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PRODUCTION OF ASSISTANTS FOR THE TEXTILE AND RELATED INDUSTRIES

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The present invention relates to the production of assistants for the textile and related industries.

We have found that valuable products which are suitable for example as impregnating, dissolving, wetting, cleansing, tanning, softening, emulsifying, dispersing and like agents, are obtained by condensing saturated organic compounds containing halogenated long aliphatic chains (with at least 5 carbon atoms) or mixtures mainly consisting of these compounds with organic nitrogenous bases containing at least one reactive hydrogen atom connected with a nitrogen atom and if desired causing organic or inorganic non-oxidizing acids, or derivatives of such acids chemically reacting like the free acids, to react simultaneously or subsequently with the products obtained to form salts therewith, or esters with strong polybasic mineral acids, or, with strong sulphonating agents, to form sulphonic acids of the resulting amines or their salts.

As examples of initial materials may be mentioned halogen derivatives of hydrocarbons containing one or more halogen atoms in the long aliphatic chains, or of mixtures or substitution products of the same, as for example products from halogenating the different petroleum fractions (benzines, illuminating oils, ligroins, lubricating oils, paraffin oils, solid paraffins and their oxidation products containing acids, alcohols and similar oxygenated compounds) or the products from the liquefaction of coal, brown coal tar oils and the like, or montan wax, fatty acids, such as the acids of vegetable or animal fats or oils, higher alcohols, as for example decyl alcohol, and ketones, such as di-amyl ketone or still higher ketones. Organic compounds which, in addition to the halogenated long aliphatic chains, contain aromatic, hydroaromatic or heterocyclic radicles are also suitable. The preparation of the halogen-containing organic compounds may be carried out according to methods already known, as for example by the direct action of halogens on the said organic compounds while heating.

The treatment of the halogen compounds with the organic bases may be carried out in the presence or absence of organic solvents or diluents such as benzine, chlorbenzene, nitrobenzene, ligroin, tetrachlorethane, cyclohexanol or cyclohexanone if desired at a pressure above atmospheric pressure and/or in the presence of condensing agents capable of splitting off hydrohalic acids such as metallic copper and salts of copper, aluminium chloride, zinc chloride, or alkaline agents as for example soda or caustic soda sodium acetate and the like.

As the organic bases which contain at least one reactive hydrogen atom may be mentioned primary or secondary aliphtic, aromatic or hydroaromatic amines as for example methyl or dimethylamines, aniline, cyclohexylamine, chlorocyclohexylamine and the like and heterocyclic nitrogen-containing compounds such as piperidine and also urea, guanidine, acid amides and the like.

In so far as the resulting reaction products still contain free hydroxyl groups they may be subsequently converted into esters with phosphoric or sulphuric acids or other strong polybasic mineral acids or with aliphatic mono- or polycarboxylic acids of low molecular weight such as oxalic, acetic, lactic and like acids. The said acids may be also employed for converting the amides into salts. When compounds are formed which are capable of forming salts, they may be converted into their ammonia or alkali metal salts and used for the purposes hereinbefore specified. In cases when employing sulphuric acid, or an agent furnishing the same, the process may be carried out so that the sulphur atom is not combined in the form of sulphuric ester but that the sulphur atom is directly combined with a carbon atom, so that true sulphonic acids are formed as can be obtained on working with sulphur trioxide, or like strong sulphonating agent, if desired in the presence of inert solvents such as carbon tetrachloride.

The resulting products may be employed for a great variety of purposes and frequently have a good wetting, washing and emulsifying action, but generally speaking they have a whole series of advantages which soaps do not have, as for example high stability to acids, insensitiveness to alkaline earth salts and the like, so that, contrasted with soaps, their field of employment is considerably extended. They are suitable for example for the production of preparations for the treatment of textiles as for example oil emulsions for spinning, greasing leather or im-

pregnating but they are also useful as assistants in the production of drilling greases or as drilling oils. They may be used as substitutes for fats, oils and mineral oils, they are suitable as levelling agents in the dyeing of materials of all kinds, for the preparation of very finely divided pigments and the like. The resulting acid esters as well as the free acids and salts are excellent washing and cleansing agents even for acid liquors, the amines being valuable solvents and dispersing agents.

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

Example 1

1000 parts of a paraffin wax having a melting point of from 46° to 50° C. are treated at 60° C. with chlorine until an increase in weight of 250 parts has taken place. After passing a powerful current of air through the reaction product in order to remove gaseous hydrogen chloride, the resulting product is heated to 180° C. with 500 parts of monoethanol amine for 5 hours. The oil obtained is washed with water and then converted, by heating for twenty-four hours at from 50 to 60° C. with 2500 parts of chlorosulphonic acid, into the acid sulphuric ester which after dilution with water constitutes a good cleansing and wetting agent. Similar products are obtained by employing 150 parts of diethanol amine instead of the monoethanol amine.

Instead of the halogenated paraffin wax, halogenated fatty acids, halogenated montanic acid, halogenated naphthenic acid and similar products may be employed with equivalent results.

