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㊹ Electro-rheological fluids and methods of making and using the same.

㊼ Disclosed are electro-rheological fluids including vermiculite treated with a quaternary ammonium salt, methods of making the same, propylene carbonate adsorbed on the solid phase, butyl benzoate added for the liquid phase, and methods of using the frequency response of electro-rheological fluids to vary apparent viscosity and to compensate for temperature changes in the fluids.

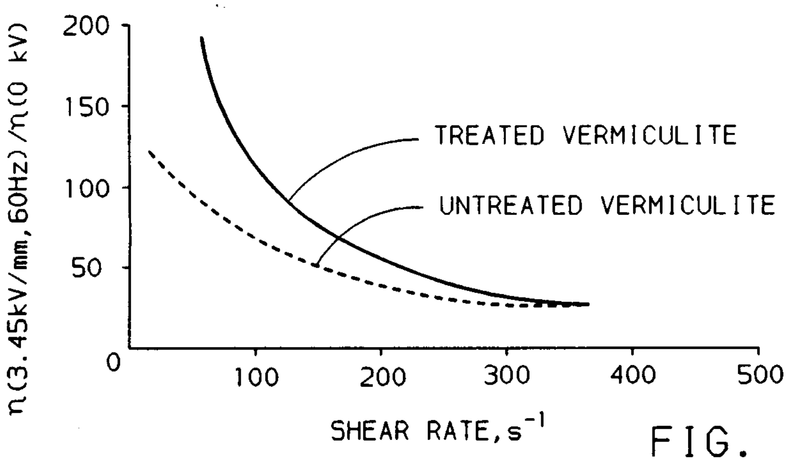

FIG. 2

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The present invention relates to fluid compositions which demonstrate significant changes in their fluid properties in the presence of an electrical field.

Electro-rheological response is a phenomenon in which the rheology of a fluid is modified by the imposition of an electrical field. Fluids which exhibit significant changes in their properties of flow in the presence of an electrical field have been known for several decades. The phenomena of electro-rheology was reported by W. M. Winslow, U.S. patent No. 2,417,850, in 1947. Winslow demonstrated that certain suspensions of solids in liquids show large, reversible electro-rheological effects. In the absence of electrical field, electro-rheological fluids generally exhibit Newtonian behavior. That is, the applied force per unit area, known as shear stress, is directly proportional to the shear rate, i.e., relative velocity per unit thickness. When an electrical field is applied, a yield stress phenomena appears and no shearing takes place until the shear stress exceeds a yield value which generally rises with increasing electrical field strength. This phenomenon can appear as an increase in apparent viscosity of several, and often many orders of magnitude. The response time to electrical fields is frequently in the order of milliseconds. This rapid response characteristics of electro-rheological fluids makes them attractive to use as elements in mechanical devices.

A complete understanding of the mechanisms through which electro-rheological fluids exhibit their particular behavior has eluded workers in the art. Many have speculated on the mechanisms giving rise to the behaviour characteristics of electro-rheological fluids. A first theory is that the applied electrical field restricts the freedom of particles to rotate, thus changing their bulk behavior. A second theory describes a change in properties to the formation of filament-like aggregates which form along the lines of the applied electrical field. One theory proposes that this "induced fibrillation" results from small, lateral migrations of particles to regions of high field intensity between gaps of incomplete chains of particles, followed by mutual attraction of these particles.

A third theory refers to an "electric double layer" in which the effect is explained by hypothesising that the application of electrical field causes a layer of materials adsorbed upon the discrete phase particles to move, relative to the particles, in the direction along the field toward the electrode having a charge opposite that of the mobile ions in the adsorbed layer.

Yet another theory proposes that the electrical field drives water to the surface of discrete phase particles through a process of electro-osmosis. The resulting water film on the particles then acts as a glue which holds particles together.

Criticism of a simple fibrillation theory has been made on the grounds that the effect is much too rapid for such intensive structure formation to occur. Workers in the art have observed a time scale for fibrillation of approximately 20 seconds, which is vastly in excess of the time scale for rheological response of electro-rheological fluids. Some workers suggest the sequence of events as a possible mechanism include: ionic migration, subsequent electro-osmosis of moisture to one pole of the particle (presumably the cationic region) and, in consequence, surface supply of water sufficient for bridging. This moisture bridge mechanism is not the lone process by which electro-rheological effects occur. The advent of anhydrous electro-rheological fluid means that water-bridging is not an essential mechanism and may indeed not be operative at all.

Despite the numerous theories and speculations, it is generally agreed that the initial step in development of electro-rheological behaviour involves polarization under the influence of an electrical field. This then induces some form of interaction between particles or between particles and the impressed electric or shear fields which results in the rheological manifestations of the effect. See Carlson, U.S. patent No. 4,772,407; and Block et al "Electro-Rheology", IEEE Symposium, London, 1985. Despite this one generally accepted mechanism, the development of suitable electro-rheological fluids and methods of improving the same remains largely unpredictable.

