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METAL WORKING LUBRICANT COMPOSITION

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ABSTRACT OF THE DISCLOSURE

Soluble oil compositions and aqueous emulsions thereof having improved extreme pressure properties and being useful in metal working operations are disclosed. The soluble oil compositions comprise lubricating oil, non-cationic emulsifier, overbased alkali metal or alkaline earth metal sulfonate, chlorinated hydrocarbonaceous component, a coupling agent and a stabilizing amount of water. The aqueous emulsions are formed by combining a major amount of water with a minor amount of the composition described above.

This invention relates to soluble oil metal working compositions. More particularly, this invention relates to soluble oil metal working lubricating compositions which have improved extreme pressure properties.

It is customary to use a coolant and/or lubricant composition while working metal. Among the metal working operations in which such a composition is normally used are cutting, grinding, drilling, boring, broaching, milling, metal shaping, bending and the like. Among the compositions used for these metal working operations are soluble oils and aqueous emulsions thereof.

Under severe conditions, the metal working composition may include an extreme pressure agent. One such agent which has been used in the soluble oil metal working compositions of the prior art is the chlorinated paraffinic type, such as chlorinated waxes and the like. However, these chlorinated components do not always give satisfactory extreme pressure properties to the metal working compositions. For example, it has been found that when a soluble oil composition containing chlorinated paraffin as the extreme pressure agent is used in an operation involving metal bending or shaping of aluminum coated steel automobile exhaust and tail pipes, an usatisfactorily large portion of the resulting pipes must be rejected because of galling and smearing of the aluminum coating and crushing of the pipe.

Therefore, one of the objects of the present invention is to provide soluble oil metal working compositions with improved extreme pressure properties. Another object of the present invention is to provide aqueous emulsions of soluble oil metal working compositions with improved extreme pressure properties. Other objects and advantages of the present invention will become apparent hereinafter.

It has now been found that the incorporation of the combination of at least one chlorinated hydrocarbonaceous component and at least one alkali metal or alkaline earth metal overbased sulfonate into a soluble oil metal working composition provides the composition with unexpectedly improved extreme pressure properties. Therefore, in one aspect, the present invention is a soluble oil composition which comprises from about 10% to about 90% by weight of a oil of lubricating viscosity; at least one non-cationic emulsifier in an amount sufficient to render

the composition emulsifiable with water; from about 1% to about 30%, preferably from about 3% to about 20%, by weight of at least one overbased sulfonate selected from the group consisting of alkali metal sulfonate, alkaline earth metal sulfonate and mixtures thereof, overbased as the corresponding carbonate and mixtures thereof; from about 1% to about 40%, preferably from about 5% to about 40%, more preferably from about 15% to about 40%, by weight of at least one chlorinated hydrocarbonaceous component which comprises at least about 5%, preferably at least about 20%, by weight of chlorine, said overbased sulfonate and chlorinated hydrocarbonaceous component each being present in amounts sufficient to improve the extreme pressure properties of said composition; at least one coupling agent present in an amount sufficient to improve the emulsibility of the components of said composition; and a stabilizing amount, preferably from about 0.1% to about 5%, and more preferably from about 0.5% to about 3.5% by weight, of water. Additional components may be contained in these soluble oil compositions to improve various properties. It is, of course, understood that more than one oil, emulsifier, overbased sulfonate, chlorinated hydrocarbonaceous component and coupling agent can be used in combination in a single soluble oil and such a composition is within the scope of the present invention. The proportions given herein apply to the total amounts of oil, emulsifier, etc., regardless of how many of each are used. Various components of the soluble oil compositions defined above, for example, the emulsifier, overbased sulfonate, chlorinated hydrocarbonaceous component and coupling agent, may be prepared and maintained in an oil or other solvent carrier. However, the composition proportions given previously are on a solvent-free basis. The soluble-oil compositions of the present invention may be prepared by blending the various components together at conditions, e.g., slightly elevated temperature, to insure product uniformity.

