

Recovery of Fats From Press Wastes¹

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Introduction

SPENT fuller's earth, carbon, and filter aid are wastes or by-products produced in the bleaching, winterizing, and filtration of animal, vegetable, and fish fats and oils. Industrial practice varies considerably in the use of bleaching and filter aids, and the wastes may contain fuller's earth, carbon, or filter aid alone or in any combination. This material contains about 30% oil, and it is estimated that in the processing of oil for edible purposes, more than 1% of the original oil is lost at this source.

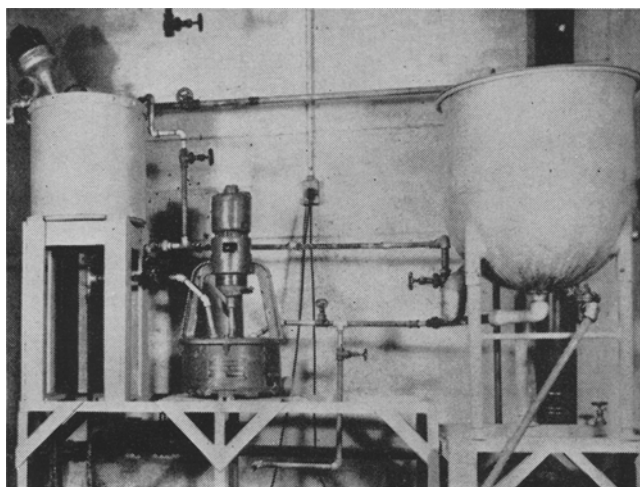


FIG. 1. Soap tank, centrifuge, and hot water tank.

A number of processes have been suggested for recovering the fats which are normally lost in this manner. Solvent extraction has been the most popular method. Sieck (9) describes a solvent extraction plant for treatment of three tons of waste per day, estimating the cost to be \$15 per ton of waste material treated. If wastes are to be collected from several locations, the additional freight expense would make such a process uneconomical except when fat prices are high. Hilditch (5) suggests the recovery of adsorbed fat by solvent extraction of spent earth while it is still in the filter press. There are several objections to this procedure. Many oil processors look with disfavor upon the introduction of solvents into their operations. Also there is the danger of solvent contamination of the oils.

Henkel (4) claims recovery of linseed oil from spent bleaching earth by heating the earth in a water solution of alkali silicate. The linseed oil is emulsified and eventually forms a layer of oil on the surface; the fuller's earth settles to the bottom of the tank. Another process described by Lochle (6) claims recovery of oils by adding water in an amount sufficient to effect surface displacement. Homogenization is carried out in a ball mill and is followed by agitation with water and centrifugation. Slocum (10) mixes

spent bleaching clay with a water solution of a soluble salt of Fe, Mn, Cr, Al, or a water-soluble salt of an amphoteric metal. The mass is covered with water, and the fats rise to the surface while the solids settle out.

Attempts have been made to recover the fats by boiling with sodium hydroxide in open kettles and allowing the solids to settle (1). The problem of removing traces of finely divided solids from the soap solution makes this method cumbersome. In addition, a large dilution is necessary to obtain rapid sedimentation by gravity.

Theory of Operation

In the process to be described the fats, still mixed with the solids, are converted to water-soluble soaps by boiling in a sodium hydroxide solution. The soap solution, containing from 4 to 10% of anhydrous soap, is then separated from the solids in a centrifuge. The centrifuge may be either of the perforate-bowl or imperforate bowl type, depending on the solid and on the nature of the impurities in the fat. The filtrate may be treated with acid to recover the fatty acids or with salt to recover the soap in the form of a curd. The solids, which are retained in the centrifuge bowl, are washed with hot water and discarded.

Several kinds of waste bleaching earths and filter aids were obtained from a number of different sources and treated as described on a laboratory scale. In this manner data were obtained on yields, properties of the product, and ease of operation for the various waste materials. These data permit an evaluation of the advantages and limitations of the method.

Equipment

The equipment is shown in Figure 1. A Tolhurst suspended perforate-basket centrifuge, 12 in. in diameter, was used for separating solids from the soap solution. It operated at 1,725 revolutions per minute, developing a force of approximately 525 times gravity. This machine was obtained as a loan from the Tolhurst Centrifugal Division, American Machine and Metals, Moline, Illinois.

