

ALIEN PROPERTY CUSTODIAN

PROCESS FOR PREPARING WATER-SOLUBLE CONDENSATION PRODUCTS

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It is already known from French Patent Specification No. 674,517 to treat fats, fatty acids or resins together with hydrocarbons or substitution products thereof, and in addition substances which contain the groups OH, COSH, COOH, CO, O, OC, CO, with chlorosulphonic acid.

According to the present invention sulphonated condensation products are made from esters of fatty acids, low molecular or high molecular, monobasic or polybasic, more particularly liquid esters, with monohydric alcohols, low-molecular and high-molecular, of aliphatic, aromatic or hydroaromatic nature, or phenols, naphthols or the like, together with polynuclear hydrocarbons, their hydrogenation products or derivatives such as naphthols, chlornaphthalene, naphthylamine. By simultaneous sulphonation and condensation the esters can be bound to the hydrocarbons with formation of true condensed sulphonic acids. Preferably operations are carried out by treating the mixture of hydrocarbon and ester with sulphonating agents such as oleum, chlorosulphonic acid, concentrated sulphuric acid, SO₃, in a stirring arrangement whilst thoroughly admixing.

The reaction may take place at elevated or reduced temperature; if the temperature is too high relatively dark products are obtained. Preferably operations are carried out with both reaction components present dissolved in one another or molten; both components also may be dissolved in a solvent. However, it is not absolutely necessary for both components to be dissolved in one another at the beginning of the reaction; but naphthalene, for example, if sulphonation is effected at relatively low temperature, may still be in the crystallised state at the commencement and may form a homogeneous mixture only as the reaction progresses. The reaction can be carried out at temperatures of 10-100° C. At relatively low temperatures of course preferably liquid hydrocarbons are taken, such as tetraline, or operations are carried out in the presence of solvents. At temperatures above 100° C dark products are obtained. The reaction may also be facilitated by adding condensing agents, such as phosphorus pentachloride, thionyl chloride, sulphuryl chloride, phosphorus oxychloride, during the addition of the sulphonating agent. In this way sulphonating agents are economised or better condensation is obtained more rapidly. As esters are used more particularly waxes, such as sperm oil, bottle-nosed whale blubber, or artificially prepared waxes. These artificially prepared waxes may for example be prepared by treating natural glycerides or waxes with low-

molecular alcohols in the hot under pressure in the presence of carbonic acid or sulphurous acid, when as a result the starting material is re-esterified in such a way that in place of the glycerin of the glycerides or of the alcohols of the wax the added low-molecular alcohol enters into the compound and forms an artificial ester. Large-surfaced bodies, such as active carbon, clay and so forth, as well as emulsifying agents may also be added in the conversion. However, the esters may also be prepared directly by uniting acids and alcohols in the hot under pressure in the presence of the gaseous acid anhydrides CO₂ or SO₂; here again large-surfaced substances may be added. Moreover the reaction water forming is removed as well. Useful products are also obtained with sperm wax or montan wax. Similarly, other esters of fatty acids and monohydric alcohols or phenols, as well as such as are made by the above-mentioned methods, may also be used. Also mixtures of different esters and mixtures of different hydrocarbons and mixtures of these mixtures may be used. As multi-ring hydrocarbons pre-eminently naphthalene, tetrahydronaphthalene and anthracene are used.

The products obtained are oils, pastes, or after evaporating down or spraying solid substances, according to the starting materials.

The sulphonic acids which form cannot be split even after boiling for days with concentrated acids or alkalis. They are stable towards the hardness formers of water. The products can be neutralised with organic or inorganic bases. The reaction products are used as outstanding washing, wetting, emulsifying and scrooping agents. Emphasis is to be laid on the softening action of the products. Further they show good emulsifying, dispersing and dye-dissolving action. The sulphonated condensation products have special importance for the softening and preparation of cellulose wool. They may however also be employed for treating other fibrous substances.

Example 1

90 parts of sperm oil and 35 parts of naphthalene are condensed with sulphonation at 40° C with 150 parts of 25% oleum in a stirring arrangement. The acid mass is allowed to stand over-night, washed next day with concentrated common salt solution and neutralised with concentrated soda lye. The reaction time amounts to three hours. Washing with concentrated common salt solution can also be omitted and in place thereof the sulphonate diluted with some water be directly neutralised. The product is ad-

justed to 33% sulphonate former, but may also be adjusted to any other sulphonate former content. The product serves for the purposes mentioned in the specification, especially as washing and softening agent.

Example 2

100 parts of 25% oleum are allowed to run slowly into a mixture of 100 parts of sperm oil and 50 parts of tetrahydronaphthalene at a temperature of 30° C whilst thoroughly stirring. After addition of the oleum 100 parts of chloresulphonic acid are added, likewise whilst stirring, at the same temperature, and on the next day, after washing with concentrated common salt solution, neutralisation is effected with triethanolamine. The reaction takes eight hours. The neutral product obtained dissolves readily in water and is absolutely stable to acid. The product may be used with advantage for preparing raw cellulose wool. It makes this loose and soft.

Example 3

50 parts of the phenyl ester of oleic acid and 50 parts of naphthalene are condensed with sulphonation with 200 parts of 25% oleum at about 50° C. The sulphonation takes about three hours. The sulphonate is allowed to stand overnight. The sulphonate is neutralised with soda lye either directly or after washing with common salt solution. The soda lye may be wholly or partly replaced by pyridine, triethanolamine or some other organic base. The product is adjusted to 33% sulphonate former.

Example 4

80 parts of naphthalene are sulphonated with 100 parts of 25% oleum. The naphthalene sulphonic acid is prepared at about 70° C. The temperature is then lowered and 120 parts of sperm oil at 40° C. are added and 150 parts of 25% oleum, likewise at 40° C. The product is neutralised after standing over-night and washed with com-

mon salt solution. The reaction time altogether amounts to about five hours. Adjustment is effected to 33% sulphonate former.

Example 5

90 parts of sperm oil and 35 parts of naphthalene are treated with 125 parts of oleum and 20 parts of phosphorus trichloride under the conditions of Example 1. The reaction time amounts to eight hours. The whole is allowed to stand over-night, washed and neutralised with soda lye and/or an organic base such as triethanolamine.

Example 6

100 parts of sperm oil and 50 parts of naphthalene are sulphonated at 100° C. with 100 parts of oleum. The oleum is added in the course of two hours. The reaction product is diluted with water and neutralised. A light brown paste is obtained. The acid sulphonate can of course also be washed and after neutralisation be adjusted to a definite sulphonate former content.

Example 7

90 parts of sperm oil and 35 parts of naphthalene are sulphonated at 35-40° C. with 125 parts of 25% oleum. During the sulphonation 20 parts of thionyl chloride are added. The reaction time amounts to about three hours. After the reaction is completed the whole is allowed to stand for some time, then washing is effected with common salt solution and neutralisation with soda lye, a part of the soda lye being replaced if desired by triethanolamine.

Example 8

100 parts of sperm oil and 25 parts of naphthalene are sulphonated at 30° C. with 150 parts of 25% oleum. The reaction time amounts to three hours. Washing is then effected with common salt solution, followed by neutralisation.

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