The compositions of the present invention may be used as the soluble oil compositions defined above, although it is preferred to use the present compositions in the form of aqueous emulsions. Therefore, in another aspect, the present invention is an emulsion which comprises (A) water in an amount from about 40% to about 99.5%, preferably from about 50% to about 99.5%, more preferably from about 50% to about 98%, and still more preferably from about 60% to about 95%, by volume of the total emulsion; and (B) a soluble-oil composition of the present invention as previously defined in an amount from about 0.5% to about 60%, preferably from about 0.5% to about 50%, more preferably from about 2% to about 50%, and still more preferably from about 5% to about 40%, by volume of the total emulsion. The aqueous emulsions defined above can be prepared by bringing together water and the soluble oil compositions of the present invention.

The compositions of the present invention, either as the soluble oil compositions or the aqueous emulsions, can be used by maintaining (or causing to be maintained) a lubricating amount of the composition on the metal surface being worked. These compositions can be used in metal working operations such as cutting, grinding, boring, broaching, milling, metal shaping, bending and the like. The compositions of the present invention are of particular usefulness when maintained on the surface of alu-

minum or aluminum coated metal, or example, automobile tail pipes, being worked, for example, bended.

Typical examples of oils suitable for use in the present invention are lubricating oils. Although mineral oils are preferred, synthetic oils may be used. Suitable oils include petroleum mineral oils, such as Refined Coastal Oils and refined Mid-Continent Oils. The petroleum oils may be refined by acid treatment, solvent extraction, hydrogenation and other procedures. Although oils of widely varying viscosities can be used in the products of the present invention, it is preferred to use an oil with a viscosity from about 30 to about 1200 SUS at 100° F., more preferably about 30 to about 150 SUS at 100° F.

The non-cationic emulsifiers suitable for use in the present invention are of the conventional type. The total amount of non-cationic emulsifier in the soluble oil compositions of the present invention and in component (B) of the aqueous emulsions is that amount required to render the composition or component emulsifiable with water. The concentration of emulsifier is preferred to be from about 1% to about 40%, more preferably from about 5% to about 30%, by weight of the total composition or component.

Among the suitable emulsifiers are those of the conventional type which include less than 200 carbon atoms per molecule. Included among these emulsifiers are the alkali metal petroleum sulfonates and sulfates; the alkali metal, alkaline earth metal and amine soaps of fatty, naphthenic and resin acids and the glycerol mono esters, such as glycerol monostearate. Also included among the non-cationic emulsifiers suitable for use in the present invention are the following compounds: glycerides of fat forming fatty acids, such as lactylated mono- and diglycerides of fat-forming fatty acids; alkyl aryl polyether alcohols; sorbitan and polyoxyalkylene sorbitan fatty and resin acid esters, such as sorbitan monopalmitate, sorbitan monolaurate, sorbitan trioleate, polyoxyethylene sorbitan monostearate and monooleate, polyoxyethylene sorbitan tristearate and trioleate; polyoxyalkylene fatty, naphthenic and resin acids, alcohols, ethers and esters, such as polyoxyethylene fatty acid and alcohol, and polyoxyethylene lauryl ether and polyoxyethylene monooleate; polyoxyalkylene phenols, such as polyoxyethylene alkyl phenol; and mixtures thereof.

The preferred emulsifiers for use in the present invention are selected from the group consisting of the alkali metal, more preferably sodium, sulfonates and mixtures thereof derived from petroleum sources (i.e., petroleum sulfonates) which contain from about 12 to about 200, more preferably from about 12 to about 50, carbon atoms per molecule.

The overbased alkali and alkaline earth metal (i.e., sodium, potassium, lithium, magnesium, strontium, barium and calcium) sulfonates useful in the present invention comprise: (1) alkali or alkaline earth metal salts of sulfonic acids; and (2) dispersed particles of the corresponding carbonate. These overbased materials may contain from about 50 mole percent to about 900 mole perment, preferably from about 100 mole percent to about 800 mole percent and more preferably from about 200 mole percent to about 750 mole percent, of basicity in excess of that needed to neutralize the precursor sulfonic acid.

