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WATERPROOFING COMPOSITION AND
SIMILAR EMULSION

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The present invention relates to improvements in emulsions and methods of making the same, and particularly relates to an improved waterproofing emulsion.

5 In the United States Patents 2,015,864 and 2,015,865 there are disclosed positively charged emulsions containing water insoluble materials, such as waxes, fats, oils, and so forth in disperse phase, which disperse phase is positively charged and maintained by protective colloids and/or emulsifying agents.

10 It is among the objects of the present invention to provide improved emulsions and methods of making the same particularly designed for waterproofing purpose in which the disperse phase will have high stability in concentrated or diluted form under varying atmospheric conditions and upon storage.

15 Another object is to provide an improved waterproofing emulsion which will much more effectively treat fabrics, paper, cloth, furs and other materials to give them waterproof properties and which will be of high efficiency even in low concentrations.

20 Another object is to prepare an improved emulsion, particularly designed for waterproofing purposes, which will give a waterproofing of particularly high effectiveness, and which is devoid of foaming characteristics and will not tend to form bubbles upon fabric or other materials and which, furthermore, will insure a uniform effect upon the fabrics without spotting or staining.

25 Another object is to provide an improved emulsion of the character described, having an increased surface tension and particularly designed for waterproofing purposes which will be devoid of complex organic protective colloidal materials such as glue, gelatine, casein, albumin, gums, 40 starches and so forth, which might tend to degrade or disintegrate or become modified upon standing, particularly in acidic environment.

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

45 It has now been found that particularly stable emulsions, suitable for waterproofing purposes and containing an oil, wax or fat, or similar water-repellant materials, may be prepared by combining certain types of basic aliphatic compounds with these waxes, oils or fats and a positive charging material such as a bivalent or trivalent metallic salt of a weak acid.

50 Among the preferred compounds which may be utilized are those formed by the combination of an aliphatic amine and a high molecular weight aliphatic compound, preferably containing more than eight carbon atoms, the preferred compounds containing between 14 to 28 carbon atoms.

60 Among the preferred compounds, which may

be utilized are alcohols, ketones, acids, and esters containing one or more amido or amino groups, one or more sulphate or sulphonate groups and one or more of the following radicals, namely, stearyl, abietyl, oleyl, linoleyl, ricinoleyl, ceryl, cetyl, myricyl, palmityl, dodecyl, octadecyl and so forth.

It has been also found satisfactory in some cases to utilize acids derived by the splitting of various vegetable or animal oils, fats and waxes, such as the fatty acids which may be obtained by splitting or hydrolysis of the following oily materials, namely, olive oil, menhaden oil, palm oil, mustard seed oil, pea seed oil, coconut oil, cottonseed oil, rape seed oil, castor oil, neat's-foot oil, peanut oil, lard oil, tallow oil, elaine oil, corn oil, sperm oil, fish oil, and/or cod oil or mixtures thereof.

These oils, or fatty acids, alcohols, ketones or aldehydes containing about the same number of carbon atoms may also serve as the waterproofing materials.

Among the preferred fatty or aliphatic acid materials which may be employed are stearic acid, oleic acid, palmitic acid, linoleic acid, linolenic acid, ricinoleic acid, lauric acid, myristic acid, abietic acid, and so forth.

These acids are preferably combined with organic amino compounds preferably of an aliphatic nature which amino compounds may also be hydroxylated or otherwise substituted.

Among the preferred amino compounds are those containing from two to ten carbon atoms and from one to five amino groups.

Among the compounds which may be preferred are diethylene-triamine, triamino-propane, diamino-ethane, triaminopentane, mono-di- or triethanolamine, diamino-propanol, other mono- or poly-amino-alkanes, imino- or alkylamino alkanes, alkenes and alkynes, which may be also substituted by groups, such as the —NO group, the —NO₂ group and so forth.

These amines may first be combined with the acids to form the ammonium salts or esters following which the combination may be dehydrated with removal of water to form an amido combination.

These combinations are referred to in the specification and claims as "amido fatty acids" or as "fatty acid amides."

However, if desired, the amino compound or compounds may be substituted directly in the aliphatic chain, whether the aliphatic chain be that of a fatty alcohol, ketone, aldehyde or acid and this amino group may be combined with a low molecular weight acid followed by amidization.

In all of these cases the esters of the fatty acids or alcohols may be employed as may also their sulphonates where the sulphonic groups are at-

tached to the aliphatic chain or the sulphate-esters may be utilized.

