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BLENDING METHOD AND APPARATUS

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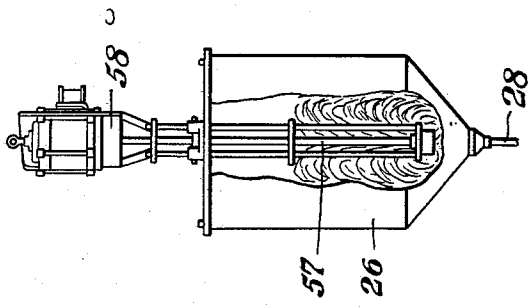


Fig. 2.

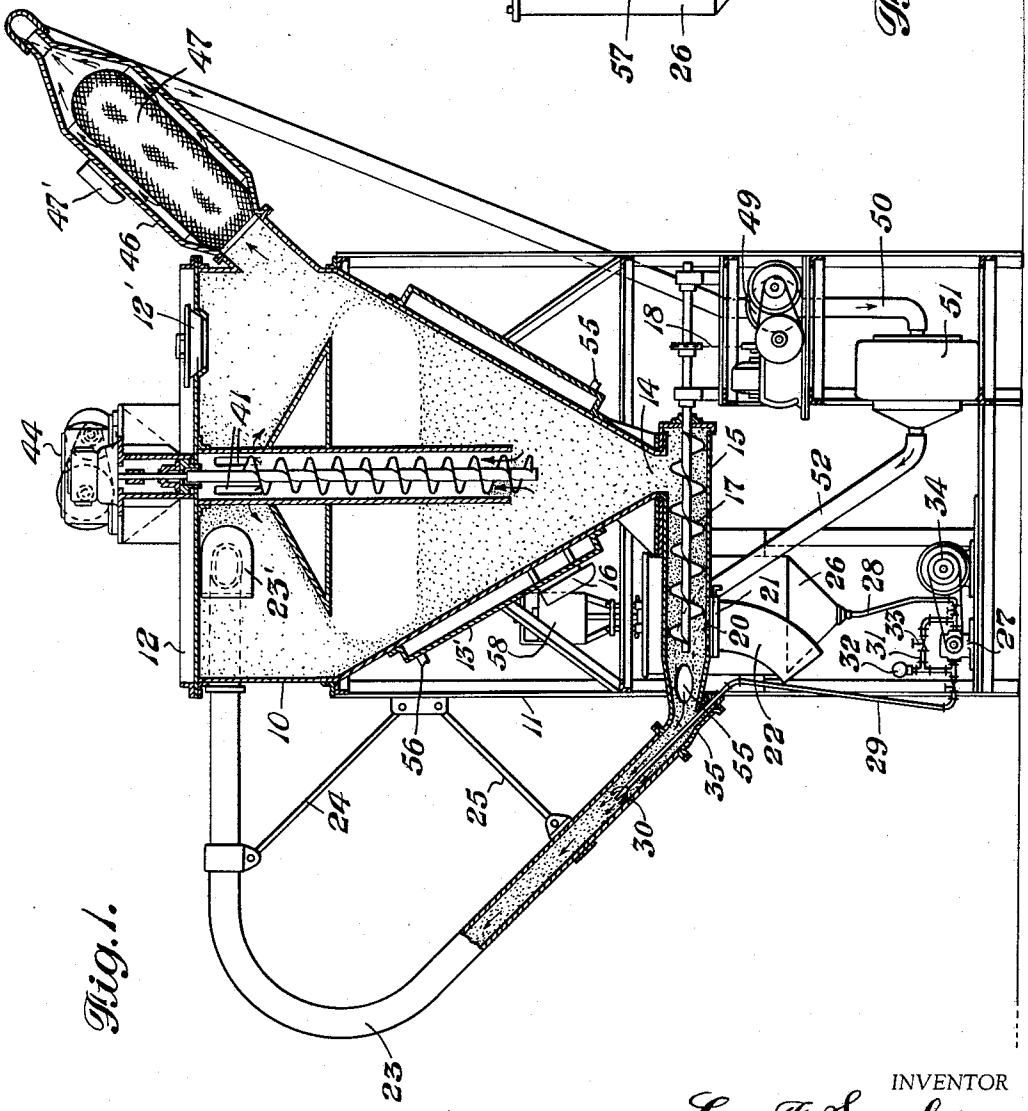


Fig. 1.

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BLENDING METHOD AND APPARATUS

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The present invention relates to the treatment of solid materials in particle form and more particularly to a method and apparatus for adding a fluid material to a powdered or granular material to obtain a uniform dispersion of the fluid in the solid material.

This case is a continuation-in-part of copending case Serial No. 177,670, filed August 4, 1950, now abandoned, entitled Blending Method and Apparatus.

