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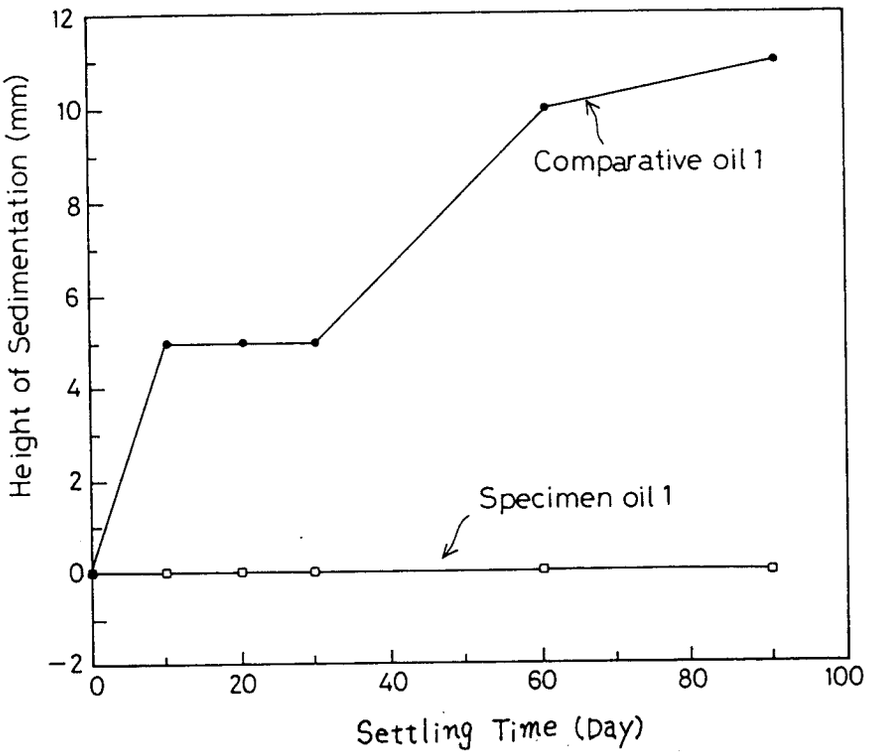
(54) **ELECTROVISCOUS FLUID.**

(57) An electroviscous fluid prepared by dispersing fine solid particles in an electrically insulating fluid. The fine solid particles have a specific gravity equal to, or greater than, that of the elctrically insulating fluid, and have a hydroxyl group on the surface thereof. The hydroxyl group is covalently bonded to a nonionic surfactant having a hydroxyl group and/or an - NH group as a hydrophilic group through a silane coupling agent, or polystyrene is covalently bonded thereto. The fluid is excellent in response, reproducibility, durability and thickening effect, particularly in dispersibility and shelf life stability, and can be utilized for electrical control of vibration control machines such as variable damping dampers, engine mounts, bearing dampers, clutches, valves, shock absorbers, precision equipment, acoustic instruments, etc., and for display devices.

EP 0 555 487 A1

FIG. 1


12
10
8
6
4
2
0
−2
Height of Sedimentation (mm)
0
20
40
60
80
100
Settling Time (Day)
Comparative oil 1
Specimen oil 1

FIELD OF THE INVENTION

The present invention relates to an electroviscous fluid, in which viscosity can be controlled by applying voltage and which can be used for electrical control of vibration control machines and devices such as variable damper, engine mount, bearing damper, clutch, valve shock absorber, precision machine, accoustic machine and also of display element, and in particular, it relates to an electroviscous fluid with improved dispersion property.

BACKGROUND TECHNIQUE

Electroviscous fluid (or electro-rheological fluid), in which viscosity of fluid changes when voltage is applied has been known since many years (Duff, A. W. Physical Review, Vol. 4, No. 1, (1896), 23). Early study on electroviscous fluid was concentrated on a system containing liquid only, and the effect was also insufficient. Subsequent studies have been performed on electroviscous fluid of solid dispersion system, and it has become possible to obtain considerable electroviscous effect.

With regard to the mechanism to give thickening effect (ER effect) in electroviscous fluid, Klass attributed this to the fact that the particles dispersed in electroviscous fluid induce polarization of the double layer in electric field (Klass, D. L., et al., J. of Applied Physics, Vol. 38, No. 1 (1967), 67). Explaining this from electric double layer, ions adsorbed around dispersed substances (such as silica gel) are uniformly arranged on outer surface of dispersoid when E (electric field) = 0, while ion distribution is deviated when E (electric field) = finite value, and particles exert electrostatic effect to each other in the electric field. Thus, each of the particles forms bridge between electrodes, and shear resistance is generated against stress, i.e. ER effect occurs.

