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PROCESS FOR THE MANUFACTURE AND PRODUCTION OF DETERGING, EMULSIFYING, WETTING AND DISPERSING AGENTS

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This invention relates to a process for manufacture and production of deterging, emulsifying, wetting and dispersing agents which has peculiarity in acting sulphonating reagents such as concentrated sulphuric acid, fuming sulphuric acid, chlorsulphonic acid on fatty material which obtained by saponification of wool fat with acting agents, such as caustic alkali, acids, or by hydrolysis under pressure, or on those products which obtained by substitution of ester radicals of the said fatty material with alcohols. The object of this invention is to make a product by extracting the whole matters of wool fat as sulphuric compound without using any special organic solvents and to obtain the product which has remarkable acting power in deterging, wetting, emulsifying and dispersing against acidic solution, alkaline solution, hard water, and seawater.

It is already known to produce higher fatty alcohol sulphonate by acting sulphonating reagent on general waxes using special organic solvent, also there are some literature on the sulphonation of wool fat. However, it has not been clear about the component of wool fat and it has been considered that the wool fat has the same component as the other waxes, but, if you apply the former method on wool fat, the component of wool fat which has the carboxyl group be removed by solving into the solvent and be avoided uselessly not coming into the product, on the other hand, if you apply the latter method acting only sulphonating reagent, the acidic saponification of wax ester is very difficult and some of the component remain as wax ester in the product and it spoils the quality of the product.

However, I, the inventor of this subject, after having made many experiments on the components of wool fat, have discovered that wool fat has a chemical property that is remarkably different from other general waxes. The proper components are special quality oxy-acids, and cholesterin, iso-cholesterin, that do not contain long chain radicals. The oxy-acids are not only contained abundantly in wool fat but the sulphuric acid ester or sulphonic acid or salts are quite different from the sulphuric acid ester or sulphonic acid that were already known, and it has eminent effect as deterging, wetting, emulsifying and dispersing agents. Besides, it was discovered that you can sulphonate the sterol in these component easily by substituting the ester radical with methyl alcohol, ethyl alcohol, or saponifying it with alkali, acids, or by making it fatty material, by demand mixing mineral acid or hydrolysis under pressure. Thus the invention was accomplished on the ground of those discovery and the details are as follows.

When you substitute ester radical of crude or refined wool fat with methyl alcohol, ethyl alcohol, alcohol combines with carboxyl group of wool fat at the same time hydroxy group that has been combined with carboxyl group become free. On that condition if you act sulphonating reagents such as concentrated sulphuric acid or chlorsulphonic acid, the sulphate radical combine with hydroxy group in the wool fat to produce sulphonate ester or sulphonic acid. Also, after saponified the said wool fat with caustic alkali or acids under certain pressure, or by elevating pressure, mix mineral acid like sulphuric acid, or making it fatty material by hydrolysis under pressure, then if you act directly the sulphonating reagent such as concentrated sulphyric acid, fuming acid, chlorsulphonic acid, the proper component of wool fat almost combine with sulphonating reagent and be obtained as perfectly sulphuric compound.

Also, the inventor has discovered that the many components that obtained from wool fat by the abovementioned methods, have different action for sulphonating reagent, so that for the object of use of the said each product you can make it excellent product which suits each use by mixing it more differently than natural quantity. Consequently when you put this invention in practice you can obtain the most appropriately harmonized product by acting sulphonating reagent on the component of the said fatty material after changing the rate of each component, or after sulphonating the component separately mix it as demanded combining rate. Now, the experiments in each case are as follows:

1. To the 100 kg. of wool fat of which saponification value 115.0, acid value 10.5, adding 40% caustic soda 50 kg. and saponify it stirring for about 10 hours under 95° C. Then heat it stirring and adding abt. 200[1] of water and make the anticipated fatty material separate combining sulphuric acid. The saponification value of this material is 113.0, acid value is 96.0 and the decomposition rate is 85.7%. In the fatty material obtained thus, drop 15 kg. of sulphuric acid stirring, then, after sulphonating it by dropping fuming sulphuric acid 5 kg. add water 100[1] and stir it and make it standing and remove the acidic waste that come separate in the lower layer and use the higher layer, i. e. sulphuric compound

as it were, or after dried it neutralizing with alkali.