The potential usefulness of electro-rheological (ER) fluids in automotive applications, such as vibration damping, shock-absorbers, or torque transfer, stems from their ability to increase, by orders of magnitude, their apparent viscosity upon application of electrical field. This increase can be achieved with very fast (on the order of milliseconds) response times and with minimal power requirements. Although ER-fluids have been formulated and investigated since the early 1940's, basic limitations have prevented their utilization in practical devices. The most severely restrictive of these limitations are (1) that the suspensions be stable, i.e., should be readily re-dispersible upon standing, even if settlement occurs and (2) they not suffer from the limitation imposed by the presence of water so that, at extended temperatures, i.e., outside of 0-100 degrees C., service and durability can be achieved. This latter requirement is particularly restrictive in that most fluid compositions require water as an ER "activator" so that in completely dry systems the ER-effect is entirely absent or so small that it is not effectively useful.

An object of this invention is to formulate a stable, substantially water-free, or non-aqueous ER-fluid with improved properties. In other words, one goal of this invention to remove the water without compromising the electro-rheological effect.

This invention generally includes electro-rheological fluids comprising particles treated with polar salt such as an amine salt and a non-conducting or dielectric fluid. Preferably, the particles are vermiculite and are chemically-defoliated and are plate-like. The preferred polar salts are quaternary ammonium salts. Preferably the fluid is non-aqueous. Preferably the liquid phase of the fluid is substantially free of quaterany ammonium salt not bound to the particles and substantially free of dispersing agents.

These and other objects, features and advantages of this invention will be apparent from the following detailed description, claims and appended drawings, in which:

Figure 1 is a graphic illustration of the effect on viscosity of an ester additive to an electro-rheological fluid.

Figure 2 is a graphic illustration of the effect on viscosity of treating a solid phase of an electro-rheological fluid with an amine salt.

Figure 3 is a graphic illustration of the effect on viscosity of changing the frequency of an applied field and the temperature of an electro-rheological fluid.

Figure 4 is a graphic illustration of the effect of varying the temperature of an electro-rheological fluid and varying the frequency of an applied field to maintain constant viscosity.

Figure 5 is a graphic illustration of the effect on viscosity of adsorbing propylene carbonate on a solid phase of an electro-rheological fluid.

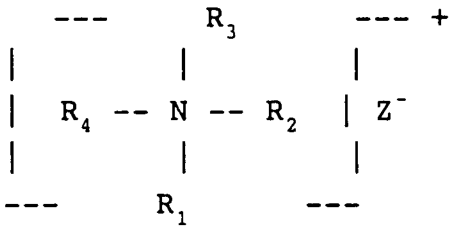
Figure 6 is a graphic illustration of the effect on viscosity of absorbing propylene carbonate on the solid phase and of an ester additive to an electro-rheological fluid.

Vermiculite is a gold-coloured mineral having the formula $3MgO(FeAl)_2O_3,3SiO_2$. Chemically-defoliated vermiculite, in a plate-like Al-Mg sheet silicate form, is commercially available from W. R. Grace, Inc., U.S.A., under the trade name MICROLITE 903^{tm}. The term "plate-like Al-Mg sheet silicate form" means that the particles are made up of multiple-layered planes ("sheets") consisting of extensive Si-O linkages (silicate). The planes are ionically-bonded to one another via cations such as Al^{3^+} and Mg^{2^+}. The particles of vermiculite are partially-conductive particles. In this commercial form, the vermiculite is hydrophilic which makes it a) difficult to disperse in typical base electro-rheological fluid phases such as silicon oils and hydrocarbons and b) is not likely to stay dispersed. If the vermiculite does not stay dispersed, it settles and forms a cake material. This is undesirable because considerable mechanical energy must be expended to continually re-disperse the solid in order for the material to function as an electro-rheological fluid. The present invention provides a method of treating this vermiculite to achieve advantageous results.

In one embodiment of this invention, chemically-de-laminated vermiculite plates are surface-treated with an organic amine salt. An ionic bond is formed between the vermiculite and the amine salt. The amine salt serves two purposes. Firstly, the amine salt allows the vermiculite, after proper drying, to form a very stable dispersion with silicon oil or other non-aqueous liquids such as hydrocarbon liquid phase materials. Secondly, the amine salt allows the individual plate-like vermiculite particles to be polarized in an electrical field without the presence of water. The polarization of the particles is a necessary requirement for a system to demonstrate an ER behaviour. The geometry of the plate-like vermiculite particles produces a greater polarization in an electric field than other shapes. Preferably, the plate-like vermiculite has an average face diameter range from about 1 to about 30 micrometres, and a particle thickness of about 6 to about 10 nanometres (about 60 to about 100 Angstroms). The vermiculite may be present in an amount range from about 5 to about 50, and preferably about 10 to about 30 percent by weight of the composition.

