I hereby apply for a patent on which is described an invention which is described in the following:

Title of Invention: Preparing a Particle Suspension in Emulsion

We request that this application be considered as a basic application in the countries on the list below:

- [ ] Country of Residence
- [ ] Country of Invention

Dated this [date]

William Street

My/Our Address

The Commissioner of Patents
The present invention relates to a method of preparing a stable suspension of micrroized solid particles in low viscosity liquid oil-in-water emulsion. This method thus provides a stable
COMMONWEALTH OF AUSTRALIA

DECLARATION IN SUPPORT OF A CONVENTION OR NON-CONVENTION
APPLICATION FOR A PATENT OR PATENT OF ADDITION

In support of the application No. (a) made by (b) CORIVAERK AS

for a patent/patent of addition for an invention entitled (c)
A METHOD OF PREPARING A STABLE SUSPENSION OF MICRONIZED SOLID
PARTICLES IN THE OIL PHASE OF AN OIL-IN-WATER EMULSION SUCH
AS A PIGMENTED STAIN

1. (d) Jørgen A. Houmann (Managing Director)
of (e) Birkemosevej 1
DK-6000 Kolding
Denmark

do solemnly and sincerely declare as follows:

1. (g) I am authorized by the abovementioned applicant for the patent/patent of
addition to make this declaration on its behalf.

2. The basic application(s) as defined by Section 141 of the Act were made in the
following country or countries on the following date(s) by the following applicant(s)
namely:

in (i) Denmark on (j) June 25 1976
by (k) CORIVAERK AS

in (l) on (m) 19
by (n) on (o) 19
by (p) on (q) 19

3. (l) I am/we are the actual inventor(s) of the invention.

4. The basic application(s) referred to in paragraph 2 of this Declaration were the
first application(s) made in a Convention country in respect of the invention the
subject of the application.

Declared at Kolding this 14th day of June 1977

To: The Commissioner of Patents,
Commonwealth of Australia.
thereafter. This method, however, is limited to
moment of application and for a very short period
low value favourable to the penetration at the very

PATENT OFFICE ACT.

INSPECTED AT THE
AND MAY BE
FOR REPRODUCTION
ARE UNSUITABLE
THIS APPLICATION
LODED WITH
DOCUMENTS
a stable emulsion or a stable emulsion and protective colloids, such as alkylaryl- and alkylsulfonates, amine and metal salts thereof, such as carboxylic acids and polycarboxylic acids.
(54) METHOD OF PREPARING A STABLE SUSPENSION OF MICRONIZED SOLID PARTICLES IN THE OIL PHASE OF AN OIL IN WATER EMULSION

(71) ORIVÆRKS AS

(21) 26 388/77 515 153 (22) 23.6.77

(23) 23.6.77 (24) 25.6.76

(31) 2886/76 (32) 25.6.76 (33) DK

(43) 4.1.79 (44) 19.3.81

(51) C09C 1/02 C09D 17/00 C09D 7/14 C09D 15/00

(72) Bentsen, A.T.

(74) HRA

(56) 46 292/72 473 499 B01F

53 481/64 291 144 B01F

18 982/62 270 528 B01F

(57) Claim 1. A method of preparing a stable suspension of micronized solid particles in the oil phase of an oil-in-water emulsion, wherein the micronized solid particles are ground in an oil phase in the presence of one or more dispersion aids capable of rendering the surface of the particles oleophilic and one or more steric and/or electrostatically stabilizing dispersion agents to form a stabilized suspension, after which said suspension is emulsified in an aqueous medium in the presence of one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.
example silicon dioxide, carbon black, aluminium powder and zinc dust, as well as organic compounds, for instance heliogen green, phthalocyanine blue, benzidine yellow and others.

In practice, the following steps are carried out:

1. Preparation of a suspension of solid particles in the oil phase of an oil-in-water emulsion using the invention described above.
2. The liquid to be polished is applied to the material to be polished, as a wood stain or the like, in the form of the suspension of solid particles in the oil phase of an oil-in-water emulsion.
3. After application of the suspension to the material to be polished, the liquid is allowed to penetrate into the material, thus forming a continuous film of the pigment on the surface of the material.