2. In the abovementioned wool fat 100 kg. pour 40% caustic soda 50 kg. and putting it in a closed apparatus, then saponify for 6–7 hours under 120°–130° C., pressure 50 lbs. per square inch, then make it fatty material by adding mineral acid. The saponification value of this material is 120.0, acid value 109.7, decomposition rate 98.0%, then make it sulphuric compound by acting sulphonating reagent, and use it as it is or after dried it neutralizing with alkali.

3. With the abovementioned wool fat 3 kg. combine ethyl alcohol 3 kg., caustic soda 0.05 kg., and substitute the ester radical by heating and stirring, then distil excessive alcohol and remove it, then neutralize caustic soda and saponified alkali with sulphuric acid, in this product, fatty material, drop stirring concentrated sulphuric acid, then after washing neutralize with caustic soda and dry it. But, to combine alkali when we substitute ester radical is to make the said reaction more effective using it as catalyzer, and we get almost the same result with organic solvent like petroleum benzine.

4. First stir wool fat 100% and 40% caustic soda 50% for about 10 hours under 95° C., then get fatty material by combining sulphuric acid heating together with water 100%. Then treat this product with petroleum ether and extract soluble part from it and sulphonate dropping concentrated sulphuric acid. Use this product as it is or after drying it neutralizing with alkali. This product is good for absorbing agent.

5. Adding 5% concentrated sulphuric acid finely to wool fat 100% heating 90°–120° C. and stir for about 3 hours, then divide it soluble part and insoluble part using alcohol, and sulphonate them separately and get each homogenous sulphonating reagent. We get the product from this sulphonating reagent as it is, or combining in several rate, e. g. the product which contain much soluble part is effective as deterging agent.

6. To wool fat 100% add zinc oxide, calcium oxide 3% or without adding them, act hydrolysis by steaming in an autoclave. Then, divide the fatty material which was got thus, in distillable part and not distillable part by vacuum distillation and after sulphonate each part separately use the product as it is or combining in any rate.

The product which obtained by this invention is far superior in the operation of deterging, emulsifying, wetting and dispersing than the many products which sulphonated using this as raw materials or using lanolin acid or higher alcohol under the wrong knowledge of past.

That is (A) the product which obtained by separating acid part from wool fat and sulphonate, then neutralize it, with caustic soda, (B) the product which obtained by separating not saponifiable part from wool fat and sulphonate it, then neutralize with caustic soda, (C) the product neutralized with caustic soda after sulphonating the whole components of wool fat by the process mentioning (instance of experiment 1). Then, making these three samples phenol phthalene neutrality and determined with dyne centimeter the surface tension which is fundamental property to compare the superiority or inferiority of the action of deterging, wetting, dispersing, emulsifying, and etc. under the same condition, we got the results as mentioned below, but the method of testing them is made by taking 0.1% solution from each sample and acted by dropping number calculation under 30° C., 40° C. and 50° C.

Sample	30° C	40° C	50° C
(A)	52.56	53.57	53.90
(B)	64.11	63.51	59.61
(C)	46.42	43.88	41.52

As you see in the above table the surface tension of the product (C) made by the process mentioning is weaker about 28%–31% comparing to (B) and about 12%–23% comparing to (A). Next, in testing quality of each sample (A) is good in solubility but less colloidal, (B) is bad in solubility and more colloidal, (C) is good in solubility and more colloidal, too.

After all, this invention is to extract the whole main components of wool fat without any loss as a sulphonating product, the product which obtained by substituting ester radical is colloidal and superior in wetting action.

Now, the product which obtained by this invention can be not only made as a perfect product which is proper in the every use as the fibre industrial agent like leather industry, textile industry, etc., but also it can not be acted by hard water or dissolve easily in sea water and shows the many actions as mentioned before. Also, if you combine this in the dental powder (or paste) it makes the quality superior and it is very effective to make the detergent action of mouth perfect, by double decomposition with base as calcium carbonate, magnesium carbonate which were contained in the usual, soap without the fear of producing insoluble salts like calcium soap, magnesium soap.

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