The chemically-defoliated vermiculite is surface-treated by exchanging lithium on the surface of an amine cation. The amine cation may be primary, secondary, or tertiary, and preferably is a quaternary ammonium salt. Suitable treating materials are amine salts including at least one selected from the group consisting of alkyl ammonium halides, preferably with an alkyl group having 2 to 18 carbons, and most preferably 12 carbons or dodecylamine salt. Suitable amine salts, and particularly quaternary ammonium salts, contemplated include those listed in Bosso et al, U.S. Patent No. 3,839,252 which is hereby incorporated by reference.

A quaternary ammonium salt is a type of organic nitrogen compound in which the molecular structure includes a central nitrogen atom joined to four groups (the cation) and an anion, the structure as indicated as:



$$\left[\; R_4 - \overset{\overset{\displaystyle R_3}{|}}{\underset{\underset{\displaystyle R_1}{|}}{N}} - R_2 \;\right]^+ Z^-$$

wherein R_1, R_2, R_3 and R_4 are alkyl or aromatic groups or hydrogen, and wherein at least one of the R-groups has from 2 to 18 carbons and the other R-groups have from 2 to 18 carbon atoms.

Particularly suitable quaternary ammonium salts include at least one selected from the group consisting of octadecyldimethylbenzyl ammonium chloride, hexamethonium chloride, and lauryl pyridinium chloride.

Typically, the vermiculite is placed in a solution of amine salts comprising the amine salt and de-ionized water as a solvent. The equivalent of amine in solution to vermiculite may range from 5 to 1, preferably 2 to 1 and most preferably 1 to 1. The time period for which the vermiculite is treated may range, depending on temperature, from 3 to 24 hours, preferably 3 to 12 hours and most preferably 6 to 12 hours at room temperature. Higher temperatures require less time to treat the particles. The attached hydrocarbon chain on the treated material will render it substantially hydrophobic, thereby increasing its inherent dispersity as well as its stability towards coagulation.

Next, excess amine is removed from the treated material by washing with ethanol. The solid is filtered and dried under vacuum at a temperature ranging from 60 ° C to 110 ° C, preferably 75 ° C to 110 ° C and most preferably 100 degrees C., which is less than that which will cause change in the surface treatment but high enough to promote removal of residual water in a reasonably short time period. The dried, treated vermiculite is substantially free of water. The term "substantially free of water" means less than 1% by weight water adhering to the vermiculite. Preferably, the amount of water adhering to the vermiculite is less than that required (approximately 6-10% by weight) for the water to be an "activator" of ER-response. That is, the amount of water adhering to the vermiculite is not sufficient to create water bridges between particles in the influence of an electrical field. This drying is preferably carried out under vacuum to a constant pressure ranging from about 13.3322 to 66.661 Pa (100 to 500 mTorr), preferably 13.3322 to 33.3305 (100 to 250) and most preferably at least 19.9983 Pa (150 mTorr).

The resultant treated, dried materials are then dispersed in a base fluid composition by ball-milling for 22 hours. The ball-milling substantially reduces the average face diameter to at least the range of from about 5 to 25 micrometres, preferably 1-5 micrometres, more preferably about 1 micrometre to about 3 micrometres, and most preferably less than 1 micrometre which also promotes suspension stability and dispersibility. The ball-milling base fluid may comprise any suitable fluid known in the art, and is preferably 75% silicon oils/25% butyl benzoate. Other suitable ball-milling fluids include mineral oils or a material that is to be used as the liquid phase of the ER-fluid.

Suitable liquid-phase materials are disclosed in Block et al, "Electro-Rheology", IEEE Symposium, London, 1985, which is hereby incorporated by reference. A suitable silicone oil is commercially available from Dow Corning Corporation, U.S.A., under the trade name Dow Corning 200 Fluid (20cS)^{tm}.

The following example illustrates one embodiment of the present invention:

EXAMPLE I

A defoliated vermiculate suspension is prepared by adding about 7 to about 15 grams of chemically-defoliated vermiculite to about 1 to about 100 ml of de-ionized water. A suitable chemically-defoliated vermiculite is MICROLITE 903^{tm}. The aqueous defoliated vermiculite suspension is added drop-wise to an aqueous solution of excess amine hydrochloride solution, mechanically agitated for six hours, and then filtered. The amine hydrochloride solution may be 6.2% by weight of octylamine hydrochloride or 3.0% of dodecylamine hydrochloride in an aqueous solution. The solid is re-dispersed and filtered twice with ethanol to remove any excess amine hydrochloride. The solid is then dried in a vacuum at 100 degrees C. until at least a 19.9983 Pa (150 millitorr) vacuum is reached. The amine-treated vermiculite is ball-milled with a base fluid (e.g., 75% silicon oil/25% butyl benzoate) for 24 hours.

The above procedure was used to prepare a vermiculite treated with octylamine or dodecylamine hydrochloride. Carbon analysis showing the efficiency of surface treatment is listed in Table I. "Efficiency of surface treatment" indicates the percentage of cation exchange, and is derived from dividing the experimental value of the percentage of cation exchange by the corresponding theoretical value of the percentage of cation exchange. Thus, from the values listed in Table 1, the cation exchange efficiency ranges from about 58 percent for the octylamine-treated vermiculite particles to about 95 percent for the dodecylamine-treated vermiculite particles.