The invention is a method for preparing a stable suspension of micronized solid particles in the oil phase of an oil-in-water emulsion such as a pigmented stain. It is useful for protecting and finishing materials, particularly wood. The suspension is applied to the material to be polished and allows the liquid to penetrate into the material, forming a continuous film of the pigment on the surface.
The present invention relates to a method of preparing a stable suspension of micronized solid particles in low viscosity liquid oil-in-water emulsion. This method thus provides a stable suspension of solid particles in a low viscosity liquid, which does not have a paste-like consistency. The invention will be explained in greater detail below with reference to penetrating pigmented stains for protection of wood and wood products, said stains showing no tendency or only a slight tendency towards stratification or sedimentation of the pigments or other suspended solid particles, but may also be applied for example in the preparation of abrasives, polishing agents and cleaning agents.

When applying stain to a porous surface, such as a wood surface, one of the conditions of obtaining good penetration is a relatively low viscosity of the liquid compared to the rate of evaporation.

In practice, a short drying time is desired, and the absolute viscosity must consequently be low. If the stain is coloured wholly or partly by means of pigment particles, these particles will precipitate on standing according to Stokes' law, i.e. the rate of sedimentation is a function of particle size and viscosity. This implies that the stain must be strongly stirred before use, and such compositions are therefore usually sold in buckets and similar containers offering good stirring possibility. If the stain is not sufficiently stirred, colour faults will occur with the product.

It is known that colloidal dispersions, such as ink, insignificantly differ from mixtures; the only apparent preparation is the stirring and long time of standing.

Another, with addition of polar substances, is eliminated, be particularly the pigment which even be soluble. By said flocculation, the actual penetration time is reduced.

A third possibility is the viscosity of the oil phase, such as polyacrylate, for types of oil. The viscosity of the oil phase can be decreased by the addition of water-soluble or water-dispersible binders, for example polyacrylate dispersions, styrene-butadiene-polymer dispersions or polyvinylacetate dispersions.
occur with regard to the tint and colour intensity when the
product is used.

It is known to obviate this problem in several ways:

The simplest method is a reduction of the particle size to
colloidal dimensions whereby the sedimentation will be
insignificant even after long periods of time. This method
is only applicable to specific expensive products as the
preparation of such a pigment dispersion is very costly.

Another, widely used method is flocculation of the pigment by
addition of so-called "anti-settling agents", like various
colloidal silicic acid, etc. for types of stain based on organic solvents. In this manner
viscosity may be kept high on standing while attaining the

For preparing, by way of example, a pigmented stain, a paste
of the following composition will generally be ground first:
low value favourable to the penetration at the very moment of application and for a very short period thereafter. This method, however, is limited to the cases where very high shear forces arise during the application process, for example in application by brush, and provides no possibility of obtaining sufficiently deep penetration, as the lowering of viscosity is in practice of very short duration.

It is the object of the present invention to provide a method that does not suffer from the abovementioned drawbacks and where, as far as stains are concerned, a product having good penetration ability and good protective effect can be obtained, said product showing no appreciable tendency towards stratification or sedimentation of the pigment even on prolonged standing. More generally, it is the object of the invention to provide stable suspensions of micronized solid particles in the oil phase of an oil-in-water emulsion without noticeable tendency towards sedimentation.

This object is achieved by the method according to the invention which is characterized in that the micronized solid particles in the oil phase of an oil-in-water emulsion wherein the micronized solid particles are ground in an oil phase in the presence of one or more dispersion aids capable of rendering the surface of the particles oleophilic and one or more sterically and/or electrostatically stabilizing

**EXAMPLE 1**
Preparation of a pigmented stain.
dispersion agents to form a stabilized suspension, after which said suspension is emulsified in an aqueous medium in the presence of one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents.

and 30 g of 15% polyvinylmaleic acid alkylglycolesterammonium salt ("Thickner LN"), which is an anionic stabilizer acting also as chelating agent and thickener and which was first incorp

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and said dispersion aid or aids. Thus, the particles are incorporated and retained in the internal phase of a stable oil-in-water emulsion whose external continuous phase is thus composed of a liquid of low viscosity.

It has thus surprisingly been found that partly by imparting suitable oleophilic surface properties to the particles and partly by providing suitable steric and/or electrostatic forces between the particles and between the particles and the boundary layer of the water phase of the emulsion, stable suspensions may be obtained showing no tendency or only an extremely slight tendency towards sedimentation of the particles on standing.

