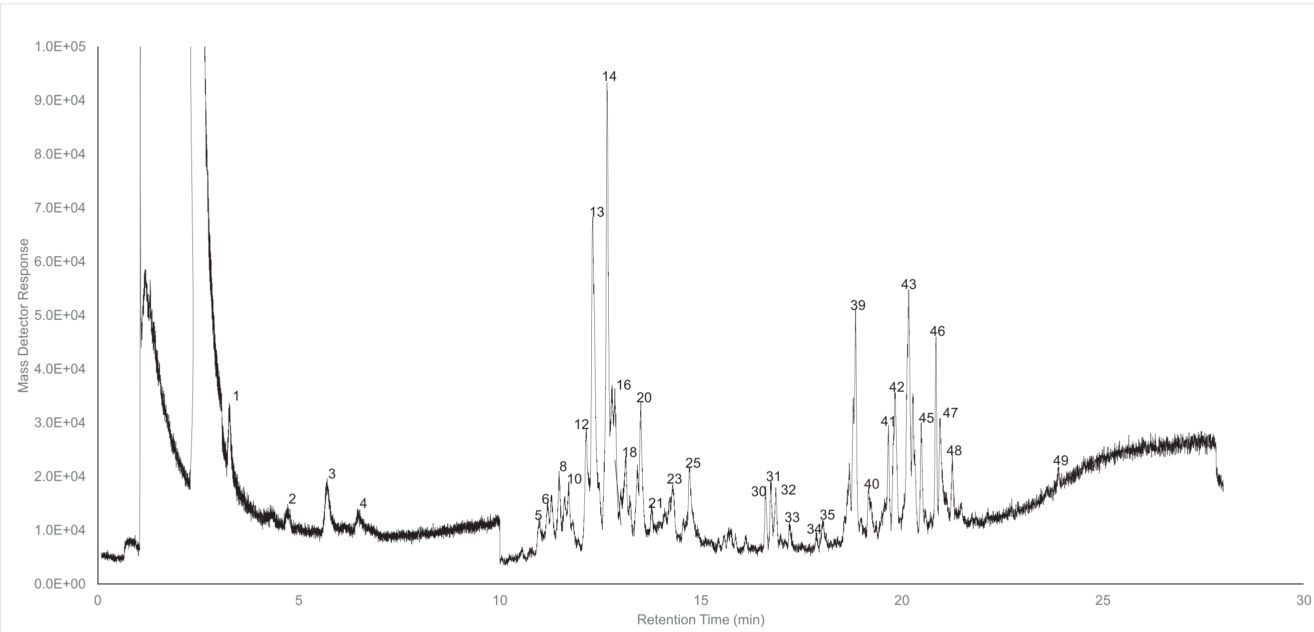

Figure 5. Total ion chromatogram of VOCs from headspace of an American vodka from corn (distilled six times, filtered through activated carbon) by SPME-MDGC-MS-O.