TABLE I

CARBON ANALYSIS*		
Material	Theoretical	Experimental
Octylamine-treated	9.67%	5.6%
Dodecylamine-treated	13.73%	13.0%
Untreated vermiculite	-	0.41%

*Analysis performed on LECO Corp. Model CS-444 Carbon/Sulfur Analyzer

For solid phases which require water to achieve polarization under electrical fields, the electro-rheological effect decreases when the water is removed. This effect may be restored, and in particular formulations greatly enhanced, by blending into the fluid phase of the electro-rheological composition an additional fluid such as a high-boiling ester. Suitable esters include at least one selected from the group comprising benzoates, preferably alkyl, or alkyl adipates. The alkyl group may range from C_1 to C_{18} and preferably the ester is n-butyl benzoate. Preferred adipates include di-isononyl adipate and dioctyl adipate. The amount of additional liquid may comprise from about 5 to about 75%, preferably 5 to about 50%, and most preferably about 5-25% by volume of the electro-rheological fluid. The additional fluid adds to the inherent stability and dispersibility of the treated solid phase as well as acting to lower quite substantially the base fluid viscosity and hence, the zero-field viscosity of the suspension. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,750, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

The primary basis for the utility of electro-rheological effect is the change in shear stress (i.e., increase in apparent viscosity) with applied electric field. At zero-field, an electro-rheological fluid composition comprising 10% vermiculite treated with dodecylamine, 75% silicon oils/25% butyl benzoate prepared in a manner described above has a viscosity at a shear rate of 400/seconds (which will be standard conditions for the purposes of illustration) of 28mPa sec (cP). At a field strength of 3.45kV/mm (AC, 60Hz), the fluid has an apparent viscosity of 1198mPa sec which is 43 times the zero-field value. This increase in apparent viscosity is greatly magnified as the shear rate decreases. The ratio of viscosity at 3.45kV/mm to viscosity at zero-field as a function of shear rate, is shown in Figure 1. Also shown for comparison in Figure 1 is the same plot for the same composition but without butyl benzoate. A comparison of these two plots emphasises (1) the significant enhancement of electro-rheological effects which is achieved by the addition of butyl benzoate and (2) the minimal electro-rheological effect exhibited by the non-aqueous system without n-butyl benzoate.

Figure 2 is a plot of the viscosity ratio as a function of shear rate (3.45kV/mm to zero-field). Here the above-described electro-rheological composition (illustrated by Figure 1) is compared to the same composition but in which the vermiculite particles are not treated with an amine to form the dispersed phase. Although the electro-rheological effect for these two systems is comparable, the composition containing the vermiculite particles not treated with an amine is basically unstable to the extent that, upon repeated application of an electric field, large particle aggregates form and precipitate out of the suspension. With time, the fluid will separate into two phases and must be subjected to ball-milling to re-disperse the solid. This is not the case with the treated vermiculite composition. After sitting for as long as six months, the solid is readily re-dispersed by shaking the composition.

Another embodiment of this invention includes a method of changing the frequency of an alternating current electric field applied to an electro-rheological fluid and temperature of the fluid to adjust the apparent viscosity of the fluid. The term "apparent viscosity" is the ratio of shear stress to shear rate. An electro-rheological fluid comprising 10% solids prepared as described above and a mixture of 25% n-butyl benzoate/75% polydimethylsiloxane fluid was evaluated for change in viscosity as a function of temperature and varying frequencies as shown in Figure 3. As shown in Figure 3, in the field-off case (lower curve designated 0 kV, 0 Hz), the normal exponential decrease in viscosity (measured at a shear rate of 400/sec) expected for a particulate suspension is observed. The other curves show significant increases/or decreases of viscosity with temperature depending on the frequency and temperature range. The invention is best illustrated by considering the vertical line at a temperature of 50 degrees C. By continuously changing the frequency, at constant applied potential, from 50Hz to 5000Hz and preferably 60Hz to 1000Hz, any desired viscosity in the range shown can be achieved without changing the applied potential. This method may be adopted to the operation of a device such as a shock-absorber, or an engine mount, which requires that the viscosity be varied continuously from the field-off value to some maximum value at a given temperature. Further, the frequency may be varied at any given operation temperature to produce a desired viscosity. Analog systems or "look-up tables" may be utilized in this regard. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,759, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

Another embodiment of this invention includes a method of changing the frequency of an alternating current electric field applied to an electro-rheological fluid to maintain a constant viscosity over varying temperatures. This embodiment can be best illustrated by Figure 4 in which it can be seen that to achieve a constant viscosity of 0.3 Pas (300cP) (measured at shear rate of 400/sec) the frequency can be adjusted from 60 Hz to 1000 Hz to compensate for variation in temperatures ranging from 10 degrees C. to approximately 85 degrees C. The data illustrated in Figure 4 is for an electro-rheological fluid prepared as described for the embodiment illustrated by Figure 3. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,747, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