Methods having, on the face of it, certain points of similarity to that described, but proving on close analysis to display decisive differences and which especially do not aim at or lead to products having the long-term stability obtainable by the method according to the invention are described in a number of publications:

German Offenlegungsschrift No 22 12 521 describes a method of preparing an electrophoresis bath in emulsion form in which a pigment suspension in a water-dissolved completely neutralized carboxylic acid is added to the bath. However, the measures stated aim at retaining the pigment in the water phase, and the method is not aimed at the preparation of products with long-term stability.

By way of comparison identical stains have been prepared, the dispersion aid characteristic of the invention, however, being omitted. In all cases the pigments were precipitated...
Swedish Patent No 139,292 teaches a method of preparing resin emulsions applicable for example as glue or for fixation of pigments on textiles. In Example 5 copper-phthalocyanine blue is distributed in a concentrated emulsifier solution to which is further added emulsifying agent, water, and an amino resin emulsion. Said emulsion, thus containing no pigment in the internal phase of the emulsion as the amino resin emulsion is prepared and added separately, is further thickened by an emulsion of petrol in an emulsifier solution of the kind mentioned above. Neither in this case must the pigment be expected in any way to become incorporated in the emulsion internal phase. The features characteristic of the invention are thus not at all present here.

Swedish Patent No 135,667 teaches a method of preparing an emulsion concentrate which must be thinned immediately before use, and the long-term stability is consequently unimportant. More exactly, the object is to prepare emulsions where water-insoluble protectants are dissolved in the internal phase by a method which endeavours to eliminate their possible negative impact on the emulsion stability on account of interaction with the external phase by means of a delicate stepwise correction for these interactions.

The possibility of dispersed active substances is nowhere described more specifically, and there is no mention of the conditions of possibly stabilizing a substance in the internal phase against sedimentation. Hence, there are no points of similarity to the characteristic features of the invention.

and was stable even in case of great additional thinning.
German Offenlegungsschrift No 22 56 757 discloses an emulsion which is particularly applicable as a spray and which may be of the water-in-oil type as well as the oil-in-water type, particles of a substance with antibacterial, insecticide, herbicide or vegetation-modifying activity being dispersed in one phase.

These liquids are ostensibly more stable than known products of the same kind, however their stability has only been documented for 48 hours, and it is particularly emphasized that the products are readily redispersible in contrast to the known products.

The specific feature of the spray is partly the adjustment of the density ratio and volume ratio between the phases within precisely defined limits, and partly the selection of a suitable non-ionic surfactant which is hydrophobic or hydrophilic, depending on whether the particles are desired to be present in the oil phase or in the water phase.

The possibility of establishing in this way, for particles of substances of the type mentioned, a stability or at least redispersibility sufficient for the purpose of application cannot be precluded, but the tests of the applicant have proved that said measures are quite inadequate for a long-term stability of pigmented oil-in-water emulsions.

Thus, none of the above-mentioned publications mention the combination characteristic of the method according to the invention.
invention of a dispersion aid capable of rendering the particle surface oleophilic, a dispersion agent capable of stabilizing the internal phase of the emulsion, and an emulsifier system compatible therewith which is capable of stabilizing the emulsion itself.

In an oil-in-water emulsion of the kind described in the present invention the retention of the particles in the internal phase and hence the stability of the suspension may be obtained, according to the invention, more specifically in the following manner:

To render the particles, for example the pigment particles, oleophilic, i.e. they are moistened more readily by oil than by water, they are ground according to the invention in the presence of suitable surfactants so that they are brought into said oleophilic state, which is a condition of the actual incorporation into the oil phase.

In this place and in the following these agents are called dispersion aids and may also, as far as pigments are concerned, be called pigment wetting additives.

According to the invention this may be effected by treatment with one or more surfactants selected from the group of non-ionic agents, such as alkyl polyethoxylates and alkyl arylpolyglycol ethers; ampholytic agents, such as electron-neutral salts of cation-active groups, e.g. salts of fatty amines with fatty acids or polycarboxylic acids; anion-active agents, arylsulfonic acid salts, alkyl arylsulfonic acid salts, individually or in combinations or mixtures to effect the desired dispersion.

