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EMULSION CONCENTRATES

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This invention relates to the preparation of stable emulsion-soluble soaps and to preparation of stable emulsion concentrates from such soaps.

"Emulsion-soluble" soaps consist of a fatty acid soap in a water-oil-soap emulsion. They differ from conventional soluble oils in that they contain no coupling agents. One of the principal uses of emulsion-soluble soaps is for form-parting compositions for concrete. These soaps are applied in relatively dilute water emulsions to the faces of the forms before the concrete is poured and prevent the dried concrete from sticking to the face of the form, thereby assuring a smooth surface on the concrete structure.

It is common practice to prepare emulsion-soluble soaps in an emulsion-concentrate form by adding oil and diluting with water. Such concentrates are then shipped to the desired location where they are diluted to the desired dilution for use.

Difficulty has been experienced in preparing satisfactory emulsion concentrates because of instability and breakdown of the concentrate, apparently caused by entrainment of air in the mass during the soap-forming stage.

We have discovered that emulsion concentrates of unusual stability can be prepared if the acidity of the mass during the soap-forming stage is maintained at not less than about 0.1% by weight oleic acid equivalent. We have further found that the stability of emulsion concentrate is enhanced if all the oil content of the concentrate is added to the soap prior to dilution with water to the desired concentration. It is further important in preparing the concentrate that the acidity of the final concentrate be regulated to about 0.45 to 0.65% by weight oleic acid equivalent.

It is an object of our invention to provide a method for preparing emulsion-soluble soaps capable of forming stable emulsions. It is a further object of our invention to provide a method for preparing stable oil emulsions from emulsion-forming soaps. Other objects of the invention will be revealed from the following description.

In accordance with our invention, a mineral oil to be emulsified is mixed with the required amount of fatty acids so that upon reaction with an alkali a soap is formed having an acidity of not less than 0.01% by weight oleic acid equivalent, and preferably between 0.01 and 0.1%. The oil may be a mineral lubricating oil fraction, preferably having a viscosity within the range of 145 to 165 SUS at 100° F., or the oil can be an acidic extract resulting from the solvent extraction of mineral lubricating oil fractions with solvents, such as phenol, furfural and nitrobenzene, to improve the viscosity index of the mineral oil. An example of a suitable extract is a phenol extract having the following characteristics:

Acid number, approximately 4.1 to 4.6, usually 4.13
API gravity, 16 to 17°
Flash, 365 to 380° F.

Fire, 420 to 435° F.
Viscosity/100° F., 160 to 180 SUS
Viscosity/210° F., 39 to 41 SUS
V.I., −20 to −27

Among the fatty acids which are useful in preparing the soap are the usual soap-forming fatty acids containing 16 to 24 carbon atoms per molecule, including but not limited to oleic, palmitic, stearic and fish acids.

The oil and fatty acid are thoroughly mixed at ambient temperature, that is, existing room or atmospheric temperature. After the components are thoroughly mixed, there is added thereto an alkaline reagent in an amount sufficient to react with the fatty acids to form the soap. The alkaline reagent may be a basic alkali or alkaline earth metal compound, such as calcium hydroxide, sodium or potassium hydroxide and carbonates. We prefer, however, to use either caustic soda or caustic potash.

The amount of alkaline reagent added to the mixture in the soap-forming stage is critical. Sufficient must be added in order to react with the fatty acids and form a gel-like soap structure, but the quantity of alkaline reagent must be regulated so that the mixture remains at all times on the acid side, and at the completion of the soap-forming stage the acidity of the mixture should not be less than 0.01% by weight oleic acid equivalent. The upper limit of acidity of the soap mixture is fixed only by the acidity of the final emulsion concentrate, which will hereafter be discussed. We prefer to regulate the amount of alkaline agent so that the final soap composition has an acidity of between 0.01 and 1% by weight oleic acid equivalent.

The soap-forming stage is carried out at a temperature of approximately 70° to 100° F. Lower temperatures may be used but they increase the time of the soap-forming stage. Higher temperatures should be avoided since the soap becomes dehydrated and deleteriously affects the gel structure of the soap.