According to one preferred method of carrying out the present invention, the amido compound is prepared by heating together the poly-amino alkane and the fatty acid which is to be combined with said amino compound.

Either one or both of these compounds may contain an aliphatic chain containing between 8 to 24 carbon atoms and these compounds are preferably reacted together in the proportions of 1 mol of the alkane to 1 mol of the acid although higher ratios of amines to acids are readily possible.

The mixture of the amine and acid is preferably heated up to a temperature above 150° C. until foaming subsides and until some or nearly all of the amino combination has been transformed into an amido combination.

The relative proportions of amido to amino groups may be widely varied depending upon the exact utilization of the compound.

To give one specific example, 1 mol of diethylene triamine or 103 grams thereof are added to 1 mol of molten or melted stearic acid or 284 grams thereof.

The heating and stirring together of the mixture is continued from 15 to 30 minutes with an optimum of about 20 minutes until the temperature reaches about 180 to 190° C.

Water will be liberated as a result of the amido formation and the end of the action will be apparent when the foaming subsides. After cooling, an amido stearyl diethylene amine is obtained.

If desired, the corresponding weights of palmitic, oleic, abietic or other fatty acids may be employed.

The resultant material is particularly designed as an emulsifying agent or as an emulsion stabilizer and the proportion of acid to amine may be varied depending upon the utilization.

If the material is to be utilized for emulsification of vegetable waxes, it is desirable to utilize from 1½ to 3 mols of amine of each mol of fatty acid.

Where the emulsifier is intended for the emulsification of mineral waxes, such as paraffin, the ratio of amine to fatty acid may be between 1 mol of the former to 1 to 3 mols of the latter.

This emulsifying agent may be incorporated in relatively small amounts with positively charging materials and with water insoluble fats, oils and waxes.

Among the preferred positive charging materials are metallic salts which on heating will form insoluble oxides such as the salts of zinc, aluminum or other bivalent and trivalent metals having amphoteric properties, preferably in combinations with weak acids as for example, organic acids.

Among the salts which may be utilized are the acetates, the formates, the gluconates or even the hydroxides in certain instances.

Among the water insoluble materials which can be utilized are the various oils disclosed above, as possible sources of fatty acids or as raw materials for the manufacture of fatty alcohols.

There also may be utilized various fats and waxes such as paraffin, ozokerite, ceresin, beeswax, candelilla wax, carnauba wax, japan wax, montan wax, spermaceti, chlorinated naphthalene or Halowax, petrolatum or petroleum jelly, with or without a wax mineral oil, benzene or even fatty acids such as stearic acids or the vari-

ous fatty acids and alcohols which may be derived from the oils above mentioned.

Under some conditions, paraffin oil, asphalt, bituminous substances and caoutchouc, may be used, but these materials are generally not preferred.

It is also possible to use colloidal stabilizer materials, such as gelatine, glue, albumin, casein, starch, gum, dimethyl cellulose, protalbic acid, carbohydrates and sulphite waste liquors, but it has been found generally preferable to omit these colloidal stabilizer agents from the emulsifying mixture.

In making the emulsion, the waxy or the fatty material may be mixed with a small amount, say 10% of the emulsifying material or amido compound. Then, the positive charging material may be added with stirring.

Finally, water may be added preferably at a temperature above 100° F. until a uniform mixture is obtained. This mixture is preferably passed through a colloid mill to assure thorough homogenization and combination thereof.

To give a specific example, 88 grams of paraffin wax and 6 grams of amido stearyl diethylene amine are melted together, following which 150 grams of aluminum acetate and 156 grams of water are added at a temperature of 140° F. while the mixture is being continually stirred. Then, this mixture is passed through a colloid mill.

It has been found generally desirable in making these mixtures to include only the water insoluble material or wax, that is wax, oil or fat, the amido emulsifying agent and the positive charging material, such as aluminum acetate.

Generally, the amido aliphatic emulsifying agent may constitute between 1 to 20% with a preferred range of 5 to 15% of the water insoluble material. The positive charging material may be between one tenth the weight of the water insoluble material to equal the weight of such material.

As a general rule, this combination of water insoluble material, positive charging material and emulsifier are mixed with from ¼ to 3 times their weight of water, and this emulsion then is in condition to be shipped and stored.

In case of treating fabrics, furs, leather or paper with the emulsion, water may be added in sufficient amount until the water insoluble material constitutes less than 10% and preferably less than 5% of the final emulsion.

For example, from 1 to 10% of the concentrated emulsion may be added to a water bath or from 1 to 10 parts of the emulsion may be diluted with 100 parts of water.