These agents of dispersion aids and may also, as far as pigments are concerned, be called pigment wetting additives.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

agents, such as fatty alcohol sulphates, sulfinides, alkylarylsulfonates, especially alkylbenzene sulfonates and alkyl naphthalene sulfonates, phosphate esters, metal naphthenates and metal salts of fatty acids; and cation-active agents, such as quaternary ammonium compounds, alkylimidazole salts and lecithin. These agents may also impart to the individual particles an electric surface charge which will contribute, if the original potential (zeta potential) of the particle is not sufficiently great, to an anti-floculating effect owing to electrostatic repulsion. Thus, for example, the pigment particles from the preparatory process or the storing are often surrounded by a water film which must be displaced at the grinding process in the presence of the surfactant or surfactants to obtain good dispersion. In addition to this change of the surface properties of the particles, the formed suspension of particles in the oil phase may be further stabilized according to the invention by adding to the oil phase during the grinding agents producing electrostatically active forces between the particles. These may be selected from the same types as the above-mentioned dispersion aids imparting oleophilic properties, apart from the non-ionic.

It has been found, however, that the best stability of the suspension is obtained if adding to the oil phase during the grinding oil-soluble agents which may also perform steric stabilization. Such agents are for example surface active soluble polymers with polar character, such as polymers of oxidized, unsaturated fatty acids and esters or ethers thereof, such as modified vegetable oils and fatty acids and/or esters, or has been used, for example, about 0.1 to 1, or even up to 2wt%.

It should be noted that the surfactant or surfactants just described, in addition to their surface active characteristics, are preferred to be cationic or anionic. Such agents include, for example, quaternary ammonium compounds, ampholytic agents, anion-active agents, and lecithin.

2. A method according to claim 1, wherein said dispersion aid imparting oleophilic properties comprises one or more surfactants selected from the group of non-ionic agents, ampholytic agents, anion-active agents, and cation-active agents.
thereof, partly polymerized esters of polycarboxylic acids, such as alkyds, and styrene-, isocyanate- and silicone-modified varieties thereof, acryl-modified and/or epoxidized oils and resins, aldehyde condensation products with amines and/or phenols, and polyethers and polyvinyl derivatives. It has been found that effective stabilization is generally first observed in polymers having a molecular weight of about 1500 and increases with increasing molecular weight up to about 10,000, after which the effect may diminish.

It should be noted that a number of the said types of surfactants perform both electrostatic and steric stabilization, just as they may affect the particle surface itself, and according to circumstances the same or several different surfactants may therefore be used in addition to the dispersion aid.

The concrete selection of surfactants depends, inter alia, on the type of particles and the desired end product and may be determined by the person skilled in the art through tests. A preferred combination in the preparation of pigmented stains is a dispersion aid containing a hydrophobic group of cation-active character, such as an amine, possibly in the form of an amine salt, especially with a hydrophobic anion (electron-neutral salt), or in the form of an amine ester, such as a lecithin, in combination with a sterically stabilizing surface polymer mentioned above.

It is of vital importance to the favourable progress of the

1. A method according to claim 3, wherein said alkylaryl sulfonates are selected from the group consisting of alkylbenzene sulfonates and alkyl-naphthalene sulfonates.
of the method that the particles are incorporated in a stable emulsion. This stability is provided by addition of one or more emulsifiers which produce steric and/or electrostatic forces between the individual particles of the internal phase (the oil phase) of the emulsion. In this connection it is of vital importance that said stabilization of the emulsion is not effected at the expense of the stability of the suspension in the internal phase, and it is therefore necessary to select emulsifiers that do not affect the stability of the oil phase produced by the dispersion agents and dispersion aids.

These emulsifiers, which may be of ionogenic or non-ionic type, can be added to one or both phases of the emulsion. As far as ionogenic (cationic or anionic) types are concerned, the electrostatic repulsion between the internal phase drops is of substantial importance to the stabilization, while the effect of non-ionic types depends primarily on steric repulsion and is extensively pH-independent. The stability is further affected by the distribution of the surfactant between the two phases, and especially in the case of non-ionic types a so-called HLB value (hydrophilic-lipophilic balance) is often calculated for selection of the emulsifier which is best suited for a given type of oil.

To obtain electrostatically active forces in the stabilization of the emulsion there may be added one or more surfactants acting in the external phase and in the boundary layer to the internal phase and selected from the group of ionogenic

8. A method according to claims 1 or 2, wherein said dispersion aid imparting oleophilic properties
emulsifiers and protective colloids, such as alkyl-aryl- and alkylsulfonates, amine and metal salts hereof, such as calcium alkylaryl sulfonates, carboxylic acids and polycarboxylic acids and their salts (soaps), such as polyacrylic acid salts with ammonia, amines and alkali metals, polyvinyl carboxylic acids and partial esters and/or salts thereof, cellulose derivatives as well as polycondensed inorganic acids and salts hereof, such as sodium hexametaphosphate and lithium polysilicate.

