Patented June 27, 1944 2,352,365

UNITED STATES PATENT OFFICE

2,352,365

RECOVERY OF FATTY SUBSTANCES BY FROTH FLOTATION

Robert Ben Booth, Springdale, Conn., and Adolphus Mangum Webb, Bronxville, N. Y., assignors to Chemical Construction Corporation, New York, N. Y., a corporation of Delaware

No Drawing. Application February 11, 1941, Serial No. 378,412

5 Claims. (Cl. 210—53)

This invention relates to the recovery of oils, fats, fatty acids, and similar fatty materials from emulsions and particularly to the recovery of wool grease and its constituents from wool steeping and scouring liquors.

It has been known in the past that fatty aqueous emulsions could be treated by froth flotation to recover the values therefrom and such a process is described in the U. S. Patent No. 1,505,944. A process of recovering the fatty constituents of emulsions by froth flotation in which improved results are obtained by means of a froth reducing substance is described in the copending application for U. S. Letters Patent of R. B. Booth, Serial No. 373,937, filed January 10, 1941. It is also known that fatty substances can be recovered from emulsions by the use of centrifuges. While all these prior art processes have been found to be useful in recovering values from fatty emulsions, they all possess the disadvantage that varying percentages of the fatty values remain in the tailing liquors and are passed to waste.

Our invention is particularly suitable for the recovery of wool grease from wool scouring liquors, and this appears to be one of its important commercial uses.

In the centrifuge methods the recoveries have been uniformly unsatisfactory in that liquors containing only relatively high percentages of wool grease could be treated successfully and usually resulted in the production of tailings containing about 0.5% or more of wool grease. Similarly while the flotation processes in some instances, and particularly when froth controlling substances are used, result in improved results over the centrifuge processes, they nevertheless result in the production of tailing liquors containing 0.20% or more of wool grease.

In wool treating processes the dessuint liquors usually contain less than 1% wool grease and the scouring liquors contain higher amounts of wool grease. In the prior art process the wool grease from the dessuint liquors was frequently not recovered and hence was a total loss.

The scouring liquors from wool washing processes are usually discharged at a temperature of about 120° F., and these hot liquors contain most of the wool grease from the wool which may be as high as 3%. When these liquors are subjected to a centrifuging process, which is standard procedure, the percentages of wool grease in the liquors are frequently only reduced to about 1%, that is to say, at least 33% of the wool grease is lost in the tailings from the centrifuge. When the scouring liquors containing a high percentage of wool grease (2 to 3%) are subjected to a froth flotation, even according to the improved process of the previously identified copending application of R. B. Booth, in the presence of a substance such as kerosene, the recoveries are somewhat better than those obtained by the centrifuge process, but still produce tailing liquors containing 0.20% or more of wool grease.

Accordingly, there remained a definite need for a recovery process which is capable of recovering a major portion of the oils, fats and grease which previously have been lost. It is, therefore, the principal object of the present invention to develop such a process which is not subject to the limitations of the prior art. In addition, it is also desirable to develop such a process which is simple, inexpensive to operate and requires little or no additional apparatus.

Surprisingly, it has now been found that the desired objects of the present invention may be readily and simply accomplished by treating the liquors with a grease-liberating agent. This may be done either prior to or during a froth flotation operation.

While it is not necessarily a limitation on the invention, it is thought that the excessive losses in the prior art process have been due to a large portion of the wool grease being held in the continuous phase of an oil-water emulsion. It has been found that the use of any agent capable of converting the wool grease to the discontinuous phase before or during flotation will accomplish the desired object. Such agents are referred to in the instant specification as "liberating agents."

In carrying out the process in accordance with the present invention, emulsions, such as for example liquors containing wool grease, are treated with liberating agents and the liquors subjected to a froth flotation operation to recover the wool grease constituents. As liberating agents any liquid, solid, or gas may be used which causes or aids the separation of the wool grease constituents from the containing liquors. Preferably, the liquors are cracked with a mineral acid and the wool grease constituents concentrated by flotation treatment.

While any of the various mineral acids such as hydrochloric, sulfuric and the like may be employed, sulfuric acid, because of its efficient liberating action, availability and cheapness, is the preferred mineral acid. The amount of acid required varies with the nature of the liquor to be treated and the grease content thereof so that no specific amounts can be set out which are

applicable to all cases. When using sulfuric acid, amounts ranging from about 5 to 25 pounds per ton of liquor to be treated were found to give satisfactory results.