Winslow proposed an electroviscous fluid using paraffin, silica gel powder, and also water to turn the system to slightly electroconductive. (Winslow, W.M., J. of Applied Physics, Vol. 20 (1949), 1137). By this study of Winslow, the electroviscous effect of the electroviscous fluid is called "Winslow effect".

In the electroviscous fluid of solid dispersion system, specific gravity of electrically insulating fluid as base oil is normally 0.78 - 0.92 (15 °C), while specific gravity of solid particle, e.g. silica gel powder, is about 2.2. If there is big difference in specific gravity, it is not desirable for keeping ER effect constant because electroviscous effect varies or deteriorated during operation due to high sedimentation of dispersoid even if dispersing agent is used.

It is an object of the present invention to provide an electroviscous fluid having excellent dispersion property by giving dispersion property to silica particles, which serve as dispersoid.

DISCLOSURE OF THE INVENTION

The electroviscous fluid of the present invention comprises solid particles dispersed in electrically insulating fluid, said solid particles have specific gravity equal to or higher than that of the electrically insulating fluid and have hydroxyl group on the surface thereof, said hydroxyl group is bonded by covalent bond to non-ionic surface active agent having hydroxyl group and/or -NH group as hydrophilic group by using silane coupling agent.

Also, the electroviscous fluid of the present invention comprises solid particles dispersed in electrically insulating fluid, said solid particles have specific gravity equal to or higher than that of the electrically insulating fluid and have hydroxyl group on the surface thereof, and polystyrene and/or polyisoprene with polymerization degree of 5 - 320 is bonded to said hydroxyl group by covalent bond.

When silica particles for example are dispersed in electrically insulating fluid in an electroviscous fluid, dispersing agent is normally used. In such electroviscous fluid, dispersing agent is physically adsorbed on silica particles, and it appears that dispersing effect is generated by repellent action between the adsorbed dispersing agents themselves. In this case, the stability of dispersion is low, and sedimentation of silica particles decreases electroviscous effect.

The dispersoid in the present invention has specific gravity equal to or higher than that of the electrically insulating fluid, and solid particles having hydroxyl group on the surface are bonded by covalent bond to non-ionic surface active agent having hydroxyl group and/or -NH group as hydrophilic group by using silane coupling agent, or it is bonded to polystyrene by covalent bond, and this provides stable dispersion property in the electrically insulating fluid.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a diagram showing dispersion stability of an electroviscous fluid of the present invention;
Fig. 2 is a diagram showing dispersion stability of the electroviscous fluid of the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

There is no specific restriction on the electrically insulating fluid. For example, mineral oil and synthetic lubricating oil may be used. More concretely, paraffin type mineral oil, naphthene type mineral oil, and oils such as poly-α-olefine, polyalkylene glycol, silicone, ester, diester, polyol ester, phosphoric acid ester, silicon compound, fluorine oil, alkylbenzene, alkyldiphenyl ether, alkylbiphenyl, alkylnaphthalene, polyphenyl ether, synthetic hydrocarbon, etc may be used. The substance having viscosity of 5 to 300 cSt at 40 °C can be used.

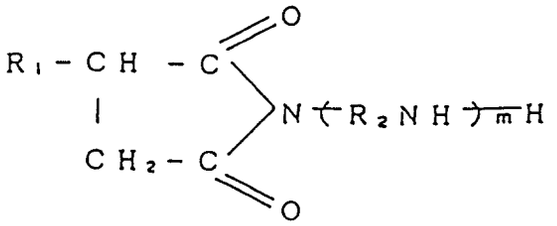
Solid particle should have specific gravity equal to or higher than that of the electrically insulating fluid and should have hydroxyl group on the surface. More concretely, silica gel, zeolite, etc. may be used. For example, silica gel particle having specific gravity of 2.2 (15 °C), particle size of 0.01 to 200μm, surface area of 100 to 700 m^2/g (BET method), and silanol group density of 1 to 10 SiOH/100$Å^2$ can be used.

First, description will be given on solid particle, to which non-ionic surface active agent is bonded to its surface.

The non-ionic surface active agent bonded to the surface of the solid particle by covalent bond must have hydroxyl group and/or -NH group as hydrophilic group. More concretely, polyalkenyl succinimide, succinimide ester, polyoxyethylenealkyl ether, polyoxyethylenealkylaryl ether, or fatty acid ester of polyhydric alcohol having hydroxyl group may be used.

