

1

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DETERGENT COMPOSITIONS

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This invention relates to detergent compositions, suitable for use in the removal of oily materials from surfaces and particularly suitable for the removal of mineral oils or sludges from the hands or other parts of the body.

According to the invention, a detergent composition consisting of metal alkyl sulphates, of low solubility in water, is obtained by the sulphation of a distillation fraction of shale oil boiling in the approximate range 270° C.-360° C. to which is added a filler, such as kieselguhr.

Preferably the composition also comprises a binding agent, for example, a clay, such as bentonite. We have also found that the composition may advantageously have incorporated therein a proportion of refined mineral or fatty oil, for example, a spindle oil. This addition of oil is particularly advantageous where removal of tarry matter is involved, and it aids in producing a smooth paste.

It is preferred that the composition should contain not more than 30% by weight of the filler, 5-20% by weight of the binding agent and not more than 15% by weight of oil.

The metal alkyl sulphates for use in the detergent composition of the invention are preferably prepared as follows: A shale oil distillate boiling in the approximate range 270° C.-360° C. and preferably derived from Scottish shale, is given a preliminary treatment with dilute sulphuric acid and with aqueous soda solution for the removal of nitrogen bases and phenols, and thereafter contacted with strong sulphuric acid.

The sulphuric acid used is preferably 96-100% strength and in an amount constituting from 10% to 20%, and preferably about 12-13% by volume of the oil feed. The sulphuric acid is intimately contacted with the oil while maintaining the temperature of the reactants below 20° C. and preferably at about 5° C.-20° C.

The reaction mixture containing alkyl hydrogen sulphates and dialkyl sulphates is separated into acid and oil layers in any convenient manner, as by gravity or by centrifuging.

The alkyl hydrogen sulphates and dialkyl sulphates are converted to metal alkyl sulphates by treatment with a metal hydroxide or carbonate, preferably sodium carbonate. This may be accomplished by adding the acid oil layer, after removal of all traces of acid sludge, to an excess of sodium carbonate in aqueous solution (for example a 16% solution). It is also an advantage to stir the resulting emulsion under a reflux condenser to avoid losses of the more volatile portions of the oils, for an extended period of time, for example 5 hours at 95° C. The surface active agents may be recovered from this emulsion by adding an aqueous alcoholic solution with stirring. It has been found that a solution containing 10-30% by weight of iso-propyl alcohol in water gives satisfactory results, but that other alcohols, for example, methanol, may be used. The quantity of alcoholic solution added should be sufficient to give two clear layers

2

in 15 to 30 minutes. The amount of oil retained in the lower layer after breaking the emulsion is considerably influenced by the proportions of alcohol and water relative to detergent, and by the composition of the alcohol.

A treatment of the emulsion with an equal volume of aqueous alcoholic solution containing between 15 and 25 parts by weight of isopropyl alcohol suffices to break the emulsion in a reasonable time. The upper oily layer after extraction, has been found to contain alcohols of the order of 1-5% by weight, which may be recovered by distillation. The alcohol free oil may be redistilled after suitable refining treatment, such for example as distillation with steam and lime, or catalytic desulphurisation. Alcohol free unreacted oil may alternatively be blended back with the feed to the process, as it has been found to contain appreciable quantities of sulphatable olefins.

It has been found that the proportion of residual oil in the alcoholic detergent solution after emulsion break is about 12% by weight of the sodium alkyl sulphate solution after evaporation of the alcohol.

The separated alcoholic detergent solution is evaporated to a thin paste or emulsion containing about 12½ by weight of sodium alkyl sulphates (calculated as  $C_{20}H_{41}SO_3ONa$ ) and admixed with filler, and if desired a binding agent, mineral oil, and other auxiliary ingredients, customarily employed in the art, such as antiseptic, deodorant and colouring matter.

The invention is illustrated but in no way limited by the following examples:

Example 1

A sodium alkyl sulphate aqueous paste prepared from a Scottish shale oil distillate fraction boiling in the range 270-360° C., as described above, and containing approximately 12½% by weight of sodium alkyl sulphates, was admixed with bentonite and kieselguhr in the following proportions by weight:

|                                                | Per cent |
|------------------------------------------------|----------|
| Sodium alkyl sulphate paste                    | 68       |
| Bentonite                                      | 8        |
| Kieselguhr (7-9 microns average particle size) | 24       |

Example 2

A sodium alkyl sulphate aqueous paste as in Example 1, and containing 12½% by weight of sodium alkyl sulphates, was admixed with bentonite, kieselguhr, refined mineral oil and water in the following proportions by weight:

|                                                 | Per cent |
|-------------------------------------------------|----------|
| Sodium alkyl sulphate paste                     | 40       |
| Bentonite                                       | 15       |
| Kieselguhr (3-10 microns average particle size) | 26       |
| Refined mineral oil                             | 9        |
| Water                                           | 10       |

The resulting paste in each case was a good detergent for the removal of oily contaminants, and was found particularly suitable for cleansing the hands of mineral lubricating oils and sludges. The cleansing action for mineral oil was not reduced by use with hard or salt water. The detergent paste was equally effective if the dirt contained weak acids or metallic salts (such as zinc chloride) which normally render ordinary soap-bearing hand cleansers useless. Owing to the fine state of division of the mineral matter, the paste could be used in the cleaning of painted surfaces without the fear of scratching.

