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## SURFACE ACTIVE AGENTS

Murray Senkus, Terre Haute, Ind., assignor to  
Commercial Solvents Corporation, Terre  
Haute, Ind., a corporation of Maryland

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My invention relates to new and useful surface, or capillary active substances and more particularly it pertains to the utilization of certain amines to greatly reduce the surface tension of various aqueous mixtures and thereby increase the wetting properties thereof.

There exists in numerous industries a growing need for certain types of chemical compounds, usually employed in small quantities, to secure an effect which is principally the result of a wetting action such as the effect obtained by the presence of a surface active agent at a water-oil interface. For example, in the textile and leather treating industries, there are many operational steps where wetting or detergent action is highly desirable. As a result many different agents have been proposed to reduce surface tension and promote the wetting power of various types of aqueous mixtures. The materials previously used, however, although satisfactory in some respects, were not entirely free from certain material disadvantages.

In an effort to furnish materials to the textile and related industries free from the undesirable properties of ordinary soaps and other similar commonly known surface active agents, there have been developed as detergent and wetting agents products obtained by the sulfonation of certain fatty acids occurring in oils and fats. Such fatty acids are characterized by the presence of unsaturated linkages and/or hydroxyl groups in the molecule. On sulfonating such acids, products are obtained which contain sulfuric acid ester groups in the molecule and, of course, carboxylic acid groups are attached to the terminal carbon atoms. The products, however, in general possess weaker wetting and detergent properties than soaps. Such products are also relatively unstable and are decomposed by alkaline hydrolysis.

Other proposed wetting agents comprise the semi and completely esterified products resulting from the esterification of a higher alcohol with an unsaturated polybasic acid. After the unsaturated esterified acid is produced, it is converted into a more soluble form, ordinarily by treatment with sodium bisulfite to yield the corresponding sulfo derivatives of the esterified polybasic acid. While such materials have met with a considerable degree of success in various in-

dustries where the need for surface active materials exists, they also possess certain disadvantages. Obviously, surface active agents of this type are not stable to alkali and in some instances, especially in the textile field, much difficulty has been experienced with the tendency of the textile treating baths containing such materials to foam.

I have now discovered that certain amines serve as highly satisfactory wetting agents in wetting and assisting in the spreading of various solutions on solid surfaces, particularly, where prior wetting and detergent agents, especially of the type mentioned above, are of little or no value. Specifically the amines which I have found operative are 1-methylheptylamine, N-(2-aminoisobutyl)-1-methylheptylamine, N-(2-aminoisobutyl)hendecylamine, N-(2-aminoisobutyl)piperidine, and N-(2-aminoisobutyl)-2,4-dimethylaniline. The wetting agents of the present invention, for example are not decomposed either by hard waters or solutions containing appreciable quantities of alkali. My new wetting agents possess the further advantage in that aqueous solutions thereof do not foam. In addition these materials, which are generally water white liquids, are soluble in most of the common organic solvents.

One of the outstanding and surprising features of my invention is the fact that amines of the above type exhibit surface active properties. To my knowledge such amines have never before been known to possess such characteristics. A further surprising factor is that while the aforesaid amines have been found to be excellent surface active agents, numerous of their close homologues have been tested and were found to possess substantially no surface active properties whatever.

In general, the quantity of the new wetting agents which may be employed will be found to vary with the type of wetting operation under consideration as well as with the material to be wetted. For most purposes, however, I have found that these wetting agents may be utilized in amounts of from about 0.1 to 0.6% by weight of the treating bath.

The wetting power of the amines of the present invention is demonstrated by the data appearing in the table below and was measured by the method of Draves and Clarkson (Amer. Assoc. Textile

Colorists and Chemists Year Book, vol. 17, pp. 216-22 (1940)), which determines the sinking time required for a 5 g. skein of unboiled 2-ply cotton yarn in varying concentrations of aqueous solutions of the amines at a temperature of 30° C. The data below also show the comparative wetting powers of the wetting agents of the present invention and Santomerse No. 1 (a sulfonated alkyl di-phenyl), a commercially available wetting agent.