As polyalkenyl succinimide, mono type succinimide given by the following formula (1) is used:

Formula (1):



(where R_1 is an olefin oligomer group having 30 or more carbon atoms, R_2 is an alkylene group having 2 to 4 carbon atoms, and m represents an integer of 1 to 10.)

In this polyalkenyl succinimide, polyolefine polymer given by R_1 has 30 or more carbon atoms, or more preferably 40 to 400 carbon atoms with average molecular weight of 500 to 5,000. As olefin to produce this, α-olefin having 2 to 8 carbon atoms such as ethylene, propylene, 1-butene, isobutylene, 1-hexene, 2-methylpentene-1,1-octene, etc. can be used. Polyolefin polymer is preferably polypropylene, or polyisobutyrene.

As polyalkylenepolyamine, the substance having m (repeating unit number) of 1 to 10 in the above formula is preferably used. Polyethylenepolyamine, polypropylenepolyamine, polybutyrenepolyamine, etc. are used, and polyethylenepolyamine is particularly preferable.

To prepare this dispersoid, silane coupling agent are mixed in a solvent by 50 - 400 weight parts, or more preferably 80 to 200 weight parts, to 100 weight parts of silica particles to bond silane coupling agent to silanol group in the silica particles. Further, under reflux condition, the product of the above reaction and polyalkenyl succinimide are mixed by 100 to 1,000 of polyalkenyl succinimide, or more preferably, by 200 to 800 weight parts to 100 weight parts of silica particles. Thus, polyalkenyl succinimide can be bonded to silica particles by using silane coupling agent. In this case, quantity of polyalkenyl succinimide bound to silica particles can be increased by adding alcohol such as n-butanol or phenol as catalyst. Such catalyst may be added by 20 to 300 weight parts, or more preferably by 50 to 200 weight parts, to 100 weight parts of polyalkenyl succinimide.

As silane coupling agent, it is appropriate to use γ-chloropropyltrimethoxysilane, β-(3,4-epoxycyclohexyl)-ethyl-trimethoxysilane, γ-glycidoxypropyl-trimethoxysilane, γ-isocyanate propyltriethoxysilane, γ-glycidoxypropyl-methyldiethoxysilane, etc.

Next, description will be given on solid particles with polystyrene and/or polyisoprene on its surface.

To bond polystyrene and/or polyisoprene with polymerization degree of 5 to 320 on the surface of solid particles by covalent bond, silica particles and thionyl chloride are treated to chlorinate silanol group in silica particles. On the other hand, n-butyl lithium and styrene are polymerized by anionic polymerization to prepare anionic polystyrene, and this anionic polystyrene and chlorinated silica particles prepared above react in solvent.

Chain length of polystyrene can be easily controlled by adjusting reaction temperature, and polymerization degree of polystyrene in the present invention is 5 to 320, or more preferably 130 to 280. When polymerization degree is less than 5, it is not desirable because lipophilic property is too low and silica particles. If it is more than 320, aggregate lipophilic property decreases. The percentage of polystyrene in dispersoid depends upon the quantity of silanol group and it is normally 14 to 16 weight %. In the above, description has been given to the case where styrene is used, but the same applies to isoprene. When polyisoprene is bonded to silica particles by covalent bond, the effect similar to the case of polystyrene can be obtained. Also, polystyrene and polyisoprene may be mixed and used.

The dispersoid (solid particles) thus prepared is used by 0.1 to 50 weight % to the entire electroviscous fluid.

It is recommendable to add polyhydric alcohol or its partial derivative in the electroviscous fluid of the present invention, as polarization promoting agent which promotes polarization of solid particles and increases electroviscous effect.

As the polyhydric alcohol, dihydric or trihydric alcohol such as ethylene glycol, diethylene glycol, triethylene glycol, tetraethylene glycol, polyethylene glycol, glycerine, propanediol, butanediol, pentanediol, hexanediol, etc. may be used.

As partial derivative of polyhydric alcohol, partial derivative of polyhydric alcohol having at least hydroxyl group, partial ether, in which some of terminal hydroxyl groups of the above polyhydric alcohol are substituted by methyl group, ethyl group, propyl group, alkyl-substituted phenyl group (The number of carbon atoms in alkyl group substituted with phenyl group is 1 to 25.), and partial ester, in which some of terminal hydroxyl groups are esterized by acetic acid, propionic acid, butyric acid, etc.