I claim:

1. A process for the production of a detergent composition suitable for the removal of oily materials from surfaces and particularly for the removal of mineral

3

oil and sludges from the hands and other parts of the body, which comprises sulphating a shale oil distillation fraction boiling within the approximate range 270° C.-360° C. with strong sulphuric acid in an amount constituting 10-20% by volume of said distillation fraction, neutralising the product with an alkali metal base to produce alkali metal alkyl sulphates of low solubility in water and admixing the said alkali metal alkyl sulphates with kieselguhr as a filler.

2. A process according to claim 1 in which the sulphuric acid is of 96-100% concentration.

3. A process according to claim 1 in which the sulphuric acid is employed in an amount constituting 12-13% by volume of the distillation fraction reacted therewith.

4. A process according to claim 1 in which the distillation fraction is reacted with sulphuric acid at a temperature below 20° C.

5. A process according to claim 1 in which the distillation fraction is reacted with sulphuric acid at a temperature in the range 5-20° C.

6. A process according to claim 1 in which the alkali metal base is sodium carbonate.

7. A process according to claim 1 in which the product obtained by reacting the distillation fraction with sulphuric acid is stirred with an aqueous solution of an alkali metal base for a prolonged period of time.

8. A process according to claim 1 in which the filler is employed in a finely divided form.

9. A process for the production of a detergent composition suitable for the removal of oily materials from surfaces and particularly for the removal of mineral oil and sludges from the hands and other parts of the body, which comprises sulphating a shale oil distillation fraction boiling within the approximate range 270° C.-360° C. by reacting said fraction with strong sulphuric acid in an amount constituting 10-20% by volume of said distillation fraction while maintaining the temperature of the reactants below 20° C.; separating the reaction mixture into acid and oil layers, the acid layer comprising some oil; converting the alkyl hydrogen sulphates and dialkyl sulphates in the acid-oil layer into alkali metal alkyl sulphates by treating said acid-oil layer with an aqueous solution of an alkali metal base whereby an emulsion is formed; breaking the emulsion formed into an oil-alcohol fraction and a solution of detergent and alcohol by agitating the emulsion with an aqueous alcoholic solution; separating the alcoholic detergent solution from said oil-alcohol fraction; evaporating said solution to a thin paste; and, mixing said paste with kieselguhr as a filler to form the desired detergent composition.

10. A detergent composition suitable for the removal of oily materials from surfaces and particularly suitable for the removal of mineral oils and sludges from the

4

hands and other parts of the body, comprising alkali metal alkyl sulphates of low solubility in water, obtained by the sulphation of a distillation fraction of shale oil boiling in the approximate range 270° C.-360° C. using 10-20% of the volume of said fraction of 96-100% concentration by weight sulphuric acid at a temperature in the range 5-20° C. and neutralizing the product with sodium carbonate, and, in admixture with said sulphate, kieselguhr as a filler.

11. A process according to claim 1 wherein the alkali metal alkyl sulphates in the form of an aqueous paste containing about 12.5% by weight of the alkali metal alkyl sulphates are admixed with the filler.

12. A process according to claim 1 in which the kieselguhr filler constitutes not more than 30% by weight of the alkali metal alkyl sulphates.

13. A process according to claim 1 in which bentonite is added as a binding agent in a proportion constituting 5 to 20% by weight of the alkali metal alkyl sulphate.

14. A process according to claim 1 in which mineral oil is incorporated with said alkali metal alkyl sulphates and filler.

15. A process as defined in claim 14 in which the mineral oil is employed in a proportion constituting not more than 15% by weight of the alkali metal alkyl sulphates.

16. A process as defined in claim 14 wherein the mineral oil is spindle oil.

17. A detergent composition prepared in accordance with the process of claim 1.

18. A detergent composition prepared in accordance with the process of claim 11.

19. A detergent composition prepared in accordance with claim 12.

20. A detergent composition prepared in accordance with claim 13.

21. A detergent composition prepared in accordance with claim 14.

22. A detergent composition prepared in accordance with claim 15.

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