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[54] **METHOD OF SPRAYING A POWDER PAINT SLURRY**

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[51] **Int. Cl.²** **B05D 1/02**

[58] **Field of Search** 117/104, 21, 31; 427/421, 427/372, 385

[56] **References Cited**

UNITED STATES PATENTS

2,972,553	2/1961	Hess	117/104 X
3,089,783	5/1963	Carlson et al.	117/104 X
3,287,157	11/1966	Brown et al.	117/31 X
3,437,072	4/1969	Levinson	117/21 X
3,446,652	5/1969	Smith	117/104 R
3,502,493	3/1970	Forestek	117/31 X
3,687,885	8/1972	Abriss et al.	117/161 UZ
3,713,872	1/1973	Porter et al.	117/104 R X
3,770,482	11/1973	Millar et al.	117/21 X
3,787,230	1/1974	Hoffman et al.	117/104 R X

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[57] **ABSTRACT**

A method of painting with a composition of a pumpable, sprayable powder paint slurry comprising powder paint, water and a surfactant, having sufficient surfactant to reduce the surface tension of the aqueous portion to less than 35 dynes/cm, having a minimum concentration of water in excess of the interstitial volume of the powder, and having as a maximum concentration of water that necessary to produce, when sprayed onto a substrate, a film of powder and water in which the water is less than the interstitial volume of the powder.

6 Claims, 5 Drawing Figures

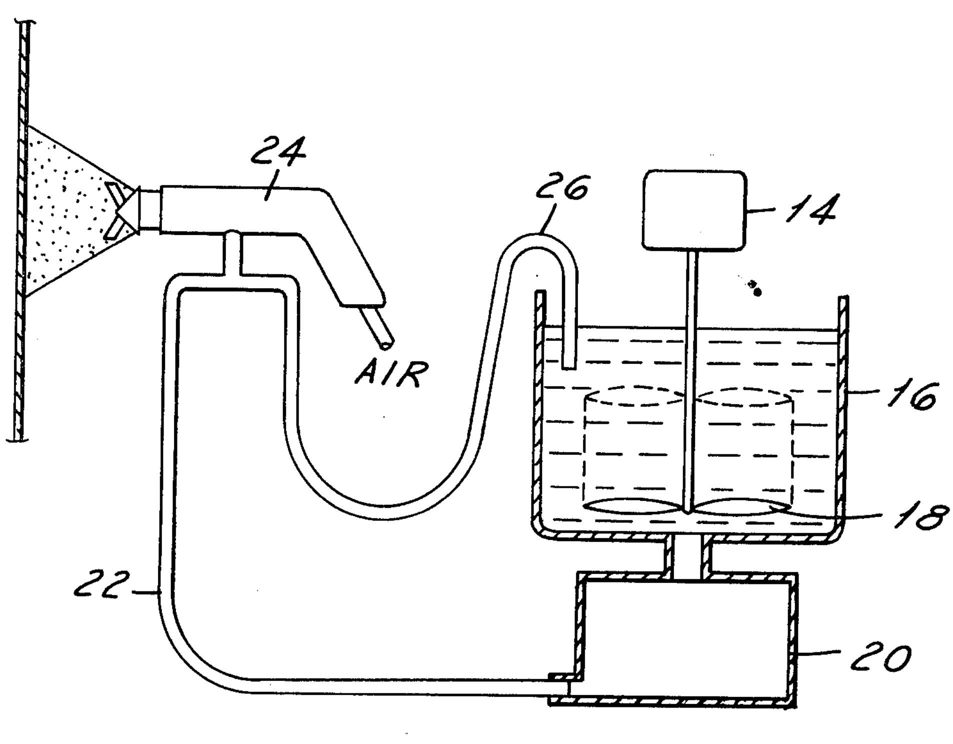


FIG. 3

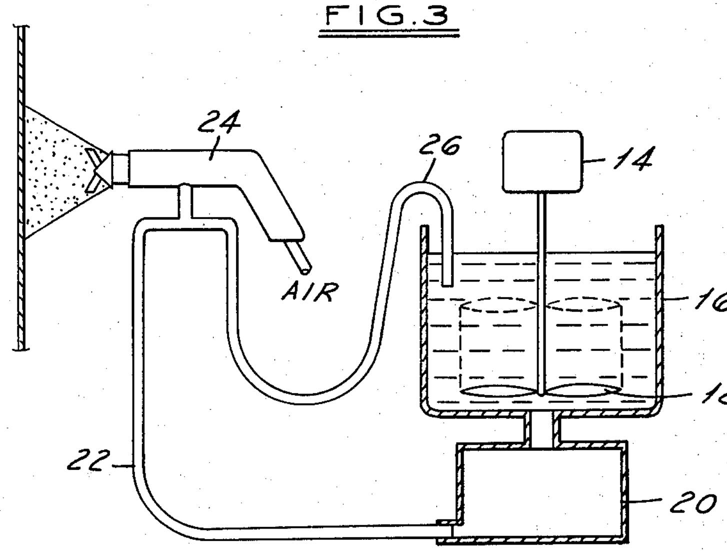


FIG. 1

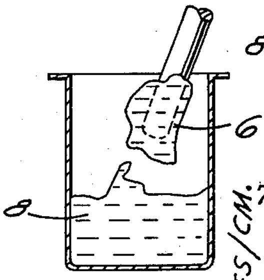


FIG. 5

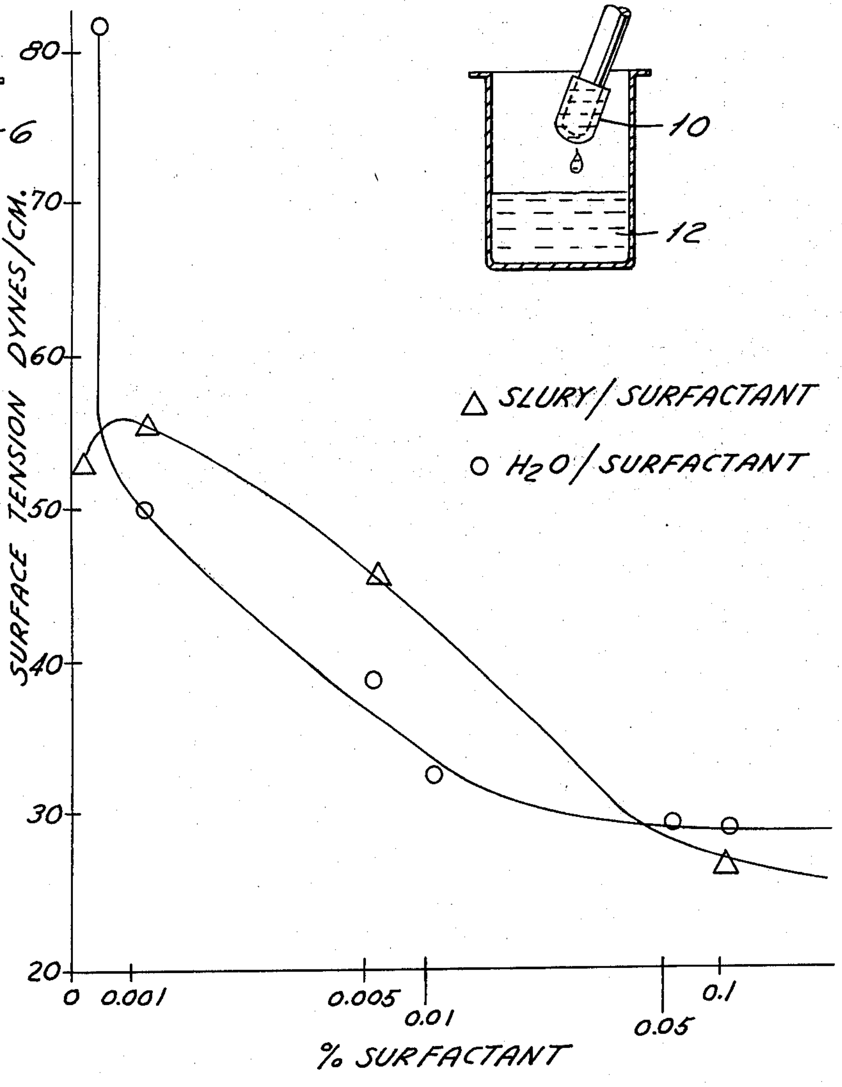
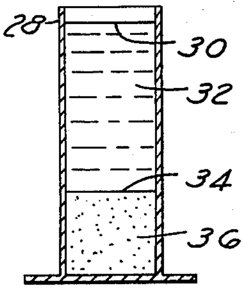


FIG. 2

FIG. 4



METHOD OF SPRAYING A POWDER PAINT SLURRY

BACKGROUND OF THE INVENTION

Powder paint has been increasing in popularity as an industrial coating material at a rapid rate. The reasons for the widespread acceptance of powder paint are: virtual absence of air pollution, recovery and reuse of powder lost by overspray, and improved quality of cured out paint films. The application of a powder paint coating is essentially a two-step process involving first, applying a layer of powder, and second, treating the powder layer to cause it to coalesce, flow and cure into a desired paint film.

Previous powder paint coating technology has applied powder paint in a dry form using electrostatic spraying equipment or fluidized beds. Both methods employ specialized equipment to apply the powder which require large capital expenditures and which obsolete existing liquid paint spraying and dipping equipment.

It is an object of this invention to facilitate the use of existing liquid paint spraying equipment in the application of powder paint.

It is the further object of this invention to provide a sprayable pumpable slurry of powder paint and water which can be pumped and sprayed using essentially conventional liquid paint equipment and which when sprayed will produce a film of powder paint and water which can then be treated to form a conventional paint film, thereby obviating the necessity for large capital expenditures and obsolescence of existing painting equipment.

Many industries, notably the ceramic and enamel coating technologies, apply aqueous slurries of particulate matter using spray equipment similar to that used in spray painting. The slurries used in those industries invariably contain various additives such as gums, resins, and glues as suspending and dispersing agents and to impart strength to the wet film before it is fired. The use of such additives in the concentrations required is not possible in applying an aqueous slurry of powder paint as they will adversely affect the film properties of the powder paint. Also, the ceramic and enamel frits which are used to form slips or slurries are hydrophilic and are therefore easily wettable in forming the slurries. Powder paint is, however, hydrophobic and does not wet when a slurry is formed. This difference in the physical properties of the powder paint and ceramic and enamel frits is evidenced by the problems involved in forming a slurry of powder paint which can be pumped and sprayed to produce a layer of powder and water.

When an aqueous slurry of powder paint and water, without any additives, is pumped through conventional spray equipment, the pumps, guns and paint lines clog up after short periods of time and the equipment must be completely cleaned before spraying can be resumed. Pursuant to the invention disclosed herein an aqueous slurry of powder paint can be formed which is pumpable and sprayable without clogging and plugging the spray equipment and which will form a film of powder paint and water when sprayed.

SUMMARY OF THE INVENTION

In accordance with the present invention a pumpable, sprayable, aqueous slurry of powder paint is prepared in which the aqueous portion has a surface tension of less than 35 dynes/cm, and preferably between 29 and 35 dynes/cm. Further, the minimum amount of the aqueous portion should be greater than the interstitial volume of the powder, and the maximum amount of the aqueous portion should not exceed that amount which, when the slurry is sprayed onto a substrate, will produce a film of powder and water in which the water portion is less than the interstitial volume of the powder. In other words, there must be evaporated out of the slurry during the painting such an amount of water as will reduce the aqueous portion to below the interstitial volume of the powder portion.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 illustrates a slurry of powder paint and water having a volume of water less than the interstitial volume of the powder;

FIG. 2 illustrates a slurry of powder paint and water having a volume of water greater than the interstitial volume of the powder;

FIG. 3 illustrates a spraying apparatus for use in spraying a slurry of powder paint and water onto a substrate;

FIG. 4 illustrates a method of determining the interstitial volume of a powder; and

FIG. 5 is a graph of the effect of a surfactant on the surface tension of water, and a powder slurry.

DESCRIPTION OF THE PREFERRED EMBODIMENT

In painting with an aqueous slurry of powder paint and water, the water acts as a vehicle in a conventional liquid paint. It functions as a carrier in which the film forming component, in this case powder paint, is dispersed and which is then applied to a substrate to form an adherent wet coating of water and powder. The liquid carrier is then evaporated to leave an adherent coating of powder on the substrate which can subsequently be treated to cause the powder to coalesce, flow and cure into a desired paint film.

In painting with such an aqueous slurry there are several interrelated problems that can only be solved in relation to each other. They are: the slurry must be pumpable and sprayable from essentially conventional spray painting equipment without clogging or plugging the equipment; the applied slurry coating of powder and water must adhere to the substrate sufficiently to produce a coating with substantially no sagging, running, or creeping; and the adherent slurry coating of powder and water must not contain any substances in such concentration that they would have a deleterious effect on the resulting cured paint film.

Pursuant to this invention it is possible to form a slurry of powder paint and water which can be sprayed onto a substrate to form an adherent film of powder and water, and which can be treated to remove the water and cause the powder paint to form a fixed and adherent paint film thereon.

One of the important properties of a free flowing slurry is the surface tension of the aqueous phase. I have found that a satisfactory sprayable and pumpable slurry of powder cannot be formed unless the surface tension of the aqueous portion is less than 35 dynes/cm and preferably between 29 and 35 dynes/cm. At a surface tension above 35 dynes/cm the powder slurry will clog and plug the paint spraying equipment. Examples 1 and 2 illustrate the effect of surface tension on a powder slurry, and how when a slurry is formed with a surface tension of greater than 35 dynes/cm it will clog the paint spraying equipment, (Example 1) and (in Example 2) when the surface tension of the slurry is reduced to less than 35 dynes/cm a free flowing pumpable, sprayable slurry is produced.

FIG. 5 illustrates the concentration of surfactant necessary to reduce the surface tension of water alone, and water and powder paint, to below 35 dynes/cm. In FIG. 5 the use of Aerosol OT as a surfactant is illustrated. It will be observed that the concentration of Aerosol OT necessary to sufficiently reduce the surface tension is less than 0.1 percent. It is desirable to use a surfactant which will reduce the surface tension below 35 dynes/cm at concentrations of less than one percent. Armeen SZ and Aerosol OT have been found to be satisfactory surfactants when used according to this invention. Any commercially available surfactant would be satisfactory as long as it is capable of reducing the surface tension of the aqueous portion of the slurry to less than 35 dynes/cm, and has no deleterious effect on the powder particles or on the desired characteristics of the paint film to be formed.

In FIG. **1** I have illustrated in a simplified way the physical characteristics of a slurry which produces an adherent film, and in FIG. **2** a slurry which is free flowing or pumpable and sprayable. FIG. **1** illustrates a slurry which has a paste-like appearance, resulting from mixing a large quantity of powder with water. If a spatula is used as an agitator and then withdrawn, the paste does not return to a smooth horizontal surface **8**; and the paste forms an adherent film on the spatula which will not run off **6.** Such a paste-like composition will clog up pumps, guns and other equipment after spraying for a short period of time. It will, however, when applied, form an adherent film of powder which does not run, sag or creep.

Further addition of water to the paste-like slurry of FIG. **1**, while maintaining the surface tension of the aqueous phase at or below 35 dynes/cm, will result in a "free flowing" slurry as illustrated in FIG. **2.** If a spatula is used as an agitator and then withdrawn the slurry will return to its own smooth horizontal surface or form its own miniscus **12,** and the slurry will not adhere to the spatula but instead will run off **10.** Such a slurry would be sprayable from a spray gun, but if a slurry of this character hits a substrate it will run off and will not form an adherent film.

If a sufficient amount of free flowing slurry's liquid component has evaporated while the atomizing spray travels from the gun to the substrate, so that the arriving spray has the characteristics pictured in FIG. **1,** then a well adhering film of powder and water forms on the substrate. Thus, pursuant to this invention, we are able to determine for each individual paint powder a critical amount of liquid which governs the transition from a paste-like state to a free flowing state. I have determined that the critical amount of liquid is greater than the interstitial volume of a particular powder. When an amount of water is provided in a slurry which is greater than the interstitial volume an essentially free flowing slurry will result. When an amount of water is provided which is less than the interstitial volume then a paste-like slurry will result. Example 2 illustrates the preparation of a free flowing powder paint slurry and example 3 illustrates a slurry which does not possess free flowing characteristics.

I have defined the interstitial volume of a powder as the volume of the interstices between a given weight of powder particles as determined by the method set forth herein. FIG. **4** schematically represents an interstitial volume determination. In a weighed graduated cylinder **28,** a weighed amount of paint powder **36** is mixed with several times its weight of water **32,** the surface tension of which has been reduced to below 35 dynes/cm through the addition of a small quantity of surfactant. The powder is then allowed to settle, and the sedimentation volume **34** of the powder is determined. The total volume confined by the upper miniscus **30** is read after any foaming has subsided or has been eliminated. The total weight of the graduate, water and powder is determined. The weight of the graduate is then subtracted giving the weight of water and powder in the graduate. Making the not unreasonable assumption that density of the supernatant liquid is unity within the precision of the determination, then further computation is simplified, since one ml of supernatant can be set equal to 1 gram. If so, the total volume 30 minus the sedimentation volume 34 = the volume of supernatant = weight of supernatant. Then the weight of the supernatant is added to the weight of the powder, and is then subtracted from the total weight of the powder and water to give the weight equal to the volume of fluid between the powder particles, conveniently called the "interstitial volume". It is advisable to take readings and then to re-agitate the mixture, to let settle again, to take another set of data, etc. I then express the "critical composition" as weight percent of water in the sediment when the water content is equal to the interstitial volume of the powder.

$$\frac{(i \times 100)}{g + i} = \text{critical composition} = \%\ \text{water in sediment}$$

wherein i is the interstitial volume and g is the weight of powder.

A free flowing slurry is one which contains more fluid than the interstitial volume, as in FIG. **2,** and a well adhering sprayed-on film contains less fluid than the interstitial volume, as in FIG. **2.**

The relative amounts of water and powder which results in free flowing slurries vary widely for different powders; apparently depending upon the particle size and shape.

If the powder particles could be perfect spheres a sprayable slurry could be obtained when enough liquid is provided to cover the settled powder. The other extreme is exemplified by powder particles which are flat square plates. These would tend to settle like "bricks in a wall" with a minimum of interstices. Yet to provide enough space for their random motion enough liquid would have to be provided to allow for the rotation of their longest dimension, that is a spherical space of a diameter equal to the body diagonal of the square plate.

Actually the shapes of the powder used in my experimentation were — on microscopic exam — neither perfect spheres nor plates.

Thus well sprayable slurries were obtained by use of liquid volumes not too much larger than the powder's interstitial volumes. These minimum liquid volumes ranged from about 1.2 to about 2.5 times the interstitial volume of the individual powders.

It is believed that by providing a volume of water which is greater than the interstitial volume of the powder the powder paint particles are provided with sufficient space in which to move so they do not become aggregated into a mass of powder particles which could then clog up a paint gun or line. Sufficient space is provided to allow for the random motion of the powder particles which provides the free flowing character of a slurry produced according to this invention.

The maximum amount of water surfactant mixture which can be added to produce a free flowing pumpable slurry is that amount in excess of the interstitial volume, but in excess by less than the evaporative spray loss during spraying of the slurry onto a substrate to be coated. Referring to FIGS. **1** and **2**, the slurry pictured in FIG. **2** may be diluted with surfactant-water mixture to the extent that during the spraying of the slurry onto a substrate a sufficient amount of water can be evaporated to produce an adherent layer of powder paint and water with the characteristics of FIG. **1.** The volume of water contained in the sprayed on layer of powder and water must be less than the interstitial volume of the powder, for an adherent film to be formed which will not sag, run nor creep. The volume of water added to produce a free flowing slurry can be increased above the interstitial volume to any amount necessary as long as during the spraying of the slurry onto a substrate a sufficient amount of water can be evaporated to produce a layer of powder paint and water having a volume of water less than the interstitial volume of the powder.

Another characteristic of a free flowing sprayable slurry is its behavior when not agitated. Slurries which settle "hard" as the painter calls it, will tend to clog up those parts of the spray equipment which lack agitation. "Hard settling" can be controlled by the addition of antisettling agents such as Ben-A-Gel EW. Any commercially available anti-settling agent can be used as long as it prevents hard settling of the powder paint in the slurry, and has no deleterious effect on the powder particles or on the desired characteristics of the paint film to be formed. Examples **4** and **5** illustrate the use of an antisettling agent with a powder which has hard settling characteristics. The addition of the anti-settling agent to the slurry increases the interstitial volume of the powder which requires a larger concentration of water to be provided to form a free flowing slurry.

The following examples will illustrate the invention:

EXAMPLE 1

The interstitial volume of 5 grams of Monsanto MA **1000 L58004** powder manufactured by Monsanto, Plastics Products and Resins Division, 190 Grochmal Avenue, Indian Orchard, Mass. was determined according to the procedure outlined above on two successive days, as follows. The liquid portion of the slurry contained 0.05% surfactant (5.2 ml of 1% Aerosol OT and 94.8 ml water). The surfactant used was Aerosol OT, is described by the manufacturer, American Cyanamid Co., Process Chemicals Dept., Wayne, N.J. as **75** weights sodium dioctyl sulfo succinate, **20** weight water, **5** weight of a lower alcohol to provide fluidity.

Weight of Powder	5.0 g	5.0 g
Weight of Powder and Liquid	21.48g	21.43g
Total volume of Liquid and Powder	20.1 ml	20.0 ml
Sediment Volume of Powder	6.1 ml	6.1 ml
Supernatant Volume	14.0 ml	13.9 ml
Supernatant and Powder (Weight)	19.0 g	18.9 g
Interstitial Volume		
Total weight of liquid and powder		
− weight of supernatant and powder		
= weight of interstices or volume	2.5	2.5
Critical concentration	33.3	33.3
$\frac{\text{Interstitial volume} \times 100}{\text{Interstitial volume and weight of powder.}}$		

To prepare a slurry in which the volume of water is equal to the interstitial volume for this powder will require a slurry containing **33.3%** water and **66.7%** powder.

A slurry was prepared as follows:		
300.0 g	Powder	
0.13g	Aerosol OT	(0.19g 75% in water)
250.0 g	Water	(45.5% water)

The liquid phase filtered from the slurry, showed a surface tension of **41.8** dynes/cm. The water concentration was **45.5%** which is in excess of the critical concentration, or in excess of the interstitial volume. The slurry was placed into spray equipment as in FIG. **3** consisting of a quart size paint pot (**16**) with a motor driven (**14**) agitator revolving at about **100** rpm (**18**), a centrifugal pump (**20**) submerged in the slurry, with about a two-foot length of translucent rubber latex tubing (**3/16** inch ID, **1/16** inch wall) (**22**) leading to a spray gun (**24**), Binks model No. **62** fitted with No. **66** nozzle and a **66** SH cap. Another two-foot length of rubber latex tubing provides the return line to the paint pot (**26**). The rate of rotation of the agitator was regulated by a Rheostat. The slurry is circulated from the pump to the gun and back to the paint pot at a rate of **1000** ml per minute when the slurry is not being sprayed. When the gun is spraying the slurry the rate of circulation is reduced to **700** ml per minute.

After a few minutes of pumping the slurry the stream slowed down and the gun clogged up. Without agitation, a fluffy sediment of wetted powder was observed in the paint pot. The fluffy sediment in the paint pot indicates that this powder did not have hard settling characteristics.

EXAMPLE 2

Two slurries were prepared using the same powder as in Example **1**, but with the addition of sufficient surfactant to lower the surface tension of the aqueous phase to less than **35** dynes/cm, as follows:

300.0 g of Powder
0.26 g Aerosol OT
250.0 g Water.

The liquid phase, when filtered from the slurry, showed a surface tension of 30.7 dynes/cm. The concentration of the water was 45.5% which was in excess of the critical concentration of 33.3%.

Both slurries were individually placed in the above equipment, and a well atomized spray produced good test panels without any clogging of the spray gun. The pumping was continued for one hour with no clogging and then another panel was sprayed with good results.

After each spray test the remaining slurry was drained from the pump and placed in a plastic bag inside a sealed container. One of the slurries was stored at room temperature for one week, the other was frozen solid for the week. The frozen slurry was thawed and both slurries were again sprayed. Each slurry produced good spray panels and did not clog the pump. The test was continued for two subsequent weeks and both slurries produced good test panels through the third week. The above test procedure is A.S.T.M. method D-2243 which is a general method to test the freezer-thaw stability of a sample.

Examples 1 and 2 illustrate that by lowering the surface tension of the aqueous portion of the slurry to less than 35 dynes/cm a sprayable, pumpable slurry was produced which did not clog the spray gun.

EXAMPLE 3

Another slurry of the same powder used in Examples 1 and 2 was prepared using water containing 0.1% Aerosol OT and having a surface tension of less than 35 dynes/cm, as follows:

300.0 g Powder
0.12 g Aerosol OT
123.0 g Water

The slurry contained 29.1% water which is less than the critical concentration of the powder of 33.3%.

The slurry had a paste-like appearance, as in FIG. 1 of the drawings, and when placed in the above spray equipment would not spray, and pumped for less than one minute before it clogged up the pump.

Examples 2 and 3 illustrate that even when the surface tension of a slurry is reduced below 35 dynes/cm, there must be provided an amount of liquid in excess of the interstitial volume of the powder to form a sprayable, pumpable, free flowing slurry of powder paint.

EXAMPLE 4

The interstitial volume of 5 grams of Grow GP-16-D1 powder manufactured by the Grow Chemical Corp., 14100 Stansbury, Detroit, Mich. was determined according to the procedure outlined above on two successive days, as follows.

The liquid portion of the slurry contained 0.10% surfactant (10 ml of 1% Armeen SZ and 90 ml water). The surfactant used was Armeen SZ, described by the manufacturer, Armour Industrial Chemical Co., Box 1805, Chicago, Ill. as sodium-N-coco-2-Aminobutyrate.

Weight of Powder	5.0 g	5.0 g
Weight of Powder and Liquid	21.8 g	21.8 g
Total Volume of Powder and Liquid	21.0 ml	21.0 ml
Sediment Volume of Powder	9.5 ml	10.1 ml
Supernatant Volume	11.5 ml	11.0 ml
Supernatant and Powder Weight	16.5 g	16.0 g
Interstitial Volume		
Total weight of liquid and powder		
− weight of supernatant and powder		
= weight of interstices of volume	5.3	5.8
Critical concentration	51.5	53.8
Interstitial volume × 100 / Interstitial volume and weight of powder		

A slurry of the above powder was prepared as follows:

300.0 g Powder
0.45 g Armeen SZ
445.0 g Water (59.8% of water)

The liquid phase, filtered from the slurry, showed a surface tension of 31.4 dynes/cm.

Placed in the above equipment (Example 1) excellent pumping characteristics were observed. However, the gun clogged up. Without agitation, a hard, somewhat brittle sediment was found in the paint pot which would be characterized as hard settling by a paint sprayer.

EXAMPLE 5

Another slurry was prepared using the same powder as in Example 4, with the addition of an anti-settling agent to prevent hard settling.

The liquid portion of the slurry contained 13.1 ml of 1% Armeen SZ (see Example 4), 3.75 ml Ben-A-Gel EW (described by the manufacturer, National Lead Company, 111 Broadway, New York, N.Y. as a specially processed magnesium montmorillonite powder of very fine ultimate particle size), and 83.15 ml H_2O.

Weight of Powder	5.0 g	5.0 g
Weight of Powder and Liquid	22.61g	22.56g
Total volume of Liquid and Powder	21.3 ml	21.5 ml
Sediment volume of Powder	11.6 ml	11.6 ml
Supernatant volume	9.7 ml	9.9 ml
Supernatant and Powder Weight	14.7 g	14.9 g
interstitial Volume		
Total weight of liquid and powder		
− weight of supernatant and powder		
= weight of intersticies of of volume	7.9	7.7
Critical concentration	61.2	60.5

A slurry was prepared as follows:

200.00g Powder
0.70g Armeen SZ
0.20g Ben-A-Gel EW
535.00g Water (75.2% of water).

The liquid phase, filtered from the slurry, showed a surface tension of 34.4 dynes/cm.

Placed in the above spray equipment a well atomized spray produced good test panels, and did not clog the spray gun. Without agitation, a fluffy sediment of wetted paint powder was observed in the paint pot with no hard settling. The addition of the anti-settling agent therefore produced a free flowing, pumpable, sprayable slurry of powder paint.

The slurry deposit on the test panels weighted 1.650 grams prior to heating to remove the water. After removal of the water a deposit of powder weighing 0.673 gram was obtained. The deposited coating of powder paint and water was thus 59.2% water, which is less than the interstitial volume of water as calculated above. During the spraying of the slurry containing 75.2% water, sufficient water had been evaporated to reduce the volume of water in the slurry deposit to less than 61.2%, the interstitial volume, and thus produce an adherent coating of powder and water.

What is claimed is:

1. The method of spray painting an aqueous slurry of powder paint, comprising the steps of: preparing a slurry of the powder paint and water having a surface tension of the aqueous portion of less than 35 dynes/cm and having a minimum amount of water greater than the interstitial volume of the powder; spraying the powder slurry onto a substrate and evaporating from the slurry during the spraying of the slurry onto the substrate a sufficient amount of water to form a layer of powder paint and water on the substrate in which the amount of water is less than the interstitial volume of the powder.

2. The invention defined in claim 1 characterized by evaporating from the spray a sufficient amount of water to form a layer of powder paint and water on the substrate in which the amount of water is less than the interstitial volume of the powder.

3. The invention defined in claim 1 characterized by reducing the surface tension of the aqueous portion of the powder paint slurry to 29 – 35 dynes/cm by the addition of a surfactant.

4. The invention defined in claim 1 characterized by adding an anti-settling agent to the slurry.

5. The invention defined in claim 1 characterized by removing the water from the layer of powder paint and water on the substrate and causing the powder paint to coalesce and cure into a desired paint film.

6. The method of claim 1 wherein the minimum amount of water ranges from about 1.2 to about 2.5 times the interstitial volume of the powder paint.

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