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(54) Title: NON-SETTLING, UNIVERSAL MACHINE DISPENSABLE PEARLESCENT PIGMENT DISPERSIONS**(57) Abstract**

An aqueous, non-settling, universal nacreous pigment dispersion that can be used in dispensing machines for the custom mixing of retail and trade sales of alkyd or solvent-thinned and water-based surface coatings employs a synthetic silicate, sodium lithium magnesium silicate, as the suspending agent. Also disclosed are methods for preparing the dispersion which incorporates one or more carriers, humectants, surfactants and other optional conventional additives.

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**NON-SETTLING, UNIVERSAL MACHINE
DISPENSABLE PEARLESCENT PIGMENT DISPERSIONS**

Field of the Invention

The invention relates to an improved nacreous or
5 pearlescent pigment dispersion that is machine
dispensable for in-store tinting of aqueous and solvent-
based surface coatings.

Background of the Invention

In order to permit paint manufacturers to offer an
10 almost limitless selection of colors to their retail and
trade customers, colorant dispensing machines have been
developed to permit the so-called "in-store" tinting of
pigmented or clear bases at the point of sale. Advanced
colorant dispensing machines have been developed that
15 include up to twelve, or more, colorant reservoirs into
which each colorant is poured, and a metered pumping and
measurement system for dispensing the required amount of
one or more colorants into various sized containers of
base. Computer-controlled dispensing machines have
20 reached a level of sophistication so that only the code
associated with a desired end color need be entered, and
the machine draws from the associated memory in selecting
the colorant(s) and volume(s) required for dispensing
into the container of base paint.

25 Once filled with its complement of pigment
dispersions, the dispensing machine may not be used for
extended periods of time, i.e., from overnight to longer

holiday periods. Furthermore, particular colors or types of pigment dispersions may not be frequently used.

Of principal concern in any such system is the ability to reproducibly and consistently obtain precisely the 5 same shade and/or tint of mixed paint. Since the pigments in the colorant dispersions have a tendency to settle, the colorant reservoirs or canisters are typically equipped with intermittent stirrers, and the dispensing mechanism includes a recirculating loop that 10 permits the pigment dispersion to be periodically pumped from the reservoir through the loop and returned to the reservoir.

However, a certain portion of the colorant dispersion can remain in the dispensing nozzle between the valve in 15 the circulating loop and the nozzle orifice. In addition, the stirring and recirculating pump systems cannot totally prevent the separation and accumulation of pigments that have a tendency to settle from the colorant dispersions. However, even small changes in the 20 composition of the colorant caused by settling or separation of the pigment dispersion can result in noticeable variations in custom tinted paints. Various measures have been proposed by dispensing machine designers to minimize the settling and drying of the

pigment dispersions once they are loaded into the machines.

In USP 5,493,840, a circular trough containing water is positioned proximate the outlets of the dispensing nozzles for the purpose of providing an atmosphere that is high in water vapor in order to inhibit the drying of the colorant dispersion in the nozzles. This apparatus further complicates an already complex piece of equipment.

10 In order to reduce nozzle blockage, Canadian patent document 2,118,484 discloses a stem affixed to a valve, which in the closed position occupies the tip of the nozzle and also allows circulation of the pigment dispersion through a bypass port.

15 Since these mechanical features are not available for existing dispensing machines that have been in use for sometime, solutions to the problems of settling and nozzle blockage must be met by formulating the pigment dispersions sold for dispensing from these machines.

20 Universal colorant dispersions, i.e., those that are suitable for use with either aqueous or solvent-based surface coatings, have been well-known in the art for several decades. Universal pigment dispersions containing nacreous or pearlescent pigments are also 25 well-known to the art. Nacreous pigments, i.e.,

pearlescent pigments, are used for decorative effect. The original "pearl essence" pigments were derived from fish scales and tissue. Limited supplies of this natural resource led to the development of nacreous basic lead carbonate in the 1930's. It is not known whether the natural product is commercially available today. Inorganic nacreous pigments are available in a variety of forms and are produced most commonly by coating a naturally occurring platelet of mica or other mineral clays with titanium dioxide, an iron oxide, or both. As used herein the terms "nacreous" and "pearlescent" are understood to be synonymous.