The process is applicable to the treatment of any of the various liquors or combinations of liquors obtained in the steeping and washing and scouring of wool or recovery of wool grease. Such liquors include dessuint liquors, washing and scouring liquors, concentrate and/or tailing liquors from wool grease recovery processes such as centrifuging, aeration, flotation, wool-grease-containing liquors partially or incompletely cracked by the use of acids, other suspensions, solutions, emulsions, and liquors containing wool grease or its constituents as well as mixtures of combinations of such liquors. Such liquors may be relatively rich or dilute in wool grease depending on their previous treatment.

The operations may be conducted at temperatures ranging from room temperatures or lower up to the temperature at which scouring is carried out or higher. Operations, especially flotation, at lower temperatures, have the advantage that tailings of lower wool grease content are frequently obtained. On the other hand, it is an advantage that the scouring liquors of relatively high temperatures may also be processed, in which cases it is not necessary that the liquors be cooled. In many cases when the flotation is carried out at the higher temperatures the comparatively small wool greases losses are compensated for by the saving in time and effort required in cooling the liquors.

It is customary to operate flotation processes using a series of machines and such a process may be employed in the present invention. In many cases the use of a series of machines will result in higher over-all recoveries. The tailings may be passed from one machine to another in either a diluted or undiluted condition and subjected to further treatment to recover additional wool grease values. The dilution of the liquors and additional further quantities of reagents usually result in improved results because it appears that the dilution sometimes aids in breaking the emulsion and at the same time reduces the temperature of the solution. The liberating operation may be conducted prior to and/or during the flotation process. Also, the liberation-flotation process may be carried out simultaneously in one and the same flotation machine or in stages in a series of such machines.

Unfortunately, however, when ordinary methods of froth flotation are applied to wool grease liquors such as those with which the present invention is concerned, it is found that they possess a very marked tendency to overfroth. This has two difficulties. The process is difficult to handle and produces a froth concentrate in which the grease is in too diluted a form. As was pointed out in the previously identified copending application of R. B. Booth, this tendency to overfroth can be overcome by the use of petroleum distillates such as gasoline, kerosene, light fuel oil and the like. Oils of this class having the specific gravity of from about 30–90° Bé. have been used satisfactorily. Amounts required vary from 1 to 2 pounds per ton of liquor to be treated up to about 10 pounds per ton. The exact amount required depends somewhat upon the characteristics of the liquor to be treated.

The grease containing liquors may be partially stripped of their wool grease content by various methods (e. g., centrifuging, aeration, flotation) and then subjected to the liberation operation and treated by flotation to recover the products of the liberation operation. Thus, improved recoveries of the wool grease constituents of the liquors are effected. In either batch or continuous operations, one or more flotation machines arranged in parallel or in series provide a very convenient apparatus for conducting the entire recovery operation, thus improving the mechanical efficiency of the recovery process by eliminating the use of several different types of apparatus. The Fagergren type flotation apparatus, well known in mineral flotation art, is well suited for carrying out the process. However, the process is not limited by the type or form of the equipment employed.

It will be apparent that the great advantages of the present process are in its simplicity and flexibility. The essential features are; the liberation of grease from the continuous phase of the emulsion; the recovery of the liberated grease by froth flotation; and the control of the excessive frothing by the use of the petroleum hydrocarbon distillants. The other features of the process may be widely varied. For example, the process is not limited to any particular liquors. It is applicable both to untreated liquors and those which have been processed according to earlier known procedures. The process may be conducted over a wide range of temperatures. The amounts and quantities of reagents used are not particularly critical. The process may be carried out in a variety of standard apparatus, although the use of froth flotation machines is preferred.

In the use of the various types of flotation machines, a simple and highly efficient apparatus is provided for conducting the liberation and recovery processes. Such equipment is very flexible and may be readily adapted to batch or continuous operations or may be used with other types of apparatus. Thus, it is possible to conduct the liberation and recovery process in one and the same apparatus and thus improve the over-all mechanical efficiency of the process or to entirely divorce the liberation and flotation processes from each other, as desired. It is further possible to treat a liquor first by flotation and then by the liberation-flotation method or first to treat by the liberation method followed by the flotation process to recover the liberated values. It is immediately apparent that the type of product resulting from such treatment can be varied within limits as may be desired by the operator; for example, by the use of this process, it is possible to recover a large portion of wool grease content of a liquor as neutral wool grease by first applying flotation and then further increasing the recovery of the wool grease constituents by applying the liberation-flotation treatment to the tailings produced by the first flotation treatment.

