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FLUSHING PIGMENTS

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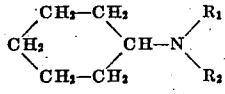
10 Claims. (Cl. 106—262)

The present invention relates to improvements in the manufacture of non-aqueous pigment dispersions in a liquifiable vehicle, and particularly to the transfer of aqueous pigment pulps directly into organic vehicles by an operation or process known to those skilled in the arts as "flushing." It has special reference to agents for effecting and/or facilitating the transfer.

It is well known that some pigments may be flushed with oil without an added flushing agent. For example, aqueous white lead is readily flushed into linseed oil by simply mixing the two materials. Pigments in this class are usually hydrophobic in character. It is also known that certain agents facilitate the flushing operation, or even bring it about in the case of certain combinations of pigment and receiving vehicle. Pigments which do not flush readily or which do not flush without an added flushing agent are usually hydrophilic in character. Various explanations are given for the success of such agents or class of agents and for the failure of chemically similar compounds. Some of these explanations involve the physical nature of the molecule as well as its chemical reactivity.

The present invention is based upon the discovery that a group of amines not heretofore known as flushing agents, are useful as such. The value of the invention lies in the facts that only a small quantity is needed to effect the transfer, and that the flushing agent is relatively inexpensive.

The object of the present invention is to use as flushing agents, amines or their soaps with fatty acids, having the essential grouping



where R_1 and R_2 may be hydrogen, methyl, ethyl, phenyl or cyclohexyl groups or combinations thereof.

It is also an object of the invention to form pigment characterized by the presence of a cyclohexylamine flushing agent.

Other and ancillary objects and advantages will appear from the following description and explanation of the invention.

The invention may be used with both inorganic and organic pigments. The act of flushing involves intimate mixing of the aqueous pigment pulp and the receiving vehicle at a considerable expenditure of time and power. A flushing agent quickens the process and saves time and money. There is a frequent tendency of the vehicle and the water to emulsify in such operations. Hastening the process minimizes emulsification. The formation of emulsion decreases the efficiency of the flushing operation, because more water is retained in the mass, which water must be removed by other means. One use of the flushing agent is to minimize or prevent the formation of emulsion.

Pigments particularly suitable for flushing in accordance with this invention are iron blues, chrome yellows, organic lakes and pigment dyestuffs.

The vehicle may be any water-immiscible liquid or solid which is or becomes liquid at the temperature of operation. It may be selected from coal tar solvents, mineral spirits, or hydrogenated naphthas, representing the volatile vehicles, or from linseed oil, China wood oil, soya bean oil, perilla oil, other vegetable and animal oils, oleoresinous varnishes or synthetic varnishes representing the drying vehicles, or mineral oil, petrolatum, castor oil or waxes, representing the non-drying vehicles. All these may be used in the present invention. The drying oils ordinarily have an acid number, representing a content of fatty acid which may form a soap with the amine flushing agent. The formation of the soap does not render the amine ineffective, for the amine agent may be added effectively as a soap or as the free amine. The amine may be condensed with soap-forming fatty acids such as oleic, stearic, palmitic, linolenic, or ricinoleic acid, or with soap-forming resin acid, such as rosin or abietic acid, to form the amine soap flushing agent.

Because the free amine or its soaps may be used with vehicles which are volatile or which possess no acid number, it is known that both the free amine and its soaps are flushing agents. While soap formation is not essential for the practice of this invention, slight acidity in neutral vehicles adds to the effectiveness of the amine flushing agent, where the acid is of a soap-forming character. Thus, fatty acids, or other soap-forming acids may be added.

Examples are given below to illustrate this invention. While these examples deal mainly with iron blue pigments, its usefulness is not restricted to such. Iron blue (ferric ferrocyanide) is highly hydrophilic and is one of the most difficult pigments to employ in a flushing process. Therefore, the invention is illustrated largely by reference to it, as it is the severest test found to measure the comparative effectiveness of the flushing agents.

EXAMPLE I.—*Printing base ink*

	Pounds
Water-wet iron blue presscake containing 500 lbs. of the pigment (dry basis)	2000
Litho varnish (a heat-bodied linseed oil varnish)	500
Cyclohexylamine	13

About one-third of the iron blue presscake and 250 lbs. of litho varnish are mixed in a machine of the dough-mixer type, equipped for vacuum drying, heating and cooling. Any device that gives intimate mixing will serve the purpose. To this mixture is then added 7 lbs. of cyclohexylamine and mixing continued. In a short time, usually less than twenty minutes, the water in the presscake will separate or "flush out" and the iron blue is taken up by the varnish-oil vehicle. The water is decanted off or removed by other means, and about one-third of the remaining presscake is added to the mixer. 50 lbs. of litho varnish and 2 lbs. of cyclohexylamine are also added. On further mixing, more water is flushed out. The remaining presscake is flushed out in two charges in the above manner, using 2 lbs. of cyclohexylamine and 50 lbs. of litho varnish in each charge. When all the presscake is flushed, steam is turned on and the temperature of the mass raised to 50°–60° C. Vacuum is applied to the mixer and mixing is continued under reduced pressure until the pigment-oil paste is substantially water-free. The mass is cooled and the remaining 100 lbs. of litho varnish added to make a base ink containing one part of pigment to one part of varnish. This is a typical procedure for the practice of this invention, but it is not limited to such. Those skilled in the art may vary it for their particular need. Instead of vacuum drying, the paste may be finished on a roller mill. The time cycle for this procedure is about four hours.