It is preferable to use such polyhydric alcohol or its partial derivative by 1 to 100 weight % of dispersoid, or more preferably by 2 to 80 weight %. If it is added by more than 1 weight %, ER effect is low. If it exceeds 100 weight %, it is not desirable because electric current easily flows. It is needless to say that water may be used together with the polyhydric alcohol in such degree as not to hinder ER effect.

Further, acid, salt or basic component may be added. As such acid component, inorganic acid such as sulfuric acid, hydrochloric acid, nitric acid, perchloric acid, chromic acid, phosphoric acid, boric acid, etc. or organic acid such as acetic acid, formic acid, propionic acid, butyric acid, isobutyric acid, valerianic acid, oxalic acid, malonic acid, etc. may be used.

As salt, compound comprising metal or basic radical (such as NH_4^+, $N_2H_5^+$) and acid radical can be used. Above all, it is preferable to use the compound, which is dissolved and dissociated in a system of polyhydric alcohol or partial derivative of polyhydric alcohol, e.g. the compound to form typical ionic crystal such as halide of alkali metal or alkali earth metal, or alkali metal salt of organic acid. As the salt of this type, there are LiCl, NaCl, KCl, $MgCl_2$, $CaCl_2$, $BaCl_2$, LiBr, NaBr, KBr, $MgBr_2$, LiI, NaI, KI, $AgNO_3$, $Ca(NO_3)_2$, $NaNO_2$, NH_4NO_3, K_2SO_4, Na_2SO_4, $NaHSO_4$, $(NH_4)_2SO_4$, or alkali acid metal salt of formic acid, acetic acid, oxalic acid, succinic acid, etc.

As base, hydroxide of alkali metal or alkali earth metal, carbonate of alkali metal, amines, etc. are used, and it is preferable to use the compound, which is dissolved and dissociated in a polyhydric alcohol, partial derivative of polyhydric alcohol or in a system of polyhydric alcohol and/or partial derivative of polyhydric alcohol and water. As the base of this type, there are NaOH, KOH, $Ca(OH)_2$, Na_2CO_3, $NaHCO_3$, K_3PO_4, Na_3PO_4, aniline, alkylamine, ethanolamine, etc., and the salt and the base as described above may be used together.

Acid, salt and base are to increase polarizing effect. When used in combination with polyhydric alcohol and/or partial derivative of polyhydric alcohol, the polarizing effect can be increased more, and these substances may be used by 0.01 to 5 weight % to the entire electroviscous fluid. If it is less than 0.01 weight %, ER effect is too low. If it exceeds 5 weight %, it is not desirable because electric current easily flows and power consumption increases. When acid, salt or base component is added to the electroviscous fluid of the present invention, it is necessary that partially esterized compound of polyhydric alcohol is not hydrolyzed.

To the electroviscous fluid of the present invention, antioxidant, corrosion inhibitor, anti-wear agent, extreme pressure agent, defoaming agent, etc. are added as additives when necessary.

The purpose of adding the antioxidant is to prevent oxidation of the electrically insulating fluid, and also to prevent oxidation of polyhydric alcohol, partial derivative of polyhydric alcohol, etc. used as polarization promoting agent.

As the antioxidant, the compound inactive to polarization promoting agent, dispersoid, etc., and phenol type or amine type antioxidant commonly used may be used. More concretely, phenol type antioxidant such as 2,6-di-t-butylparacresol, 4,4'-methylene-bis-(2,6-di-t-butylphenol), 2,6-di-t-butylphenol, or amine type antioxidant such as dioctyldiphenolamine, phenyl-α-naphthylamine, alkyldiphenylamine, N-nitrosodiphenylamine, etc. may be used. Such substance can be added to the electroviscous fluid by 0.01 to 10 weight %, or more preferably by 0.1 to 2.0 weight %. If it is less than 0.01 weight %, there is no antioxidation effect. If it exceeds 10 weight %, problems arise such as hue change, generation of turbidity or sludge, increase of consistency, etc.

Corrosion inhibitor may be added, and it is advised to use the substance inactive to polarization promoting agent, dispersoid, etc. More concretely, nitrogen compound such as benzotriazole and its derivative, imidazoline, pyrimidine derivative, etc. and the compound containing sulfur and nitrogen such as 1,3,4-thiadiazole polysulfide, 1,3,4-thiadiazoril-2,5-bisdialkyldithiocarbamate, 2-(alkyldithio)-benzoimidazole, etc., or β-(o-carboxybenzylthio)-propion-nitrile or propionic acid may be used. Such substance is used by 0.001 to 10 weight %, or more preferably by 0.01 to 1.0 weight %, to the entire electroviscous fluid. If it is less than 0.001 weight %, there is no corrosion inhibitive effect. If it exceeds 10 weight %, problems arise such as hue change, generation of turbidity or sludge, increase of consistency, etc.