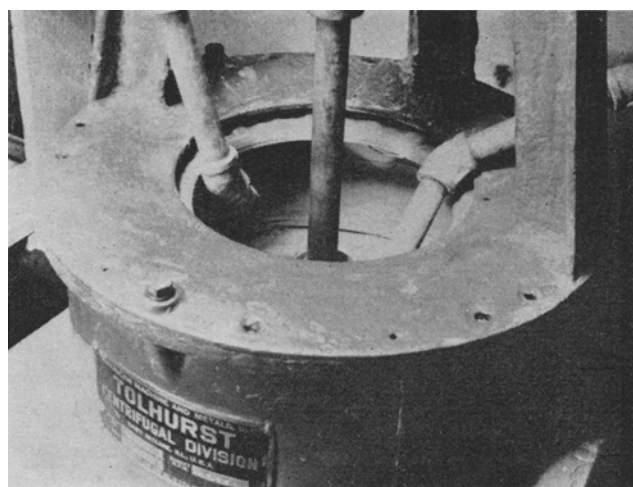


FIG. 2. Closeup of centrifuge, showing feed lines.

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TABLE I
Press Residues Used in Tests

Sample Number	Material	Supplier	Type of Fat	% Fat
1.....	Filter aid from winterizing	Penick and Ford, Cedar Rapids, Iowa	Corn Oil	58.0
2.....	Filter aid from expeller oils	Penick and Ford, Cedar Rapids, Iowa	Corn Oil	59.0
3.....	Carbon and fuller's earth	Darling and Co., Chicago, Ill.	Stearic Acid	44.0
4.....	Fuller's earth and filter aid	Wilson and Co., Cedar Rapids, Iowa	Edible Lard	30.5
5.....	Fuller's earth and carbon	Lever Brothers, Hammond, Ind.	Tallow and Coconut Oil	29.5
6.....	Filter aid	Rath Packing Co., Waterloo, Iowa	Edible Lard	30.5
7.....	Fuller's earth and filter aid	Armour and Co., Chicago, Ill.	Cottonseed Oil	27.5
8.....	Filter aid, sugars, and protein from starch hydrolysis	A. E. Staley Mfg. Co., Decatur, Ill.	90% Palmitic Acid	31.5
9.....	Fuller's earth and filter aid	Armour and Co., Chicago, Ill.	Inedible Grease	30.0
10.....	Filter aid from winterizing	A. E. Staley Mfg. Co., Decatur, Ill.	Corn Oil	37.5
11.....	Filter aid from winterizing	A. E. Staley Mfg. Co., Decatur, Ill.	Soybean Oil	33.5
12.....	Fuller's earth and filter aid	A. E. Staley Mfg. Co., Decatur, Ill.	Corn Oil	27.0
13.....	Fuller's earth and filter aid	Geo. A. Hormel Co., Austin, Minn.	Edible Lard	34.5
14.....	Fuller's earth and filter aid	John Morrell and Co., Ottumwa, Iowa	Inedible Grease	24.0
15.....	Filter aid from winterizing	Corn Products Refining Co., Argo, Ill.	Corn Oil	26.0
16.....	Fuller's earth and carbon	Corn Products Refining Co., Argo, Ill.	Corn Oil	31.0
17.....	Fuller's earth and filter aid	Wilson and Co., Chicago, Ill.	Inedible Grease	30.5
18.....	Filter aid	Wilson and Co., Chicago, Ill.	Edible Lard	29.5
19.....	Fuller's earth and carbon	Spencer Kellogg, Edgewater, N. J.	Castor Oil	58.0
20.....	Fuller's earth	Spencer Kellogg, Edgewater, N. J.	Linseed Oil	31.5
21.....	Fuller's earth	Spencer Kellogg, Edgewater, N. J.	Soybean Oil	35.5
22.....	Fuller's earth	Planters Cotton Oil Mill, Weatherford, Texas	Peanut Oil	72.0
23.....	Carbon and filter aid	Spencer Kellogg, Edgewater, N. J.	Coconut Oil	37.5
24.....	Fuller's earth and carbon	Swift and Co., Chicago, Ill.	Cottonseed Oil	34.5

A 15-gallon tank for saponification of the fats was located to the left and above the centrifuge in such a position that the slurry could flow to the centrifuge by gravity. The slurry was agitated during saponification by a mixer, and by open steam, which also provided heating. The slurry entered the centrifuge basket near the bottom and in the direction of basket rotation, as shown in Figure 2. The rate of feed was controlled by a gate valve.

Hot water was obtained from a 40-gallon steam-jacketed aluminum kettle located above and to the right of the centrifuge. Hot water was used for heating the centrifuge before a run and for rinsing the cake and cleaning after a run. The centrifuge and pipes leading to it could also be heated by open steam. Filtrate left the centrifuge through an opening in the casing and was collected and weighed. A cold water spray was located above the saponification tank for quenching in case the reaction became too vigorous.

Authorities disagree as to whether the suspended basket centrifuge should be loaded while rotating at full speed (3, 7, 8). Preliminary trials failed to show any noticeable vibration when the centrifuge was loaded at full speed. Also several attempts were made to load the bowl with slurry at low speed and then bring the bowl up to full speed. In every case there was much vibration. It was concluded that it was preferable to feed the centrifuge while it was operating at full speed, and this practice was followed.

