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PURIFICATION OF SULFATED HIGHER ALIPHATIC ACID ESTERS

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The present invention relates to a novel process for purifying a sulfated partial higher aliphatic acid ester of polyhydrc alcohol, as hereinafter described and claimed.

In the manufacture of such material, the final product may contain a variable amount of free higher fatty acid material as a by-product depending upon the mode of preparation and the specific processing conditions employed therein. One method of removing the higher fatty acid material is to solvent extract the impure sulfated ester material with ether, but this method has the disadvantages inherent or common to ether extraction processing and the like.

It has now been discovered that impure sulfated partial higher fatty acid ester of polyhydric alcohol may be treated with an anion exchange resin in order to selectively or preferentially remove the higher fatty acid material and to recover the sulfated ester material in purified form. A preferred embodiment comprises a process for the separation of higher fatty acid from higher fatty acid monoglyceride sulfate salt by treating such a mixture in aqueous solution with an anion exchange resin and separating a solution of said monoglyceride sulfate salt in purified form.

The higher fatty acid monoglyceride sulfate salts are well-known in the art and include such soluble salts as the alkali metal (e.g. sodium, potassium), ammonium, mono-, di- and tri-ethanolamine, monoisopropanolamine and ethylamine salts. The soluble alkaline earth metal salts such as calcium or magnesium salts may be employed also. The higher fatty acid radicals are derived usually from fats, oils, waxes or fatty acids of natural or synthetic origin and have usually from 8 to 22 carbon atoms and preferably from 12 to 18 carbon atoms. Examples of suitable materials are the sodium, potassium and lithium coconut fatty acid monoglyceride monosulfate salts and their hydrogenated derivatives, ammonium lauric, myristic and palmitic monoglyceride monosulfate salts, potassium and sodium tallow fatty acid monoglyceride monosulfate salts, and the like. While the present invention is applicable to any mixture of the higher fatty acid material with the monoglyceride monosulfate salt, it is effective with other sulfated salts of partial fatty acid esters also. The corresponding sulfate salts of partial higher fatty acid esters of other lower aliphatic polyhydric alcohols such as ethylene glycol, propylene glycol and butylene glycol containing said impurities may be treated in accordance with the present invention.

This purification process is particularly applicable to a neutralized reaction product of a sulfonating agent, a fatty acid material and a polyhydric alcohol containing a minor proportion of free higher fatty acids. A preferred process for forming the impure reaction product cited by way of illustration involves the interaction of such components as glycerine, higher fatty acid material (whether fatty oils, fats, waxes or fatty acids) and a substantial excess of a strong sulfonating agent to form the sulfuric acid ester of the higher fatty acid monoglyceride. The resulting acidic reaction product is neutralized with an alkaline neutralizing agent such as sodium hydroxide or the like to form the corresponding salts. Such procedures are known in the art as illustrated by Muncie, U.S. Patent Nos. 2,130,361; 2,130,362; 2,242,979; etc. Depending upon the conditions during hydrolysis, esterification, and particularly during neutralization, there may be formed varying amounts of free higher fatty acid material, such as by saponification of the ester. The acidic reaction product is neutralized usually to a pH from about 5 to 9, and preferably about 6 to 7, whereby the sulfated ester is in the form of its salt and the higher fatty acid material is present primarily as free higher fatty acid.

As a result of such processing, the final reaction mass comprises usually about 30 to 35% of the organic sulfate salt, and the remainder comprises mainly inorganic sulfate salt such as sodium sulfate with a minor proportion of free higher fatty acids. If desired, this detergent base may be treated in known manner, such as by alcohol extraction, to substantially remove the inorganic salts as illustrated by Russell, U.S. Patent Nos. 2,303,582 and 2,316,719. This monoglyceride sulfate base, either with or without the presence of the inorganic salt, may be purified in accordance with the present invention.

The impure monoglyceride sulfate salt containing the higher fatty acid may be purified in a simple and practical manner by treatment with an anion exchange resin which preferentially adsorbs the higher fatty acids. These anion exchange resins are known water-insoluble materials and are generally synthetic resinous amine or quaternary ammonium compounds. For example, Dowex 1 is a strongly basic quaternary ammonium resin having the trimethyl benzyl ammonium active group on a cross-linked copolymer of styrene and divinylbenzene. Dowex 2 differs only in that one of the methyl groups is replaced by an ethanol group. Other anion exchange resins can be made in known manner also by condensing aromatic amines, e.g. aniline or m-phenylene diamine, with formaldehyde. Alkyl groups may be incorporated into the resins during preparation to form the quaternary ammonium bases or co-condensation with aliphatic polyamines may be employed. Permutit S–1 is a highly basic quaternary amine anion exchange resin which has been found to be particularly suitable in the present invention. Other suitable anion exchange resins are Permutit CCG; Amberlite IRA—410, 400 and 4B; Nalcite SAR and WBR; and Duolite A–4, A–40 and A–41, etc. These resins have exchangeable anions associated with each quaternary ammonium or polyamine active group such as the chloride, sulfate, hydroxide anions and the like.