A wide variety of anti-settling or suspension agents have been employed in preparing pearlescent pigment oxide dispersions for use in surface coatings to reduce the tendency of the pigments to settle. Commercially acceptable formulations include at least three such agents. These suspension or anti-settling agents can include bentonite and other organo-clay mineral (hectorite) products, magnesium aluminum silicate and hydroxyethyl cellulose. The formulations employing these anti-settling agents are used for in-plant tinting of pre-mixed surface coatings where settling during shipment and storage prior to use is not a concern. This is because the shipping drums containing the pigment

dispersions are equipped with efficient agitators that can maintain the dispersions as a reasonably uniform mixture and any remaining pigment can be scraped from the empty drum and added to the batch of base.

5 The number of colors of ready-mixed pearlescent paints must necessarily be limited by the costs of warehousing and retail shelf storage and display. Pearlescent pigment dispersions are also available in artist supply stores, but are sold only in small tubes and are 10 relatively expensive.

A wide variety of chemical compounds have been developed for use as suspension agents and for preparing thixotropic compositions. For example, it is known from USP 5,270,399 to employ the synthetic silicate LAPONITE® 15 RD, which is a thixotropic agent, in preparing finished surface coating compositions employing colored pigment dispersions in water dispersible base coat compositions.

It has also been known to use synthetic silicate compositions including those sold commercially under the 20 trademark LAPONITE® in the automotive industry with metallic and pearlescent pigments. However, it is understood that the automotive finishes are also prepared in large batches that are comparable to the in-plant manufacture of surface coatings.

However, prior to this invention, it had been found that pearlescent colorant dispersions had a tendency to settle much more rapidly than conventional dispersions and efforts to produce a non-settling nacreous pigment dispersion were unsuccessful. For this reason, 5 pearlescent dispersions have not been available for use in the dispensing machines used for in-store tinting.

Some of the advantages and capabilities that are provided by a universal machine dispensable pearlescent 10 pigment include (1) ready availability to consumers and to the trade at an affordable price; (2) a greatly expanded range of colors and tints; (3) the availability of custom colors for use in connection with faux and fantasy finishing techniques for residential and 15 commercial decorating or redecorating to achieve effects that have not heretofore been possible.

It is therefore an object of this invention to provide a non-settling, universal, machine dispensable pearlescent colorant dispersion.

20 It is a further object of the invention to provide a method and process for the manufacture of a non-settling, universal, machine dispensable pearlescent dispersion.

It is another object of the invention to provide a 25 nacreous or pearlescent pigment dispersion that can be employed in existing dispensing machines used for the

custom tinting of aqueous and solvent-thinned paint bases.

It is yet another object of this invention to provide such a dispersion and method for its manufacture that 5 requires only one thixotropic agent to effect a non-settling nacreous pigment composition.

Another object of the invention is to provide a universal dispersion that contains no volatile organic content ("VOC"), as defined by current government 10 environmental regulations.

Summary of the Invention

In accordance with the objectives of the invention an improved non-settling, universal nacreous pigment dispersion for use in dispensing machines for addition to 15 surface coatings comprises a nacreous pigment component and a water-containing liquid carrier for said pigment component, where the liquid carrier component includes a dispersion of hydrous sodium lithium magnesium silicate as a thixotropic agent for suspending the pigment. The 20 invention thus provides a nacreous or pearlescent pigment dispersion which eliminates the need for blending a plurality of suspending agents, and instead employs only a single additive for this function.

Moreover, the method of preparing the improved pearlescent pigment dispersion is simple and efficient and insures a consistent and acceptable product. The method also minimizes shear stress applied to the 5 pigments which are subject to mechanical damage and loss of reflectivity.