To obtain sterically acting forces when stabilizing the emulsion there may be added one or more ionogenic surfactants of the type described above under electrostatic stabilization and/or non-ionic agents, such as fatty acid- and fatty alcohol polyglycol ethers, polyethoxylated alkyl phenols, polycarboxylic acid esters and ethers, such as fatty alcohol succinates, sorbitol esters and ethers, sorbitan ether polyethoxylates, polyvinyl alcohols, polyethylene oxide and ethers and esters hereof as well as cellulose derivatives, such as hydroxyethyl cellulose.

It has been found that a combination of a non-ionic emulsifier of the correct HLB value (for example of the alkylphenol polyethylene oxide adduct type) added to the internal phase, and a minor amount of anionic surfactant of the protective colloid type, for example a polyacrylate or a polyvinylmaleinate, added to the external phase is particularly suitable for stabilization of the emulsion itself as it functions satisfactorily for example in the suspension of pigments in the internal

15. A method according to claim 11 wherein a water soluble or water dispersible pigment binder is further added to the emulsion.
phase of emulsions of vegetable oils and their synthetic analogs or derivatives thereof and does not affect the particle stability in the inner phase adversely. Said anionic component may also act to adjust a suitable viscosity and for chelation of heavy metal ions which may otherwise as contaminants destroy the electrostatic stabilization, however these functions may also be performed by separate components of cationic, anionic or non-ionic nature, such as sodium tripolyphosphate.

To improve stability, especially when the suspension is employed in highly diluted state involving a risk of stratification in the case of great differences in density, there may also according to the invention be added to the oil phase one or more density-equalizing agents, including light liquids, such as aliphatic and aromatic hydrocarbons, and heavy liquids, such as halogenated hydrocarbons, for example ethylene dichloride, tris-2,3-dibromo propylphosphate and chloro paraffins. It is also possible to add solid substances, for example light substances, such as wax and microscopic hollow glass balls (microspheres), and heavy substances, such as solid chloro paraffin and antimony trioxide. These may serve at the same time as flameretardants.

Applicable pigments are for example oxides and insoluble salts of earth alkali metals and metals in the transition series, for example iron oxides, chromium oxides, zinc oxide, barium sulphate, titanium dioxide, calcium carbonate, aluminium and magnesium silicate; other oxides and elements, for example zinc dust, heliogen green and perylene critical to the oil.

Finally, other components may be in the form of pyrethroid, and derivatives and pyridin-thi insecticide agents and pigment binder. Oxidative distribution is very small.
example silicon dioxide, carbon black, aluminium powder and zinc dust, as well as organic compounds, for instance heliogen green, phthalocyanine blue, benzidine yellow and perylene red. The type of pigment, however, is not critical to the invention.

Finally, other protectants of various kinds may be added to the oil or water phase, and these may for example also be in the form of micronized solid particles. Examples of other protectants include biocides, such as fungicides, for instance tetrachloroiso-phthalonitrile, copper-8-hydroxyquinoline, tributyl tin oxide and derivatives thereof; preservatives, such as sodium pyridin-thion-1 and 2,2-dibromoglutarodinitrile; insecticides, such as lindane or endosulfane, IR-reflecting agents and specific UV-absorbing agents, just as oil soluble pigment binders may be added to the oil phase, for instance oxidative drying, possibly modified oils. However, it has surprisingly been found that the requirement for specific UV-absorbing agents in the pigmented stains described is very small as they cause extremely good penetration and distribution of the pigment in the treated wood surfaces.

To the water dispersible styrene-butadiene dispersions, catalyst (sic) and the equilibrium external phase, desired a sticative.

The stable pigmented stain invention presents for oil-in-water emulsions a precise and simple manner by mixing first the oil phase, as well as by the method is eliminate co-flocculation. The mixture within wide not least on the oil-in-water phase by weight of 30 percent b...
To the water phase can be added water soluble or water dispersible binders, for example polyacrylate dispersions, styrene-butadiene-polymer dispersions or polyvinylacetate dispersions. If desired, there may also be added an oxidation catalyst (siccative) which is complexed in a form so that the equilibrium concentration of the free catalyst in the external phase is so small that the stabilizing system of the emulsion is not affected noticeably hereby, and, if desired, a stabilizer and/or an antioxidant to stabilize the siccative.

