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METALLIC POWDER AGGREGATE

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This invention relates to the production of metallic pigments in a form convenient for mixing with the desired vehicle, which effects a more efficient covering power and leafing power of the metallic powder, eliminates the dusting nuisance of metallic pigments in their customary powdered state, forms a product stable physically so that it does not separate into two or more phases, and possesses the property of retaining its original leafing power without appreciable loss indefinitely. Moreover, the apparent density is very much greater than that of either the metallic powders or of the pastes which now constitute commercial products, thus effecting economies in storage and shipping.

It possesses the further important advantage that it can be used in any vehicle desired, whether varnish, lacquer or aqueous vehicle. Because of its substantially dry condition, economies in packing are secured, for instead of cans, some variations of the new product can be shipped in a wrapper of metallic foil or coated paper, etc. In addition, another object of this invention is, to combine with the above mentioned properties that of ready miscibility with the customary vehicles. When brushed out in a film after mixing with a vehicle, the product shows a smooth brilliant surface with a satisfactory color. Another important advantage of this invention is that powders, which when mixed with 30 to 40% of liquid ingredient to form a paste which soon loses its original leafing power, may be used according to this method to form stable dustless aggregates, in which the leafing power remains indefinitely in its original degree.

Heretofore, metallic pigments have been produced in two forms; powders and pastes containing about 35% liquid. The objections to the powders are: they dust, they do not wet easily or completely, they form paints of various kinds which are likely to contain lumps, and, because of the difficulty of complete wetting, a considerable proportion of the inherent leafing and covering powers are not utilized. The objections to the pastes are: they often separate into two phases or layers, they often possess a transient leafing power probably due to the solution of some or all of the polishing film, which is non-metallic in character, by the liquid added to form a paste, in which event the original leafing power may decrease to a small fraction of that obtained when freshly mixed.

I have found that when polished metallic powders which may vary in size from 200 to 325 or more per linear inch, known in trade practice as

bronze powders, are treated with an amount of liquid so small that there is no sensible moistening of the powder, as for example, from $\frac{1}{2}$ per cent of the weight of the powder to not more than 10% of its weight, and then slowly compressed into a briquette by means of a hydraulic press or other suitable apparatus, that a briquette or dustless aggregate is obtained which, under suitable conditions has a leafing power of the same metal content in excess of that shown by either a paste made from the same powder or by the powder itself. This aggregate retains the leafing power shown by the fresh product.

The pressure is applied to a mass of slightly moist particles. Each of these particles is in the form of a flake having a crinkled surface. In other words one dimension of the particle is very much smaller than the other two, and its surface is in a roughly corrugated condition. As the pressure is applied, these particles arrange themselves at right angles to the direction of the pressure, the result being a mass of particles in flake form substantially all of which are parallel to each other. This may cause the briquettes to cleave at right angles to the direction of the pressure applied. As a result of such cleavage the briquettes tend to break up into smaller masses or clumps of laminated flakes. The pressure also causes the corrugations or striations in the particles to straighten out to a greater or less extent, this accounting at least partially for the observed increased leafing power. Another and important result of such pressure is to cause the liquid to disperse itself evenly between the flakes, resulting in a homogeneous product. In cases where the liquid consists of a solution of a highly volatile solvent and a non-volatile solid, uniform dispersion of the liquid ultimately results in a solid mass with the flakes separated from each other by a non-volatile solid after the solvent has been allowed to evaporate.

The use of a small amount of liquid is an important feature of this invention. The polishing film of bronze powders consists of substances soluble in the liquid constituents of paint vehicles. It has frequently been noticed in the art that solution of this polishing film destroys or lessens the leafing power of the powder. The greater the amount of solvent liquid in contact with the polished powder, the greater is the likelihood that the polishing film will be dissolved. The present invention permits the use of so small an amount of liquid that solution of the desirable film is minimized or eliminated.

Bronze powders, being covered with a film of

grease, which often consists of a mixture of such materials as stearic acid and aluminum stearate, are themselves wetted only by those vehicles which are solvents for this film, such as varnishes and lacquers. Such powders therefore have the disadvantage that aqueous vehicles, such as water dispersible gums, wet such powders only with difficulty. Pastes made from bronze powders, and containing about 35% water-immiscible liquid such as a petroleum distillate are not only immiscible with aqueous vehicles but are objectionable when added to lacquer, because the addition of about one pint of petroleum distillate or similar material to 1 gallon of lacquer which is necessary in order to incorporate the required amount of metallic pigment, tends to and often does effect a precipitation of the cellulose nitrate which is both insoluble and non-dispersible in petroleum fractions.

