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FATTY DERIVATIVES OF ALKYLATED AMINES

Jacob Katz, Providence, R. I., assignor to Warwick Chemical Company, West Warwick, R. I., a corporation of Rhode Island

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The present invention relates to the manufacture of fatty derivatives of alkylated amines and particularly relates to the manufacture of fatty acid amides which are valuable as assistants in the textile, leather and allied industries.

The invention further relates to an improved finishing process for textiles wherein novel effects are produced giving the textile an improved hand and enhanced texture, said improved finishing being accomplished by the use of alkylated amine derivatives.

The usual type of assistants or emulsifying agents which are utilized in the textile, leather, paper and allied industries are of the nature of soaps or sulphonated compounds having high molecular weight fatty acid groups which are combined with alkali metals, the usual compounds being the sodium salts of the fatty acids or the sodium salts of sulphonated or sulphated fatty acids, alcohols or ketones.

These compounds in the presence of water or in water solution tend to split into positively charged sodium ions and negatively charged high molecular weight fatty ions.

As a result, these materials are of little value in baths containing substantial concentrations of heavy metal or alkaline earth metal ions since the fatty materials tend to combine with the heavy metals and alkaline earth metals to form insoluble materials which are precipitated.

Moreover, the presence of substantial amounts of acid in the bath tend to split out the fatty matter and cause precipitation of the fatty material.

It is among the objects of the present invention to produce new chemical compounds having the valuable assisting or emulsifying properties of the nature of soaps and sulphonated compounds, above referred to, which at the same time will be relatively stable in baths containing substantial quantities of alkaline earth and heavy metal materials and/or substantial amounts of acid materials.

Another object of the present invention is to produce new and inexpensive dispersing agents, wetting agents, emulsifying agents and softening agents of wide application and of general utility in textile, leather, electro-chemical, metal and allied industries.

It is further desired to produce compounds of the character above described which, when applied to fabrics or other textiles, or other materials, for softening or for conditioning purposes will remain substantially permanent upon such fabrics even though said fabrics be worn or vigorously washed many times and it is among the further objects of the present invention to produce compounds of this character and method of treating fabrics which will produce these results.

Other objects will be obvious or will appear during the course of the following specification.

In accomplishing the above objects, it has been found that certain types of non-hydroxy-aliphatic amino compounds may be combined or condensed with fatty acids or high molecular weight saturated or unsaturated aliphatic acid compounds to produce amido compounds of unusual properties and of great value.

It has been found most satisfactory to utilize an aliphatic amine preferably containing from two to twenty carbon atoms, which may contain two or more amino groups, which in the preferred embodiment are attached to different carbon atoms. These amines are included in the claims by the expression "low molecular weight aliphatic polyamine."

Although these amino groups may be substituted by methyl, ethyl, propyl or other alkyl or aryl or aralkyl groups, it has been found most satisfactory to employ primary amino groups.

In respect to the fatty acids, they may contain from eight to thirty carbon atoms and preferably from ten to twenty carbon atoms and if desired, they may contain one or more unsaturated bonds or one or more hydroxy groups. These fatty acids are included in the specification by the expression "high molecular weight fatty acid."

The carboxy group is preferably at the end of the carboxy chain although in certain instances it may be positioned at intermediate portions of the aliphatic chain.

In certain cases, sulpho, boro, or phospho groups may be attached to the aliphatic chain at the ends or intermediate portions thereof which groups may be amidized at the same time as the carboxy groups.

In some instances, the boro-phospho- or sulpho groups themselves may be amidized and the carboxy groups may be omitted or eliminated or they may be converted into ester groups or even into alcohol, ketone or aldehyde groups, by esterification or reduction processes.

It is an important feature of the present invention that there are utilized at least two mols of a fatty acid for every mol of a poly-amino non-hydroxy aliphatic compound having two or more primary amino groups.

Among the amines which may be employed are ethylene diamine, diethylene tramine, triethylene tetramine, glycine amide, and various di, tri,

tetra and poly amines of normal, iso or tertiary propanes, butanes, pentanes, hexanes, cyclanes or unsaturated olefines, having from two to eight carbon atoms.

These aliphatic compounds may in some instances contain ether, keto, nitro, carboxyl or sulpho groupings.