Materials

Twenty-four samples of spent fuller's earth, carbon, and filter-aid wastes were received from 12 proc-

essors of fats and oils. The sources of these materials and the nature of the residue are shown in Table I. The samples were stored in tightly closed containers at a temperature of 36°F. to minimize oxidation and were processed as soon as possible after they were received.

The preliminary investigation was made of these samples on a laboratory scale. The fat content was determined by extraction with carbon tetrachloride. The data obtained are shown in Table I. The solvent was removed from the extract, and the fat was examined by inspection. In most cases it appeared somewhat inferior in color to the original oil, which was processed by the filter aid or bleach, but with one exception no odor of oxidized oil could be detected.

A 300 g. sample was saponified with sodium hydroxide in water to give a soap solution of from 4 to 10% concentration. The slurry at 210°F. was then filtered by suction on a 12-cm. Buchner funnel, using a single filter paper. The full capacity of the vacuum system was not used since under high vacuum there was much flash cooling which caused the filtrate to jell. The filtering rates indicated are relative to a rate of 300 g. per minute, which is considered excellent.

Dry sodium chloride was added to a portion of the boiling filtrate until the brine contained 15% sodium chloride by weight. The observations on this procedure and color of the soap are shown in Table II. Dilute sulfuric acid was added to a portion of the filtrate until the aqueous layer was slightly acid. The filtrate was then boiled to collect the fatty acids in a clear layer. When excessive quantities of unsaponifi-

able material were present, a clear layer was not obtained. In this case the soap was first salted out, then acidified.

The results of these tests are shown in Table II. They served to indicate whether or not the slurry could be filtered, what soap concentration should be used, and what was the nature of the filtrate. The amount of samples 19, 20, 21, and 22 obtained were not large enough to make the test described. Sample 24 was highly oxidized and could not be tested.

Procedure

An amount of waste material was chosen for each run which would deposit a cake from one inch to two inches in thickness in the centrifuge basket. The material was mixed with water in the saponification tank and heated with open steam. The required amount of 50% sodium hydroxide was added, and mixing and heating continued for three hours. The slurry was allowed to stand until saponification was complete. It was then reheated to about 180°F. and fed to the centrifuge. Agitation was continued during the centrifugation to maintain the solids in uniform suspension. The filtrate was collected and weighed at five-minute intervals. The soap was acidified and the yield of fatty acids measured.

Results

The results of eight experiments on different types of materials are shown in Table III. The choice of perforate or imperforate operation depended on the nature of the sample. Wastes which did not contain carbon were well adapted to solid-bowl or imperforate operation, where the rate of through-put was much greater. This did not give a good separation when carbon was present because of the low density of the carbon. Even with perforate filtration, finely divided carbon gave the filtrate a gray color and was concentrated in the fatty acid layer to give it a black color. Use of a fine cloth or filter aid might have prevented this.

Filter-aid from the meat packing plants contained much protein material. This caused the filter cloth to become plugged, reducing the rate of perforate centrifugation to a very low value. Imperforate operation permitted rapid filtration, but it was necessary to salt out the soaps before acidification. Enough protein was carried over with the soap solution to cause emulsification of the fatty acids when this was not done. The fatty acids obtained by salting out, redissolving the soap, and acidification of wastes containing protein material were very dark brown in color.

