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UNIVERSITY OF LOUISVILLE

ACTIVATED CARBON TREATMENT OF RAW WHISKEY
"

A Thesis

Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

By

Earl A. Fallin
"

1942

ACTIVATED CARBON TREATMENT OF RAW WHISKY

Earl A. Fallin

Approved by the Examining Committee

Director _____

April 1, 1942

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ABSTRACT

There has been some controversy on the actual chemical changes that take place when the distillate from an alcohol fermentation process is treated with activated carbon.

This investigation determines the chemical changes and changes in taste characteristics that take place when such a distillate as raw whiskey is treated with each of several activated carbons and in one case over a range of temperatures from 20° to 80° Centigrade.

Tables of results are included with figures illustrating the percentage change effected by the treatment.

There was no outstanding difference in effect between the different activated carbons used at room temperature. A definite demarcation is noted, however, at elevated temperatures.

INTRODUCTION

In the production of whiskey, adsorption methods are often used to remove impurities, particularly those present at relatively low concentrations. In a liquid mixture or solution some constituents are often selectively attracted to the surface of different adsorbents to the almost complete exclusion of others.

In the distillation industries, raw whiskeys are treated with activated carbon to remove undesirable products that are formed during fermentation. At present, these treatments are generally made at room temperature with an arbitrarily chosen kind of activated carbon. Very little is generally known of the actual chemical changes effected by the treatment.

It is the purpose of this research to determine some of these more important chemical changes in the raw whiskey that are affected by treatment with different kinds of activated carbons; and to determine the effect of temperature on one of the carbon treatments.

A taste preference is intended to show a definite correlation with the extent of the chemical change. Such a relationship would substantiate the effects of the various treatments.

HISTORICAL

In the revival of a comparatively old process industry, by science, little has been done as yet toward the actual determination of chemical changes that take place in the distillate from an alcohol fermentation during storage or ageing.

The chemical changes involved are many and complex. Different amplitudes are affected according to the kinds of material used as containers during storage, as has been shown by laboratory investigations of the Bureau of Internal Revenue (11). They found upon storing raw whiskey for four years in charred whiteoak barrels that the most rapid increase of acids, esters, solids, and color took place in the first six months. During the four years, acids gained from 24.9 mg per 100 ml to 56 mg per 100 ml, while esters gained from 7.4 mg per 100 ml to 21.3 mg per 100 ml. Fusel Oil, however, dropped from 69 to 58.4 mg per 100 ml.

It was found, too, that raw whiskey changes while standing in glass. The acids, aldehydes, and furfural content decreased while the esters and color increased. After four years the whiskey had lost most of its green or slop taste and odor.

It has been the practice to treat raw whiskey with charcoal to remove certain undesirable green characteristics. In 1908, William Dudley (4) found that a charcoal made by burning sugar-maple wood, when used to filter raw whiskeys, would remove fatty oils and other substances which are insoluble in a whiskey distillate. He also asserted that soluble constituents are removed partly by adsorption but principally by diffusion into the charcoal, where particles of higher molecular weight are held longer while the lighter materials pass more rapidly.

Thomas and Hochwalt (6) patented a process for producing an

improved beverage substantially free of usual green characteristics by subjecting the liquor to a hydrogenation treatment in the presence of a catalyst of finely divided platinum, nickel, and cobalt. By this method, it is maintained the odor and taste characteristics are permanently improved.

Mr. G. Chabot (2) found that beer worts treated by activated carbon were greatly improved in taste and foam retention properties, and that protein turbidity was removed.

Fritzweiler and Dietrich (5) purified absolute alcohol by percolation through carbon. Vuorinen and Lauren (12) report that birch-wood charcoal removes a large number of aldehydes from alcoholic solutions, formaldehyde being an exception. In some cases they found that this was more easily accomplished in lower proof alcohols.

Chaney (1) states, from a study of the properties of activated carbon in industrial application, that the adsorption power for particles of colloidal dimensions is a mathematical function of the ratio of its activity to its apparent density, fineness being constant. The adsorption is most effective if the carbon carries an opposite electric charge.

The United States Army (7) has shown, in revealing its method of preparation of activated charcoal, that ordinary wood, dried, compressed, and slowly heated, replaces coconut and peach pits for efficiency in adsorption.