The preferred fatty acids are lauric, myristic, palmitic, ricinoleic, oleic, stearic, eleostearic, hydrogenated abietic, cyclo carboxylic or other acids in which the chains or rings are homogeneous or heterogeneous containing from two to twenty-four carbon atoms and one or more carboxy groups and are also in some instances containing as elements of the chain or ring —CH_2—, —O—, —S—, —CO—, —N— and other groupings.

The preferred compounds, however, are those formed from ethylene diamine, triethylene tetramine and the fatty acids, lauric, myristic, palmitic, oleic and stearic.

The condensation reaction between the two or more mols of the acid and the one mol of the polyamine may be conducted at atmospheric or reduced pressures, reduced pressures producing lighter colored products.

The temperature reaction may range between 50° and 200° C. and sometimes up to 250° C. and the temperatures are gradually increased as the condensation proceeds from the formation of the amino esters to the formation of the amides.

As a general rule, the residual amides which are obtained after the water of the reaction has been removed are insoluble in water and are yellowish waxy products having melting range from 50° to 150° C., the materials are insoluble in water, but may be put in solution either by relatively strong bases or acids because of their amphoteric properties.

Among the acids which may be employed for solubilizing the final amides are acetic, formic, lactic, propionic, boric, sulphonic acids, alkyl phosphoric acids, and so forth.

The amide being insoluble and not readily emulsifiable, may be deposited upon textile fibers or fabrics, whether woven or knitted, by modification, elimination or removal of the solubilizing agent, with assurance that it will remain in position and give an unusual and distinct feel to the fabrics.

For example, acetic or formic acids may be readily volatilized away leaving the amide upon the fabric. This deposition may be carried out after the fabrics have been impregnated or sprayed with the solution of the amide and the amides may be or consist of other amides than those specifically disclosed, such as compounds of the type R_3CONX_2 where R_3 is an alkyl having from eight to thirty carbon atoms, and X is hydrogen, or an aliphatic or aromatic grouping.

The compounds are particularly satisfactory inasmuch as they will not precipitate in the presence of acids, or heavy metal or calcium salts.

In forming the final compounds, there is first an interaction of an alkyl polyamine containing at least two amino (NH_2) groups with as many mols of fatty acid as there are amino groups.

This reaction is as follows:

(1) $H_2NR_1.NHR_2.NH_2 + 2R_3COOH \longrightarrow$
Polyamine, fatty acid
$R_3COOH.NH_2R_1.NHR_2.NH_2HOOCR_3$
Addition product

R_1, R_2 and R_3 represent the same or different alkyl groups, R_3 preferably being higher and R_1 and R_2 lower alkyl groups.

In the second reaction there is the formation of amides by heating the above addition product until the calculated quantity of water is split out of the molecule.

This reaction is approximately as follows:

(2) $R_3COOH.NH_2R_1NHR_2NH_2.HOOCR_3 \xrightarrow{125\text{-}300°C.}$
Addition product
$R_3CONHR_1.NHR_2NHOCR_3 + 2H_2O$
Fatty acid amide + water

As typical examples which are given by way of illustration and not by way of limitation, since many changes and substitutions may be made, all within the scope of the present invention.

Example I

568 parts (2 mols) of stearic acid are melted in a 2 liter Calissen distilling flask (the side arm equipped with a water cooled condenser) and raised to a temperature of 90° C. Whereupon 103 parts (1 mol) of diethylene triamine are slowly added. The reaction is strongly exothermic and the heat of reaction raises the temperature to 115 to 120° C. The product which at first had turned to a yellow-orange jelly becomes liquid and heating is again resumed. When the mass has been raised to 160° C., the product suddenly begins to froth and foam and the second amide-forming reaction takes place. Heat is continually applied, the temperature remaining practically constant while the water of reaction is collected in the drip. When the frothing subsides the reaction is over and the temperature begins to rise.

The drip contains 36 grams (2 mols) of water (as calculated) together with a small quantity of the alkyl amine. The resulting product is a light yellow waxy solid-melting point about 100 to 105° C. (depending on the heat treatment) and insoluble in water.

Example II

One mol of cocoanut oil fatty acid and one mol of Japan wax fatty acid are reacted as above, with one mol of ethylene diamine. The reaction is carried out as in Example I, except that frothing begins at about 140 to 145° C. The resulting product is much darker than the stearyl derivative in Example I, and lacks the hard waxy appearance. It is insoluble in water.