The overbased salts derived from sulfonic acids which include preferably from about 12 to about 200 and more preferably from about 12 to about 50 carbon atoms per molecule are of particular usefulness in the present invention. Among the suitable sulfonic acids are the following: mahogany sulfonic acids, petroleum sulfonic acids, mono- and polywax substituted naphthalene sulfonic acids, phenol sulfonic acids, diphenyl ether sulfonic acids, diphenyl ether disulfonic acids, naphthalene disulfide sulfonic acids, naphthalene disulfide disulfonic acids, diphenyl amine disulfonic acids, cetyl-phenol mono-sulfide sulfonic acids, cetoxy caprylbenzene sulfonic acids, di-cetyl thianthrene sulfonic acids, such as cetyl chloro-benzene sulfonic acids, cetyl-phenol sulfonic acids, cetyl-phenol disulfide sulfonic acids, cetyl-phenol mono-sulfide sulfonic acids, cetoxy capryl-benzene sulfonic acids, di-cetyl thiathrene sulfonic acids, di-lauryl beta-naphthaol sulfonic acids, and di-capryl nitronaphthalene sulfonic acids, aliphatic sulfonic acids such as paraffin wax sulfonic acids, unsaturated paraffin wax sulfonic acids, hydroxy substituted paraffin wax sulfonic acids, tetraisobutylene sulfonic acids, tetra-amylene sulfonic acids, chloro-substituted paraffin wax sulfonic acids, nitroso paraffin was sulfonic acids, etc., cycloaliphatic sulfonic acids, such as petroleum naphthene sulfonic acids, cetyl-cyclopentyl sulfonic acids, lauryl-cyclohexyl sulfonic acids, bis - (diisobutyl)-cyclohexyl sulfonic acids, mono- and poly-wax substituted cyclohexyl sulfonic acids, etc.

With respect to the sulfonic acids, it is intended herein to employ the term "petroleum sulfonic acids" to cover all sulfonic acids which are derived at least in part from petroleum products. Additional examples of sulfonic acids and/or the alkali and alkaline earth metal salts thereof which can be employed as starting materials are disclosed in the following U.S. Pats.: 2,174,110; 2,174,560; 2,174,508; 2,193,824; 2,197,800; 2,202,791; 2,212,786; 2,213,360; 2,228,598; 2,233,676; 2,239,974; 2,263,312; 2,276,090; 2,276,097; 2,315,514; 2,319,121; 2,321,022; 2,333,568; 2,333,788; 2,335,259; 2,337,552, 2,346,568; 2,366,027, 2,374,193 and 2,383,319.

Because of their availability and effectiveness in the compositions of the present invention, the preferred overbased sulfonates are the alkaline earth metal sulfonates and mixtures thereof overbased as the corresponding carbonate, and mixtures thereof, and, in particular, the calcium sulfonates overbased as calcium carbonate. Calcium sulfonates overbased as calcium carbonate can be obtained by passing carbon dioxide through a mixture of neutral calcium sulfonates, mineral oil, lime and water. The formation of the overbased sulfonates can be aided through the use of "promoters," such as phenols, aromatic amines, sucrose and lower aliphatic alcohols. Many patents have been issued which disclose processes for making calcium carbonate overbased sulfonates. Among these are U.S. Pats. 2,865,956 and 2,956,018. The other overbased sulfonates useful in the present invention can be prepared by methods analogous to that given above for the overbased calcium sulfonates.