Zaharia, Angelescu, and Motoc (13) state that animal charcoal removes, in varying amounts, the different impurities in commercial alcohols when the adsorption is performed under conditions precluding

oxidation. This point is important because activated carbon has been mentioned as a catalyst for the oxidation of alcohols to aldehydes and acids (3). Several patents (8), (9), (10) have been issued involving the use of air and charcoal for the quick ageing of whiskey which utilize this catalytic effect.

THEORETICAL

The treatment of a solution with an adsorbent such as activated charcoal results in a common phenomenon consisting of the adhesion of molecules of a gas or dissolved substances to the surfaces of the solid bodies, resulting in a relatively high concentration of the gas or solution at the place of contact, while the carrier solution is freed of these substances.

Activated carbon has a large surface area per unit volume which is greatly increased with a reduction in particle size. Some kinds of activated carbons, too, are more porous than others and have a larger surface area per unit volume. This surface area is capable of holding the adherent molecules. The amount adsorbed increases with the length of time allowed for the treatment until mechanical equilibrium is reached. Agitation of the solution being treated will accelerate the contact with the adsorbent and thus reduce the time required for reaching mechanical equilibrium.

A change in temperature will affect the size of the pores of the carbon and increase the surface area, thus varying the amount adsorbed. A temperature change will also alter the rate of circulation and diffusion.

The removal of the substances thus adsorbed will disturb the chemical equilibrium of the solution, and additional chemical changes will take place. Any variation in temperature or pressure will affect both the rate of chemical change and the solubility factors involved. The chemical and mechanical changes will be accelerated as the temperature and pressure are increased.

The presence of air in the pores of an adsorbent will oxidize some solutions. The raw whiskeys treated in this research contain alcohol,

acids, esters, and aldehydes which will be oxidized by the air present, but these substances will also be adsorbed by the carbon. Either oxidation or adsorption may predominate according to the kind of activated carbon used and the temperature and pressure maintained during the treatment.

MATERIALS

The distillate from an alcohol fermentation process of a 51% rye, 39% corn, 10% small grain mash was stored in one-gallon glass bottles. This whiskey was clear, 100 proof alcohol and had no visible colloidal suspended material.

A 100 proof, raw bourbon whiskey from a grain mash of 51% corn and 49% small grain was also used. This, too, was clear and had no colloidal suspension perceptible to the naked eye.

For treating raw whiskeys in this experiment, several different kinds of activated charcoal of various particle sizes were used, as well as activated alumina and silica gel as adsorptive agents.

APPARATUS AND PROCEDURE

For the first part of the colorimetric analyses a Duboscq Colorimeter was used, but as the standards were not always made up of the same particular kinds of aldehydes, esters, etc., the shades of color were too far different to allow accurate reading. For this reason a photo-electric colorimeter was substituted and the previous analyses repeated. Preliminary determinations were made of standard whiskeys in order to establish the author's accuracy and technique.

Part I, Analytical Procedures:

The samples used were tested for proof by an appropriate hydrometer which had been checked with a National Bureau of Standards hydrometer. The temperature was noted and the proof found in the tables of the United States Gaugers' Manual.

Permanganate time was accepted as an indication of the amount of reducing compounds present. The method consists of bringing 50 ml samples to 13° C. in Nessler tubes and adding 1 ml of standard potassium permanganate solution. The time which elapses from the adding of the permanganate until the solution turns from pink to yellow is the permanganate time. The permanganate solution was prepared by distilling 500 ml of water from a solution containing at least 0.5 gm of permanganate and adding 0.1 gm potassium permanganate to this distillate.

A volumetric method of determining aldehydes by back titration with iodine solution was tried. The results, however, could not be consistently reproduced, though all precautions were taken.

Finally the procedure for determining aldehydes was established and the analysis made by cooling 10 ml samples of 100 proof whiskey to

15° C. in a refrigerator. Then 5 ml of previously prepared fuchsine solution of 15° C. were added and the sample shaken thoroughly. After it had been standing for 15 minutes, the sample, which had turned to a reddish color, was compared to a similarly treated standard containing approximately the same amount of aldehyde. The reading of the unknown divided by the reading of the standard (that is, the colorimetric ratio) times the amount of aldehyde in the standard represents the aldehyde in the sample.