It is not necessary to conduit the process in stages as above described, but such procedure is more efficient than in conducting the entire operation on all the materials, as is well known. The removal of water may or may not remove some of the flushing agent. Some of the forms are soluble in water, but also more soluble in the flushing vehicles, so that with the amounts given for the procedure given, there is sufficient of the agent present.

EXAMPLE II

	Pounds
Water-wet iron blue presscake containing 600 lbs. of the pigment (dry basis)	2400
Linseed oil	600
N-methyl cyclohexylamine	20

The procedure is similar to that of Example I.

EXAMPLE III

	Pounds
Water-wet iron blue presscake containing 500 lbs. of the pigment (dry basis)	2000
Castor oil	500
Cyclohexylamine	12

Procedure is similar to that of Example I.

EXAMPLE IV

	Pounds
Water-wet phospho-tungstate rhodamine lake containing 600 lbs. of the pigment (dry basis)	2000
Litho varnish	600
N-ethyl cyclohexylamine oleate	4

Procedure is similar to that of Example I.

EXAMPLE V

	Pounds
Water-wet peacock blue (a standard organic lake) presscake containing 500 lbs. of the pigment (dry basis)	2000
Mineral oil	500
Cyclohexylamine oleate	25

Procedure is similar to that of Example I. The mineral oil and cyclohexylamine oleate are added, as needed, to flush the pigment.

EXAMPLE VI

	Pounds
Water-wet light chrome yellow presscake containing 1500 lbs. of the pigment (dry basis)	3000
Cyclohexylamine	10
Linseed oil	600

Procedure is similar to that of Example I.

EXAMPLE VII

	Pounds
Water-wet iron blue presscake containing 600 lbs. of the pigment (dry basis)	2000
Mineral spirits	600
Glycerol-phthalate varnish, 100% solids	800
Cyclohexylamine	25

The pigment is flushed in the glycerol-phthalate varnish with cyclohexylamine similar to Example I. The mass is vacuum dried and cooled, and the mineral spirits added.

EXAMPLE VIII

	Pounds
Water-wet toluidine red toner presscake containing 600 lbs. of the pigment (dry basis)	2000
Ester gum varnish (100% solids)	600
Mineral spirits	400
Phenyl-cyclohexylamine oleate	10

EXAMPLE IX

	Pounds
Water-wet Lithol Red R presscake containing 500 lbs. of the pigment (dry basis)	2000
Japan wax	500
Dicyclohexylamine ricinoleate	15

Procedure is similar to that of Example I.

EXAMPLE X

	Pounds
Water-wet iron blue presscake containing 600 lbs. of the pigment (dry basis)	2500
Litho varnish	600
N-dimethyl cyclohexylamine	25

Procedure is similar to that of Example I.

The foregoing examples establish that the amine may be primary, secondary, or tertiary, so long as it has at least one cyclohexyl radical. This establishes that the effective grouping is $C_6H_{11}N=$ in a free amine or in an amine soap. The agents may have solubility in water, but are generally more soluble in the liquid vehicle. The removal of water from the mass after a flushing operation may remove some of the agent. However, where the operation is conducted in one step, its main function has been performed. Where the operation is carried out in several steps, more of the agent is added, so that any removal of some of the agent in the withdrawn water cannot be effective to deplete the process of the agent.

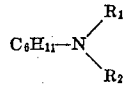
The pastes prepared in the manner described are suitable for the manufacture of all types of protective and decorative finishes and for allied uses.

The quantity of flushing agent required for a given result varies with the apparatus used, the pigment, the pulp concentration, the receiving vehicle and other factors. It is a small quantity, commonly less than 5% of the dry weight of pigment.

The practice may be varied in accordance with the scope of the invention as expressed in the appended claims.

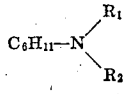
I claim:

1. The method of flushing pigment particles from aqueous paste form into a liquid vehicle immiscible with water to release water, which comprises agitating a mass of aqueous pigment paste with a receiving liquid vehicle which is immiscible with water, in the presence of a small quantity of a cyclohexylamine compound having the grouping therein with the formula



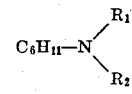
in which R_1 and R_2 may be selected from the group consisting of hydrogen, and the radicals: methyl, ethyl, phenyl and cyclohexyl.

2. The method of flushing pigment particles from aqueous paste form into a liquid vehicle immiscible with water to release water, which comprises agitating a mass of aqueous pigment paste with a receiving liquid vehicle which is immiscible with water, in the presence of a small quantity of a cyclohexylamine compound of the formula



in which R_1 and R_2 may be selected from the group consisting of hydrogen, and the radicals: methyl, ethyl, phenyl and cyclohexyl.

3. The method of flushing pigment particles from aqueous paste form into a liquid vehicle immiscible with water to release water, which comprises agitating a mass of aqueous pigment paste with a receiving liquid vehicle which is immiscible with water, in the presence of a small quantity of a cyclohexylamine soap of a cyclohexylamine of the formula



in which R_1 and R_2 may be selected from the group consisting of hydrogen, and the radicals: methyl, ethyl, phenyl and cyclohexyl, and a soap-forming acid selected from the group of soap-forming resin acids and soap-forming fatty acids.

4. The method according to claim 1 in which the receiving liquid contains a drying vehicle for pigment.

5. The method according to claim 1 in which the receiving liquid is a non-drying vehicle for pigment.

6. The method according to claim 1 in which the receiving vehicle is volatile.

7. The method according to claim 1 in which one of the attachments R_1 and R_2 is hydrogen.

8. The method according to claim 1 in which the compound is cyclohexylamine.

9. The method of claim 1 in which the receiving liquid is litho varnish.

10. The method of claim 1 in which the pigment is iron blue.

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