Patented May 21, 1940 2,201,944

UNITED STATES PATENT OFFICE

2,201,944

PROCESS FOR THE PRODUCTION OF CAPILLARY-ACTIVE SULPHONATION PRODUCTS

Martin Luther and Bruno v. Reibnitz, Mannheim, Germany, assignors to I. G. Farbenindustrie Aktiengesellschaft, Frankfort-on-the-Main, Germany

No Drawing. Application April 7, 1938, Serial No. 200,800. In Germany April 15, 1937

4 Claims. (Cl. 260—458)

The present invention relates to the production of sulphonation products from the neutral constituents of oxidation products of high molecular non-aromatic hydrocarbons.

We have found that valuable sulphonation products suitable as foaming, washing, wetting and dispersing agents can be prepared in a technically simple manner by separating from the unsaponifiable (neutral) constituents of oxidation products of high molecular non-aromatic hydrocarbons the compounds containing from 10 to 18 carbon atoms and one oxy or oxo group and sulphonating these compounds, if desired, after hydrogenating.

The crude oxidation products of high molecular non-aromatic hydrocarbons may be subjected to a known treatment for removing the saponifiable compounds. The compounds containing from 10 to 18 carbon atoms and one oxy or oxo group can be easily separated from the other unsaponifiable products particularly the di- and polyoxy and di- and polyoxo compounds and the main amount of the high molecular non-attacked hydrocarbons by simply distilling. The said fraction represents a mixture which mainly consists of primary or secondary alcohols, aldehydes and ketones. In some cases also small amounts of polyoxy or polyoxo compounds may be present. If desired the unsaponifiable oxidation products may be subjected before distilling to an extraction with organic solvents, such as low molecular aqueous alcohols or esters, as it is described, for example, in the German Patent 570,952 or the U. S. Patent 1,921,381.

The distillation can be carried out in any suitable manner if desired while introducing steam preferably under diminished pressure. The boiling limits of the fraction to be employed as initial material depend in each case on the nature or the composition of the oxidation products employed. For example while carrying out the distillation under a pressure of 15 mm. the fraction boiling between 100° and 220° C. and while working under a pressure of only 2 mm. the fraction boiling between 65° and 175° C. is employed.

When using a column apparatus, preferably a bell-tray column, a particularly good separation of the desired fractions is obtained. Distilling plants which are suitable for working in continuous process may be employed with advantage. The higher boiling constituents which also contain the main amount of the non-attacked hydrocarbons can be removed after finishing the distillation or they can continuously be separated as residue. The said residues may also be subjected to a distillation before again oxidizing. Since the oxidation of the high molecular non-aromatic hydrocarbons is preferably carried out when at most 70 per cent of the hydrocarbons have been oxidized or the oxidation is carried out under mild working conditions, the amount of the unattacked hydrocarbons is often considerable.

The desired fraction of the neutral oxidation products containing from 10 to 18 carbon atoms obtained in the distillation possesses only small amounts of unattacked hydrocarbons. These may be removed by the extraction while using organic solvents, for example according to the process of the U. S. Patent 1,921,381. Preferably the removal of the hydrocarbons may be carried out after sulphonating the fraction and neutralizing the sulphonation product with alkali according to the process described in the U. S. Patent 1,933,375.

The neutral oxidation product to be employed as initial material can be subjected before or after the distillation to a hydrogenation which may, for example, be carried out according to the process described in the U. S. Patent 2,048,662. In this case preferably the oxo groups are converted into hydroxy groups.

The sulphonation is carried out with any suitable sulphonating agent, such as sulphuric acid, oleum or chlorosulphonic acid. The amount of the sulphonation agents to be employed depends on the nature of the sulphonating agent. Generally the sulphonating agent is employed in an amount equivalent to the hydroxy value of the said unsaponifiable oxidation products containing from 10 to 18 carbon atoms. If desired, solvents or diluents, for example diethylether, trichlorethylene, carbon tetrachloride or similar solvents and/or water-binding substances, such as anhydrides or chlorides of organic or inorganic acids, for example acetic anhydride or phosphorous pentoxide may be employed. The sulphonation is preferably carried out at temperatures from 5° below zero C. to 30° C. As soon as the sulphonation product is soluble in water it is worked up in usual manner, for example by introducing into ice-water and neutralizing the mixture with an alkali, ammonia or an amine. The salt solutions obtained may be evaporated to dryness.

The products obtained possess capillary active properties, particularly a high foaming, washing, wetting and dispersing power.

The following example will further illustrate the nature of the present invention but the invention is not restricted to this example. The parts are by weight.

Example

A product obtained by oxidizing hard paraffin (crude scale wax) with air is freed from saponifiable constituents. 100 parts of the remaining unsaponifiable constituents which contain about 60 per cent of unattacked paraffin hydrocarbons are distilled under a pressure of 15 mm. The fraction which distils up to about 100° C. which consists of about 3 per cent of the unsaponifiable matter is separated. The fraction boiling between about 100° and 200° C. (for about 30 per cent of the unsaponifiable matter) contains practically all the monooxy and monooxo compounds with from 10 to 18 carbon atoms in the molecule and from 30 to 35 per cent of unattacked paraffin hydrocarbons. This fraction is subjected to a catalytic hydrogenation under the working conditions as described in Example 4 of the U. S. Patent 1,948,662. The hydrogenation product thus obtained is sulphonated at 20° C. while employing 42 per cent of chlorosulphonic acid. After neutralizing the sulphonation product with a caustic soda solution the unsulphonated constituents are removed by extracting while employing benzine and alcohol according to the process of the U. S. Patent 1,933,375. After separating the organic solvents and evaporating the aqueous solution, about 30 parts of a product having an excellent foaming, washing, wetting and dispersing power are obtained.

The constituents distilling above 200° C. may, if desired, be again subjected to the oxidation process.

What we claim is:

1. The process of producing sulphonated compounds which comprises subjecting the unsaponifiable constituents of the oxidation products of high molecular non-aromatic hydrocarbons to a distillation under subatmospheric pressure, recovering the fraction boiling under a pressure of 15 millimeters (mercury gauge) between 100° and 220° C. and sulphonating this fraction.

2. The process of producing sulphonated compounds which comprises subjecting the unsaponifiable constituents of the oxidation products of high molecular non-aromatic hydrocarbons to a distillation under subatmospheric pressure, recovering the fraction boiling under a pressure of 15 millimeters (mercury gauge) between 100° and 220° C., hydrogenating and then sulphonating this fraction.

3. The process of producing sulphonated compounds which comprises hydrogenating the unsaponifiable constituents of the oxidation products of high molecular non-aromatic hydrocarbons, then subjecting the hydrogenated products to a distillation under subatmospheric pressure, recovering the fraction boiling under a pressure of 15 millimeters (mercury gauge) between 100° and 220° C. and sulphonating this fraction.

4. The process of producing sulphonated compounds which comprises subjecting the unsaponifiable constituents of the oxidation products of high molecular non-aromatic hydrocarbons to a distillation under subatmospheric pressure, recovering the fraction boiling under a pressure of 15 millimeters (mercury gauge) between 100° and 220° C., then subjecting this fraction to an extraction with a water-insoluble solvent and sulphonating the fraction.

MARTIN LUTHER.
BRUNO v. REIBNITZ.