

## UNITED STATES PATENT OFFICE

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FAT SPLITTING AGENT AND METHOD OF  
PRODUCING SAME

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1 Claim. (Cl. 252—182)

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The present invention relates to the hydrolysis of fats and fatty oils and to agents for promoting same, and relates more particularly to the use of sulfonic acids of relatively high boiling polyalkylated benzenes in the splitting of such fatty materials.

This application is a division of my copending application Serial No. 482,933, filed April 13, 1943, entitled Process and Agent for Fat Splitting.

An object of the present invention is the production of fat splitting agents comprising sulfonic acids or salts of sulfonic acids of alkylated aromatic hydrocarbons, and particularly of alkylated aromatic hydrocarbon mixtures containing substantial amounts of polyisopropyl benzene.

A further object of this invention is the use, in the production of fat splitting agents, of high boiling alkylated aromatic hydrocarbons produced as a by-product in the manufacture of cumene or monoisopropyl benzene.

Cumene is currently being manufactured on a large scale for use as a high antiknock blending agent for gasoline. In the process of manufacture, benzene or commercial benzol containing minor amounts of toluene and xylene is catalytically alkylated with propylene or hydrocarbon gases containing predominantly propylene, together with small quantities of methane, ethane, ethylene, propane, butanes and butenes. During the alkylation reaction, cumene is produced as the primary product, other alkylated benzenes being obtained in minor amounts. The cumene may be separated from the other alkylated benzenes by fractional distillation, and these alkylated benzenes may be further separated into fractions, if desired.

It is a purpose of the present invention to employ as a charge stock for the production of sulfonated fat splitting agents, a mixture of alkylated benzenes higher boiling than cumene, and produced as a by-product of the cumene process. For example, in the operation of the cumene process there may be obtained a yield of 90% cumene (boiling range 290° F. to 310° F.), 3% secondary and tertiary butyl benzenes (boiling range 335° F. to 350° F.), 4% diisopropyl and dibutyl benzenes (boiling range 390° F. to 410° F.) and 3% of polyalkyl benzenes including polyisopropyl and polybutyl benzenes (boiling range 410° F. to 550° F.). It will be understood, however, that depending upon the charge stock and the conditions of operation, the yield of cumene may be lower in some cases, with a corresponding increase in higher boiling alkylated benzenes. In accordance with this invention, all of the material higher boiling than cumene may be used as a charge stock for the production of the sulfonated fat splitting agents, or any of the high boiling fractions comprising this material may

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be used individually as the charge stock. It has been found that a charge stock comprising the bottoms or residual material from the fractional distillation of cumene boiling within the range of 330° F. to 400° F., and which contains a substantial amount of butyl benzenes, diisopropyl benzene and dibutyl benzene, will produce upon sulfonation, a product having fat splitting properties. However, a more satisfactory and efficient product is obtained by sulfonating a residual material boiling within the range of 400° F. to 550° F., and preferably 430° F. and 550° F., and comprising the polyisopropyl benzenes and polybutyl benzenes.

In accordance with the present invention, cumene bottoms or distillation residue having a boiling range of 400° F. to 550° F. and comprising diisopropyl benzene, dibutyl benzene, and the polyisopropyl and polybutyl benzenes is thoroughly agitated with a sulfonating agent, for example, 75% to 100% by volume of 98% sulfuric acid at a temperature between 120° F. and 160° F. for a period of about fifteen minutes, whereby sulfonation of a substantial portion of the hydrocarbons is effected. The sulfonation mixture may be permitted to settle and stratify, and any unsulfonated hydrocarbons as well as unreacted sulfuric acid which settles out may be removed from the crude aromatic sulfonic acids. By this procedure, there is obtained an upper layer comprising up to about 6% of unsulfonated hydrocarbons, a middle layer comprising sulfonic acids containing about 25% to 30% by volume of the sulfuric acid initially used, but of a concentration of about 85%, and a lower layer comprising about 50% by volume of the sulfuric acid initially used, but of a concentration of about 85%.

However, in accordance with the preferred method of treatment, the sulfonation mixture is diluted with a small quantity of water, i. e., of the order of 5% to 10% by volume, and preferably about 7%, whereby there is obtained upon settling and stratification, an upper layer of sulfonic acid containing traces of hydrocarbons and about 15% to 20% by volume of the sulfuric acid initially used, but of a concentration of about 80% to 85%, and a lower layer of spent sulfuric acid of about 75% to 80% concentration. The layers are separated from one another and the upper layer may be used directly as a fat splitting agent. Or, if desired, the upper layer may be neutralized with a suitable base such as sodium or potassium hydroxide, lime, or calcium hydroxide, with or without dilution with water, to form the corresponding water-soluble sulfonic acid salts.

Various other modifications may be made in the sulfonation process, including the pretreatment of the charge stock at ordinary tempera-

tures with sulfuric acid of 70% to 95% concentration to remove traces of olefines or other undesirable materials. The sulfonation process may also be carried out using sulfonating agents other than 98% sulfuric acid, such as chloresulfonic acid, sulfur trioxide, or fuming sulfuric acid, provided the temperature of sulfonation is maintained at such a level as to prevent excessive formation of tarry material. The quantity of sulfonating agent to be used may vary over a wide range, and may advantageously be applied in a plurality of treatments, the aromatic sulfonic acids being separated from the unsulfonated hydrocarbons between each application of the sulfonating agent. The time required to effect sulfonation will depend upon the nature of the charge stock, the temperature of sulfonation, the concentration and activity of the sulfonating agent, and the efficiency with which the sulfonating agent and charge stock are brought into contact with one another. The sulfonation time, using 98% sulfuric acid at about 140° F., may range from about 5 minutes to 15 minutes. However, at lower temperatures, the time may be longer.

