

ALIEN PROPERTY CUSTODIAN

PRODUCTION OF SULPHONATION PRODUCTS

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No Drawing. Application filed March 9, 1937

The present invention relates to the production of sulphonation products.

In U. S. Patent No. 1,993,375 there is described a process for the production and purification of sulphonation products by causing sulphoning agents to act on high molecular alcohols, neutralizing the resulting products and mixing with water, incorporating the aqueous mixture with a water-soluble solvent and subjecting the resulting mixture to a treatment with a water-insoluble solvent, selected from the class consisting of liquid hydrocarbons and chlorinated hydrocarbons, thereby freeing the neutralized products not reacted with and not capable of reacting with sulphoning agents.

I have now found that highly valuable sulphonation products from mixtures containing alcohols of high molecular weight and other organic substances may also be obtained by at first converting the etherifiable constituents contained in the said mixtures into the corresponding polyalkylene glycol ethers, then treating the mixture with sulphoning agents and after neutralizing extracting the mixture in the presence of water with hydrocarbons or chlorinated hydrocarbons of low boiling point while adding low-molecular alcohols or ketones.

As mixtures suitable for the present process, i. e. those containing alcohols of high molecular weight and other organic substances, such as fatty acids, hydroxy acids, esters, aldehydes, ketones and the like, I may mention for example the alcohol mixtures, resulting in the reduction of fatty acids having a high molecular weight or their esters, as for example palmitic acid, stearic acid, oleic acid, ricinoleic acid, palm kernel oil, castor oil, tallow, sperm oil or other oils, fats or waxes. The said reduction products contain besides the mentioned esterifiable constituents generally esterlike waxes. Furthermore may be mentioned as suitable mixtures the oxidation products of paraffinic hydrocarbons, from which may or may not be removed the acid or the saponifiable constituents.

The introduction of the polyalkylene glycol radicles into the etherifiable constituents of the mixtures is effected in known manner for example by treating with alkylene oxides, preferably with ethylene oxide. It is also possible to introduce into the alcohols or carboxylic acids polyalkylene glycol ether radicles as such, as for example by condensing with polyglycol ethers having reactive atoms or atomic groups, such as beta-chlor-beta'-hydroxydiethylene ether. Thus polyalkylene glycol ether radicles of the general for-

mula $-(O-R)_n-OH$, wherein R stands for an alkylene radicle, such as ethylene, propylene, or a butylene radicle and n is a whole number. For example by causing ethylene oxide to act on the said mixtures polyglycol ether radicles containing 1, 2, 5, 10 or more $(O-C_2H_4)$ -groups may be introduced into the etherifiable compounds contained in the said mixtures. The mixture containing the polyalkylene glycol ethers formed is sulphonated in any desired manner.

The process is carried out for example as follows:

Mixtures containing alcohols of high molecular weight and other not etherifiable substances are treated with alkylene oxide in such an amount that polyalkylene glycol ether radicles of any desired molecular size enter into the molecules of the alcohols or the acids. The ether like compounds are then sulphonated and the resulting sulphonation mixture is neutralized by means of aqueous alkaline liquors. To the neutralized mixture there is added an alcohol of low molecular weight, as for example methyl alcohol or isopropyl alcohol, and then an aliphatic hydrocarbon, as for example benzine. After thoroughly mixing and standing for some time, layers are formed which are then separated from each other. The proportion of low molecular aliphatic alcohols to benzine hydrocarbons may be varied within certain limits, depending on the material to be treated and its content of water; it amounts to from about 1:1 to 2:4. The extraction may be repeated several times with somewhat the same amount of the hydrocarbon. The addition of the aforesaid solvents is made at room temperature or slightly elevated temperature, preferably between 20° and 50° C. The aqueous layer obtained is then freed from water and the alcohol by evaporation. From the distillate the alcohol may be recovered. Thus a sulphonation product remains which contains no or practically negligible amounts of undesirable by-products.

The following examples will further illustrate how the present invention may be carried out in practice, but the invention is not restricted to the said examples; the parts are by weight.

Example 1

The oxidation product obtainable according to German Patent No. 405,850 from paraffin-like middle oil with the boiling ranges between 260° and 320° C, is hydrogenated by means of hydrogen at 220° C under a pressure at 200 atmospheres in the presence of a copper-magnesium catalyst. 100 parts of the hydrogenation product

having a hydroxyl number of 160 and consisting chiefly of aliphatic alcohols having a high molecular weight, after an addition of 0.5 part of sodium hydroxide, are reacted with 37.5 parts of ethylene oxide at 140° C. The condensation product of ethylene oxide thus obtained is sulphonated at about 20° C with such an amount of chlorosulphonic acid as is theoretically necessary, the said amount being calculated with reference to the hydroxyl number. The sulphonation mixture is then neutralized with 10 per cent aqueous caustic soda solution until the reaction has turned alkaline (determined with phenolphthalein). The solution is then extracted at 35° C by the addition of 150 parts of isopropyl alcohol and 150 parts of benzine. After separating the layers formed the aqueous layer is evaporated. The residue consists of about 80 parts of a high-grade sulphonation product having a good washing power which gives no undesired precipitates when employed in water containing lime.

Example 2

A product obtained by oxidizing paraffin wax with air is hydrogenated, after removing the saponifiable constituents by the treatment with caustic soda solution and, after freeing it from any unattacked initial material, by extracting with methanol, and then subjected to distillation in vacuo. One half of the distillation product, having a hydroxyl number of 210, is then treated with so much ethylene oxide at 150° C, after adding 0.5 per cent of sodium ethylate, that the ethylene oxide absorbed amounts to about 3 molecular proportions for each hydroxyl group. The resulting product is then dissolved in benzine and then sulphonated at 15° C by treatment with a mixture of equal parts of ether and chlorosul-

phonic acid. The sulphonation mixture is then neutralized by means of 20 per cent caustic soda solution while well cooling, 100 parts of 50 per cent ethyl alcohol are then added whereupon the solution is thrice extracted at 45° C each time by means of 150 parts of benzine. By evaporating the aqueous alcoholic solution there are obtained about 120 parts of a sulphonation product containing about 15 per cent of inorganic salts, the said product having a good washing power, especially for linen, as well as an excellent dispersing power for lime soaps.

Example 3

A product obtained from paraffin wax by oxidation with air, having an acid number of 50 and a hydroxyl number of 36, is distilled in vacuo until 50 per cent of the product are distilled off. 15 parts of ethylene oxide are then caused to act on 100 parts of the distillation residue after addition of 1 part of sodium ethylate. The reaction product is dissolved in benzine and then treated at 50° C with chlorosulphonic acid in an amount as theoretically required (calculated on the hydroxyl number). After neutralizing the sulphonation product with 10 per cent caustic soda solution, 50 per cent ethyl alcohol are added in an amount corresponding to the caustic soda solution added. The solution is then extracted at from 40° to 50° C three times with 200 parts of benzine each time. The aqueous alcoholic solution obtained is evaporated to dryness, thus 140 parts of a sulphonation product being obtained which has an excellent foaming, washing and dispersing power and which is especially suitable for dispersing calcium soaps.

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