Another embodiment of this invention includes a method of activating (i.e., turning-on or producing a desired electro-rheological effect) or de-activating (i.e., turning-off or eliminating an electro-rheological effect) an electro-rheological response of a fluid under the influence of a substantially constant alternating current electric field comprising varying the frequency of the field to achieve the desired result. The method may be accomplished without substantially varying the temperature of the field. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,759, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

Another embodiment of this invention may be characterized as a method of producing a predetermined change in the viscosity of an electro-rheological fluid including the steps of applying, for a predetermined period, a substantially constant alternating current electric field to the electro-rheological fluid and changing the frequency of the electric field from a first level, corresponding to a first viscosity, to a second level, corresponding to a second viscosity at a given shear rate. The difference between the first and second viscosities would be equivalent or equal to the predetermined change in viscosity desired. In such a method, the strength of said alternating current electric field ranges from 1 to 5 kV/mm, said first level of frequency is greater than 10Hz, and said second level of frequency is less than 10 kHz. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,759, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

Another embodiment of this invention includes improvements in performance of the electro-rheological fluid achieved by absorption of an activator, preferably propylene carbonate, directly onto the surface of a solid phase. The solid phase of electro-rheological fluid is prepared in the manner described above with the additional step of adsorbing an activator directly onto the surface of the solid. Propylene carbonate, which is insoluble in silicon oils, such as polydimethylsiloxane, is adsorbed onto the solid in specific amounts by weight. For example, pre-weighed amounts of a solid such as vermiculite of about 10 to about 50 percent by weight is immersed in ethanol solution containing about 1 to about 25 percent by weight of propylene carbonate. After thorough mixing, ethanol is removed from the solid by heating at about 100 degrees C. under vacuum for 24 hours. These conditions were chosen to maximize removal of ethanol, leaving a maximum amount of propylene carbonate adsorbed onto the solid. The specific amount of adsorbed propylene carbonate was determined by weighing the treated solid. In this fashion, solids were prepared in amounts of adsorbed propylene carbonate ranging from 1.4 to 16% by weight. Preferably the amount of adsorbed propylene carbonate for enhanced electro-rheological response ranges from about 9% to about 16%, and preferably about 9 to about 12% by weight. As shown in Figure 5, little enhancement of the electro-rheological effect (measured as a difference in field-on [2.07kV/mm, 60Hz AC] to field-off shear stress divided by the field-off value) is observed when a total amount of propylene carbonate less than 9% by weight is adsorbed onto the solid. With amounts of 9% by weight or greater adsorbed onto the solid, an increase in electro-rheological effect is registered over the entire shear rate range. The use of an activator adsorbed on the solid phase does not compromise the non-aqueous nature of the fluid. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,749, entitled "Electro-rheological Fluids and Methods of Making and Using the Same", filed on the same date as the filing date of this application.

It has been surprisingly found that the combination of using an adipate such as dioctyladipate (DOA) in the liquid phase and absorbing propylene carbonate on the surface of the solid phase produces more than an additive effect. Electro-rheological fluids were prepared having a solid content of about 10% by weight. A first fluid was prepared containing amine-treated vermiculite having 6% by weight propylene carbonate adsorbed on the vermiculite and dispersed in silicone oil. The ER-response is represented by a first curve of Figure 6. A second material was prepared with amine-treated vermiculite in a liquid phase containing 65% by volume DOA and 35% by volume silicone oil. This ER-response of the second fluid is represented by a second curve of Figure 6. A third fluid was prepared having amine-treated vermiculite having 6% propylene carbonate adsorbed on the vermiculite and a liquid phase containing 65% by volume DOA and 35% by volume silicone oil. The ER-response of the third fluid is represented by a third curve of Figure 6. The three fluids were subjected to an electric field strength of 2.07 kV/mm. The ER-response of the fluids is plotted as shear stress ratio (that is, the ratio of excess shear stress under field to that same fluid at zero field) versus shear rate. As can be seen, the third fluid has a greater ER-response at 50/sec shear rate than the first and second fluids added together. Aspects of this embodiment are claimed in our co-pending European patent application No. corresponding to USSN 684,750.

The various embodiments may be combined and varied in a manner within the ordinary skill of persons in the art to practice the invention and to achieve various results as desired.

Where particular aspects of the present invention are defined herein in terms of ranges, it is intended that the invention includes the entire range so defined, and any sub-range or multiple sub-ranges within the broad range. By way of example, where the invention is described as comprising about 1 to about 100% by weight of component A, it is intended to mean that the scope of the invention also includes, for example, about 5 to about 25% by weight of component A, and about 50 to about 75% by weight of component A. Likewise, where the present invention has been described herein as including $A_{1-100}B_{1-50}$, it is intended to include within the scope of the invention compositions such as $A_{1-60}B_{1-20}$, $A_{60-100}B_{25-50}$ and $A_{43}B_{37}$.