Example 2

Chlorine is led into 200 parts of an American illuminating petroleum (boiling point from 150° to 300° C.) at from 60° to 70° C., until an increase in weight of about 50 parts has taken place. Hydrogen chloride formed is then removed by passing a powerful current of air through the reaction product. 1000 parts of the chlorinated petroleum are heated in an autoclave for 10 hours at 160° C. with 250 parts of piperidine and a small amount of copper powder. The resulting oil yields readily soluble salts with acids such as hydrochloric, sulphuric, phosphoric or acetic acids; these salts give well foaming solutions having an action similar to soap in water.

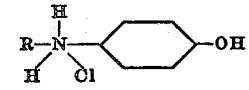
Example 3

Chlorine is led at 80° C. into 250 parts of a brown coal tar oil boiling between 200° and 350° C. until an increase in weight of 35 parts has taken place and the remainders of chlorine and the dissolved hydrogen chloride are expelled with a powerful current of air. The resulting chlorinated product is heated to 160° C. for 10 hours in an autoclave with 80 parts of normal butylamine and a trace of copper powder. A basic oil is obtained which is very suitable as a softening agent for leather and as a dispersing agent for dyestuff printing pastes. With acids the product yields readily soluble salts which have good foaming and saponaceous properties even in acid solutions.

Similar products are obtained by employing 140 parts of di-n-butylamine or 60 parts of ethylene diamine instead of the normal butylamine.

Example 4

250 parts of a chlorinated petroleum obtained according to Example 1, are heated to 200° C. for 10 hours with 109 parts of para-aminophenol and a trace of copper powder. A thick oil is obtained which mainly consists of the following compound:



in which R is a hydrocarbon radicle containing long carbon chains. This compound is soluble in water and the solutions have strong dispersing properties.

160 parts of 1.2-, 1.7- or 2.7-aminonaphthols may be employed instead of the para-aminophenol with the same effect.

The resulting product may be sulphonated and then yields a readily soluble sulphonic acid which is very suitable as a wetting agent.

Instead of the free aminophenol, the halogenated petroleum may be treated with one or a mixture of the isomeric aminophenol sulphonic acids. Water-soluble products are then directly obtained.

Example 5

350 parts of montan wax bleached with chromic acid and mainly consisting of high molecular carboxylic acids are chlorinated at 80° C. until a chlorine content of 15 per cent is obtained. The chlorinated product is freed from hydrogen chloride and any chlorine which may still be present and is heated for 15 hours at 200° C. with 300 parts of stearic acid amide. The resulting high-molecular acid amide may be converted into a water-soluble sulphonic acid having good saponaceous action by treatment with 1000 parts of fuming sulphuric acid containing 10 per cent of sulphur trioxide at from 80° to 100° C. The halogenated paraffin waxes hereinbefore described, varieties of petroleum and the like may be employed instead of the chlorinated montan wax with equivalent results.

What we claim is:—

1. The process for the production of assistants in the textile and related industries, which comprises condensing halogenated paraffin with an organic nitrogenous base, containing at least one reactive hydrogen atom connected with a nitrogen atom.

2. The process for the production of assistants in the textile and related industries, which comprises condensing chlorinated paraffin wax with a primary alkylolamine.

3. The process for the production of assistants in the textile and related industries, which comprises condensing chlorinated paraffin wax with a primary alkylolamine and acting on the resulting mixture of amines with a strong non-oxidizing acid.

4. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic compounds each of which contains at least five carbon atoms, with a heterocyclic organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom.

5. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon

atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, in the presence of a condensing catalyst capable of splitting off hydrohalic acids.

6. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, and acting on the resulting mixture of amines with a strong non-oxidizing acid.

7. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, and acting on the resulting mixture of amines with a strong polybasic mineral acid.

8. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, at least one of the said reaction components containing at least one hydroxyl group, and acting on the resulting mixture of amines with a strong non-oxidizing acid until esters are formed.

9. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, and sulphonating the resulting mixture of amines by treatment with a sulphonating agent.

10. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom, and acting on the resulting mixture of amines with a strong sulphonating agent until sulphonic acids of the amines contained in the said mixture are formed.

11. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons of natural origin each of which contains at least five carbon atoms, with an organic nitrogenous base containing at least one reactive hydrogen atom connected with a nitrogen atom.

12. The process for the production of assistants in the textile and related industries which comprises condensing a mixture of saturated halogenated aliphatic hydrocarbons each of which contains at least five carbon atoms with a mono-alkylol-amine.

13. A mixture of different ethanolamines each of which corresponds to the formula $HO.CH_2—CH_2—NH.R$, in which R represents a saturated aliphatic radicle containing a number of carbon atoms corresponding to that of the constituents of paraffin.

14. The products obtainable by carrying out the process set forth in claim 4.

15. The products obtainable by carrying out the process set forth in claim 6.

16. The products obtainable by carrying out the process set forth in claim 11.

17. Preparations suitable as assistants in the textile and related industries comprising essentially a mixture of amines each of which corresponds to the formula $R_1—NH—R_2$, wherein R_1 denotes a saturated aliphatic radicle containing at least 5 carbon atoms and R_2 denotes an alkylol radicle.

18. Preparations suitable as assistants in the textile and related industries comprising essentially a mixture of amines each of which corresponds to the formula $R—NH—CH_2—CH_2OH$, wherein R denotes a saturated aliphatic radicle containing at least 5 carbon atoms.

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