As indicated, the resin particles are contacted with an aqueous solution of the water-soluble higher fatty acid monoglyceride sulfate salt or the like containing higher fatty acid impurities. The solution may contain additional liquid solvent medium such as a lower aliphatic alcohol, e.g. ethyl alcohol, provided that it does not substantially adversely affect the adsorption. Any suitable technique for contacting solutions with anion exchange resins may be employed, such as the batch or column method. In the former, the resin particles are mixed with the treating solution and, after equilibrium, the resin and the solution are separated by filtration or other means of liquid-solid separation. Alternatively, the process may conducted using column operation wherein the resin bed is fixed or moving and the feed solution flows past the resin in any desired direction. In practice of this operation, it is convenient usually to use a fixed bed such as a vertical cylinder filled with the anion exchange resin particles with downflow operation.

This purification process may be conducted under vary-

ing conditions. The process may be performed at room temperature or with moderate cooling or elevated temperatures, such as up to the boiling point of the solution. The purification may be accomplished in a batch, semi-continuous or continuous manner as desired. The treating solution may be a dilute or concentrated solution of the monoglyceride sulfate base or the like, and may even be employed in slurry form though it is preferred to use concentrations from about 1 to 50% by weight. The resin particles may be of any practical size such as from about 10 to 400 mesh though it is preferred to use particles from about 20 to 200 mesh. After the resin has been exhausted or when desired, it can be regenerated readily by passage of alkali salt solution through the anion exchanger resin. The anion of the alkali salt solution should correspond to the anion in the resin. If desired, the resin particles may be washed with water to remove any organic sulfate salt possibly adsorbed thereon.

This process results in the purification of the monoglyceride sulfate salt and is independent of variation in the free fatty acid content of the original base in general. While the amount of free fatty acids is variable, it will be generally less than 50% by weight of the organic matter or solids, and usually from about 1 to 20% by weight of the mixture of fatty acids and organic sulfate salt. The filtrate or effluent containing the purified monoglyceride sulfate base may be analyzed for its higher fatty acid content as indicative of the efficiency of the adsorption treatment. If desired, the filtrate may be subjected to further treatments with the resin particles to further lower the fatty acid content. As a result of this purification, the impure monoglyceride sulfate salt or the like containing minor proportions of higher fatty acids can be treated so as to render the product substantially free from the fatty acids. The higher fatty acid content of the purified product can be lowered to less than 5%, and preferably less than 1%, on an organic solids basis in a simple and practical operation using the resin particles to remove the higher fatty acids from a polar medium such as the aqueous solution of the impure monoglyceride monosulfate salt.

The filtrate or effluent may be treated in any suitable manner to recover the purified monoglyceride sulfate salt from the liquid medium if desired. This purified material may be subjected to any conventional means of further treatment if desired, such as by washing, drying, solvent extraction or crystallization.

The following specific examples are further illustrative of the nature of the present invention, but it is to be understood that the invention is not limited thereto. All amounts of the various ingredients are by weight unless otherwise specified.

Example I

200 grams of an anion exchange resin known as Permutit CCG are mixed with 600 cc. of water and the mixture is permitted to stand overnight. The excess water is then decanted and the residue is washed with dilute sodium hydroxide until no reaction for chloride ion is obtained. The resin is then washed with distilled water until the wash liquor is neutral. This moist resin is mixed with 100 ml. of a 38.5% solids aqueous solution of sodium hydrogenated coconut monoglyceride monosulfate salt having a pH of about 6.5. This detergent base contains 2.5% free fatty acids on a solids basis and is essentially free of inorganic salts. The resulting mixture is then stirred mechanically for 6 hours and permitted to stand overnight. The supernatant liquid is then filtered and a clear supernatant filtrate is collected. An analysis of the purified filtrate discloses that the free fatty acid content has been decreased to 1.1% on a solids basis.

Example II

The procedure of Example I is repeated using an anion exchange resin known as Permutit S–1. The resulting filtrate had a reduced free fatty acid content of 1.4%.

Example III

The procedure of Example I is repeated using an aqueous solution of the ammonium salt of coconut monoglyceride monosulfate with similar results.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications of this invention can be made and that equivalents can be substituted therefor without departing from the principles and true spirit of the invention.

Having thus described the invention, what is claimed is:

1. A process for the purification of an impure water-soluble sulfated partial higher fatty acid ester of a polyhydric alcohol admixed with higher fatty acid which comprises contacting an aqueous solution of said impure sulfate ester material with an anion exchange resin to remove preferentially said higher fatty acid, and separating said sulfate ester in purified form from said resin.

2. A proces for purifying a water-soluble higher fatty acid monoglyceride monosulfate salt admixed with higher fatty acids which comprises contacting an aqueous solution of said mixture of monoglyceride sulfate salt and higher fatty acid with an anion exchange resin whereby said fatty acids are preferentially adsorbed, and separating the solution of said monoglyceride sulfate in purified form from said anion exchange resin.

3. A process for purifying a water-soluble higher fatty acid monoglyceride sulfate salt admixed with a minor proportion of higher fatty acid which comprises forming an aqueous solution of said monoglyceride sulfate salt and higher fatty acid, treating said solution with an anion exchange resin to adsorb fatty acid, and recovering said higher fatty acid monoglyceride sulfate salt in purified form.

4. A process in accordance with claim 3 wherein said monoglyceride salt is the sodium salt of coconut fatty acid monoglyceride sulfate.

5. A process in accordance with claim 3 wherein the salt is the ammonium salt of coconut fatty acid monoglyceride sulfate.

6. A process in accordance with claim 3 wherein the salt is the sodium salt of hydrogenated coconut fatty acid monoglyceride sulfate.

References Cited in the file of this patent

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