The stable pigment suspensions prepared according to the invention present the additional advantage that they are well suited for obtaining reproducible colour tints. When co-grinding pigments of different colours in the oil phase a co-flocculation has previously been observed, which impedes a precise and reproducible shading. It has been found that by mixing finished pigment suspensions of the type described as well as by grinding together the pigments in the oil phase by the method according to the invention, this co-flocculation is eliminated, whereby a desired tint can be obtained in a simple manner.

The mixture proportion between the constituents may vary within wide limits depending on the desired end product and not least on the kind of solid particles.

The oil-in-water emulsion may thus contain from 1 to 70 percent by weight of oil, but will usually contain from about 20 to 30 percent by weight.
For preparing, by way of example, a pigmented stain, a paste of the following composition will generally be ground first:

\[
\begin{align*}
&1 - 75 \text{ parts by weight of pigment} \\
&0.1-10 \text{ " " " dispersion aid (pigment wetting agent)} \\
&5 - 45 \text{ " " " stabilizing agent (stabilizer)} \\
&0 - 45 \text{ " " " density-equalizing agent}
\end{align*}
\]

the sum of the constituents being 100 parts by weight.

For 50 parts by weight of this paste the following is subsequently used:

\[
\begin{align*}
&0 - 650 \text{ parts by weight of pigment binder} \\
&10 - 50 \text{ " " " emulsifier (stabilizing agent for the emulsion)} \\
&0 - 5 \text{ " " " anticracking and stabilizer therefore} \\
&0 - 10 \text{ " " " biocides and other secondary materials.}
\end{align*}
\]

This oil phase is emulsified in a water phase which may contain:

\[
\begin{align*}
&0.1 - 0.5 \text{ parts by weight of stabilizer which may also perform other functions (calculated as active substance)} \\
&0 - 400 \text{ " " " pigment binder, however not exceeding 50 percent by weight of the water phase,} \\
&0 - 10 \text{ " " " biocides and other secondary materials}
\end{align*}
\]

as well as demineralized water up to 1000 parts by weight.

The invention is illustrated in greater detail by means of the examples stated below:
EXAMPLE 1
Preparation of a pigmented stain.

By dispersion on a pearl mill there was prepared an orange paste by grinding 40 parts by weight of transparent iron oxide red (e.g. "VN 188", which is an orange hydrophilic pigment sold by Siegle & Co.), 5 parts by weight of dispersion aid (pigment wetting agent) e.g. "Disperbyk" which is an electro-neutral salt prepared by reacting an alkylol amine salt with a polycarboxylic acid, sold by Byk-Mallinckrodt, and 55 parts by weight of soybean oil alkyd having an oil length of 65% (sterically stabilizing dispersion agent for the pigment). The dispersion took place at ambient temperature to a particle size of max 20 μm.

50 g of this paste was mixed at ambient temperature for about 10 minutes with 200 g of 100 percent (solvent-free) linseed oil alkyd having an oil length of about 80% which acts as binder, 12 g of nonylphenoldodecaethoxylate (sterically stabilizing non-ionic emulsifier), 1.2 g of cobalt naphthenate (6% Co - siccative), 0.8 g of a 38% solution of o-phenanthroline in butyl glycol, sold by Vanderbilt Co. under the trade name "Activ 8" (stabilizer for siccative against hydrolysis) as well as 2 g of methylethylketoxime (volatile antioxidant acting as anti-skinning agent).

The 266 g mixture thus obtained, which may be regarded as a fluid oil paint, was dispersed at ambient temperature for about 10 minutes in a mixture of 210 g of demineralized water and 30 g of salt ("TiO₂ water. By dispersion was added which binder was also added.

The stain was thinned by 12 cP, pH 7.2 and at least applied to the test material in summer. It is stable for at least 5 months. For example, test conditions.

In the summer with other pigments and phthalate lecithin "Texaphob®" and castor oil obtained...
and 30 g of 15% polyvinylmaleic acid alkylglycolester ammonium salt ("Thickener LN"), which is an anionic stabilizer acting also as chelating agent and thickener and which was first mixed for about 5 minutes with another 60 g of demineralized water. By said process there was obtained 566 g of pigment dispersion having the character of an emulsion paint to which was added while stirring 434 g of demineralized water, to which biocides and other protective agents may have been added.