The overbased sulfonates useful in the present invention may either be the clear type, i.e., the overbased sulfonate when dispersed in an oil carrier is clear to the naked eye, or the cloudy type, i.e., the overbased sulfonate-oil mixture has a cloudy or hazy appearance. Clear type overbased sulfonates can be characterized in that substantially all the dispersed carbonate particles are less than about 60 A. in size when the overbased sulfonate is dispersed in an oil carrier. The cloudy type overbased sulfonates can be characterized in that a major portion of the dispersed carbonate particles have a maximum dimension greater than about 60 A., and preferably greater than about 100 A., in size when the overbased sulfonate is dispersed in an oil carrier. (The term "maximum dimension" as used herein refers to the largest straight line dimension of the particle, be it length, width, diameter or thickness. The term does not refer to the circumference or perimeter of a spherical, cylindrical or multi-sided particle.) Of course, the carbonate particles should not be so large as to interfere with the quality of the soluble oil compositions into which they are to be incorporated. Because of this practical consideration, it is preferred that the maximum dimension of the carbonate particles of the cloudy type overbased sulfonate be limited to about 3 microns, more preferably to about 1 micron. Therefore, the preferred maximum dimension range for the major portion of carbonate particles of the cloudy type overbased sulfonate is from about 100 A. to about 3 microns, more preferably from about 100 A. to about 1 micron when the overbased sul-

fonate is dispersed in an oil carrier. Specific examples of the cloudy type calcium sulfonates preferred for use in the present invention are products which include a major portion of the calcium carbonate particles with a maximum dimension in the range from about 700 A. to about 900 A. (when the overbased sulfonate is dispersed in an oil carrier).

Although both the clear and cloudy type overbased sulfonates are suitable for use in the present invention as extreme pressure agents in combination with the chlorinated hydrocarbonaceous component, it is preferred to use the cloudy type materials. This preference is because of the improved corrosion properties of the soluble oil compositions (and aqueous emulsions thereof) containing the cloudy type overbased sulfonates. Corrosion inhibition is always an advantage in metal working operations and may become critical in certain applications.

The chlorinated hydrocarbonaceous component suitable for use in the present invention may vary widely in structure and composition provided that the chlorine content of this component is at least about 5%, perferably at least about 20%, by weight. Included among the suitable chlorinated components are chlorinated paraffin, (including paraffin wax, kerosene and the like) chlorinated olefin and polyolefin, chlorinated aromatics including chlorinated naphthenes, chlorinated esters of fatty, naphthenic and resin acids as well as chlorinated microcrystalline wax and the like and mixtures thereof which contain less than about 70 carbon atoms per molecule. Of course, more than one chlorinated component may be used in a single composition, and such a composition is within the scope of the present invention. It is preferred to use chlorinated paraffin, including paraffin wax and kerosene, chlorinated olefin, chlorinated polyolefin, and the like and mixtures thereof which contain less than about 70, preferably from about 20 to about 40 carbon atoms per molecule. Still more preferably, chlorinated paraffin containing from about 20 to about 40 carbon atoms per molecule can be used. The chlorinated components useful in the present invention may be prepared in any conventional manner, such as, for example, contacting molecular chlorine with the hydrocarbonaceous material to be chlorinated. By "hydrocarbonaceous material" is meant those materials (e.g., paraffins, waxes, olefins, polyolefins, esters and the like) which are composed mainly of hydrogen and carbon, and include such materials which contain, in addition, minor amounts of substituents, such as oxygen, sulfur, nitrogen, etc., which do not substantially affect their hydrocarbon character.

To improve the emulsifiability of the various components, at least one coupling agent is added to the composition of the present invention. The use of these agents in soluble oil compositions and aqueous emulsions is conventional and well known to the art. These agents function to couple the aqueous portion of the neat soluble oil composition to the continuous oil phase so as to give a uniform product and also to couple components (A) and (B) in the aqueous emulsions of the present invention. Among the well-known coupling agents usable in the compositions of the present invention are the aliphatic ether-alcohol compounds containing from about 1 to about 30 carbon atoms, for instance, diethylene glycol; ethyl Cellosolve, Carbitol and butyl Cellosolve; the polyether glycols with terminal ether groups; the polyoxyalkylene glycols, as well as ethylene glycol, propylene glycol and the like; and the alcohols, such as ethyl alcohol, propyl alcohol, butyl alcohol and the like. The preferred coupling agent for use in the present invention is selected from the group consisting of ethyl Cellosolve, Carbitol and butyl Cellosolve. Preferably the coupling agent comprises from about 0.5% to about 10%, more preferably from about 3% to about 10%, by weight of the soluble oil compositions and component (B) of the aqueous emulsions of the present invention.