The method of the invention comprises (a) forming a mixture of the hydrous sodium lithium magnesium silicate in a major portion of the water used to formulate the 10 liquid carrier; (b) agitating the mixture at high speed for 10 to 20 minutes to effect full hydration of the silicate; (c) adding the remaining ingredients comprising the liquid component, which can include surfactants, a vehicle, at least one humectant, a biocide and defoamer; 15 (d) adding the pearlescent pigment component; (e) adjusting the pH of the composition to 8-9; and (f) continuing to mix the ingredients at a low shear until a uniform dispersion is formed.

In accordance with one embodiment of the invention, a 20 universal, non-settling, machine dispensable pearlescent pigment dispersion is prepared by forming an aqueous mixture of an anti-settling agent that is a synthetic silicate, namely, hydrous sodium lithium magnesium silicate, which synthetic silicate is capable of forming 25 a stable aqueous sol, and adding to said aqueous mixture

the following: a C₂ or C₃ olefinic glycol or polyglycol, a combination of surfactants compatible with aqueous and solvent-based or alkyd surface coatings, a viscosity reducer, a biocide, an optional defoamer, an aqueous 5 alkali solution as required to adjust the pH of the dispersion to a pH of about 8-9, pearlescent oxide pigments having a particle size in the range of from about 1 to about 180 microns, but preferably in the range of from about 10 to about 60 microns, and a surfactant 10 for wetting the pearlescent pigments.

The invention also contemplates a conventional commercial dispensing machine adapted for the in-store tinting of latex and alkyd-based surface coatings having a plurality of dispersion canisters, in which at least 15 one of said canisters contains the improved pearlescent pigment dispersion as described above.

The pigments having utility in the invention are commercially available from several sources in the United States. One useful type of pigment consists of flat mica platelets coated with titanium dioxide, iron oxide or a combination of these oxides. The length of the platelets range from about 5 microns to 10 microns; the larger particles produce relatively greater reflectance, or 20 pearlescent effect, and less opacity, and the smaller

particles produce more of a satin luster having greater opacity.

Suitable pigments are sold by the following companies:

5 BASF of 3000 Continental Drive Mt. Olive, NJ 07828 under the brand name "Paliocrom";

EM Industries, Inc. of 5 Skyline Drive, Hawthorne, NY 10532 under the trademark AFFLAIR;

10 Mearle Corporation of 41 E. 42nd Street New York, NY 10017, under the trademark "Mearlin Luster Pigment"; and by

Semo Chemical Company, Ltd of Korea, whose products are distributed by Royale Pigments of 12 Route 17N, Paramus, NJ 07652 under the trademark "Semo Pearl Pigments".

15 Other forms of pearlescent pigments are known in the art. For example EP-A-0 268 918 and EP-A-0 342 533 disclose titanium dioxide pigments having a hydrated zirconium oxide coating that exhibit improved weathering properties in non-aqueous surface coatings. A silicon dioxide coated pearlescent pigment is disclosed in USP 20 5,472,491, where the base pigment can be a platelet shaped mica, kaolin or glass that is coated with titanium dioxide, iron oxides (II)/(III), chromium oxide, zirconium dioxide, tin dioxide or zinc oxide, and has 25 particular utility in water-borne coating systems.

Nacreous pigments derived from natural sources such as fish scales and tissue are also believed to be suitable for use in the practice of the invention.

Pigment oxides useful in the invention are those that produce metallic effects, including silver, gold and bronze, as well as those producing so-called earthtone colors. The dramatic effects created by the incorporation of these pigments are best achieved in surface coating bases that do not contain titanium dioxide pigments. The maximum effect is obtained in mass tone applications where the base is clear.

The pigments should be mixed using a mixer or agitator with low shear forces to avoid or minimize fracturing of the platelets. Mixing should be discontinued once a uniform dispersion has been obtained.