In the following, the present invention is explained referring to some examples.

[Example 1]

(Preparation of dispersoid)

A mixture containing 10 g of silica particles (particle size: 1.4 μm), 20 ml of water, and 200 ml of dioxane is mixed for 60 hours in a ball mill. Then, 10 ml of γ-glycidoxypropyltrimethoxysilane was added, and the mixture was further mixed for 12 hours.

Solid particles were centrifuged (14,000 rpm; 60 min.) and were added to 300 ml of dioxane, and this was mixed for 5 hours in a ball mill. Then, 100 ml of dioxane was removed by distilling for 30 minutes.

To the remainder, 60 g of polyalkenyl succinimide and 100 ml of dioxane were added, and the mixture was refluxed for 10 hours.

After reflux, it was dispersed in toluene, and unreacted polyalkenyl succinimide was removed by centrifugation. After settling it for 12 hours, sedimentation was removed, and supernatant was dried under reduced pressure for 12 hours at 70 °C. Thus, 12 g of solid particles, to which polyalkenyl succinimide was bonded was obtained. The conversion ratio was 76%.

The silica particles were analyzed by infrared spectroscopy, gas chromatography, etc. to confirm that polyalkenyl succinimide was bonded to silica particles.

(Preparation of electroviscous fluid)

Electroviscous fluid having the following composition was prepared and was used as the specimen oil 1 (viscosity: 40 cSt (40 °C)):

(1)	Alkylbenzene (viscosity: 25 cSt (40 °C); specific gravity 0.88)	92.5 weight parts
(2)	Dispersoid prepared in the above	5.5 weight parts
(3)	Triethylene glycol	2.0 weight parts

[Example 2]

Dispersoid was prepared by the same procedure as in Example 1, except that γ-chloropropyltrimethoxysilane was used in the same quantity instead of γ-glycidoxypropyltrimethoxysilane,and electroviscous fluid (specimen oil 2) was prepared by the same procedure as in Example 1.

[Example 3]

(Preparation of dispersoid)

A mixture containing 10 g of silica particles (particle size: 1.4μm), 20 ml of water, and 200 ml of dioxane was mixed for 60 hours in a ball mill. Then, 10 ml of γ-glycidoxypropyltrimethoxysilane was added, and the mixture was further mixed for 12 hours.

Silica particles were centrifuged (14,000 rpm; 60 min.) and were added to 300 ml of dioxane, and this was mixed for 5 hours in a ball mill. Then, 100 ml of dioxane was removed by distilling for 30 minutes.

To the remainder, 60 g of polyalkenyl succinimide and 100 ml of dioxane were added. Further, 150 ml of n-butanol was added as catalyst and refluxed for 10 hours.

After reflux, it was dispersed in toluene and centrifuged to remove unreacted polyalkenyl succinimide. This was settled for 12 hours to remove sedimentation. The supernatant was dried under reduced pressure for 12 hours at 70 °C and 12 g of solid particles, to which polyalkenyl succinimide was bonded, was obtained. Conversion ratio was 76%.

The silica particles were analyzed by infrared spectroscopy and gas chromatography, and it was confirmed that polyalkenyl succinimide was bonded to silica particles.

Using the dispersoid thus prepared, electroviscous fluid (specimen oil 3) was prepared by the same procedure as in Example 1.

[Comparative example]

Electroviscous fluid [viscosity 40 cSt (40 °C)] having the following composition was prepared, and this was regarded as the comparative oil 1:

(1)	Alkylbenzene (viscosity: 25 cSt (40 °C); specific gravity 0.88)	89.0 weight parts
(2)	Silica gel (particle size: 1.4 μm)	4.0 weight parts
(3)	Triethylene glycol	2.0 weight parts
(4)	Succinimide	5.0 weight parts

After the specimen oil 1 and the comparative oil 1 were agitated well respectively, 12.5 g of each was placed into a test tube of 13 mm in inner diameter and 10 cm in height, and sedimentation quantity of porous solid particles was determined.

The results are shown in Fig. 1 with settling time (day) as axis of abscissa and height of sedimentation (mm) as axis ordinate.

As it is evident from the figure, sediment in the specimen oil 1 is very low and dispersion is excellent. When sedimentation quantity of porous solid paraticles was measured in the specimen oils 2 and 3 by the same procedure as in the specimen oil 1, the results were equal to or higher than the specimen oil 1. In particular, no precipitation occurred after about one year in the specimen oil 3.