Claims

1. A composition containing a solid phase suspended in a liquid phase, for producing an electro-rheological response in the presence of an electric field, characterised in that the composition is an anhydrous composition in which the liquid phase comprises a non-conducting or dielectric fluid; and the solid phase comprises chemically-defoliated vermiculite particles present in an amount ranging from 5 to 50 weight percent of the total composition, treated with a quaternary ammonium salt so that the cation exchange efficiency ranges from about 58 percent to about 95 percent, effective to produce an electro-rheological response to an electric field.

2. A composition according to claim 1, in which said vermiculite particles have a plate-like shape.

3. A composition according to claim 1, in which said vermiculite has an average face diameter ranging from about 1 to about 3 micrometres.

4. A composition according to claim 1, in which said vermiculite has an average face diameter of less than 1 micrometre.

5. A composition according to claim 1, in which said quaternary ammonium salt is prepared from an alkyl ammonium halide.

6. A composition according to claim 1, in which said quaternary ammonium salt comprises octylamine hydrochloride.

7. A composition according to claim 1, in which said quaternary ammonium salt comprises dodecylamine hydrochloride.

8. A composition according to claim 1, in which said liquid phase is at least one material selected from the group consisting of polydimethylsiloxane and mineral oil.

9. A composition according to claim 1, in which said liquid phase comprises silicone oil.

10. A method of preparing a composition according to claim 1, in which the chemically-defoliated vermiculate particles are treated with said quaternary ammonium salt to obtain said range of cation exchange efficiencies; the treated vermiculite is dried at a temperature and pressure sufficient to produce substantially water-free treated vermiculite; and a sufficient amount of said substantially water-free treated vermiculite is added to said non-conducting or dielectric fluid so that said composition produces said electro-rheological response in the presence of an electric field.

11. A method according to claim 10, which further comprises the step of: ball-milling the dried, treated, substantially water-free vermiculite to a particle length ranging from about 1 to about 3 micrometres.

12. A method according to claim 11, in which said step of ball-milling is carried out so as to produce a vermiculite particle having a length of less than 1 micrometre.

13. A method according to claim 11, in which the liquid phase comprises silicone oil, and a ball-milling fluid is added which comprises at least one selected from the group consisting of silicone oil and n-butyl benzoate and mixtures thereof.

14. A method according to claim 13, in which said ball-milling fluid is 75 percent by weight silicone oil and 25 percent by weight n-butyl benzoate.

15. A method according to claim 10, in which about 7 to 15 grams of vermiculite particles are suspended in about 100 ml of water; and said suspension is added dropwise to an aqueous solution comprising about 3.0 to about 6.2 percent by weight of said quaternary ammonium salt.

16. A method according to claim 10, in which said quaternary ammonium salt is either octylamine hydrochloride or dodecylamine hydrochloride.

17. Chemically-defoliated vermiculite particles for use in forming a composition according to claim 1, characterised in that the vermiculite particles are treated with a quaternary ammonium salt.

18. Chemically-defoliated vermiculite particles according to claim 17, in which said vermiculite particles are substantially free of water.

19. Chemically-defoliated vermiculite particles according to claim 17, in which said quaternary ammonium salt is either octylamine hydrochloride or dodecylamine hydrochloride.

