THE FILTRATION OF ALCOHOLIC LIQUIDS THROUGH WOOD CHARCOAL.

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The processes usually involved in the manufacture of distilled liquors from starchy materials consist in (1) rendering the starch soluble; (2) hydrolyzing the starch; (3) fermenting the saccharine fluid; (4) distilling the beer; and (5) aging the distillate. The composition of the product depends on the kind and mixture of the raw materials used, and on the details of the treatment during each stage of the manufacture.

The product known as "Kentucky" or Bourbon whiskey is made by the regulation method, of a mixture of corn, rye and malt (either barley or corn malt or both) in widely varying proportions.

What is known as "Tennessee" whiskey is made of the same grains as the Bourbon whiskey, by the process outlined above, except that immediately after distillation it is passed through wood-charcoal filters. The charcoal is made on brick floors in the open air, preferably of sugar-maple wood, and is thoroughly burned so that it contains no volatile matter. The charcoal is ground up into pieces somewhat larger than grains of corn and packed firmly, by ramming it down layer upon layer with a wooden tamp, into wooden "leaching tubs" or percolators, about nine feet high, six feet in diameter at the top and five feet in diameter at the bottom, holding about eighty bushels of charcoal.

The new whiskey is run directly from the still into a cooling tank, from which it is distributed to the leaching tubs. Owing to the presence of alkali in the charcoal, the first runnings from the leachers are pumped back to the beer still. It requires the percolation of about twelve barrels through each leaching tub to wash out all of the alkali. After the alkali is washed out, the runnings go to the cistern from which the product is barreled. It is then aged in the usual way.

The leachers are frequently renewed. The distiller carefully watches the percolate and as soon as it loses its pleasant, characteristic odor, the use of the filter is discontinued, emptied and charged with fresh charcoal. When the leaching tub is properly packed, it takes about 72 hours for the first runnings to appear, and after the alkali is washed out, it will satisfactorily leach about 80 barrels of whiskey, *i. e.*, it requires about one bushel of charcoal to a barrel of whiskey. At the Cascade distillery there are seven leaching tubs, five of which are constantly in use, while one is being emptied and the other is being charged and washed. The five filters leach 17 barrels, or about 800 gallons, a day. The loss is found to be from 12% to 15%.

Any new whiskey which is passed through charcoal has an entirely different odor and flavor from an unleached whiskey made of the same

materials, and of course it ages differently. Although the grains used in the mash of Tennessee whiskey are the same as for Bourbon, yet it is as different from it in composition and flavor, as any other variety of whiskey; in fact in everything which serves to distinguish whiskeys. The difference is wholly due to filtration through wood charcoal.

All new whiskey, as it comes from the still, has a rank and disagreeable odor, but when it is filtered through wood charcoal it smells clean and sweet. *New Bourbon, rye, corn and malt whiskies* have quite *characteristic odors*, but if they be leached through charcoal, they have essentially the *same odor*. Rye whiskey retains its characteristics more persistently than any of the others. This proves (1) that the odors of new whiskies are due to *volatile products, peculiar to the grains* from which they are made, carried over during distillation, and (2) that these products are removed, some mechanically and some physically, by charcoal. There is no evidence of oxidation or other chemical action beyond that which may occur with the first runnings which are pumped back to the still.

Grain oils and other extraneous matter are carried over mechanically during distillation. This material is undoubtedly in part responsible for the odor and flavor of new whiskey and also of the aged product. I find that it is removed mechanically by the charcoal and the fatty matter is found in the spent charcoal from the leachers. Most new whiskies become turbid on dilution with water, but not so with whiskies which have been leached through charcoal.

A study was made of the effect of wood-charcoal filtration on the alcohols and other soluble constituents in Tennessee, Bourbon, rye and malt whiskies and the results divulged some interesting facts. The effect on the "fusel oil" was first investigated. The analyses were made by the Roese-Herzfeld method, the technique being that described in my paper[1] on this method. The results are as follows:

KIND OF WHISKEY.—FUSEL OIL, PARTS BY VOL. IN 100,000 OF 100 PROOF ALCOHOL.

	Unleached	Leached.	Per cent. fusel oil removed.
1. Rye	314.8	265.2	16
2. Malt	296.3	254.1	14
3. Artificial	290.9	248.6	13
4. Tennessee	276.2	265.2	4
5. Bourbon	237.6	221.0	7
6. Rye	226.5	182.3	19
7. Bourbon	165.7	111.6	33
8. Malt	298.3	243.0	19
9. Tennessee	243.0	220.9	9

It will be noted that in every case some of the fusel oil is removed by leaching through charcoal, but the percentage removed varies widely.

[1] THIS JOURNAL, 30, 1271 (1908).

This I found to be due to the variation in the length of the column, speed of filtration and the fineness of the charcoal. I found it impossible to get concordant results on a small scale because of the impossibility of maintaining uniformity in the size of the particles and uniformity in packing. On a commercial scale the results should be quite uniform. In leaching the samples, I used percolators nine inches high, two and a quarter inches in diameter at the top and one and a half inches at the bottom.