Next, in order to evaluate electroviscous effect, the following measurements were executed on the specimen oils 1 to 3 prosecuted and the comparative oil 1 at 40 °C and 90 °C, using voltage-applicable rotational viscometer.

- Responsiveness: Evaluated by the (second) up to stabilization of viscosity when AC electric field is changed from 0 to 2.0×10^6 (V/m).
- Reproducibility: Evaluated by variation of viscosity at electric field of 2.0×10^6 (V/m) when AC electric field is subjected to repeated cycles of $0\rightarrow2.0\times10^6\rightarrow0$ (V/m).
- Durability: Evaluated by change (%) over time of viscosity AC electric field is kept constant at 2.0×10^6 (V/m); (Measuring time: 50 hours) (viscosity decrease occurs because sedimentation of porous solid particles is high.)
- Thickening effect: Evaluated by multiplication factor of viscosity when AC electric field is 1.4×10^6 - (V/m) compared with the time when electric field is 0 (V/m). The results are summarized in Table 1.

Table 1

		Responsiveness (sec)	Reproducibility (%)	Durability (%)	Thickening effect (×)
Specimen oil 1	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	98	10
Specimen oil 2	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	98	10
Specimen oil 3	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	97	10
Specimen oil 1	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	97	10

It is evident that the electroviscous fluid of the present invention is by no means inferior to the comparative oil in responsiveness, reproducibility, durability and thickening effect.

[Example 4]

(Preparation of dispersoid)

Under nitrogen flow, 10 g of silica particles (particle size: 1.4μm) and 300 ml of thionyl chloride were refluxed for 48 hours. Then, thionyl chloride in excess was removed under reduced pressure by distilling, and the remainder was further dried under reduced pressure at 140 °C for 60 hours.

On the other hand, 50 g of styrene was dropped into tetrahydrofuran containing 0.1 g of n-butyl lithium while maintaining reaction temperature at -78 °C, and anionic polystyrene (degree of polymerization: 250) having average molecular weight of 26,064 was obtained.

Into the anionic polystyrene thus obtained, chlorinated silica particles prepared in the above were mixed, and this was allowed to react for 18 hours. After reaction has been completed, small quantity of methanol was added to deactivate macromonomer.

The reaction dispersion system thus obtained was washed by three times by centrifugation using benzene to remove unreacted substances, and the particles were dried under reduced pressure. Invert ratio was 88%.

Then, particle size was examined under electron microscope, and it was the same before and after the reaction.

The silica particles were analyzed by IR and combustion TCD detection method, and it was confirmed that polystyrene was bonded to silica particles.

(Preparation of electroviscous fluid)

Electroviscous fluid having the following composition was prepared, and this was used as the specimen oil 4 (viscosity: 45 cSt (40 °C)):

(1)	Alkylbenzene (viscosity: 25 cSt (40 °C); specific gravity 0.88)	91.5 weight parts
(2)	Dispersoid prepared in the above	6.5 weight parts
(3)	Triethylene glycol	2.0 weight parts

[Comparative example 2]

Electroviscous fluid having the following composition (viscosity 45 cSt (40 °C)) was prepared, and this was used as the comparative oil 2:

(1)	Alkylbenzene (viscosity: 25 cSt (40 °C); specific gravity 0.88)	86.5 weight parts
(2)	Silica gel (particle size 1.4 μm)	5.5 weight parts
(3)	Triethylene glycol	2.0 weight parts
(4)	Succinimide	6.0 weight parts

By the same procedure as in Example 1, sedimentation quantity of porous solid particles was determined. The results are given in Fig. 2.

As it is evident from Fig. 2, the electroviscous fluid of the present invention is very low in sedimentation quantity and has superb dispersion property.

On the specimen oil 4 and the comparative oil 2, evaluation was made as electroviscous fluid by the same procedure as in Example 1. The results are summerized in Table 2.

Table 2

		Responsiveness (sec)	Reproducibility (%)	Durability (%)	Thickening affect (×)
Specimen oil 4	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	98	10
Specimen oil 2	40 °C	1 or less	±2	100	2
	90 °C	1 or less	±2	97	10

As it is evident from the table, the electroviscous fluid is by no means inferior to the comparative oil in responsiveness, reproducibility, thickening effect and durability.