Table

| Amine                                        | Grams of amine per liter | Sinking time, sec. |
|----------------------------------------------|--------------------------|--------------------|
| 1-methylheptylamine.....                     | 2                        | 0                  |
| Do.....                                      | 1                        | 133                |
| N-(2-aminoisobutyl)-1-methylheptylamine..... | 2                        | 0                  |
| Do.....                                      | 1                        | 21.8               |
| N-(2-aminoisobutyl)-hendecylamine.....       | 1                        | 49.8               |
| N-(2-aminoisobutyl)-piperidine.....          | 2                        | 19.3               |
| Do.....                                      | 1                        | 33.2               |
| N-(2-aminoisobutyl)-2,4-dimethylaniline..... | 2                        | 45.6               |
| Do.....                                      | 1                        | 77.9               |
| Santomerse No. 1.....                        | 2.5                      | 12.9               |
| Do.....                                      | 1.25                     | 34.0               |

While 1-methylheptylamine can be synthesized in any of several ways known to the art, the remaining compounds are most conveniently prepared in accordance with the procedures of my copending application, U. S. Serial No. 455,932, filed August 24, 1942. By the procedure there described and claimed, amines of the latter type are synthesized by subjecting the corresponding nitro amine to catalytic hydrogenation in the liquid phase at superatmospheric temperatures and pressures. The nitro amines utilized in this process are prepared in accordance with the procedure described in my copending application U. S. Serial No. 455,931, filed August 24, 1942. According to this procedure, a primary or secondary aliphatic amine is reacted with formaldehyde to form the corresponding N-hydroxymethyl mono- or dialkylamine which is in turn reacted with an equimolecular quantity of a secondary nitroparaffin to produce the desired nitro amine. In the event an aromatic amine is employed an alkaline catalyst should be used. The preparation of such nitro amines is preferably carried out at temperatures of from between about 25 and 30° C. Temperatures above this range may be used but in doing so care should be exercised to avoid temperatures which cause the formaldehyde to be volatilized from the reaction mixture.

The wetting agents of the present invention may be used alone or in combination with other wetting agents or detergents for cleansing and scouring vegetable and animal fibers when removing fatty or oily materials therefrom. They may also be used as penetrants in flax retting baths. The penetrating power of these new compositions may be utilized with advantage when

they are added to baths containing starchy ferments which are employed for removing sizing from textile materials. These products function as useful wetting, cleansing, and penetrating agents in bleaching liquors such as those used in the kier boiling of cotton goods and may be added to the lye liquors used for mercerizing cotton goods. They can also be used to assist in twist setting of yarn and in processes of stripping colors. Such compounds may also be used as assistants in processes of stripping dyes from dyed textile material. A second class of uses of these new surface active agents is as assistants in the application of dyestuffs. They may be used as dye leveling agents owing to their wetting and penetrating properties in neutral or alkaline dyeing baths and are capable of facilitating dyeing with developed dyes, the dyeing of acetate fibers with insoluble dyes, the dyeing of animal fibers with vat dyes, the dyeing of various synthetic fibers and the dyeing of leather.

It will readily be seen from the foregoing discussion that by means of the present invention, a group of new surface active materials has been made available and that such agents are adapted for use in numerous industrial processes which require surface active substances. It will also be apparent to those skilled in the art that many widely different embodiments of the present invention exist and that such embodiments may be utilized without departing from the scope of said invention.

My invention now having been described what I claim is:

1. An aqueous bath for the wet treatment of organic fibrous materials comprising an amine selected from the class consisting of N-(2-aminoisobutyl)-1-methylheptylamine, N-(2-aminoisobutyl)-hendecylamine, N-(2-aminoisobutyl)piperidine, 1-methylheptylamine, and N-(2-aminoisobutyl)-2,4-dimethylaniline.

2. An aqueous bath for the wet treatment of organic fibrous materials comprising from about 0.1 to 0.6% by weight of the treating bath of an amine selected from the group consisting of N-(2-aminoisobutyl)-1-methylheptylamine, N-(2-aminoisobutyl)-hendecylamine, N-(2-aminoisobutyl)piperidine, 1-methylheptylamine, and N-(2-aminoisobutyl)-2,4-dimethylaniline.

3. An aqueous bath for the wet treatment of organic fibrous materials comprising N-(2-aminoisobutyl)-1-methylheptylamine.

4. An aqueous bath for the wet treatment of organic fibrous materials comprising N-(2-aminoisobutyl)hendecylamine.

5. An aqueous bath for the wet treatment of organic fibrous materials comprising 1-methylheptylamine.

MURRAY SENKUS.