For these aldehyde determinations it is necessary to prepare aldehyde-free alcohol for making the standards. This was done by refluxing a good grade of 100 proof alcohol for 12 hours in a ground-glass jointed apparatus. The volatile constituents were intermittently sucked out at the top, and the alcohol was then distilled. Only that portion giving a negative test to a fuchsine solution was retained.

The acetaldehyde used for the standards, was first redistilled in an apparatus consisting of a column 400 mm long packed with 4 mm glass beads.

One gram of this acetaldehyde was added by means of a small Dumas bulb to 100 ml of the 100 proof, aldehyde-free alcohol and a solution of approximately 100 gm per 100 liter was obtained. The actual concentration was calculated by considering the weights of both the acetaldehyde and the alcohol. This solution was further diluted with alcohol to obtain a 10 gm per 100 liter solution from which the standards of 5, 2.5, 1.25, and 0.625 gm per 100 liter were made.

A suitable fuchsine solution was prepared by dissolving 0.25 gm of medicinal fuchsine in 200 ml of hot water and the solution filtered.

After cooling the filtrate, 1.77 gm of C.P. sodium sulfite and 0.75 ml of concentrated sulfuric acid were added. By diluting to 500 ml and storing in a refrigerator this solution could be kept for some time.

Total acids were determined by titration with 0.05 N sodium hydroxide. Air was displaced from a 125 ml ground-glass-stoppered pyrex Erlenmeyer flask with carbon dioxide free air. The carbon dioxide free air was prepared by passing compressed air through a 20% solution of sodium hydroxide, then through a dilute solution of sulfuric acid. Fifty millilitres of a sample to be analyzed were placed in the flask. By the use of a phenolphthalein indicator, the sample was neutralized with sodium hydroxide, and the acids were reported as acetic.

To this neutralized solution 10 ml excess of sulfuric acid were added and the solution let stand for 10 hours at 30° C., then titrated back with 0.05 N sulfuric acid. The same titration was made with a 10 ml sodium hydroxide blank. The difference was reported as ethyl acetate esters.

For the fusel oil determination, the sample was diluted with four parts of water, and 1 ml of this solution was pipetted into a 50 ml pyrex test tube. While the contents of the tube were cooled in an ice bath, first 10 ml of concentrated sulfuric acid and then 1 ml of salicylaldehyde (0.8 ml salicylaldehyde to 100 ml of fusel oil-free alcohol) were slowly added. The solution was mixed and the tube placed immediately in boiling water for exactly 15 minutes; after that it was again immersed in an ice bath and diluted with 10 ml of 1:1 sulfuric acid. It was then compared with a similarly treated standard in a Klett Summerson colorimeter.

The fusel oil standard was prepared by weighing 1 mg of pure fusel oil into a fusel oil-free alcohol. By subsequent dilutions standards containing 200, 250, 275 and 300 mg per 100 ml were obtained. These were then diluted with four parts of water.

A residual nitrogen determination was made by running a Kjeldahl analysis on 500 ml samples of both treated and untreated whiskeys which had been evaporated to dryness. The evaporation was carried on in a 100 ml Kjeldahl flask equipped with a distilling head and condenser. Attached to the head was an inlet tube in order that the whiskey might be fed continuously into the flask. The dry residue of the whiskey was digested with concentrated sulfuric acid and a $\text{CuSeO}_3 \cdot 2\text{H}_2\text{O}$ catalyst. The nitrogen was thus converted to ammonia so that it could be distilled off and titrated with 0.02 N sulfuric acid.

Part II, Carbon Treatment of Liquor:

Activated carbon (0.4 grams) was made into a thin paste and gradually diluted with a portion of the one liter of raw whiskey to be treated. The remaining part of the liter was added and the solution placed into a one-gallon, glass-stoppered bottle. The bottle was shaken for 30 minutes, and the sample then was divided into two parts. One part was filtered through a Busch asbestos filter, and the other through ashless double-acid washed filter paper. Since there was no appreciable difference in the two methods of filtering as far as inorganic material was concerned, the two parts were poured together, filtered again through filter paper, and stored in a one-liter pyrex, glass-stoppered bottle. Thereafter only the filter paper was used, each sample being filtered twice. This same

method was used for each separate carbon treatment.