The thixotropic or suspension additive which has been found to be most effective as an anti-settling agent is hydrous sodium lithium magnesium silicate. When formulated in accordance with the invention, use of this synthetic silicate will maintain the nacreous pigments in the universal dispersion and permit use of the dispersion in dispensing machines for in-store mixing with appropriate bases. As used hereinafter the term "synthetic silicate" will be understood to mean hydrous sodium lithium magnesium silicate. It has properties which permit the formation of stable aqueous sols which resist[s] gelling, or which can be maintained as a flowable liquid under low shear conditions. A synthetic

silicate composition of this type is sold under the brand name LAPONITE® by Southern Clay Products of 1212 Church Street, Gonzales, TX 78629.

The grade of the LAPONITE® silicate which has been found to be satisfactory for use in the invention is LAPONITE-RD. A second form of this synthetic silicate containing a peptiser, or peptising agent, is available under the designation LAPONITE-RDS. The preparation of one form of this synthetic silicate composition is described in USP 3,654,176. The viscosity modifying properties and storage capabilities of the peptiser-containing silicate identified as LAPONITE®-RDS were found to provide no particular advantages in the practice of the invention.

In the examples which follow, the synthetic silicate is added to tap water in a vessel equipped with a Cowles mixer and agitated for approximately 15 minutes in order to effect complete hydration of the synthetic silicate. Immediately thereafter, the remaining ingredients of the formulation are added, while continuing agitation. The order of addition can include an additional carrier or vehicle; one or more humectants, such as diethylene glycol monomethyl ether and hydroxyethylethylene urea ("HEEU"); conventional surfactants of the types customarily employed in universal dispersions, to include

a surfactant to facilitate wetting and blending of the nacreous pigment or pigments; a biocide; an anti-foam agent and the pigment(s). As certain surfactants, such as nonyl phenol, and its ethoxylates, are known to cause 5 foaming, its addition can be delayed until after addition of the pigments. The pH of the composition is brought to about 8-9 using a dilute aqueous alkali, such as a 50% potassium hydroxide solution. Mixing is continued for the minimum time required to achieve a uniform 10 composition.

Suitable formulations for preparing universal non-settling nacreous pigments for use in dispensing machines can contain ingredients in the ranges indicated, all in parts by weight:

15	Water	1-50 parts
	Synthetic silicate	0.1-2.0 parts
	Carrier (glycol)	8-50 parts
	Humectant	3-15 parts
	Surfactants	1-15 parts
20	Nacreous Pigment(s)	1-50 parts
	Base (for pH adj.)	As needed
	Biocide (optional)	0.05-0.2 parts
	Defoamer	As needed

In a preferred embodiment of the invention that is 25 adapted to provide a dispersion of pearlescent pigments for incorporation in architectural finishes and surface coatings of both the alkyd and water-borne types by way of in-store dispensing machines, the non-settling

dispersion comprises the following ingredients in the ranges indicated all being in part by weight:

	Water	35-50 parts
5	Synthetic silicate	1.0-2.0 parts
	Carrier (glycol)	8-15 parts
	Humectant	3-8 parts
	Surfactants	7-10 parts
	Nacreous Pigment(s)	10-33 parts
10	Base (for pH adj.)	As needed
	Biocide (optional)	0.05-0.2 parts
	Defoamer	As needed

Under present regulatory standards, ethylene glycol and propylene glycol are deemed to contribute to the VOC of products in which they are incorporated. Polyethylene glycol ("PEG") and higher polyclycols can be substituted for some or all of the other glycols to reduce or eliminate these VOC constituents. PEG having an average molecular weight of from about 200 to about 700 can be utilized.

20 The following universal nacreous pigment dispersions were prepared using pigments that produced a silver and gold pearlescent effect when the dispersions were added to water-borne and alkyd clear bases to which other colorant dispersions were also added.