The effect of leaching on the proof of the samples was as follows:

	Proof.		
Sample.	Unleached.	Leached.	Loss, per cent.
1	98.20	96.60	1.63
2	99.94	95.78	4.16
3	103.10	101.60	1.45
4	114.42	103.46	9.58
5	99.80	98.08	1.72
6	100.44	98.20	2.23
7	100.50	98.38	2.11
8	107.80	101.30	6.03
9	112.28	104.80	6.66

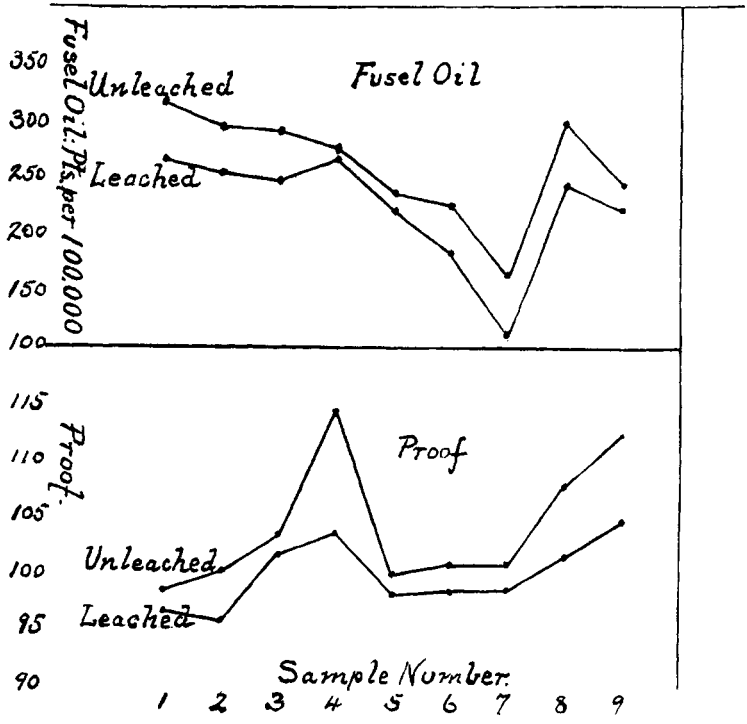

Fig. 1.

The reduction of the proof shows that the ethyl alcohol is absorbed by the charcoal to a greater extent than the water. The curves in Fig. 1 show the reduction of proof and the removal of fusel oil.

The curves bear no apparent relationship. This is to be expected because the amount of fusel oil present is too small to have any appreciable effect on the proof. The reduction in proof is therefore, almost wholly due to the removal of the ethyl alcohol by the charcoal.

A test of a day's run at the Cascade distillery made on June 8, 1908, gave the following result:

Proof of mixed unleached whiskey in the cooler	128.
Proof of the above after leaching	116.
Reduction of proof by leaching	9.375%

The acids, esters and aldehydes are removed to a greater or less extent, but the furfurol in every instance is completely removed. The removal of notable quantities of the acids would naturally be expected because a small amount of alkali would surely remain in the charcoal even after the washing which the first runnings give it. The removal of some of the esters can be accounted for in the same way, but the alkali remaining in the charcoal would soon be neutralized, and therefore this would not account for the removal of such a large amount as was found to be taken out in most instances. Furthermore, I have found that when the charcoal is washed with water until no trace of alkali can be found in the washings, it has the same effect.

Some samples of new unleached and leached whiskies were sent to Dr. H. W. Wiley, and analyzed under the direction of Mr. L. M. Tolman, with the following results:

PARTS BY VOLUME IN 100,00 OF 100 PROOF ALCOHOL.

	Sample.	Acids.	Esters.	Aldehydes.	Furfurol.	Fusel oil.
1.	Tennessee, unleached	5.9	21.4	2.4	None	113.0
	Tennessee, leached	2.2	25.4	2.5	None	108.3
2.	Bourbon, unleached	1.4	21.4	6.3	None	48.5
	Bourbon, leached	2.2	23.5	4.8	None	46.1
3.	Rye, unleached	21.9	90.0	3.6	18.2	74.0
	Rye, leached	2.9	70.6	3.6	None	62.0
4.	Rye, unleached	33.6	83.6	26.7	24.3	149.0
	Rye, leached	4.3	63.0	23.3	Trace	131.0
5.	Malt, unleached	18.2	62.0	4.2	None	105.1
	Malt, leached	4.3	37.6	4.8	None	104.5
6.	Bourbon, unleached	16.2	64.6	23.17	None	101.5
	Bourbon, leached	4.3	23.5	19.4	None	97.5

Dr. Wiley's results on the fusel oil were obtained by the Allen-Marquardt method. The results on the acids in No. 2, the esters in No. 1 and 2 and the aldehydes in No. 5, are anomalous and I cannot explain them except by assuming that the slight difference in the figures is within the limits

of analytical error. Sample No. 1 was leached at the distillery and without doubt it was taken from a leaching tub in which the charcoal was about spent. The other samples were leached by me in the laboratory. The results are shown graphically in Fig. 2.