The stain (1000 g) thus obtained has at 20°C a viscosity of 12 cP, pH 7.8, specific gravity 1.083. It is easily prepared and at low cost, it has excellent penetration ability when applied to wood surfaces and causes no air pollution in use. It is stable on standing and shows no tendency towards sedimentation of the pigment, even after a storing period of 14 months. The stain also has excellent freeze-thaw stability. For example, no product changes were observed after a standard test comprising 3 freezings to -18°C with intermediate thawings.

In the same manner stains have been prepared using pigments with other colours, such as "VN 088", yellow iron oxide pigment and "VN 288" red iron oxide pigment, carbon black and phthalocyanine blue, and other dispersion aids, such as "Texaphor 963" (Texaphor is a Registered Trade Mark), "Stabilizing Electro Neutral Pigment Wetting Agent" and lecithin, as well as other dispersion agents, such as tall oil alkyds. Stains of the same excellent quality were obtained.
By way of comparison identical stains have been prepared, the dispersion aid characteristic of the invention, however, being omitted. In all cases the pigments were precipitated after standing for a short period.

The protective effect of a number of stains prepared as stated above for treatment of wood surfaces has also been tested. Under accelerated test conditions as regards weather the products described have exhibited a surprisingly high degree of weather resistance and have proved to be superior to current types of stain on alkyd-solvent base as well as on acryl dispersion base under otherwise identical conditions as regards degree of pigmentation and concentration of binder, etc.

EXAMPLE 2
Preparation of a pigmented stain.

By dispersion on a pearl mill a black paste was prepared from (Printex is a Registered Trademark), 15 parts by weight of carbon black ("Printex 300")/4 parts by weight of bentonite (thickener, 10% in white spirit), 1 part by weight of dispersion aid ("Disperbyk"), 35 parts by weight of linseed oil alkyd (dispersion agent), and 45 parts by weight of white spirit (density-equalizing agent).

50 g of this paste was treated analogously with the paste prepared in Example 1, and there was obtained a stable stain having a specific gravity of 1.03, pH 7.8, and viscosity 12 cP. Said stain also showed excellent penetration ability.
and was stable even in case of great additional thinning.

EXAMPLE 3

A polish was prepared by grinding together 1 part by weight of diatomite, 8 parts by weight of dearomatized white spirit, 1 part by weight of alkyd, 0.1 parts by weight of linoleyltrimethylene diamine-dioleate (dispersion aid), 0.5 parts by weight of polyethylene glycol dioleate (PEG 400-dioleate), which was subsequently emulsified in 89.4 parts by weight of water containing 0.0025 parts by weight of polyacrylic acid ammonium salt "Acrysol ASE95" (anionic stabilizer). The obtained odourless composition was stable on prolonged standing with no tendency towards sedimentation.
1. A solid where the spirit is diluted by water, glycerol, or other liquids by weight or volume or a solid or a dispersion where the solid is dispersed by weight or volume in an acid medium.
THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method of preparing a stable suspension of micronized solid particles in the oil phase of an oil-in-water emulsion, wherein the micronized solid particles are ground in an oil phase in the presence of one or more dispersion aids capable of rendering the surface of the particles oleophilic and one or more sterically and/or electrostatically stabilizing dispersion agents to form a stabilized suspension, after which said suspension is emulsified in an aqueous medium in the presence of one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.

2. Dispersion of the suspension of ionic cational surfactants with one or more emulsifiers that do not affect the stability of the oil phase produced by said dispersion agent or agents in the aqueous medium.

3. A method of preparing a stable suspension of micronized solid particles in the oil phase of an oil-in-water emulsion, wherein the micronized solid particles are ground in an oil phase in the presence of one or more dispersion aids capable of rendering the surface of the particles oleophilic and one or more sterically and/or electrostatically stabilizing dispersion agents to form a stabilized suspension, after which said suspension is emulsified in an aqueous medium in the presence of one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.

4. A method of preparing a stable suspension of micronized solid particles in the oil phase of an oil-in-water emulsion, wherein the micronized solid particles are ground in an oil phase in the presence of one or more dispersion aids capable of rendering the surface of the particles oleophilic and one or more sterically and/or electrostatically stabilizing dispersion agents to form a stabilized suspension, after which said suspension is emulsified in an aqueous medium in the presence of one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.
2. A method according to claim 1, wherein said dispersion aid imparting oleophilic properties comprises one or more surfactants selected from the group of non-ionic agents, ampholytic agents, anion-active agents, and cation-active agents; and said stabilizing dispersion agent comprises one or more of the abovementioned surfactants or one or more surface active soluble polymers with polar character.