The use of a small, stabilizing amount of water in neat soluble oil compositions is conventional and well known in the art. This water acts to stabilize or maintain the soluble oil composition as a dispersion or suspension suitable for shipping and handling. The amount of stabilizing water should preferably be from about 0.1% to about 5.0%, more preferably from about 0.5% to about 3.5%, by weight of the total soluble-oil composition. This stabilizing water should be distinguished from the relatively large amount of water used to form the aqueous emulsions of the present invention.

Other ingredients, such as bactericides, corrosion inhibitors, rust inhibitors, etc. may be present in the soluble oil compositions, for example, in amounts of about 1% to about 10% by weight of the total composition. When using a bactericide, the preferred agent is tetrachlorophenol; however, various other compatible bactericides can be used.

The effectiveness of the soluble oil lubricating compositions of the present invention can be tested in the following manner which is a modified version of the Socony Mobil Tapping Method, shown on page 61 of the Lubrication Engineer's Manual, Applied Research Laboratory, United States Steel, Monroeville, Pa. (May 1966). A drill press tapper is equipped to give a measurement of the amount of torque required to work a tap down an accurately sized hole. While this work is occurring, both the tap and the hole are lubricated by the oil composition (either neat or emulsified) being tested. The lower the amount of torque required, the more effective the composition is as a lubricant.

In order to insure adequate lubrication of both the tap and the hole, a lubricant ring oiler filled with the lubricant to be tested is fitted atop the hole. The tap passes through the lubricant bath and thus picks up a film of lubricant before entering the hole.

The following test conditions, i.e., size specifications, were used:

Tap size—½ in. x 13 National Course Thread
Tap style—Plug, standard—4 cams per thread
Tap hole size—15/32 in. for 60% thread
Tap r.p.m.—Approximately 162

The following examples illustrate more clearly the compositions of the present invention. However, these illustrations are not to be interpreted as specific limitations on the invention.

EXAMPLE 1

In order to establish a baseline for further testing, the following composition was prepared by blending the various components together at a slightly elevated temperature, i.e., about 100° F. to about 130° F., to insure complete mixing:

Components:	Wt. percent
Mineral oil	[1] 44.0
Sodium sulfonate	[2] 20.0
Chlorinated paraffin	[3] 30.0
Butyl Cellosolve	2.0
Water	4.0

[1] The mineral oil used had a viscosity of about 100 SUS at 100° F.
[2] About 60% by weight sodium sulfonate in mineral oil. The sodium sulfonate was derived from petroleum sources and contained an average of about 26 carbon atoms per molecule.
[3] The chlorinated paraffin used contained about 40% by weight of chlorine and about 28 carbon atoms per molecule.

One part of this composition was emulsified in four parts (by volume) of water and the resulting emulsion was tested by the tapping procedure described previously.

The results of these tests with work pieces composed of various metals are given below:

Metal:	Torque required, inch pounds
1117 Steel	192
8620 Steel	445
1100 Aluminum	119
7072 Aluminum	340

EXAMPLES 2 AND 3

These examples illustrate that the compositions of the present invention have substantially improved lubricating properties relative to the composition of Example 1.

The following compositions using the same mineral oil, sodium sulfonate and chlorinated paraffin used in Example 1, were prepared and tested in a manner similar to that of the composition of Example 1.