The present invention may be further illustrated by the examples set forth in the following table, the sulfonation treatment and recovery process being substantially the same as that described hereinbefore, and involving treatment with 98% sulfuric acid, water dilution, and separation of the sulfonic acid-sulfuric acid mixture from spent sulfuric acid. The charge stocks were fractions from cumene bottoms, such bottoms being the distillation residue from the production of cumene by alkylating coal tar benzol with petroleum refinery gas containing a substantial proportion of propylene, using a phosphoric acid catalyst at 400° F. to 500° F. under a pressure of 200 to 400 lbs./sq. in.

The products of the alkylation reaction were fractionally distilled, the cumene being taken overhead and the bottoms remaining as the distillation residue. In the examples, two different fractions of cumene bottoms were used as charge stocks, one comprising predominantly diisopropyl benzene, and the other comprising predominantly polyisopropyl benzene with minor amounts of polybutyl benzene.

In testing the sulfonic acids as fat splitting agents, the following procedure was employed.

300 cc. of fatty oil, such as cottonseed oil was refluxed at about 215° F. with 120 cc. of 1.5% sulfuric acid and 6 grams of the sulfonic acid agent, such agent comprising a mixture of 85% of polyalkylated benzene sulfonic acids and 15% of sulfuric acid of about 85% concentration. Samples of the fatty material were removed at intervals, settled, and titrated to determine the free fatty acid content, which is expressed as the acid value of the sample.

| Splitting Agent                                      | Neut. No. of Fatty Oil Layer after refluxing— |        |        |        |
|------------------------------------------------------|-----------------------------------------------|--------|--------|--------|
|                                                      | 0 hrs.                                        | 2 hrs. | 4 hrs. | 5 hrs. |
| None                                                 | 20                                            | 21     | 22     | 22     |
| Diisopropyl benzene Sulfonic acids                   | 20                                            | 49     | 107    | 124    |
| Polyisopropyl benzene Sulfonic acids                 | 20                                            | 120    | 176    | 176    |
| Sodium salt of polyisopropyl benzene sulfonic acids  | 20                                            | 56     | 100    | 121    |
| Calcium salt of polyisopropyl benzene sulfonic acids | 20                                            | 54     | 88     | 108    |

From the above data, it will be noted that the free sulfonic acids were more effective as splitting agents than the sodium or calcium salts, and that of the free acids, those derived from hydrocarbon fractions comprising predominantly polyisopropyl benzene were considerably more efficient than the sulfonic acids of diisopropyl benzene, and are therefore to be preferred.

In the hydrolysis or splitting of fats into free fatty acids and glycerol, the sulfonic acids may be employed in aqueous solution, or mixed with a dilute solution of mineral acid, such as sulfuric acid. The quantity of sulfonic acid required is usually small, i. e., of the order of 0.25% to 5% by weight of the fatty material to be hydrolyzed, and very satisfactory results have been obtained with from 1% to 2% of sulfonic acids. The hydrolysis is preferably carried out at elevated temperatures in the presence of steam, temperatures of the order of 180° F. to 220° F. being suitable. Upon completion of the hydrolyzing operation, the insoluble free fatty acids may be separated from the aqueous solution by decantation or other suitable method, and the glycerol may be recovered from the aqueous solution of sulfonic acids by distillation following the neutralization of such acids with caustic soda, lime, or other suitable base.

I claim:

30 An agent for hydrolyzing fats and fatty oils, comprising a mixture of sulfuric acid of 80%–85% concentration and sulfonic acids of a polyalkylated benzene fraction boiling between 400° F. and 550° F., said agent being obtained by alkylating benzene with a gaseous hydrocarbon mixture containing olefins in which propylene predominates in the presence of a phosphoric acid catalyst at 400° F. to 500° F. under a pressure of 200 to 400 p. s. i. to produce isopropyl benzene and higher boiling polyalkylated benzenes, separating therefrom a fraction of polyalkylated benzenes boiling between 400° F. and 550° F., sulfonating said fraction with sulfuric acid of about 98% concentration, diluting the reaction product with 5% to 10% by volume of water to form an upper and lower liquid layers, and recovering from the upper layer a mixture of polyalkylated benzene sulfonic acids and sulfuric acids of 80% to 85% concentration.

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#### REFERENCES CITED

The following references are of record in the file of this patent:

#### UNITED STATES PATENTS

| Number    | Name              | Date           |
|-----------|-------------------|----------------|
| 1,622,974 | Richardson et al. | Mar. 29, 1927  |
| 1,679,093 | Miller et al.     | July 31, 1928  |
| 1,755,179 | Gunther           | Apr. 22, 1930  |
| 1,988,832 | De Groote         | Jan. 22, 1935  |
| 2,014,766 | Isham             | Sept. 17, 1935 |
| 2,232,118 | Kyrides           | Feb. 18, 1941  |
| 2,275,312 | Tinker et al.     | Mar. 3, 1942   |
| 2,396,144 | Anderson          | Mar. 5, 1946   |
| 2,450,585 | d'Guville et al.  | Oct. 5, 1946   |

#### OTHER REFERENCES

Lewkowitsch Chemical Technology and Analysis of Oils, Fats and Waxes, vol. 1, 1909, pp. 63–67.