Preferably, the final treating bath should contain between 0.1 and 5% of the water insoluble material, whether it be oil, fat or wax or combinations thereof.

When textiles such as fabrics and yarns, whether of artificial silk, natural silk, cellulose acetate, cotton, linen, wool, and so forth, are treated with this diluted emulsion, the fabrics are dipped in the bath and permitted to remain in the bath with or without agitation for a period of 5 minutes to 1 hour.

All of the emulsions according to the above description have positively charged disperse phases and when they are subjected to electrical currents the dispersed particles will tend to move to the cathode. These emulsions are all perfectly stable in the concentrated form in which they are prepared. The non-aqueous con-

amphoteric metallic salt, said bath having been prepared by the addition of a relatively large quantity of water to a relatively small quantity of a concentrated emulsion containing said water insoluble material, said agent and said polyvalent salt.

3. For use in a process of waterproofing negatively charged textile materials, a mixture of a relatively large quantity of water and a relatively small quantity of a concentrated emulsion containing an amide combination of a high molecular weight fatty acid containing from 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, a water soluble salt of a polyvalent metal having amphoteric properties and a water-insoluble material, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

4. For use in a process of waterproofing negatively charged textile materials, a bath containing a relatively large quantity of water and a relatively small quantity of a concentrated emulsion containing an amide combination of a high molecular weight fatty acid containing 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, a water soluble aluminum salt and a paraffin wax, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

5. For use in a process of waterproofing negatively charged textile materials, a bath containing a relatively large quantity of water and a relatively small quantity of a concentrated emulsion containing a paraffin wax, aluminum acetate and an amide combination of a high molecular weight fatty acid containing from 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, the disperse phase of said emulsion being positively charged and said textile absorbing the waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

6. For use in a process of waterproofing negatively charged textile materials, a bath containing a relatively large quantity of water and a relatively small quantity of a concentrated emulsion containing an amide combination of a high molecular weight of fatty acid containing from 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, aluminum acetate, petroleum jelly and a wax, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

7. For use in a process of waterproofing negatively charged textile materials, containing a relatively large quantity of water and a relatively small quantity of a concentrated emulsion containing aluminum sulphate, mineral oil, stearyl diethylene amine and stearic acid, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse

phase from said bath by reason of its opposite electric charge.

8. For use in a process of waterproofing negatively charged textile materials, a bath containing 20 to 100 parts of water and 1 part of a concentrated emulsion, said emulsion consisting of an amide combination of a high molecular weight fatty acid containing 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, a water soluble salt of a polyvalent metal having amphoteric properties and a water-insoluble material, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

9. For use in a process of waterproofing negatively charged textile materials, a bath containing 20 to 100 parts of water and 1 part of a concentrated emulsion, said emulsion consisting of a mixture of 100 parts of waxy material, 2 to 10 parts of amido stearyl diethylene amine, 150 to 400 parts of water and 10 to 50 parts of aluminum acetate, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

10. For use in a process of waterproofing negatively charged textile materials, a bath containing 20 to 100 parts of water and 1 part of a concentrated emulsion, said emulsion consisting of 6 parts of an amide combination of a high molecular weight fatty acid containing from 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms, 700 parts of water, 30 parts of aluminum formate and 88 parts of paraffin, the disperse phase of said emulsion being positively charged and said textile absorbing said oppositely charged waterproofing material in the disperse phase from said bath by reason of its opposite electric charge.

11. For use in a process of waterproofing textiles with diluted aqueous emulsions of water insoluble oil, waxy and fatty materials as the positively charged disperse phase, said textiles being negatively charged, a concentrated emulsion with a positively charged disperse phase having a solid content of at least 15%, said solid content including the positively charged disperse phase constituting at least 20 to 50% of the emulsion, a stearyl amide as an emulsifying agent in amount from 5 to 15% of the disperse phase and a water soluble salt of a polyvalent metal having amphoteric properties in an amount ranging from 25 to 75% of the disperse phase diluted with at least 20 to 100 parts by weight of water per part of emulsion to form the bath.

12. An acidic waterproofing emulsion containing a positively charged disperse phase including a wax, a water soluble amide combination of a high molecular weight fatty acid containing from 14 to 28 carbon atoms and a relatively low molecular weight aliphatic amine having between 1 and 6 carbon atoms and a positively charging water-soluble material.

13. An acidic waterproofing emulsion containing a positively charged disperse phase including a wax, a stearic acid alkylene amide and an aluminum salt.

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