If the acidity is carefully regulated, as pointed out, the resulting soap concentrate will be in a paste or gel form without any air occlusion. The soap concentrate is cooled to ambient temperature and the remainder of the mineral oil is added while adjusting the acidity by addition, if necessary, of free acid so that the acidity of the final emulsion concentrate is between about 0.45 and 0.65% by weight oleic acid equivalent. In the event that a lubricating oil is used, it will be necessary to add free oleic acid or other fatty acid to bring the acidity of the mixture up to the desired level. Where solvent extract is used, the acidity of the mixture can be regulated by adding the proper amount of extract.

After the oil or extract has been added to the soap and thoroughly mixed, sufficient water is then added and mixed with the composition to achieve the desired soap-oil-water concentrated emulsion composition. The product is then ready for shipment.

In preparing the initial soap concentrate, we prefer to mix with the soap-forming ingredients sufficient of the oil so that the resulting soap concentrate will contain about 12–20% by weight of soap. Ordinarily, this will require about 40–60% by volume of the total oil used in preparing the emulsion concentrate. The final emulsion concentrate may contain about 6–10% by weight of soap but any desired concentration which is found economical can be used. As previously pointed out, it is essential that the reaction mixture be maintained slightly acidic during and at the end of the soap-forming stage, and preferably that sufficient acid be incorporated in the soap-forming mixture to achieve a free acidity of about 0.01 to 0.1% by weight, calculated as oleic acid, in the resulting soap concentrate. This range has been found to be particularly suitable when using an acidic oil such as solvent extract as the cutback

oil in making the finished emulsion concentrate. Because of the relatively high acidity of solvent extract, if the soap concentrate has an acidity between 0.01 and 0.1% weight oleic acid equivalent, the final concentrate can be adjusted to the desired acidity of 0.45 to 0.65% oleic acid equivalent by the addition of the proper amount of the extract oil. On the other hand, if neutral oil is used as the cutback oil, it may be desirable to prepare the soap concentrate with an acidity as high as 0.7% by weight calculated as oleic acid so that when the soap is cut back with oil and water added to the desired concentration the resulting concentrate will have an acidity equivalent to 0.45 to 0.65% by weight of oleic acid. It will be apparent, therefore, that the upper limit of acidity of the soap concentrate is not critical except insofar as it should be regulated in accordance with the acidity of the cut back oil to be used so that when the desired amount of cut back oil is added the finished concentrate will have an acidity of between 0.45 and 0.65% oleic acid equivalent.

Not only is it important to carefully control the acidity in the soap-forming stage and in the final emulsion concentrate, but we have found that the order in which the cut-back oil and water are added to the soap concentrate in order to make the finished emulsion concentrate affects the stability of the resulting emulsion concentrate. If the cut-back oil is all added to the soap concentrate prior to the addition of the water, highly stable concentrates result which do not cream upon further dilution with water. On the other hand, if the dilution water is added prior to the addition of the cut-back oil, or if the cut-back oil and water are alternately added until the desired concentration is achieved, the concentrates have a tendency to cream and separate to varying degrees upon dilution with water.

The following examples are illustrative of the invention and of the results that can be achieved by adhering to and departing from the invention.

Example I

A soap concentrate having a free alkalinity of 0.05% by weight, calculated as potassium hydroxide, was prepared from the following starting materials:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	42.17
Oleic acid	12.75
Water	42.34
Potassium hydroxide (100% assay)	2.74

The mixture was stirred for 4 hours at a temperature of 80° F. but did not form a gel paste structure. It remained liquid and entrained a very large amount of air, almost doubling in volume as a result of expansion by air. The soap was not processed further.

Example II

A neutral soap concentrate prepared in the same manner as disclosed for Example I was prepared from the following ingredients:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	42.0
Oleic acid	13.12
Water	42.15
Potassium hydroxide (100% assay)	2.73

The resulting soap concentrate was a very soft paste and some air entrainment was noted. An emulsion concentrate was formed from this soap by cutting back with additional phenol extract and diluting with water so that the resulting concentrate had an acidity of 0.4% by weight of oleic acid equivalent. This concentrate had only borderline stability. The final composition of the concentrate in terms of ingredients used in its preparation was:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	49.03
Oleic acid	7.66
Water	41.72
Potassium hydroxide (100% assay)	1.59

Example III

A soap concentrate having an acidity of 0.05% by weight oleic acid equivalent was prepared in the manner disclosed in Example I from the following ingredients:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	41.85
Oleic acid	12.9
Water	42.02
Potassium hydroxide (85% assay)	3.23