	Weight percent	
	Example 2	Example 3
Component:		
Mineral oil	17.0	25.0
Sodium sulfonate	25.0	25.0
Chlorinated paraffin	30.0	30.0
Cloudy type overbased calcium sulfonate [1]	20.0	
Clear type overbased calcium sulfonate [2]		13.0
Butyl cellosolve	7.0	7.0
Water	1.0	1.0

[1] About 36% by weight of a calcium sulfonate overbased as calcium carbonate suspended in mineral oil. The sulfonate was derived from petroleum sources and contained about 20 to about 40 carbon atoms per molecule and on the average, about 26 carbon atoms per molecule. The major portion of the calcium carbonate is in the form of platelets having a maximum dimension (across the top of the platelet) within the range from about 700 A. to about 900 A. and a thickness in the range from about 70 A. to about 90 A. These carbonate particles give the overbased sulfonate-oil mixture its characteristic cloudy appearance. The overbased sulfonate-oil mixture had a total base number of about 180. This mixture contained about 430 mole percent of basicity in excess of that needed to neutralize the precursor sulfonic acid.

[2] About 56% by weight of a calcium sulfonate overbased as calcium carbonate suspended in mineral oil. The sulfonate was derived from petroleum sources and contained, on an average, about 25 carbon atoms per molecule. Substantially all the suspended calcium carbonate particles were less than about 60 A. in size and, as a result, the overbased sulfonate-oil mixture appeared clear to the naked eye. The overbased sulfonate-oil mixture had a total base number of about 280. This mixture contained about 430 mole percent of basicity in excess of that needed to neutralize the precursor sulfonic acid.

Test results, in comparison with those from Example 1 are as follows:

	Torque required, inch pounds		
	Example 1	Example 2	Example 3
Metal:			
1117 steel	181	173	173
8620 steel	313	258	248
1100 aluminum	45	42	39
7072 aluminum	238	205	207

These examples clearly illustrate that the compositions of the present invention, which contain both a chlorinated hydrocarbon and an overbased sulfonate, have substantially improved lubricating and extreme pressure properties relative to compositions comprising only chlorinated hydrocarbon. This improvement in lubricating efficiency is apparent while working a wide variety of metals, for example, the four metals tested in the above examples. These examples demonstrate that the compositions of the present invention have utility for various metal working compositions, such as those listed hereinbefore.

While in the foregoing disclosure certain examples have been set forth which illustrate details specifying modes of applying this invention, it should be understood that such details may be varied considerably by one skilled in the art without departing from the spirit of this invention.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A composition comprising from about 10% to about 90% by weight of an oil of lubricating viscosity; at least one non-cationic emulsifier in an amount sufficient to render said composition emulsifiable with water; from about 1% to about 30% by weight of at least one overbased sulfonate selected from the group consisting of alkali metal sulfonate, alkaline earth metal sulfonate and mixtures thereof, overbased as the corresponding carbonate and mixtures thereof; from about 1% to about 40% by weight of at least one chlorinated hydrocarbonaceous component which comprises at least about 5% by weight of chlorine, said overbased sulfonate and chlorinated hydrocarbonaceous component each being present in amounts sufficient to improve the extreme pressure properties of said composition; at least one coupling agent present in an amount sufficient to improve the emulsibility of the components of said composition; and a stabilizing amount of water.

2. The composition of claim **1**, wherein said overbased sulfonate comprises (1) a metal salt of a sulfonic acid containing from about 12 to about 200 carbon atoms per molecule, and (2) dispersed particles of the corresponding carbonate, said overbased sulfonate containing from about 50 mole percent to about 900 mole percent of basicity in excess of that needed to neutralize said sulfonic acid.

3. The composition of claim **2**, wherein said chlorinated hydrocarbonaceous component comprises at least about 20% by weight of chlorine, said chlorinated hydrocarbonaceous component being present in an amount from about 5% to about 40% by weight of the total composition.

4. The composition of claim **3**, wherein said noncationic emulsifier is present in an amount from about 1% to about 40% by weight of the total composition and said coupling agent is present in an amount from about 0.5% to about 10% by weight of the total composition.

5. The composition of claim **4**, wherein said overbased sulfonate is selected from the group consisting of alkaline earth metal sulfonates and mixtures thereof, said overbased sulfonate containing from about 10 mole percent to about 80 mole percent of basicity in excess of that needed to neutralize said sulfonic acid and being present in an amount from about 3% to about 20% by weight of the total composition, and said chlorinated hydrocarbonaceous component is selected from the group consisting of chlorinated paraffin, chlorinated olefin, chlorinated polyolefien, chlorinated aromatics, chlorinated esters of fatty acids, chlorinated esters of naphthenic acids, chlorinated esters of resin acids, chlorinated microcrystalline wax and mixture thereof, said chlorinated hydrocarbonaceous component containing less than about 70 carbon atoms per molecule.