25	<u>Component</u> <u>(Gold)</u>	<u>Quantity</u>	
		<u>A (Silver)</u>	<u>B</u>
	Water	44.0	44.0
	Synthetic silicate ¹	1.6	1.6
30	Carrier (propylene glycol)	10.0	10.0
	Humectant ²	5.0	5.0

Surfactants			
	Soya lecithin	3.0	3.0
	Cationic ³	2.0	2.0
	Nonyl phenol ⁴	2.0	2.0
5	Pigment surfactant ⁵	2.0	2.0
	Biocide ⁶	0.1	0.1
	Anti-foam	0.2	0.2
	Nacreous Pigment	30.0 ⁷	30.0 ⁸

10 RD) ¹ Hydrous sodium lithium magnesium silicate (Laponite®
 2 Diethylene glycol monomethyl ether
 3 Polyoxyethylated alkylamine
 4 Add after pigment with 2-3 parts reserved water
 15 5 Potassium salt of phosphate coester
 6 NUOCEPT® 95 (Huls America)
 7 AFFLAIR® 103 (EM Industries)
 8 AFFLAIR® 309 (EM Industries)

20 The nacreous pigment dispersions of Examples A and B
 were discharged from the mixing vessel into friction top
 cans and sealed. After three weeks of heat aging at
 120°F and at ambient temperature, the containers were
 opened and some syneresis was observed. No settling was
 evident, and a uniform dispersion was produced with
 25 thirty seconds of hand stirring.

Thereafter the dispersions of Examples A and B are put
 into the reservoir canister of a standard dispensing
 machine.

30 Pearlescent pigment dispersions comparable to those of
 Examples A and B were added to the clear or transparent
 bases indicated below and thoroughly mixed with the
 conventional universal colorants indicated to produce
 surface coating compositions:

Water Based

One gallon container (116 fluid ounces) of high gloss clear

Ten ounces of pearlescent dispersion - Example A

5 Two ounces of colored pigment dispersion - thalo blue COLORTREND® 888 (Hüls America)

Alkyd/Solvent-Based

10 One gallon container (116 fluid ounces) of high gloss clear

Ten ounces of pearlescent dispersion - Example B

Two ounces of colored pigment dispersion - bone black GPD® 824 (Hüls America)

15 The surface coating compositions were applied with brush, roller and by spraying to wall surfaces to produce an iridescent or lustrous effect comparable to that obtained by the use of factory-mixed pearlescent paints.

20 The surface coatings containing the pearlescent pigment dispersions of the invention can be applied to previously painted walls and other surfaces to create faux or fantasy effects that have not previously been available to trade painters and interior decorators, let alone to the do-it-yourself category of home decorators.

25 The surface can be painted with a pastel midtone, accent or deep color, which is preferably in a flat finish. Best effects and results are achieved by using

transparent or semi-transparent colors, such as phthalo blue, phthalo green, red, black, especially bone black, violet, magenta, van dyke brown, and transparent red and yellow oxides. Inorganic colors can also be utilized, 5 but have a tendency to mask or reduce the pearlescent effects.

The surface coating composition containing the pearlescent dispersion of the invention can be applied over the base coat by sponging, rag rolling, bagging, 10 block printing, feathering and stenciling. The finished painted surface can be made to appear to be a seamless wallpaper. Unlike wallpaper which must generally be removed when replacement or redecorating is desired, the faux finishes incorporating the pearlescent dispersions 15 of my invention can simply be painted over with a flat base or primer.

Claims

1. An improved pearlescent universal pigment dispersion composition for use in surface coatings, which pearlescent pigment dispersion composition includes (a) a pearlescent pigment component and (b) a water-containing liquid component that is a carrier for said pigment component, the improvement which comprises a dispersion in said liquid carrier component of hydrous sodium lithium magnesium silicate that is a thixotropic agent for suspending the pearlescent pigment.

2. The composition of claim 1 which contains from about 10 to about 33 parts by weight of component (a), and component (b) comprises:

- (i) from about 1.0 to about 2.0 parts by weight of hydrous sodium lithium magnesium silicate;
- (ii) from about 1 to 50 parts by weight of water;
- (iii) from about 8 to about 50 parts of a glycol selected from the group consisting of ethylene glycol, diethylene glycol, polyethylene glycol and propylene glycol; and

(iv) from about 1 to about 15 parts by weight of one or more surfactants.