The soap concentrate formed a substantially air-free gel with the consistency of heavy paste within 5 minutes, in contrast to Example I, where even extensive agitation did not form the gel-paste structure. It was diluted with a small amount of water to reduce its firmness, and after all the cut-back oil was mixed with the soap, the requisite amount of dilution water to give the finished concentrated emulsion was added and mixed. The proportions of the ingredients in the finished emulsion were:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	48.93
Oleic acid	7.54
Water	41.65
Potassium hydroxide (85% assay)	1.88

The finished concentrated emulsion had a free acid content of about 0.5% by weight oleic acid equivalent. It formed stable emulsions when diluted and mixed with water in ratios of 6 and 15 parts of water per part of concentrated emulsion.

Example IV

A soap concentrate prepared in the manner set forth in Example I but having an acidity of about 0.75% by weight oleic acid equivalent was prepared from the following constituents:

Component:	Wt. percent
Phenol extract (4 Neut. No.) from 85 SUS at 100° F. neutral	38.3
Oleic acid	12.55
Water	46.2
Potassium hydroxide (85% assay)	2.95

The resulting soap was a paste-like gel substantially air-free. It was cut back with additional extract oil and emulsified with water to form a concentrated emulsion having an acidity of about 0.9% by weight oleic acid equivalent. The concentrated emulsion set into a gel within about one hour, thereby affecting its usefulness and ease of handling. Dilute emulsions were prepared from the concentrate but were found to be unstable.

Example V

A concentrated emulsion was prepared in the same manner as in Example III except that instead of adding the cut-back oil before the addition of the dilution water, the water was first added to the soap concentrate and the required amount of extract oil then added. This concentrated emulsion exhibited an undesirable amount of creaming after standing quiescent for twenty-four hours, thus showing the superiority of the product prepared by adding the cut-back oil before adding the diluent water.

Example VI

A concentrated emulsion was made in the same manner as in Example III except that instead of adding the cut-back oil prior to adding the diluent water, the water and oil were divided into four equal portions and water and oil alternately added to the soap concentrate with agitation. This concentrate exhibited an undesirable amount of creaming equivalent to the amount observed in Example V after standing for twenty-four hours.

Example VII

A concentrated emulsion was prepared in the manner disclosed in Example III except that all the dilution or cut-back oil was added to the soap concentrate prior to adding the dilution water. The resulting concentrated emulsion was milky-white and exhibited only trace creaming upon standing for twenty-four hours. Upon dilution with further quantities of water it formed stable emulsions.

It will be seen, therefore, that by carrying out the steps in accordance with this invention an emulsion concentrate is prepared which itself is stable and which forms highly stable emulsions upon dilution with further quantities of water.

We claim as our invention:

1. The method of preparing a stable emulsion-concentrate capable of forming stable emulsions upon dilution with about 6–15 parts of water per part of concentrate comprising mixing an acidic extract obtained by the solvent extraction of a mineral lubricating oil fraction with a solvent selected from the group consisting of phenol, furfural, and nitrobenzene, with a soap-forming fatty acid in an amount sufficient to form a gel upon neutralization with aqueous alkali-metal hydroxide solution, neutralizing the extract-acid mixture with sufficient aqueous alkali-metal hydroxide solution of sufficient concentration to form a gel with a free acidity of not less than about 0.01 percent by weight oleic acid equivalent, adding additional acidic extract and water to produce an emulsion-concentrate containing not more than about 10 percent by weight of fatty acid soap and not more than about 49 percent by weight of extract, and having an acidity of 0.45 to 0.65 percent by weight oleic acid equivalent.

2. A method according to claim 1 in which the quantity of extract to which the fatty acids are added is about 40 to 60 percent of the total acidic extract used to form the emulsion concentrate.

3. A method in accordance with claim 2 in which the neutralization step is carried out at a temperature not higher than about 100° F.

4. The method in accordance with claim 3 in which the extract is an acidic extract from the phenol extraction of neutral oil.

5. The method in accordance with claim 3 in which the soap-forming acid is oleic acid.

6. The method in accordance with claim 3 in which the gel is cut back with the desired amount of extract before adding water to form the emulsion concentrate.

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