My invention makes it possible to overcome these difficulties pertaining to powders and pastes. Dustless aggregates produced in accordance with this invention by use of selected liquids such as alcohols with limited solubility in water, as for example, butyl, isobutyl, methyl ethyl carbinol, and the various isomeric amyl alcohols contain the powders in such condition that they are easily wetted by any of the customary vehicles, such as lacquers, varnishes, paints, gum aqueous vehicles such as glues, silicates, gum tragacanth, tragasol, and other distempers, hereinafter referred to as pigment vehicles.

The amount of liquid added to the powder I prefer to be within the range of four to nine per cent. This liquid may consist of a single constituent, such as a petroleum distillate, benzene, toluene, xylene, cumene, a mixture of hydrocarbons known as "high flash coal tar naphtha", tricresyl phosphate, olive oil and other vegetable oils, e. g., castor oil, soy bean oil, tung oil, coconut oil, animal oils such as oleo, fish oils, tetrahydro naphthalene, hexahydrophenol, liquid fatty acids such as oleic, ethers boiling over 125 deg. C. such as diamyl ether, dihexyl ether, diheptyl ether, etc.

The small amount of liquid employed makes possible the use of plasticizers customarily used in vehicles such as tricresyl phosphate mentioned above, and various phthalate esters such as diethyl, diamyl, etc. These plasticizers are especially valuable in bronze powder aggregates intended for use with lacquers, as no ingredient is added which deleteriously affects the properties of the lacquer. For use in varnishes small amounts of drying and semi-drying oils may be used, such as linseed, tung, castor, etc.

Instead of adding the liquid to the bronze powder and mixing by agitation to form a homogeneous mixture, I often prefer to add the liquid directly to the die of the hydraulic press used and usually simultaneously with the powder. The pressure exerted on the contents of the die effects the uniform dispersion of the liquid throughout the powder, so that each particle is completely surrounded by the liquid and thus separated from all adjacent particles. This complete separation by means of a film of liquid effects complete wetting of the powder and makes every particle available for covering and leafing power. The important point is that this wetting is effected with the absolute minimum of liquid, an amount so small that no appreciable portion of the polishing film can be dissolved either at once or after storage for long periods.

The pressure used to form these briquettes or

aggregates may vary from 1000 to 8000 lbs. per square inch. Very high pressures such as 20,000 lbs. per square inch are extremely deleterious to the product. With such pressures the briquettes formed are likely to be so hard that their disintegration in the desired vehicle is extremely difficult or even impossible. If larger amounts of liquid are used to avoid this hard briquetting under pressures of this range (20,000 lbs.) a considerable amount of liquid will be squeezed out of the die. Such exudation may completely destroy the leafing power of the product, and will certainly lessen it. The reason for this is that the liquid because of having been used in greater amounts has had the effect of dissolving some or most of the polishing film. When, therefore, it runs out of the die under such high pressure it carries with it the film or part of it which is responsible for the leafing property. A pressure of 5000 lbs. per square inch has been found satisfactory with the amount of liquid specified and no oozing, exudation or spewing of excess liquid occurs.

An important reason for the slow application of pressure is that uniform wetting is obtained with greater facility when this is done. Ten to fifteen seconds should be the interval for reaching the maximum pressure, which should be maintained for fifteen seconds more. This maintenance of pressure is necessary for the following reasons: The particles align themselves parallel to each other and at right angles to the direction of the pressure. Some time interval is required for the liquid to be disseminated uniformly throughout the laminated particles. The flattening out of the corrugations and folds of the flakes also requires time and the formation of a satisfactory briquette or one possessing the optimum properties cannot be considered complete the instant the desired pressure is reached.

Instead of pure liquids as outlined above, I often prefer to use solutions of various solids in some of the liquids already listed. In certain cases I prefer to use a mixture of volatile and non-volatile liquid or a mixture of solid that is non-volatile with a volatile liquid. As examples of mixed liquids or solutions the following are cited:

25% tricresyl phosphate in benzene; 25% ethylene glycol in ethyl alcohol; 25% olive oil in benzene; 25% dibutyl phthalate in benzene; 20% gilsonite in a mixture of benzene and high flash naphtha (impure cumene); 25% resin made by chlorinating diphenyl (trade name Aroclor) in petroleum fraction boiling about 160 deg. C.; 20% castor oil in ethyl alcohol; 25% soy bean oil in petroleum distillate boiling about 40 deg. C.; 25% triphenyl phosphate in benzene; 25% stearic acid in petroleum distillate boiling about 40 deg. C.; 25% gilsonite in tetrahydro naphthalene; 25% "Aroclor" resin in toluene; 25% each of rapeseed, coconut, olive oils and oleic acid in benzene; etc., with many variations.