The treatment of raw whiskey as described above, using 0.4 grams of adsorption agent to one liter of whiskey shaken in a one-gallon, glass-stoppered bottle for 30 minutes, was made with each of the following activated carbons: Darco G-60, Lignin, Hardwood, Coconut, Lignite, G.S. Grade, Nuchar, Blood; and Activated Alumina and Silica Gel.

Only one kind of carbon, the most representative, was used during the remainder of the experiment. Instead of treating at room temperature as in the above test, all other solutions were placed in a large oven held at a constant temperature during the treating period. Treatments being made at 35°, 50°, 70°, and 80° C.

Chemical analyses as described for Acids, Esters, Fusel Oil, Aldehydes, Permanganate Time, and a reading of the proofs were made on all samples before and after treatment with each of the adsorptive agents.

It was desired to determine the effects of the treatments on the residual nitrogen content of a whiskey; therefore duplicate one-liter samples of a Bourbon whiskey were treated in the usual manner and analyses made on both before and after treatment.

Samples of the treated and untreated whiskeys were submitted to the quality laboratory of J. E. Seagram and Sons for preferential study. The samples were divided into two classes. In one of them a comparison was made between the two bourbons treated and untreated, and part of the samples were treated for the residual nitrogen determination. In another, a three way comparison was made of three rye whiskeys, one of which was untreated and the other two treated with Darco G-60 under the two different temperature conditions, namely 35° C. and 80° C.

DATA AND RESULTS

DATA SHEET

SAMPLE TREATED WITH	ACID	ESTERS	ALDEHYDES	FUSEL OIL	PERMANGA-	PROOF
	mg/100ml	mg/100ml	mg/100ml	mg/100ml	NATE TIME Seconds	
R-1 not treated	3.9	10.50	1.15	270	30	100.4
R-2 not treated	4.0	10.50	1.15	270	30	99.1
R-3 not treated	3.9	10.50	1.10	260		100.0
R-4 not treated	4.0	10.50	1.00	260	30	99.7
R-5 not treated			1.00	255		99.5
R-1 Lignin	3.45	10.50	1.35	265	75	99.2
R-2 Hardwood	4.00	9.95	1.0	260	65	98.8
R-2 Coconut	3.90	10.05	1.15	265	65	99.1
R-2 Lignite	3.90	10.40	1.15	275	60	99.8
R-3 G.M. Grade	3.85	10.40	1.00	250	45	99.5
R-3 NuChar	3.85	9.45	1.05	270	60	99.2
R-4 Blood	3.55	10.40	1.10	265	70	97.9
R-4 Act. Alumina	4.00	10.15	1.10	240	60	99.6
R-4 Silica Gel	4.00	10.05	1.05	215	30	98.9
R-1 Darco at 18°C	4.00	9.30	.95	260	60	99.0
R-5 Darco at 35°C	4.30	9.55	.80	245	105	97.2
R-5 Darco at 50°C	4.40	9.25	.75	230	120	96.6
R-5 Darco at 70°C	4.15	8.75	.65	210	165	96.7
R-5 Darco at 80°C	4.30	8.25	.60	215	240	96.6

DATA SHEET

PERCENT CHANGED BY TREATMENT

SAMPLE TREATED WITH	ACID %	ESTERS %	ALDEHYDE %	FUSEL OIL %	PERMANGA- NATE TIME %
Lignin	11.50	0	17.40	-1.85	150
Hardwood	0	-5.25	-13.05	-3.70	117
Coconut	-2.50	-4.28	0	-1.85	117
Lignite	-2.50	-0.95	0	1.85	100
G.W. Grade	-1.30	-0.95	-9.10	-3.85	50
NuChar	-1.30	-10.00	-4.55	3.83	100
Blood	11.25	-0.95	10.00	1.93	133
Act. Alumina	0	-3.33	10.00	-7.70	100
Silica Gel	0	-4.28	5.00	-17.30	0
Darco	2.50	-11.42	-17.40	-3.70	100
Darco at 35°C	7.50	-9.05	-20.00	-3.92	250
Darco at 50°C	10.00	-11.90	-25.00	-2.80	300
Darco at 70°C	3.75	-16.65	-35.00	-17.60	450
Darco at 80°C	7.50	-21.42	-40.00	-17.30	700