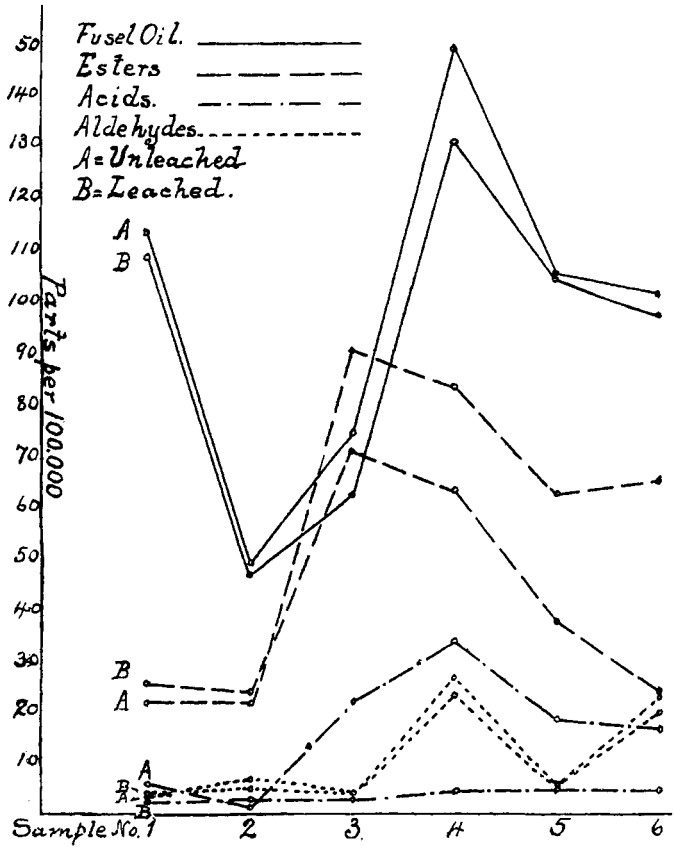

Fig. 2.

The action of wood charcoal is, in my opinion, as follows:

First.—Simple filtration removes the fatty oils and other substances which are insoluble in the distillate.

Second.—The soluble constituents are removed to some extent by adsorption, but the major part of the removal is due to diffusion into the particles of charcoal where the liquids of higher molecular weight are held longer than those of lower molecular weight which pass on more rapidly through the leacher. The length of the column of charcoal, the size of the particles, the density of the packing and the speed of filtration are important factors in the process. If the flow of the liquid

is too rapid, the proportion of the constituents of higher molecular weight which get into the charcoal is small and therefore little is held back; if the flow is too slow they will return by diffusing out of the charcoal into the whiskey. If the liquid remains in contact with the charcoal long enough, there will be nothing removed except the very small amount held by adsorption.

Third.—In the case of the furfurol, I believe it is removed wholly by adsorption, because in every instance it is completely taken out by the charcoal. The same applies to the fatty and essential oils in solution.

Fourth.—I do not believe that oxidation plays any part in the process because whatever action absorbed oxygen may have in the pores of the charcoal, it would be entirely exhausted on the portion of the distillate which is used to wash the alkali out of the charcoal.

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NOTES.

The Determination of Antimony and Arsenic in Lead-antimony Alloys.—In the March issue of the present volume of THIS JOURNAL (p. 378) the author published a paper on the above subject, and now wishes to call attention to a rather important modification which further experience has shown to be necessary.

While accurate results are obtained in many cases by using the method of dissolving described in this paper, there is always danger of losing arsenic by volatilizing, due, apparently, to the reduction of arsenic to arsenious chloride by the action of the undissolved portion. Some alloys are much more subject to this loss than others, the reason for which fact is not quite clear, as the behavior seems to be independent of the amount of arsenic present. By using a sufficiently large excess of nitric acid this loss can be avoided; but a better way seems to be to heat the alloy with hydrochloric acid alone until action ceases before adding the oxidizing agent. The lead is thus dissolved out, leaving the antimony and arsenic in very finely divided condition, and in no case have I been able to detect a trace of the latter two in solution. With the addition of nitric acid or potassium chlorate the antimony and arsenic then dissolve very readily upon shaking, without any further heating. I have not found any loss of arsenic in the subsequent boiling to get rid of free chlorine and nitrous gases.

The procedure recommended is as follows: Heat the finely divided alloy (it is a good plan to run the filings through a fairly fine sieve) with hydrochloric acid until action ceases. Remove the flask from the plate, add about 0.5 cc. of nitric acid and let it stand a few moments until the reddish color is obtained. Then shake the flask, when the antimony and arsenic will dissolve quickly and completely. Now place upon the plate