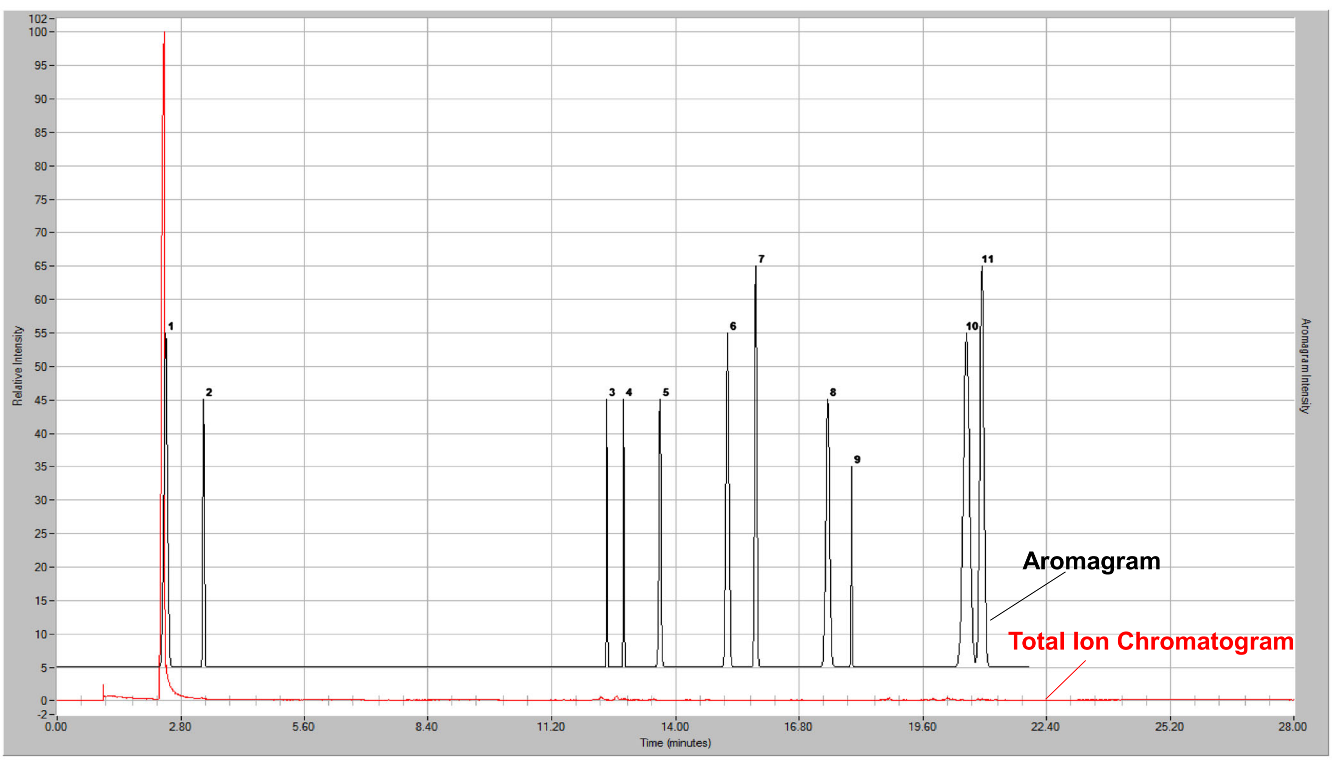

Figure 6. Comparison of aromagram and chromatogram of an American vodka from corn (distilled six times, filtered through activated carbon) by SPME-MDGC-MS-O.

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

Event no.	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	Mouldy 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	Mouldy Mushroom Resiny Unpleasant −1	60	15.76	0.13	778
8	Mushroom Mouldy 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 odour present in the headspace of each vodka sample

Rank	Brand	Country	Number of impurities	Number of odour events	Total odour[a]
1	New purified vodka	USA	0	1	798
2	Corn-based, 3× distilled, charcoal filtered	USA	8	1	1048
3	Potato-based, 4× distilled	Poland	12	4	3313
4	Grain-based	Poland	15	3	1855
5	Wheat-based	Russia	17	3	2155
6	Wheat-based	France	14	4	3196
7	Grain-based	Sweden	16	2	1846
8	Charcoal filtered	Netherlands	18	3	1646
9	Corn-based, 4× distilled 3× filtered	USA	19	4	2284
10	5× Column distilled	Sweden	19	10	4108
11	Grain-based	Finland	31	2	1896
12	Grape-based, 5× distilled	France	39	7	4000
13	6× Distilled, activated carbon filtered	USA	49	11	8620

[a]Note: total odour = sum of event areas; event area = aroma intensity × width × 100.

as detected by human olfaction (Fig. 4). Details of these 10 aromas are given in Table 3.

An American vodka from corn

Similar tests were performed on a different commercial American vodka produced from corn. Chemical analysis of this sample resulted in 49 volatile impurities in headspace and these are identified in Table 4. Sensory analysis of this sample resulted in 11 aroma notes in the headspace, as detected by human olfaction (Figs. 5 and 6). Details of these 11 aromas are given in Table 5.

The 13 vodkas can be ranked according to impurities and odour events as in Table 6. One of the vodkas, the 5× column-distilled vodka, had a much higher number of odour events than two other vodkas with a similar number of impurities. This illustrates that distillation alone did not remove the high volatile compounds that would be at the base of the odour events. However, the general trend was that the higher impurity levels resulted in more odour events.

Discussion

The source of the raw material for fermentation did not appear to play a significant role in the quality of the vodka, certainly not as quantified by the number of impurities, nor by the amount of odour 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 appear that the country of origin was not important. Packaging in glass or plastic appeared to show no difference, although there was no direct comparison made between different packaging of the same product.

Some of the observed impurities observed had high boiling points, which would lead to the expectation that these would be separated out by distillation. However, the results indicated that multiple distillation alone did not get rid of all impurities. Also, charcoal or activated carbon treatment alone did not contribute significantly to the removal of the impurities. As expected, neither did multiple filtrations. The only treatment able to remove all of the impurities was a combination of selective oxidation with ozone, GAC and a nano-noble-metal filtration, as was demonstrated with the new brand corn vodka example.

Acknowledgements

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Supporting information

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