INDUSTRIAL APPLICABILITY

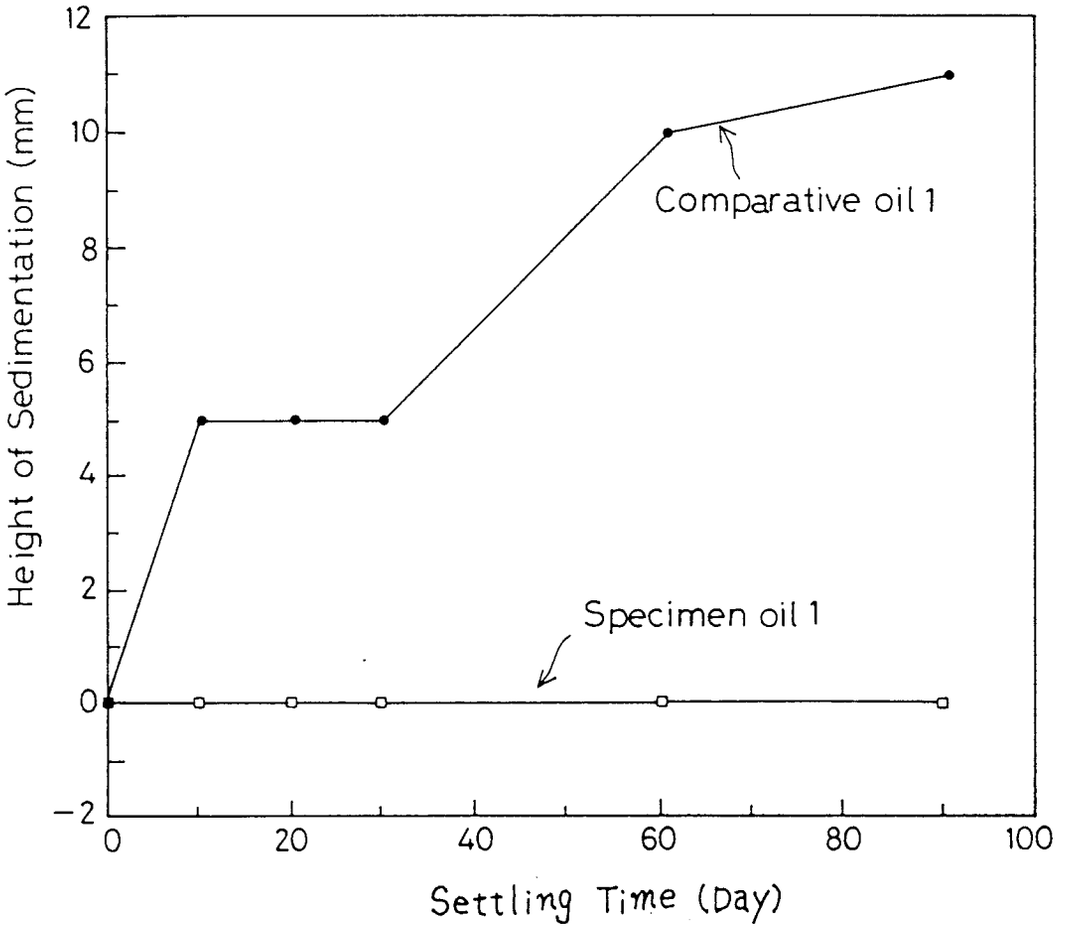
The electroviscous fluid of the present invention is an electroviscous fluid with improved dispersion property and can be applied in electrical control of vibration control machines and devices such as variable damper, engine mount, bearing damper, clutch, valve, shock absorber, precision machine, accoustic machine, etc. or electrical control of display element.

Claims

1. An electroviscous fluid, characterized in that solid particles are dispersed in an electrically insulating fluid, said solid particles have specific gravity equal to or higher than that of the electrically insulating fluid and have a hydroxyl group on surface thereof, and said hydroxyl group is bonded by covalent bond via silane coupling agent to a non-ionic surface active agent having hydroxyl group and/or -NH group as hydrophilic group.

2. An electroviscous fluid, characterized in that solid particles are dispersed in an electrically insulating fluid, said solid particles have specific gravity equal to or higher than that of the electrically insulating fluid and have hydroxyl group on the surface thereof, polystyrene and/or polyisoprene with polymerization degree of 5 to 320 is bonded to said hydroxyl group by covalent bond.