The following examples illustrate the operativeness of this process. It is to be understood, however, that the invention is not to be limited to the details set forth in the examples.

Example 1

About 54.5% of the wool grease content of a mixture of dessuint and scouring liquors was recovered by a froth flotation treatment at 120° C. in a Fagergren flotation machine. The flotation was carried out in the presence of about 3.6 lb. per ton of kerosene. The resulting flotation tailings, 2600 cc. in volume and containing 0.45%

wool grease, were treated in the flotation machine simultaneously with about 15.6 lbs. per ton of sulfuric acid and about 3.1 lbs. per ton of kerosene. Flotation was then continued for four minutes longer and the final tailing, 2180 cc. in volume, contained only about 0.13% wool grease, representing a recovery of about 71% of the wool grease in the tailings from the first flotation.

Example 2

A similar test was conducted on the flotation tailings at room temperature (20°–25° C.). The tailings, containing 0.45% wool grease, were treated in the flotation cell first with about 15.6 lbs. per ton sulfuric acid and then with about 4.6 lbs. per ton of kerosene. The resulting tailing contained 0.07% wool grease. About 84.4% of the wool grease in the feed to the cracking-flotation operation was recovered.

Example 3

A similar test was conducted on these tailings at room temperature, using about 15.6 lbs. per ton of sulfuric acid and 8.0 lbs. per ton of gasoline. The final tailing contained 0.09% wool grease. The recovery of wool grease from the original tailing liquors was about 80%.

Example 4

2600 cc. of a mixture of dessuint and scouring liquors containing 1.36% wool grease were cracked in a Fagergren flotation machine with about 22.6 lbs. per ton of sulfuric acid. Simultaneously, about 9.2 lbs. per ton of kerosene were added to the liquors which were then subjected to flotation for six minutes. The resulting tailing contained 0.04% wool grease and was 1840 cc. in volume. About 97% of the wool grease content of the mixture of liquors was recovered.

In the foregoing examples sulfuric acid was used as the liberating agent, it should be understood however that the invention is not strictly limited thereto, as similar results are obtained using the various liberating agents mentioned in the specification. Similar results are also obtained when emulsions other than wool steeping or wool washing liquors are employed.

In the specification and claims the term "wool grease" has been used generally to cover the fatty constituents of wool which are removed by the various treating processes to which wool is subjected before it is available for use as a textile material. Some of these fatty constituents are often referred to as "wool fat," and it should be understood that the term "wool grease" is used generally to cover all of the fattty constituents of wool which are removed.

In addition to the many advantages pointed out in the specification heretofore, the process of this invention very greatly aids in the solution of waste disposal problems. In the past, many waste solutions containing fat and particularly those from wool washing operations containing substantial quantities of wool grease have been passed to waste and present an acute sewage disposal problem. These fat containing liquors frequently result in the production of putrefaction products having foul odors and often form unsightly scums on the surface of streams into which they may be dumped. The tailing liquors from the present processes contain such low percentages of fat and have been so thoroughly aerated in the flotation process that they may safely be passed into sewage systems or streams without danger of contamination.

What we claim is:

1. In a process of recovering wool grease values from wool steeping and wool washing liquors containing emulsified wool grease and which overfroth when subjected to froth flotation, the steps of treating the emulsion-containing liquors with a sufficient amount of a strong mineral acid to liberate substantially all the emulsified wool grease, subjecting the acid-treated liquors to froth flotation in the presence of a petroleum distillate having a specific gravity of about 30–90° Bé. and present in amounts sufficient to prevent overfrothing but insufficient to dissolve a substantial amount of the wool grease, collecting the resultant froth concentrate and isolating the wool grease therefrom.

2. A process according to claim 1 characterized in that the mineral acid is sulfuric acid used in amounts of from about five to twenty-five pounds per ton of liquor treated.

3. A process according to claim 1 characterized in that the petroleum distillate is selected from the group consisting of kerosene and gasoline used in amounts of from about two to ten pounds per ton of liquor to be treated.

4. A process according to claim 1 characterized in that the acid-treatment is carried out in the flotation cell.

5. A process according to claim 1 characterized in that the liquors are subjected to froth flotation in the presence of the petroleum distillate before being acid treated, whereby a portion of the wool grease is concentrated in the froth and collected before the liquors are acid-treated.

ROBERT BEN BOOTH.
ADOLPHUS MANGUM WEBB.