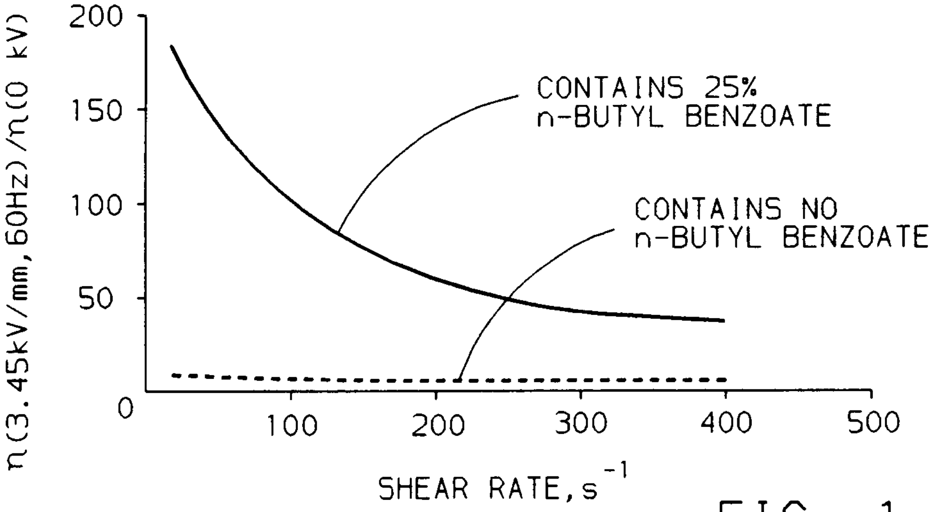


FIG. 1

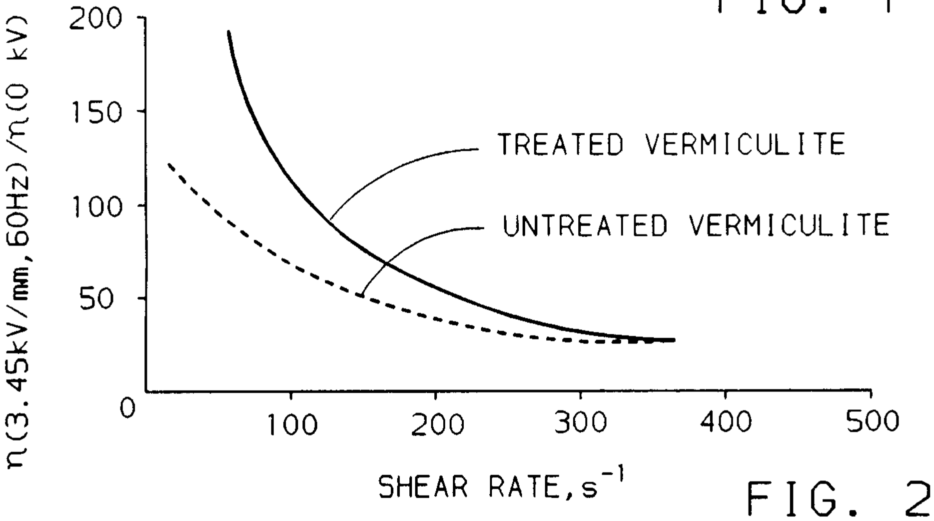


FIG. 2

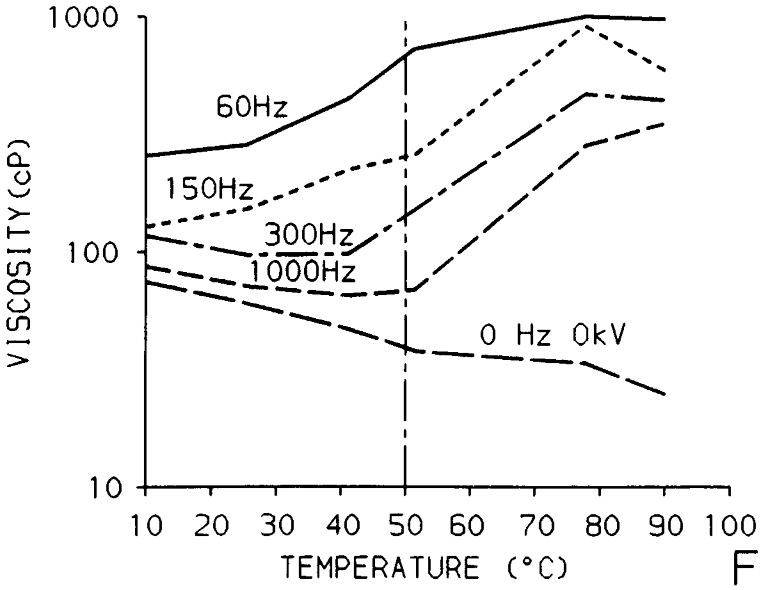


FIG. 3

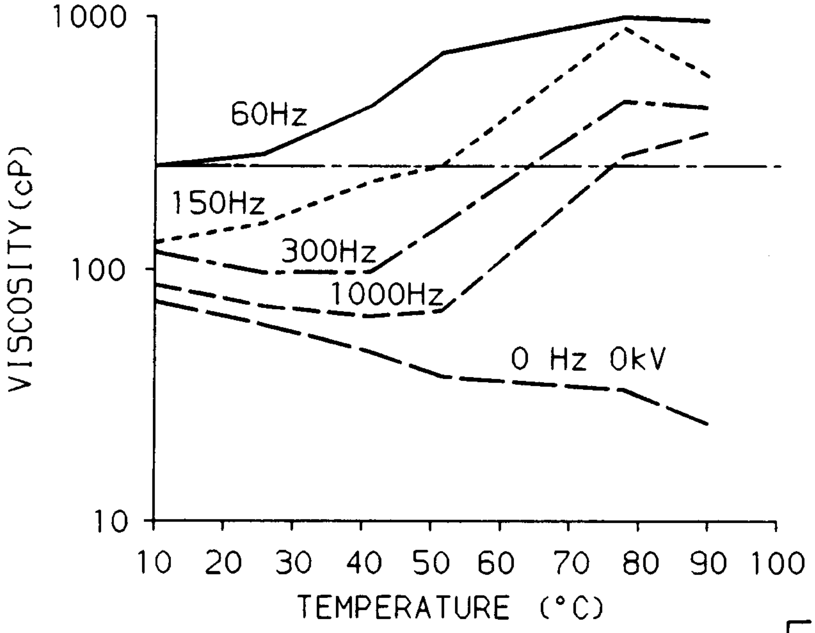


FIG. 4

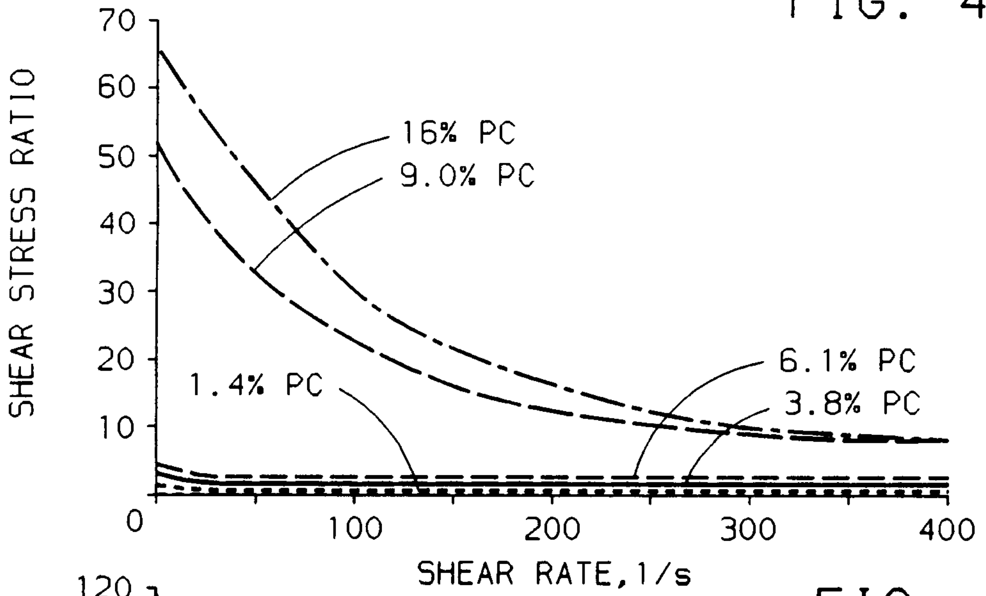


FIG. 5

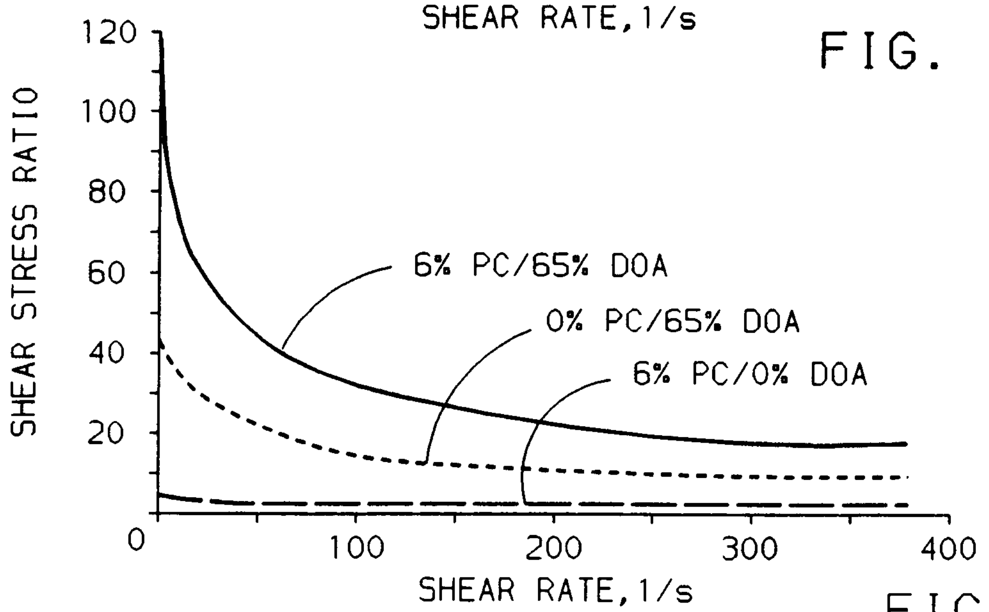


FIG. 6

European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 92 20 0905

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	US-A-3 367 872 (T.W.MARTINEK) * column 5, line 37 - line 49 * * column 49, line 50 - line 73; claims 1,10-14 * ---	1,5-10, 16-19	C10M171/00
A	EP-A-0 393 831 (ICI) * page 3, line 14 - line 17 * * page 7, line 1 - page 8, line 3; claim 1 * ---	1,2-4, 8-10	
A	EP-A-0 341 737 (MITSUBISHI KASEI CORP.) * column 4, line 19 - line 24 * ---	10-13	
P,X	EP-A-0 427 520 (THE DOW CHEMICAL CO.) * claims 1,3,7-9,12-14,18-21 * ---	1-12	
P,X	PATENT ABSTRACTS OF JAPAN vol. 15, no. 431 (C-881)(4959) 5 November 1991 & JP-A-3 181 597 (BRIDGESTONE) 7 August 1991 * abstract * -----	1-12, 16-19	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C10M

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	21 JULY 1992	ROTSAERT L.D.C.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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