RESIDUAL NITROGEN DETERMINATION

	Untreated mg/liters	Treated with Darco mg/liters	Per Cent Change
Bourbon Whiskey I	0.464	0.3712	20.0
Bourbon Whiskey II	0.476	0.2784	20.5

DATA SHEET

TASTE PREFERENCE

BOURBON WHISKEYS

Sample	Per Cent Preference	Probable Error
1. Untreated Bourbon	35.0	1.35
2. Bourbon Darco Treated at 18° C.	65.0	

RYE WHISKEYS

Sample	Per Cent Preference	Probable Error
1. Untreated Rye Whiskey	21.6	1.25
2. Darco Treated at 35° C.	34.2	
3. Darco Treated at 80° C.	44.2	2.5

FIG. 2 TEMPERATURE--PERCENT CHANGE BY TREATMENT

The aldehyde present in a sample was calculated as acetaldehyde by the colorimetric method of analysis. The colorimeter ratio, i.e., the reading of the galvanometer for the unknown divided by the reading for the standard, multiplied by the amount of aldehyde in the unknown is the amount of aldehyde in the sample.

The calculation for fusel oil was the same as for aldehyde: the galvanometer reading for the sample divided by the reading for the standard multiplied by the amount of fusel oil present in the standard equals the fusel oil present in the sample.

The acid was determined by titration with 0.05 N sodium hydroxide as acetic acid. Since 60.03 is the molecular weight of acetic acid, $60.03 \div 2 = 30.015$ gm per liter would be 1 Normal. Then 6 ml of 0.05 N sodium hydroxide would be required to neutralize 1 gm of HAC per 100 liters--thus mls of 0.05 N NaOH times 6 equals gm of acetic acid per 100 liters.

The esters were calculated as ethyl acetate gm per 100 liters. The molecular weight of this compound is 88.06, so 44.06 gm per liter would be required for a 1 N solution. Therefore, if 0.05 N sulfuric acid is used for titrating, 8.8 ml of 0.05 N sulfuric acid would be required to neutralize 1 gm of ethyl acetate per 100 liters, but 10 ml of 0.05 N sodium hydroxide was added in excess of the neutral solution. Since a blank of 10 ml of sodium hydroxide was run, the difference between the mls of 0.05 N sulfuric acid required to neutralize this blank, minus the ml 0.05 N sulfuric acid required to neutralize the sample times 8.8 equals the gm of ethyl acetate per 100 liters.

For convenience and greater accuracy, the solution for titration

with sulfuric acid was made acid by adding 10 ml of 0.05 N sulfuric acid from the same pipette used to alkalize the sample for estrification. The solution was then neutralized by titrating with the sodium hydroxide, thus permitting the use of the same burette as before. The above calculation will be:

$$(\text{ml of NaOH used in sample} - \text{ml of NaOH used in blank}) \times 8.8 =$$

gm per 100 liter.

INTERPRETATION OF DATA

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The data sheet shows that the amounts of various impurities present in the ethyl alcohol of raw rye whiskey are materially changed by treatment with activated carbon, silica gel, or activated alumina. A much greater change is shown, however, in the treatments conducted at elevated temperatures.

The chemical change taking place in each substance analyzed is plotted in Figure 2 as a per cent of that originally present in the raw sample. As compared to those tests made at elevated temperatures, the treatments conducted at room temperature showed slight change, with little difference noticeable between the various samples treated by many kinds of adsorbents.

It may be seen, however, that most carbons reduced the amount of acid present, especially those activated carbons made from lignin and from blood. Darco G-60 caused a slight increase in the amount of acid present at room temperature and a decided increase at higher temperatures, the maximum being reached at 50° C.

In no case did the esters present show any increase due to treatment, and, with the exceptions of samples treated with lignin, lignite, blood, and G. W. Grade carbon, all showed a considerable decrease in the amount of esters.