Example III

One mol of lauric acid and one mol of chlorinated stearic acid are reacted with one mol of triethylene tetramine by slowly adding the tetramine to the molten fatty acids at a reduced pressure of 100 mm. The mixture is slowly heated at 140° C. when the frothing begins. The calculated amount of water is collected in the drip and the reaction is stopped. The product is a hard yellow waxy substance insoluble in water.

It is, of course, possible to utilize other mixtures of acids than those above described, such as mixtures of oleic and palmitic acid or lauric and myristic acids, and in some cases the oils, fats or waxes themselves may be employed where the glycerine or basic part of the molecule will be split off and may be removed by distillation under vacuum or by steam, or in some cases, the split product may be left in the reaction mixture.

It is also possible to condense the amines and the fatty acids with aromatic or other compounds during the condensation reaction and this may be accomplished by adding small quantities of condensing agents such as aluminum chloride, chloro-sulphonic acids, and so forth.

The preferred condensation materials are aromatic compounds, such as cresols, xylenols, ben-

zene toluene, naphthalene and other unsubstituted or substituted mono- or poly-nuclear hydrocarbons.

The final products produced which may be sulphonated, phosphated and/or borated are most valuable agents for wetting and dispersing purposes and may be most satisfactorily employed in fulling baths, dye baths, bleaching baths and mercerizing baths.

The compounds may be utilized by themselves or in combination with other agents for the treatment of yarns or woven or knitted textiles of silk, rayon, cotton, cellulose, acetate, or wool in the treatment of leather, paper and wood, in the manufacture of ceramics, in the metal and electro-plating industries, and for many other purposes. The amides can stand high concentration of heavy metals and strong acids, such as used in pickling and electro-plating baths.

A particular valuable application of the above identified compound resides in its application to yarns or woven and knitted fabrics to give them an improved soft hand and suede-like finish which may be attained with as little as 0.05 to 0.2% of the above amido compounds.

The above mentioned and other ingredients included in the invention may be added in various combinations and proportions to produce a final product of the desired physical and chemical properties. Hence, the application is not limited to the particular chemicals, quantities, temperatures, or steps of procedure specifically mentioned, as those are given simply as a means of clearly describing the invention.

It has not been most satisfactory to use condensation products of polyamines and fatty acids having a proportion of less than two mols of the fatty acid to one of the amine, as these compounds do not give the desirable hand or suede-like finish and also in certain instances, tend to have a stripping action.

Where the preferred compounds of the present invention are solubilized or given wetting or emulsifying properties they are solubilized or concerted by treatment with acids or acidic materials.

The present specification and claims are directed specifically to the use of the preferred novel compounds of the present application, while a divisional application filed February 12, 1940, Serial No. 318,591 is directed to the novel compounds of the present application per se. The preferred materials are generally cation active.

It is to be understood that the invention is not intended to be restricted to any particular example, composition or proportions, or to any particular application, or to any specific manner of use or to any of various details thereof, herein described, as the same may be modified in various particulars to be applied in many varied relations without departing from the spirit and scope of the claimed invention, the practical embodiments herein described merely showing some of the various features entering into the application of the invention.

What is claimed is:

1. A process of giving yarns and woven and knitted fabrics an improved soft hand and a suede-like finish which comprises treating such fabrics with an aqueous bath containing between 0.05% and 2.0% of a combination of one mol of low molecular weight aliphatic polyamine and at least two mols of a high molecular weight fatty acid.

2. A process of giving yarns and woven and knitted fabrics an improved soft hand and a suede-like finish which comprises treating such fabrics with an aqueous bath containing between 0.05% and 2.0% of a combination of two mols of stearic acid and one mol of diethylene triamine.

3. A process of giving yarns and woven and knitted fabrics an improved soft hand and a suede-like finish which comprises treating such fabrics with an aqueous bath containing between 0.05% and 2.0% of a combination of one mole of low molecular weight non-hydroxy aliphatic polyamine and at least two mols of a high molecular weight fatty acid solubilized by a small amount of weak water soluble acid.

4. Textile yarns and fabrics having a not readily emulsifiable deposited surfacing of a combination of one mol of a low molecular weight aliphatic polyamine and at least two mols of a high molecular weight fatty acid, said coating giving to said yarns and fabrics a soft hand and a suede-like finish.

JACOB KATZ.