3. The composition of claim 1 where component (a) comprises metal oxide-coated mica.

4. The composition of claim 1 which contains from about 25 to 30 parts by weight of component (a) and from about 1.3 to 1.6 parts by weight of hydrous sodium lithium magnesium silicate.

5. The composition of claim 1 having a pH in the range of from about 8 to 9.

6. The composition of claim 1 which further comprises one or more of the following additives: a viscosity reducing additive, a biocide, a defoamer a pH control agent.

7. A method of preparing a universal, non-settling nacreous pigment dispersion for use in in-store dispensing machines, said method comprising the steps of:

(a) dissolving from about 1.0 to about 2.0 parts by weight of hydrous sodium lithium

magnesium silicate in from about 35 to 50 parts by weight of water to form a solution;

- (b) while agitating the solution of step (a), adding the following:
 - (i) from about 8 to about 12 parts by weight of one or more glycols selected from the group consisting of ethylene glycol, diethylene glycol, polyethylene glycol and propylene glycol,
 - (ii) from about 7 to about 10 parts by weight of one or more surfactants, and
 - (iii) from about 10 to about 33 parts by weight of nacreous pigments;
- (c) adjusting the pH of the mixture of step (b) to the range of 8 to 9; and
- (d) continuing to agitate the mixture of step (c) until a dispersion of uniform consistency is formed.

8. The method of claim 7 where step (b) further comprises the step of adding from about 3 to about 8 parts of a humectant selected from the group consisting of diethylene glycol monomethyl ether and hydroxyethylethylene urea.

9. The method of claim 7 where step (b) further comprises the step of adding from about 0.1 to about 0.3 parts by weight of a defoaming agent.

10. The method of claim 9 which further includes after step (c) adding of from about 1.5 to about 2.5 parts of nonylphenol.

11. The method of claim 7 where the nacreous pigments are metal oxide-coated mica.

12. A surface coating composition comprising (a) a base selected from the group consisting of alkyd bases and aqueous bases, (b) from about one percent to about ten percent by volume of the pearlescent pigment dispersion of claim 1, and (c) from about 0.2 percent to about 2 percent by volume of a colorant dispersion.

13. An improved aqueous nacreous universal pigment dispersion composition for use in colorant dispensing machines that is non-settling which dispersion composition comprises (a) a minor amount by weight of said composition of at least one nacreous pigment and (b) a minor amount by weight of said composition of hydrous

sodium lithium magnesium silicate that is sufficient to form a thixotropic mixture.

14. The dispersion composition of claim 13 in which the at least one nacreous pigment comprises metal oxide coated mica.

15. The dispersion composition of claim 14 where the nacreous pigments comprises mica platelets coated with titanium dioxide, or iron oxide, or both.

16. A universal non-settling nacreous pigment dispersion for use in dispensing machines, said dispersion comprising:

- (a) from about 1 to about 50 parts by weight of nacreous pigments;
- (b) from about 0.1 to about 2.0 parts by weight of a synthetic silicate that is a thixotropic agent for suspending the nacreous pigments;
- (c) from about 1 to 50 parts by weight of water;
- (d) from about 8 to about 50 parts of a glycol carrier selected from the group consisting of ethylene glycol, diethylene glycol,

polyethylene glycol and propylene glycol;
and

(e) from about 1 to about 15 parts by weight of
one or more surfactants.

17. The nacreous pigment dispersion of claim 16 where
the nacreous pigments comprise metal oxide-coated mica.

18. The nacreous pigment dispersion of claim 17 where
the mica is coated with titanium dioxide or iron oxide,
or both.

19. The nacreous pigment dispersion of claim 16 in
which the synthetic silicate composition is hydrous
sodium lithium magnesium silicate.