The removal of aldehydes seems to be large when the amount removed is compared on a percentage basis with the amount present in the raw sample. Owing to the small amount of aldehydes present at any time, however, on a weight basis the removal was no greater than that for any other impurity. Treatment with lignin carbon, blood carbon, activated alumina, and silica gel all gave about the same increase to the aldehyde

content. The others tested show some removal of that originally present.

Treatments at room temperature with the various materials showed little effect on the amount of fusel oil present, even though a high removal took place at higher temperatures.

With the exception of silica gel, all the materials used for treatment caused nearly 100% increase in the permanganate time, which indicates a considerable removal of reducing compounds such as sulfur, nitrogen, etc. A tremendous change in permanganate time was noticed for those samples treated with Darco G-60 at elevated temperatures; the higher the temperature, in fact, the greater was the permanganate time.

It is shown that the activated carbons NuChar and Darco G-60 are very similar in their effects. The change Darco G-60 produced was magnified at higher temperatures. The activated carbons from Lignin and blood also resemble each other in their effect, but are somewhat opposite to NuChar and Darco in that they increase the aldehydes and lower the acids. This latter reaction may be attributed to the presence of alkalies in these carbons.

The residual nitrogen determinations show that the untreated raw bourbon samples contained an average of 0.470 mg per liter. The treatment of the sample with Darco G-60 at room temperature removed 20% of the nitrogen in the sample.

The taste preference indicates very strongly that the treatments remove undesirable taste characteristics. As far as the treatment of the bourbon is concerned, the difference is statistically significant, as indicated by the probable error of 1.35. This means that there are approximately 85 chances out of 100 of the trend toward improvement as

indicated being duplicable.

In a similar manner the various samples in the rye whiskey are well differentiated from one another in terms of preference. Here the 35° C. treatment is significantly superior to the control, while the 80° C. treatment is reproducible to the extent of 100%, with respect to the control.

CONCLUSIONS

The adsorptive powers of the various activated carbons used at room temperature, regardless of particle size, were only slightly different in their effects on the whiskeys treated when compared to those tests made at elevated temperatures.

It has been shown that certain of the adsorptive agents used were effective in causing oxidation of the ethyl alcohol to acids and aldehydes in excess of the amount adsorbed by the carbon itself. Other agents either failed to cause oxidation, or if oxidation did take place the adsorptive powers were sufficiently great to remove the new substances thus produced as well as a portion of those impurities already present, so that to all appearances oxidation might never have taken place.

It is very probable that only a small amount of oxidation took place during these treatments since small quantities of carbon were used and they were first made into a thin paste and diluted several times before being added to the sample. As shown on Figures 1 and 2, considerable activity took place at elevated temperatures. Acids were increased, though at 70° C. there was less apparent oxidation than at other high temperatures. Fusel oils are shown to have a large reduction with a maximum reduction around 70° C. However, there was a decided and uniform decrease of aldehydes and esters as the temperature was increased.

An even more outstanding change is noticeable in the permanganate time. A 700% increase for treatment carried on at 80° C. was obtained, which would indicate a high removal of reducing compounds such as those made up of sulfur, nitrogen, etc., that were carried over in the

distillation from the fermentation process. The determination showing a removal of 20% residual nitrogen also supported this conclusion.

The tests made for taste preference showed that treatment with activated carbons removed impurities present in the raw whiskeys that are undesirable to human taste.

To conclude, these tests gave very definite evidence that the treatments carried on at elevated temperatures were much superior to those at room temperature for removing the impurities from the distillate of an alcohol fermentation process.

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VITA

Earl A. Fallin

Earl A. Fallin, the son of Walter A. Fallin and Hattie G. Fallin, was born in Springfield, Missouri, on August 9, 1914.

Mr. Fallin attended the Springfield public schools and finished his secondary education in Springfield Senior High School, in 1932. He attended Drury College from 1932 until he received a Arts and Science degree of Chemistry in the Spring of 1936.

In the years of 1936-1937, he attended the Mechanical Engineering School of Missouri University. The Fall of 1937 he transferred to the Department of Chemical Engineering where he received a degree of Bachelor of Chemical Engineering in 1938.