As specific examples of carrying out this invention I cite the following:

Example 1.—A weighed amount of polished powder is introduced into a mixing machine and 5% by weight of a petroleum fraction boiling at about 160 deg. C. is added. The mixer is started and run for two minutes. The slightly moist powder is removed therefrom and pressed into briquettes in the die of a hydraulic press. Pressure is applied and gradually increased until after fifteen seconds it reaches 5000 lbs. It is

maintained at this point for fifteen seconds, additional pressure being applied if the gauge drops below the 5000 lb. reading. The briquette is then discharged from the die.

5 *Example 2.*—A solution of 20% gilsonite is made in a mixture of benzene and high flash naphtha (impure cumene). An amount equal to 7% by weight of the amount of powder which the die of the hydraulic press will loosely hold, is weighed out. The die is filled with the polished bronze powder in about 10 portions. After each one or two portions a small quantity of the liquid is added. When the die is full, pressure is applied in a hydraulic press or a tablet making machine and the process carried out as in 1.

10 *Example 3.*—A mixture consisting of 25% stearic acid and 75% petroleum fraction boiling at about 40 deg. C. is made and the briquettes made as in Example 2. The volatile petroleum fraction is now allowed to evaporate from the cake, leaving a laminated structure the particles of which are surrounded and separated one from each other by a film of dry solid stearic acid. There is no liquid present in this finished briquette.

15 *Example 4.*—Normal butyl alcohol is used as in Example 2.

20 *Example 5.*—A solution of 25% ethylene glycol dissolved in 99% ethyl alcohol is used as in Example 2. The dustless aggregates formed in Examples 4 and 5 are easily wetted by aqueous vehicles.

25 I have described what I believe to be the best embodiments of my invention. I do not wish, however, to be confined to the embodiments shown, but what I desire to cover by Letters Patent is set forth in the appended claims.

I claim:

30 1. A compressed aggregate consisting of flaked and polished metal powder, the particles of the powder being coated with a film which lends to the particles their property of leafing in a pigment vehicle and being bound together and separated from each other by a binding substance uniformly dispersed throughout the aggregate, said binding substance being of a nature such that the same will not adversely affect the dispersion of the material of the aggregate in paint, varnish or other vehicles, or adversely affect the properties of the dried coating formed by such vehicles, the binding substance consisting of a liquid hydrocarbon the weight of the liquid being approximately $\frac{1}{2}\%$ to 10% of the weight of the powder, the aggregate having been compressed at a pressure of 1000 to 10,000 pounds per square inch.

35 2. The method of making a substantially dustless solid or plastic aggregate of metal powder which consists in mixing with said powder a

binding liquid in the amount of from $\frac{1}{2}\%$ to 10% of the weight of the powder and subjecting said mixture to pressure of from 1000 to 10,000 lbs. per square inch without destroying the leafing power of the powder.

5 3. The method of making a substantially dustless solid aggregate of metal powder, which consists in adding the dry flaked polished metallic powder to the die of a press substantially simultaneously with a predetermined amount of liquid, 10 and subjecting the mixture to a pressure of approximately from 1000 to 10,000 pounds per square inch without destroying the leafing power of the powder.

15 4. The process of enhancing the covering power of flaked and polished metal powder, and of rendering the same dustless and easily dispersible in vehicles of protective coatings, without deleteriously affecting the leafing power of such powder, which consists in uniformly dispersing throughout a quantity of such powder in dry condition, an amount of liquid just sufficient to wet all of the particles of such powder, said liquid being of a nature compatible with both the volatile and non-volatile constituents of the particular coating material in which the metal pigment is to be employed, the dispersion being accomplished by subjecting the powder and liquid to pressure in a die or mold, the amount of such pressure being sufficient to cause the dispersion 20 of the liquid throughout the powder but insufficient to cause exudation of liquid from the die or mold, the amount of liquid being from $\frac{1}{2}\%$ to 10% of the weight of the powder, the pressure being from 1000 to 10,000 pounds per square 25 inch.

30 5. A compressed aggregate consisting of flaked and polished metal powder, the particles of which may vary in size from approximately 200 to 325 per inch, said particles being coated with a film which lends to the particles their property of leafing in a coating vehicle and said particles being separated from each other by a non-aqueous organic liquid having no more than a limited solubility in water uniformly dispersed throughout the aggregate and compatible with the particular coating vehicle in which the material of the aggregate is to be used, so as to provide substantially complete wetting and separation of the individual flakes by the vehicle upon incorporation of the aggregate with the vehicle, said liquid being compatible, also, with the film formed by the drying of such coating, the amount of said liquid being from $\frac{1}{2}\%$ to 10% of the weight of the powder, the aggregate having been compressed at 1000 to 10,000 pounds per square inch.

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