3. A method according to claim 2, wherein said non-ionic agents are selected from the group consisting of alkylpolyethoxylates and alkylarylpolyglycol ethers; said ampholytic agents are electroneutral salts of cation-active and anion-active groups selected from the salts of fatty amines with fatty acids or polycarboxylic acids; said anion-active agents are selected from the group consisting of fatty alcohol sulfates, sulfimides, alkylaryl sulfonates, phosphate esters, metal naphthenates and metal salts of fatty acids; said cation-active agents are selected from the group consisting of quaternary ammonium compounds, alkylimidazole salts and lecithin; said surface active soluble polymers with polar character are selected from the group consisting of polymers of oxidized unsaturated fatty acids and esters or ethers thereof, partly polymerized esters of polycarboxylic acids, acryl-modified and/or epoxidized oils and resins, aldehyde condensation products with amines and/or phenols and polyethers and polyvinyl derivatives.
4. A method according to claim 3, wherein said alkylarylsulfonates are selected from the group consisting of alkylbenzene sulfonates and alkyl-naphthalene sulfonates.

5. A method according to claims 3 or 4, wherein said partly polymerized esters of polycarboxylic acids are selected from the group consisting of alkyds, and styrene-, isocyanate-, and silicone-modified varieties thereof.

6. A method according to claim 1, wherein said dispersion aid imparting oleophilic properties comprises a compound containing a hydrophobic group of cation-active character, and said dispersion agent comprises a sterically stabilizing surface active polymer having a molecular weight of from 1,500 to 10,000.

7. A method according to claims 1 or 2, wherein said dispersion aid imparting oleophilic properties comprises a compound containing a hydrophobic group of cation-active character selected from the group consisting of an amine, an amine salt, an amine salt with a hydrophobic anion (electroneutral salt) and an amine ester.
8. A method according to claims 1 or 2, wherein said dispersion aid imparting oleophilic properties comprises lecithin.

9. A method according to claim 1, wherein a non-ionic emulsifier is added to the oil phase for stabilization of the emulsion and a minor amount of an anionic surfactant of the protective colloid type as hereinbefore defined is added to the water phase.

10. A method according to claims 1, 2 or 9, wherein an agent equalizing the difference in density between the internal and external phases is further added to the oil phase.

11. A method according to claim 1, wherein one or more pigments are used as micronized solid particles.

12. A method according to claim 11, wherein an oil soluble pigment binder is further added to the oil phase.

13. A method according to claim 12, wherein an oxidative drying oil is used as pigment binder.

14. A method according to claim 13, wherein the oxidative drying oil is a modified oil.
15. A method according to claim 11, wherein a water soluble or water dispersible pigment binder is further added to the emulsion.

16. A method according to claim 15, wherein a pigment binder on acrylic base is used.

17. A method according to any one of claims 1, 2 or 9, wherein there is further added an oxidation catalyst (siccative) which is complexed in a form so that the equilibrium concentration of the free catalyst in the external phase is so small that the stabilizing system of the emulsion is not noticeably affected hereby.

18. A stable suspension comprising micronized solid particles in the oil phase of an oil-in-water emulsion, one or more dispersion aids capable of rendering the surface of the particles oleophilic, one or more sterically and/or electrostatically stabilizing dispersion agents and one or more emulsifiers of a type that does not effect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.
19. The suspension of claim 18, with the proviso that the oil-in-water emulsion contains from about 20 to about 30 percent by weight of oil, based on the weight of the entire composition.

20. A stable pigmented stain comprising micronized pigment particles in the oil phase of an oil-in-water emulsion, one or more dispersion aids capable of rendering the surface of the pigment particles oleophilic, one or more sterically and/or electrostatically stabilizing dispersion agents and one or more emulsifiers of a type that does not affect the stability of the oil phase produced by said dispersion agent or agents and dispersion aid or aids.

21. The stain of claim 20, with the proviso that the oil-in-water emulsion contains from about 20 to about 30 percent by weight of oil, based on the weight of the entire composition.

22. Pigmented stain, prepared by the method according to claims 1-17.

23. Pigmented paint, prepared by the method according to claims 1-17.

24. Polish or abrasive, prepared by the method according to claims 1-10.

DATED THIS 31st DAY OF JANUARY, 1981.

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By Its Patent Attorneys

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