TABLE II
Filtration of Soaps From Press Wastes

Sample	Filtration of Sodium Soap	Salting-out With 15% NaCl Brine	Color of Soap	Acidification of Sodium Soaps	Color of Fatty Acids
1	Fast, steady in 5% solution, up to 2" cake thickness	Rapid and sharp separation at 8% soap solution	Light yellow	Rapid and sharp separation	Light yellow
2	Slow in 7% solution. None at 2" cake thickness	Poor separation	Light yellow	Fair. Much non-fat material present	Dark brown
3	Fair in 5% solution. Filtrate jells quickly	Poor at 5% solution. Good at 25%. Colloidal carbon present	Black	Fair separation. Colloidal carbon present	Black
4	Fast, steady in 8% solution up to 2" cake thickness	Clean, sharp separation	Dark brown	Rapid separation; much non-fat material present	Dark brown
5	Slow, but steady in 5% solution up to 1" cake thickness	Poor separation, thin soap curd	Dark brown	Poor separation; much non-fat material present	Black
6	Very fast with 7% solution up to 2" cake thickness	Very quick, clean separation	Light yellow	Rapid and sharp separation	Light yellow
7	Slow in 9% solution. Rate increased by addition of filter aid	Good separation, rapid	Dark brown	Fair separation; much non-fat material present	Dark brown
8	Slow in 7% solution. None at 1" cake thickness	Good separation	Black	Good separation	Dark yellow
9	Fair in 4% solution. None at 1" cake thickness	Fatty acids did not collect in a layer. Heavy, viscous suspension with oxidized odor	Dark red
10	Fast in 5% solution. Slow in 8% solution	Clean, sharp separation	Light yellow	Good separation	Light yellow
11	Fast in 8% solution	Clean, sharp separation	Light yellow	Good separation	Light yellow
12	Slow in 4% solution. None at 1" cake thickness. Rate improved by addition of filter aid	Poor and slow. Carbon present	Dark brown	Fair separation	Black
13	Fast in 8% solution	Fair separation; much non-fat material in brine layer	Dark brown	Poor separation	Dark brown
14	Very slow in 7% solution. None at 1" cake thickness	Poor separation; much non-fat material in brine layer	Dark brown	Poor separation	Dark brown
15	Fast in 10% solution	Clean, quick separation	Dark brown	Good separation	Dark brown
16	Slow in 6% solution. None at 1" cake thickness	Fair separation	Dark brown	Fair separation	Brown
17	Slow in 4% solution	Slow separation of fine curd. Much unsaponifiable floc in brine layer	Dark brown	Fair separation	Dark brown
18	Fair in 5% solution	Fair separation. Much unsaponifiable floc in brine layer	Dark brown	Fair separation	Brown
23	Fair in 6% solution with 3% filter aid added	Fair separation. Good separation in 20% brine	Dark brown	Rapid separation	Dark brown

TABLE III
 Processing of Soaps With Centrifuge

Run No.	Sample No.	Type of Fat	Nature of Solid	Conc. of Soap Solution	Filtration Rate lb./min. sq. ft.	Appearance of Filtrate	Fatty Acid Yield, % of Theoretical
1	4	Edible lard	Fuller's earth and filter aid	% 6.8	.07 perforate .5 imperforate	Dark brown	% 80.1
2	4	Edible lard	Fuller's earth and filter aid	6.1	1.3 to 1.1 imperforate	Dark brown	82.6
3	10	Corn oil	Filter aid from winterizing	8.6	2.3 perforate	Light yellow	45.6
4	18	Edible lard	Filter aid	4.9	2.5 to 0.5 perforate	Light brown	79.0
5	8	90% palmitic acid	Filter aid and waste from starch hydrolysis	4.9	1.9 to 0.7 perforate 2.7 to 0.9 imperforate	Light brown*
6	6	Edible lard	Filter aid	7.2	1.9 to 1.7 perforate	Light brown	87.2
7	3	Stearic acid	Fuller's earth and carbon	3.1	0.6 to 0.2 perforate	Dark grey**
8	16	Corn oil	Fuller's earth and carbon	7.2	1.5 to 0.5 perforate	Dark grey**

* Could not be determined accurately due to highly oxidized nature of the fatty acid.

** Could not be determined accurately due to presence of carbon.

The feed rate for perforate operation was limited by the flow of the liquid through the cake and filter medium. It decreased as the cake increased in thickness. With imperforate operations the feed rate was limited by spattering which occurred at high rates. A higher rate could probably be obtained by proper design of the feed nozzle.

The filtrate from Run No. 5 was clear and light in color, but the fatty acids did not collect in a clear layer. The fatty acid layer was a mass of viscous, highly oxidized material, with a rancid odor. The soap could not be salted out. This material, a by-product of starch hydrolysis, showed normal behavior in the preliminary tests, but during 2½ months of storage at 36°F. had become so oxidized as to be useless.

Difficulty was encountered when processing materials containing a high percentage of stearic acid because of the cooling and jelling of the soap solution. Steam heating of the centrifuge during operation would probably eliminate this difficulty.

This process works satisfactorily for most materials. The fatty acids or soaps recovered can be used only for non-edible uses. Bailey (2) states that recovered oil from most sources is invariably badly oxidized and of poor quality. The fats recovered by this process gave fatty acids which were not oxidized to an extent that could be detected by odor, with the exception noted.

It is believed that satisfactory products will be obtained if residues are processed soon after being removed from the press. This means that for many oils operations should be carried out at the same plant that wastes are produced. The process just described will make this possible without the use of volatile or hazardous solvents. No highly trained help would be necessary except for existing supervisory personnel.

Conclusions

A method of recovering of the fatty material from oil-impregnated filter aids and bleaches had been

tested. It was shown to give a recovery of as much as 88% of the theoretical amount in the form of soap or fatty acids.

Compared to solvent extraction, the method described gives a lower yield and a less valuable product. Its main advantage is that it may be used where the use of solvents or the investment in solvent extraction equipment is objectionable.

Difficulties on account of the presence of carbon, protein material, and oxidation are described, and methods of overcoming them are suggested.

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