6. The composition of claim **5**, wherein said overbased sulfonate is a calcium sulfonate overbased as calcium carbonate.

7. The composition of claim **6**, wherein said overbased sulfonated contains from about 20 mole percent to about 750 mole percent of basicity in excess of that needed to neutralize said sulfonic acid and said calcium sulfonate containing from about 12 to about 50 carbon atoms per molecule and the major portion of said calcium carbonate particles have a maximum dimension of from about 100 A. to about 1 micron, when said overbased sulfonate is dispersed in an oil carrier, and said chlorinated hydrocarbonaceous component is selected from the group consisting of chlorinated paraffin, chlorinated olefin, chlorinated polyolefin and mixtures thereof, said chlorinated hydrocarbonaceous component being present in an amount from about 15%, to about 40% by weight of the total composition.

8. The composition of claim **7**, wherein said chlorinated hydrocarbonaceous component is chlorinated paraffin containing from about 20 to about 40 carbon atoms per molecule, said non-cationic emulsifier is selected from the group consisting of alkali metal sulfonates and mixtures thereof derived from petroleum sources, said sulfonates containing from about 12 to about 200 carbon

atoms per molecule and present in an amount from about 5% to about 30% by weight of the total composition, and said coupling agent is selected from the group consisting of ethyl Cellosolve, Carbitol, butyl Cellosolve and mixtures thereof, said coupling agent being present in an amount from about 3% to about 10% by weight of the total composition.

9. The composition of claim 8, wherein the major portion of said calcium carbonate particles have a maximum dimension of from about 700 A. to about 900 A. when said overbased sulfonate is dispersed in an oil carrier and said noncationic emulsifier is sodium sulfonate.

10. An aqueous emulsion comprising (A) water in an amount from about 40% to about 99.5% by volume of the total emulsion and (B) the composition of claim 1 in an amount from about 0.5% to about 60% by volume of the total emulsion.

11. An aqueous emulsion comprising (A) water in an amount from about 40% to about 99.5% by volume of the total emulsion and (B) the composition of claim 3 in an amount from about 0.5% to about 60% by by volume of the total emulsion.

12. An aqueous emulsion comprising (A) water in an amount from about 50% to about 99.5% by volume of said emulsion and (B) the composition of claim 3 in an amount from about 0.5% to about 50% by volume of said emulsion.

13. An aqueous emulsion comprising (A) water in an amount from about 50% to about 99.5% by volume of said emulsion and (B) the composition of claim 5 in an amount from about 0.5% to about 50% by volume of said emulsion.

14. An aqueous emulsion comprising (A) water in an amount from about 50% to about 98% by volume of said emulsion, and (B) the composition of claim 5 in an amount from about 2% to about 50% by volume of said emulsion.

15. An aqueous emulsion comprising (A) water in an amount from about 60% to about 95% by volume of said emulsion and (B) the composition of claim 7 in an amount from about 5% to about 40% by volume of said emulsion.

16. An aqueous emulsion comprising (A) water in an amount from about 60% to about 95% by volume of said emulsion and (B) the composition of claim 9 in an amount from about 5% to about 40% by volume of said emulsion.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,813,337 Dated May 28, 1974

Inventor(s) David B. Sheldahl

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 8, line 38; delete the number "10" and insert in place thereof ---100---.

Column 8, line 39; delete the number "80" and insert in place thereof ---800---.

Column 8, line 54; delete the word "sulfonated" and insert in place thereof ---sulfonate---.

Column 8, line 54; delete the number "20" and insert in place thereof ---200---.

Signed and sealed this 24th day of September 1974.

(SEL)
Attest:

McCOY M. GIBSON JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents