METHOD OF MAKING POWDERED PAINT

Ivan H. Tsou, Pontiac, and James W. Garner, Detroit, Mich., assignors to Grow Chemical Corp., New York, N.Y.

Filed Oct. 5, 1971, Ser. No. 186,695
Int. Cl. C08f 47/03
U.S. Cl. 260—23 R 25 Claims

ABSTRACT OF THE DISCLOSURE

A method of making powder paint is disclosed wherein there is blended with a coagulating liquid, at a rate inversely proportional to the desired particle size of the paint, a liquid paint having its solvent portion miscible with the coagulating liquid and its film-forming portion insoluble in the coagulating liquid, to precipitate the paint in powder form, and removing this precipitated powder paint from the blend.

BACKGROUND OF THE INVENTION

The use of powder coating materials, such as powdered paints, has increased greatly within the last few years. While there are many advantages in using powdered coatings rather than liquid coating materials, several disadvantages arise in the methods of manufacture of powder coatings and in particular paint powders. Currently powder coatings are manufactured by processes involving mechanical attrition, i.e. grinding of chunks of solid paint or solid components of paint. First, the equipment used for the grinding involves the capital investment of large sums; and the obstruction of current paint manufacturing equipment by some processes adds appreciably to the cost of powder coated products. Second, mechanical attrition does not permit close control over the size and distribution range of sizes of the powder particles. Third, mechanical grinding does not produce smooth, rounded paint particles but rather particles having sharp edges and odd shapes. These oddly shaped particles reduce efficiency when used in electrostatic spraying operations by necessitating higher voltages with a greater danger of electrical arcing. In the application of powder paints, the properties of the finished coating often depend heavily upon the physical properties of the powder—such as size and shape of the particles, homogeneity of composition, and uniformity of size.

The present invention eliminates all of these difficulties. The first is solved by using standard paint manufacturing equipment to make a conventional paint in which only the solvent may be altered from conventional formulas. Although some additional equipment is needed, the techniques used here require only a minimum addition of machinery. The second difficulty of conventional powder making techniques is overcome by the present process because reasonably exact control can be exercised over the size of the particles. The final difficulty is overcome because the nature of the hereinafter described process produces substantially uniformly shaped particles.

Unlike the prior art methods of powder paint manufacture wherein solid paint or solid components of paint are ground to produce the powder, the present process involves first the manufacture of conventional liquid paint, and then the controlled coagulation or precipitation of the non-solvent portion (i.e., film-forming portion) of the paint in powder form. In carrying out the process the solvent portion of the paint is selected to be miscible with a coagulating liquid which is not a solvent for the non-solvent portion of the paint. To control particle size of the precipitated powder, the rate at which the liquid paint and coagulating liquid are mixed, and the completeness of the blending of the two liquids, are of great importance. We have found that the size of the particles produced is inversely related to the rate at which the paint solvent is diluted and the range of the particle sizes is directly related to the completeness of the blending, viz., the slower the paint solvent is diluted from just prior to coagulation or the onset of precipitation until the major portion of the non-solvent portion has precipitated, the larger are the powder paint particles produced, and the less complete is the blending of the coagulating liquid and the liquid complete the greater the particles size range of the powder produced.

While precipitation of solids from liquids using reagents, and precipitation of pigmented plastics granules for subsequent extrusion or molding processes, are well known, but so far as we are aware there has been no attention focused on the manufacture of powder paints by precipitation of the non-solvent portion of the paint under conditions that will yield the requisite closely controlled particle size and shape. Examples of prior art techniques of precipitation are shown in U.S. Patents 2,020,085, 2,379,237, 2,485,287, 2,722,528, 3,215,663, 3,409,585, 3,441,530.

One object of this invention is to produce paint powders from a broad range of conventional liquid paints, and to produce these paint powders with desirable physical characteristics of size, shape and homogeneity of composition.

Another object is to manufacture powder paints while using standard and currently available liquid paint manufacturing facilities with only a modest addition or alteration of the equipment for utilization in the powder making process.

Both of these objects are achieved by using conventional equipment to produce a conventional liquid paint which is modified only in the solvent selection used as a dispersing medium for the film formers and pigments (i.e. film-forming portion).

A further object of this invention is to apply this method to any system where a homogeneous powder having particles with desired size and distribution of sizes is required for desired application of the powder.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic flow diagram of the process including many optional steps;

FIGS. 2 and 3 represent typical paint systems utilizing liquids A and B to determine the percentage of solvent added; and

FIG. 4 represents the particle size distribution for powders made at points indicated in FIG. 2.

DESCRIPTION OF PREFERRED EMBODIMENTS

In conventional liquid paints the choice of a solvent is governed by the usage of the paint and the conditions of that usage. Since powdered paint is essentially a solventless coating material, the use of any solvents during the manufacture of the powder will not affect the application of the powder. Consequently the choice of a solvent
can be tailored to the requirements of manufacturing powder having desirable physical characteristics. The use of the term "powder" in this specification indicates generically a quantity of fine paint particles each being essentially free of solvent but otherwise comprising a complete paint system of film formers and pigments. The powder may be either in a dry form or may be wet with a liquid which is not a solvent for the particles. The powder may be either hydrophilic or hydrophobic. During production of the powder as hereinafter explained it may be either dried to result in a dry powder or it may be allowed to remain wet with a liquid which is not a solvent for the particles. This latter form is preferred in applications where the paint is applied as a slurry since then no drying procedure is required in manufacture of the paint powder.

The use of the term "paint" here will also include powdered coating materials as well as more conventional paints.

The first essential step of the process is to manufacture a conventional paint using one of a select group of solvents as the liquid vehicle as described below. The property that the solvents share that make them acceptable for making powdered paint is that they are good solvents for the paint system and the precipitating or coagulating liquid.

The second essential step of the process is to suddenly and uniformly dilute the paint in a liquid medium which is miscible with the paint solvent and is not a solvent for the remainder of the paint system—the pigments and film formers. The dilution can be done in a number of ways as hereinafter described, depending upon the physical properties desired of the powder and the economics of the production process.

The remaining steps are optional, such as washing and processing the precipitated powder paint by any of numerous well known methods.

Referring to FIG. 1, the film formers and pigments (i.e., film forming portion of the liquid paint) are prepared as usual in the normal manufacture of paint. The solvent portion of the liquid paint is here called liquid A. Film formers are used here to indicate all of the elements of the paint that are soluble in liquid A. Likewise "pigments" indicate all of the components that are not soluble in liquid A. The pigments must be finely enough pulverized to remain in homogeneous suspension throughout liquid A. Although liquid A is spoken of in the singular, it is to be understood that it may be a mixture of solvents.

In addition to choosing the solvent in the liquid paint to achieve the desired powder, it is also necessary to adjust the amount of solvent in the paint, i.e., make it thinner or thicker, before precipitation to achieve optimum characteristics in the resulting powder and optimum economy of the manufacturing process. Thus, it will be readily apparent to those skilled in the art that the only difference between the paint here and an ordinary paint is in the amount and type of solvent.

Once the paint has been prepared, an optional step, which, although it is not necessary to the practice of the invention, does alter the characteristics of the resulting powder, is to gradually and uniformly mix liquid B with the liquid paint until a desired concentration of liquid B is present. If it is wished to make the powder particles as small as possible, it is helpful to increase the concentration of liquid B just short of that necessary to effect coagulation as shown by the dotted line B in FIG. 2. However, in no case can the uniformly mixed concentration of liquid B exceed the line of coagulation 4 in FIG. 2, or else premature coagulation will occur.

Again referring to FIG. 1, after the liquid B has been added (if added), the paint is then suddenly diluted and blended with liquid B. This step is important to the control of the powder particle characteristics. Once the preliminary condition of the paint and its solvent has been met, the method and the rate at which dilution and coagulation is performed will govern particle size and distribution of sizes.

One method is to agitate the liquid paint in a container and then to rapidly pour in a large amount of coagulating liquid until coagulation of the non-solvent (i.e., film forming) portion of the paint is completed. This method yields a broad range of particle sizes and is suited for batch processing. However, it is difficult to control the sizes of powder particles resulting from this method.

Another method, also suited to batch processing, is to agitate a container of the coagulating second liquid and slowly pour the prepared paint into the agitated second liquid. As such device is that the paint hits the second liquid, the pigments and film formers of the paint (i.e., the film forming portion of the liquid paint) are coagulated out of the solvent as the solvent becomes infinitely thin in the volume of second liquid in which the solvent is miscible while the pigments and film formers are not soluble. This leads to a somewhat narrower distribution of powder particle sizes, although the average size tends to be large. Again control of particle sizes proves difficult, but this method is the most convenient for making small amounts of powder for laboratory use.

A third method, and the preferred embodiment, is well suited to continuous production and allows close control over both particle size and distribution of sizes than the other two methods. In this method a container of liquid B, with a supply of liquid B being fed into the container below the paint inlet, is vigorously agitated by a rapidly rotating blade high-speed mixer. An outlet nozzle for the paint mixture is fixed beneath the surface of liquid B near the mixer blade where the agitation of liquid B is at a maximum. The paint mixture is then injected under high pressure through the nozzle into the agitated liquid B. Such agitation produces a nearly instantaneous breakup of the stream of paint into small droplets. The solvent in these droplets, since it is miscible with liquid B, then leaves the droplet causing the droplet to coalesce into a minute particle of the non-solvent portions of the paint (since they are not soluble in liquid B) which becomes the powder.

Normally because of the agitation, the coagulated particles will float on the surface as a foam or froth. Since fresh liquid B is being fed into the tank, the overflow at the top carries off the foamed powder as it is produced from the paint. Since the fresh liquid B is fed into the tank below the paint nozzle, the mixture of liquid B and the paint solvent (liquid A) also flows out the overflow thus preventing the liquid in the container from becoming saturated with solvent.

While this method is the preferred embodiment for continuous production of powdered material, it is apparent that any method of coagulation can be used provided that it permits the rapid removal, as by dilution, of liquid A from finely dispersed droplets of paint and sufficient dilution of liquid A in liquid B to prevent resolubilization and agglomeration of the particles.

After coagulation has occurred, the powder particles are removed from the liquid mixture by filtration or any other method of separating solid from liquid.

To prevent agglomeration of the particles, i.e., the clumping together of numerous small particles to form larger ones, it is desirable to wash any remaining traces of the solvent (or liquid A) from the wet powder. This can be done either by using fresh liquid B or another liquid which is miscible with the liquid A and not a solvent for the paint powder. This other liquid can be the liquid used in transporting the powder if the powder is wet packaged as described below.

The mixture of liquids A and B or the liquid that has been used to wash the powder can then be recycled in a closed system. The heat advantage of this process is that little or no solvent is lost to the atmosphere thereby avoiding air pollution. Consequently, there is no limita-
tion on the choice of solvents other than the limitations imposed by the properties of the powder paint. It is desired to employ a solvent that enables economic use of solvents which would be unacceptable for use in liquid paints.

Once the powder has been washed, it may be screened to eliminate any undesirable sizes of particles. For example, it may be desired to have only particles less than 40 microns in diameter for wet spraying application. Electrostatic spraying is more efficient when there are neither very large nor very small particles of paint in the powder, thus it may be desirable to screen out particles below 20 microns and above 80 microns in size. This wet screening can be done using any conventional method. An advantage to wet screening is that no fine paint particles enter the atmosphere as dust.

The particles that have been screened out, and are not desired for some other application, can be recycled into the original paint by redissolving them in liquid A, which they will easily do because no irreversible change has occurred in this manufacturing process.

If the powder is going to be eventually applied in a liquid form, such as in the expanded application of Edward P. Hoffman et al., attorney's file number B&P 71061-M, then the powder may be stored and packaged without drying since the powder is already wet and its final application is wet.

If the powder is to be applied in dry form such as with electrostatic spraying or fluidized bed applications, the powder can be dried by any conventional technique such as air drying or spray drying.

In order to control the particle sizes resulting from the coagulation step, it is necessary to accurately control both the mixture of the paint and the physical conditions of the coagulation.

Figs. 2 and 3 represent typical graphs which we have prepared and have found helpful in determining the mixture of the paint or composition necessary to achieve a predetermined distribution of particle sizes. The first axis x shows the weight percentage of the non-solvent portions of the paint or material (i.e., film forming portion of the liquid paint), this includes both portions soluble and insoluble in the solvent or liquid A. The soluble portions include usually plasticizers, resins, and other organic compounds; the insoluble portion includes pigments. It is not essential that there be an insoluble portion to the paint, but, if there are, then they may act as nucleation agents for the soluble portion during coagulation, thus promoting homogeneity of the composition. The second axis y shows the weight percentage of the solvent, which for convenient reference we have designated as liquid A. While a single solvent is normally used for simplicity, it is to be understood that with some material systems a mixture of more than one solvent may be desirable to manufacture a powder having a certain distribution of sizes. The third axis z represents the coagulating liquid, which is called liquid B. Again liquid B may be composed of a single liquid or a combination of liquids. While liquid A must be a solvent for at least a portion of the paint solids, liquid B cannot be a solvent, or at least must be a very poor solvent, for any of the paint solids. Further, liquids A and B must be miscible and more readily they mix with each other the easier it becomes to control the coagulated powder.

Referring more specifically to FIG. 2, the line 4 represents the point for each mixture of a liquid paint having a specific percentage of paint solids, liquid A (or solvent), and coagulating liquid B at which coagulation begins to occur. Line 6 represents the point at which the maximum, such as 95%, of the paint solids have coagulated with the slow addition of further liquid B. Similarly in FIG. 3, line 5 represents the onset of coagulation and line 7 represents the near completion, i.e., 95% of coagulation for another paint system.

It will be noted that as the percentage of liquid A increases, the lines representing onset and completion of coagulation merge. The spread between these lines where there is little liquid A present depends upon the elements in the resin or paint solids system. In general, the more complex is the resin system, the further will the two lines be separated. And the further the two lines are separated, the longer it will take, for a given mechanical system to cross these lines in dilution by liquid B. And the longer it takes to go from the onset of coagulation to the completion of the process, the larger will be the resulting powder particles. So if, as is usually the case, it is desired to obtain particles having as small size as possible, then there should be an increase in the solvent content (i.e., thin out the paint).

However, as the solvent content of the paint increases, the process becomes less efficient in that more solvent must be used, and thereafter recovered from the coagulating liquid, liquid B. Further, the more solvent there is in the paint, the less solid portion can be present and the amount of paint powder produced by a given system goes down. Thus a balance must be struck between the desired production capacity and the desired particle size of the powder produced.

For example, using the system depicted in FIG. 2, samples were manufactured having solvent content as shown at points 10, 20, 30, 40 and 50 using the same mechanical system. The powders that were coagulated were analyzed and the resulting particle size distributions are shown in FIG. 4 where point 10 corresponds to the powder made at point 10 in FIG. 2, and so forth. The logarithmic horizontal scale, μ (μu), in FIG. 4 indicates the diameter of the particles of powder in microns while the linear vertical scale shows the volume of powder in percent having diameters greater than the indicated micron size. It is apparent that if particles having sizes below 30 microns are desired, point 50 should be used with this system. Similarly, once a representation has been made as in FIG. 2 or 3, and the analysis performed of the resulting sizes as in FIG. 4, it is possible to determine the optimum amount of liquid A to be used in coagulating the paint.

This analysis applies for a mechanical system which is fixed, as well as for predetermined liquids A and B. As these factors are varied, the computation will have to be redone.

For a given paint, liquid A, and liquid B, the size of the coagulated powder can be controlled to a large extent by the mechanical system employed. The essential characteristic of the mechanical system is the rapidity with which the liquid paint is completely blended with liquid B. This in turn is believed dependent to some extent upon the size of the liquid paint droplets, as the smaller droplets would appear to more quickly and completely blend than larger droplets. Further, the size of the droplets would appear to be related to the final particle size. Therefore, the faster the blending and the smaller the size of the paint droplets, the smaller will be the resulting paint particles.

In the preferred embodiment described above as the third method, the smaller the size of the nozzle for a given pressure and agitation, the smaller will be the average size of the powder produced. Similarly, the higher the pressure, the smaller will be the average size of the powder particles although this effect is not as pronounced as the effect of the nozzle size. Agitation also has some effect upon the particle size, but this is not of great importance provided the agitation is sufficiently vigorous.

It will thus be noted that a powder made from a system using higher pressure and smaller nozzles for injection of the paint into liquid B will have the same sizes as a powder made from a paint having proportionately more liquid A to non-solvent portions of the paint with a lower pressure and larger nozzle arrangement.

One restriction on the type of paints that can be made into powders utilizing this process arises from the fact that certain film forming ingredients (plasticizers) are...
liquid at normal manufacturing and storage temperatures. When powder paint is formed containing such plasticizers and the powder remains in a liquid medium, no difficulties are presented. Thus this limitation does not apply to powders that are stored and used in the wet condition. However, when it is desired to turn that powder paint containing such film formers into a dry powder, the liquid film former tends to cause the paint powder particles to become "lacky" or to agglomerate into unusable masses.

This problem can be alleviated by restricting the amount of the liquid film formers to below a predetermined critical film fraction. For example, if di-2-ethylhexyl phthalate (DOP) is used in a paint, the problem will not arise unless more than 10 to 15% of DOP is present in the final dry powder when made and stored at room temperature. Another way to alleviate this problem is to manufacture and store the dry powdered paint at a low temperature at which the film former, which is liquid at normal temperatures, becomes solid.

Referring to FIG. 1 of the drawings, the manufacturer of a liquid paint to be used in the process is indicated at "Paint Manufacturer" and includes the conventional steps for the manufacture of liquid paint, modified only in the selection of a solvent portion, liquid A, which will be miscible with liquid B. The thus formed liquid paint may then be directly added to the liquid B in the coagulating chamber as indicated by the phantom flow arrow 100, to which liquid B is supplied as indicated at 102, or there may be a premixing of the liquid paint and liquid B short of coagulation, and then the mixture shocked by adding it to a large quantity of liquid B as indicated by flow arrow. We have found that liquid A may conveniently be acetone for a variety of liquid paint compositions and liquid B may be water.

Following coagulation or precipitation, the wet powder may be collected and passed to a filtration stage where, by conventional means, the wet powder is separated from the blend of liquids, and the blend of liquids A and B is then separated from each other for reuse. Such separation may be effected by distillation or other convenient technical. The filtered powder may be washed with either more liquid B or another liquid not a solvent for the powder, and then the powder screened for size. Undesired particles may be returned to the original liquid paint manufacturing stage for recycling. Following screening, the desired particles may be either wet packaged, or dried and then packaged.

In order to demonstrate the versatility and broad application of this method, the following examples are offered:

**EXAMPLE I**

A conventional liquid paint was made by grinding 25 grams of carbon pigment, 50 grams of Rohm & Haas Acryloid A-101 acrylic ester resin, and 125 grams of acetone in a steel ball mill for 40 hours until a pigment dispersion with a fineness of 7 measured by the Hegman grind gage was produced. This was then added along with 10 grams of acetone to the coagulated resin produced by mixing 1500 grams of Rohm & Haas Acryloid B-66 acrylic ester resin with 4500 grams of VM&P naphtha. After mixing in a food blender for one hour the paint became homogeneous. While still agitating the liquid paint, water was added as the coagulating liquid, at 500 increments until coagulation just began to occur. At this point we immediately added 2000 more grams of water to effect precipitation of the powder. The powder was then collected and separated from the liquid blend, filtered, washed, and dried.

**EXAMPLE II**

Fifty grams of the same liquid paint as described in Example I was poured by hand into 300 grams of water while agitating the water by a high speed mixer, such as the Waring Blender Model 1120. The coagulated paint was then filtered and desiccated in air for 16 hours. The screen analysis showed that approximately 21% of the powder particles were below 50 microns in size.

**EXAMPLE III**

A liquid paint was made by grinding 300 grams of titanium dioxide with 50 grams of lampblack, 25 grams of talc, 250 grams of Vinslyn 130 thermoplastic resin and 600 grams of acetone in a steel ball for 16 hours until a pigment dispersion of 7 on the Hegman grind gage was produced. To this dispersion was then added another 600 grams of Hercules Vinslyn 130 thermoplastic resin and 400 grams of acetone and such was mixed until homogeneous. The liquid paint was then pumped under a pressure of 100 p.s.i. through a nozzle having 0.028 inch diameter into a 30 gallon drum containing 20 gallons of water agitated by a saw-toothed 6-inch blade rotating at 2000 r.p.m., by a high speed mixer, such as the Shar Model S-20, and the saw-tooth blade and the nozzle were in close juxtaposition and submerged beneath the surface of the water. After filtering, rinsing, and drying the coagulated powder, the analysis showed that at least 50% of the powdered paint particles were below 50 microns in size.

**EXAMPLES IV-VIII**

In this series of examples, 70% by weight of thermoplastic acrylic polymer resin was dissolved in 30% by weight of Union Carbide's Ethyl Cellosolve. No pigment was added to this resin. Acetone was then added to portions of this solution as indicated in Table I below. These mixtures are indicated by the appropriate reference in the table to FIG. 2. Water, used as liquid B, was then uniformly added to these mixtures to bring the mixture up to the respective points 11, 21, 31, 41, 51 on line 8 just prior to coagulation. Then each mixture was pumped into a drum as described in Example III where the nozzle was of 0.065 inch diameter under a pressure of 800 p.s.i. and where the blade speed was 3200 r.p.m. The resulting powder was dried and the distribution of particle size and distribution for each powder is given by the corresponding reference in FIG. 4.

<table>
<thead>
<tr>
<th>Weight Percent</th>
<th>Reference in</th>
<th>FIG. 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>70% resin and</td>
<td>70% Ethyl</td>
<td></td>
</tr>
<tr>
<td>20% Cellosolve</td>
<td>Acetone</td>
<td></td>
</tr>
<tr>
<td>80%</td>
<td>20</td>
<td>12</td>
</tr>
<tr>
<td>60%</td>
<td>30</td>
<td>22</td>
</tr>
<tr>
<td>80%</td>
<td>30</td>
<td>32</td>
</tr>
<tr>
<td>60%</td>
<td>40</td>
<td>42</td>
</tr>
<tr>
<td>80%</td>
<td>50</td>
<td>52</td>
</tr>
</tbody>
</table>

**EXAMPLE IX**

One hundred grams of Neville Chemical Co.'s. LX1000 petroleum hydrocarbon resin was dissolved in 50 grams VM&P naphtha. This mixture was poured by hand into a high speed mixer containing acetone as a coagulant liquid B. The coagulated paint was then filtered, washed, and dried. A fine powder was produced having particle sizes essentially similar to those shown in FIG. 4.

**EXAMPLE X**

The same as Example IX except that the coagulant (liquid B) was isopropyl alcohol.

**EXAMPLE XI**

A varnish powder was made by dissolving 100 pounds of Neville Chemical Co.'s. LX1000 petroleum hydrocarbon resin in 10 gallons of linseed oil by heating the oil to 550° F. for 40 minutes. After the mixture was cooled to 100° F., a pound cobalt naphthenate (6%) was added along with 50 pounds of VM&P naphtha and thoroughly mixed. This mixture was coagulated using the equipment described in Example III under a pressure of 800 p.s.i. through a nozzle having 0.056 inch diameter into a drum containing isopropyl alcohol agitated with the blade re-
volving at 2000 r.p.m. The powder produced, after washing and drying, had powder particle diameters between 40 and 50 microns.

Families of miscible solvents and coagulants are indicated in Table II. The requirement for these families is that liquid A be a solvent for at least a part of the paint or resin system and be miscible in liquid B which is not a solvent for any element of the paint or resin.

<table>
<thead>
<tr>
<th>TABLE II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid A</td>
</tr>
</tbody>
</table>
| Xylene... | Hexane or other | Alkyd, polyesters, styrene and vinyl 
| Toluene... | para-xylene | modified poly- 
| Ethylacetate | | acrylics. |
| Alcohol... | Low molecular weight | Polyether resins, varnishes, varnishes, 
| Toluene... | acetone or ketones. | such as alcohols and aliphic 
| Hexane... | | varnishes. |
| Water... | Mineral acids in water | Useful for water soluble paints, 
| Alcohol... | | especially sodium stearates. |
| Acetone... | Water | Of greatest importance because 

As mentioned hereinbefore the term film forming portion of a liquid paint will include the film formers, and the pigments, if any, when used in the following claims.

What is claimed is:

1. The method of making powder paint comprising the steps of:
   making a liquid paint having a solvent portion and a film forming portion including a pigment, finely physically dividing the liquid paint in the presence of a coagulating liquid, which is miscible with the solvent portion but not a solvent for the film forming portion, into minute droplets of uniform composition having a size proportional to the desired powder paint particle size, diluting said droplets with a sufficient quantity of said coagulating liquid to remove the solvent therefrom and precipitate the film forming portion as powder paint particles, and separating the powder paint particles from the mixture of said solvent portion and coagulating liquid.

2. The method defined by claim 1 characterized in that the coagulating liquid is water.

3. The invention defined by claim 1, characterized in that the solvent portion of the paint is acetone and the coagulating liquid is water.

4. The invention defined by claim 1 characterized by drying the precipitated powder paint.

5. The invention defined by claim 1 characterized by separating the solvent portion and the coagulating liquid following precipitation of the powder paint, and reusing the solvent portion as aforesaid in subsequent making of liquid paint.

6. The invention as defined by claim 5 characterized by washing the separated coagulating liquid as aforesaid in subsequent making of powder paint.

7. The invention defined by claim 1 characterized by washing the paint particles, and screening the particles for size.

8. The invention defined by claim 7 characterized by washing the paint particles with the coagulating liquid.

9. The invention defined by claim 7 characterized by mixing selected sizes of the powder following screening and while in the wet state into a slurry for coating application.

10. The invention defined in claim 7 characterized by drying the powder.

11. The invention defined by claim 1 characterized by washing the paint particles, screening the wet paint particles for size, and drying the selected size paint particles to provide a dry powder paint.

12. The invention as defined in claim 1 characterized by controlling the proportion of the solvent portion to the film forming portion in the liquid paint in inverse relation to the desired powder paint particle size.

13. The invention defined in claim 12 characterized by controlling the quantity of the coagulating liquid to regulate agglomeration of the precipitated powder paint particles.

14. The invention defined in claim 13 characterized by diluting the droplets with a sufficient quantity of the coagulating liquid to precipitate the film forming portion of the individual droplets as discrete substantially nonagglomerating powder paint particles.

15. The invention defined in claim 1 characterized by controlling the quantity of the coagulating liquid to regulate agglomeration of the precipitated powder paint particles.

16. The invention defined in claim 1 characterized by diluting the droplets with a sufficient quantity of the coagulating liquid to precipitate the film forming portion of the individual droplets as discrete substantially nonagglomerating powder paint particles.

17. The invention defined by claim 1 characterized in that the step of dividing the liquid paint is carried out by subjecting the liquid paint to a rapidly moving mixer blade immersed in the coagulating liquid and diffusing the droplets through such liquid.

18. The invention defined by claim 1 further characterized by recycling the coagulating liquid and the solvent portion for reuse in the method described.

19. The invention defined by claim 1 characterized by separating from the powder paint particles those of undesired size and resolving them in a solvent which is miscible with the coagulating liquid and repeating the method described with respect thereto to make powder paint therefrom of desired particle size.

20. The invention defined by claim 1 characterized by wet screening the powder paint particles to remove particles of undesired size, resolving the particles of undesired size in a solvent which is miscible with the coagulating liquid, and repeating the method described to make powder paint therefrom.

21. The invention as defined in claim 1 characterized by agitating the liquid paint and during such agitation adding thereto a coagulating liquid which is miscible with the solvent portion but is not a solvent for the film forming portion, at a rate and in an amount to precipitate the film forming portion as powder paint particles, and separating the powder paint particles from the mixture of coagulating liquid and solvent portion.

22. The invention defined in claim 21 characterized in that the coagulating liquid is added to the agitated liquid paint in an amount less than that to cause coagulation, and while continuing the agitation suddenly further adding a sufficient quantity of the coagulating liquid to rapidly precipitate the film forming portion.

23. The invention as defined in claim 1 characterized by agitating the coagulating liquid which is miscible with the solvent portion but is not a solvent for the film forming portion, and during such agitation slowly adding the liquid paint thereto to precipitate the film forming portion as powder paint particles, and separating the powder paint particles from the mixture of coagulating liquid and solvent portion.

24. The invention as defined in claim 1 characterized by vigorously agitating the coagulating liquid, which is miscible with the solvent portion but is not a solvent for the film forming portion, by the rotary blade of a high speed mixer immersed in the coagulating liquid, injecting the liquid paint into the coagulating liquid near the blade to cause precipitation of the film forming portion as powder paint particles, and separating the powder paint particles from the mixture of coagulating liquid and solvent portion.

25. The invention as defined by claim 24 characterized by diluting the liquid paint with a coagulating liquid which is miscible with the solvent portion but is not a solvent for the film forming portion in an amount less
than that causing coagulation of the film forming portion prior to injecting the liquid paint into the coagulating liquid.

References Cited

UNITED STATES PATENTS

3,661,864 5/1972 Tapp et al. 260—96 R

3,561,003 2/1971 Lanham et al. 260—34.2
3,575,900 4/1971 Ponyik 260—34.2

ALLAN LIEBERMAN, Primary Examiner

U.S. Cl. X.R.

260—34.2, 37 R, 40 R, 41 R, 41 A, 41 B, 94.9 F, 96 R