FIG. 1


12
10
8
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4
2
0
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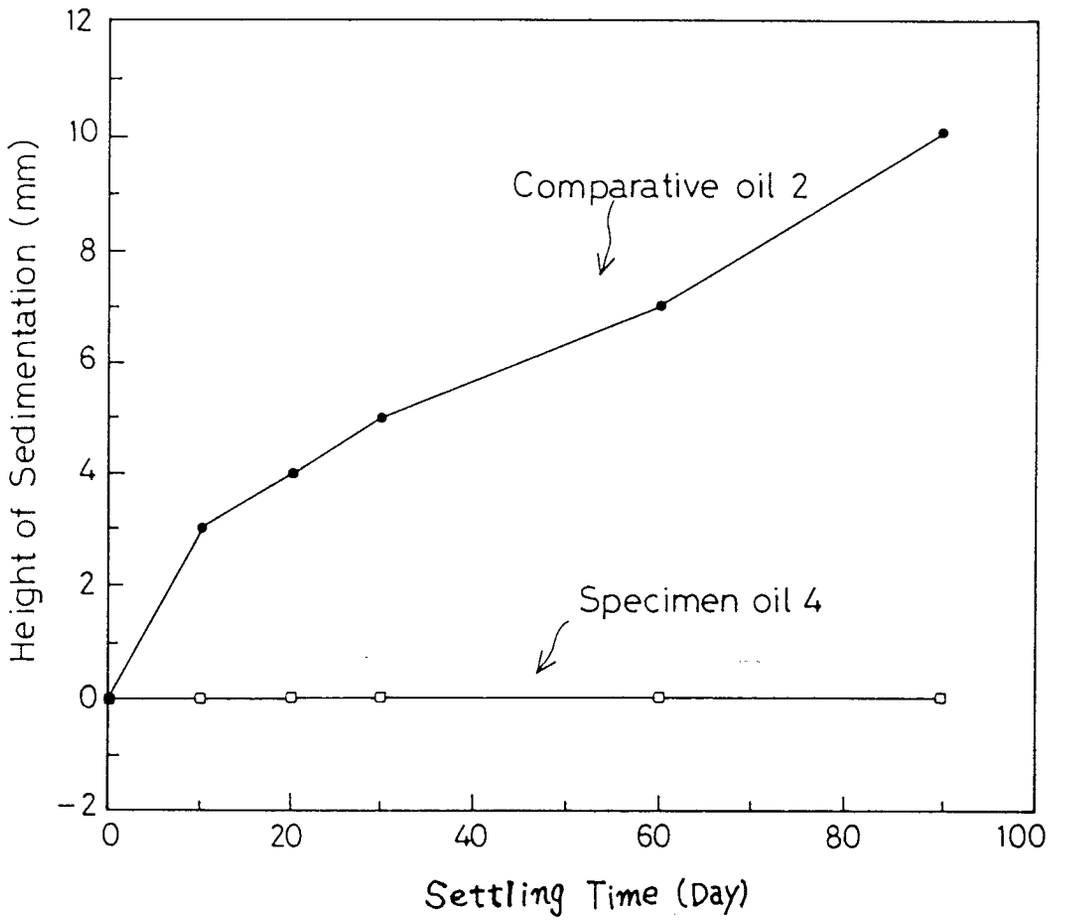
40
60
80
100
Height of Sedimentation (mm)
Settling Time (Day)
Comparative oil 1
Specimen oil 1

FIG. 2


12
10
8
6
4
2
0
-2
Height of Sedimentation (mm)
0
20
40
60
80
100
Settling Time (Day)
Comparative oil 2
Specimen oil 4

INTERNATIONAL SEARCH REPORT

International Application No PCT/JP92/00973

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int. Cl[5] C10M171/06, C10N40:14, 60:00

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
IPC	C10M171/06, C10N40:14, 60:00

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
Y	JP, A, 3-119098 (Asahi Chemical Industry Co., Ltd.), May 21, 1991 (21. 05. 91), Line 11, upper right column to line 18, lower right column, page 2 (Family: none)	2
A	JP, A, 3-137196 (Asahi Chemical Industry Co., Ltd.), June 11, 1991 (11. 06. 91), Line 12, upper left column to line 17, lower right column, page 3 (Family: none)	1
A	JP, A, 3-162494 (Ricoh Co., Ltd.), July 12, 1991 (12. 07. 91), Line 16, upper right column, page 2 to line 6, upper right column, page 4 (Family: none)	1
A	JP, A, 3-170600 (Tonen Corp.), July 24, 1991 (24. 07. 91), Line 19, upper right column to	1

* Special categories of cited documents: [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
September 21, 1992 (21. 09. 92)	October 13, 1992 (13. 10. 92)
International Searching Authority	**Signature of Authorized Officer**
Japanese Patent Office	

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

line 13, lower right column, page 2
(Family: none)

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers , because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers , because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers , because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.