In many processes it is necessary to condition solid materials which are in particle form by the addition of treating agents in fluid form wherein it is essential that the agents be uniformly distributed in the solid particles. This is particularly difficult to accomplish when the amount of agent to be added is relatively small as compared to the quantity of solid particles in which it is to be distributed.

In accordance with methods heretofore employed for this purpose, the material in particle form is dissolved in a suitable solvent to which the fluid material is added in liquid form. The materials are suitably blended in various ways during or after which the solvent is evaporated. The resultant material is then mechanically disintegrated to form the solid material again in particle form in which the fluid is uniformly dispersed. This method is relatively slow and expensive and requires the use of machinery which is heavy and expensive in relation to its capacity to produce finished material. Further, such a process cannot be successfully employed with heat-sensitive materials due to the degradation of the product because of the relatively high temperatures to which the material must be subjected during the processing.

It has also been heretofore proposed to effect the incorporation of the fluid by what may be termed an essentially dry process in which the mass of powdered material in substantially dry state is agitated while the fluid material is added. One method which has been proposed was to tumble the mass of particles in a closed rotary tumbling vessel and to introduce the liquid by pouring or spraying, depending upon the tumbling action to disperse the liquid uniformly. It has been difficult by this method to achieve uniformly satisfactory dispersion of the liquid in the solid mass.

The general object of the present invention is to provide a new and improved method and apparatus for incorporating a fluid into a mass of solid particles in such manner as to obtain a uniform dispersion of the fluid in the solid particles.

A further object of the invention is to provide a method and apparatus for incorporating a liquid into a mass of solid particles while maintained essentially dry in such a manner as to obtain a uniform dispersion of the fluid in the solid particles.

A still further object of the invention is to provide a method and apparatus for dispersing a fluid in a mass of solid particles wherein the fluid is brought into contact with the individual particles of the solid mass while the particles are in suspension in a gas.

A still further object of the invention is to provide a

method and apparatus for uniformly dispersing a liquid additive in a finely divided thermoplastic material.

Other objects and the nature and advantages of the instant invention will be apparent from the following description taken in conjunction with the accompanying drawing, wherein:

Fig. 1 is a sectional view illustrating apparatus capable of carrying the invention into effect, and

Fig. 2 is an enlarged detail partly broken away of the mixer in the liquid tank.

Although the present invention has many applications, it will be specifically described for the addition of various agents to thermoplastic compounds in particle form. The agents to be added are usually in liquid form such as, for example, plasticizers, stabilizers, pigments, dyes, and the like. The agents, if not liquids, can be either dissolved or dispersed in liquid prior to introduction into the polymer.

This invention is of particular value in the treatment of heat-sensitive materials, such as vinylidene chloride resins, where it is necessary to eliminate such operations as hot milling for blending the plasticizer into the polymer, although it is equally applicable to the incorporation of additives to any thermoplastic material.

The incorporation of pigments into vinylidene chloride resins has been a particularly difficult problem. It has been the practice to pigment vinylidene chloride resins by using dry pigments that have been hammer-milled or processed in other types of dry grinding equipment. The ground pigment is then dry blended with the polymer in such equipment as double conical blenders, ribbon mixers and the like. This procedure does not give a uniform dispersion because: the pigment particle size varies over a very wide range; if the pigment particles pick up a like static charge to the polymer particles, they repel one another; and at best, there is only a mechanical mixture of widely varying size and gravity of materials that, upon vibration attendant to subsequent handling, will separate and form striations.

In accordance with the present invention, the solid particles are suspended in a gas moving at a high velocity. The liquid to be dispersed therein, which may include plasticizers, stabilizers, pigments, dyes, and the like in solution or suspension, is sprayed into the moving suspended particles in such a manner that the liquid comes in contact with individual particles of the solid; and further, the rate of flow of the solid particles and the liquid can be so metered that the solid can be recycled several times before the total quantity of liquid has been injected into the system, thus insuring a uniform dispersion.

It is of particular importance in the coloring of thermoplastic materials which are to be extruded into filaments, that the pigment particle size be extremely small. If large size pigment particles appear in the final filament, the filament is weakened at that point. If the pigment is purchased having the proper particle size in pulp form, that is, suspended in a liquid, the liquid is removed and the pigment can be dispersed in the plasticizer. If the pigment is purchased in the usual dry form, it must be ground to an average particle size of one micron. This is accomplished by grinding the pigment in a portion of the plasticizer in a ball mill or a similar type grinding machine. The particle size can be better controlled with a more uniform product than can be achieved by dry grinding. Pigments in this form can then be dissolved or dispersed in the total quantity of plasticizer prior to the introduction of the plasticizer to the polymer. By this procedure, each individual particle of the polymer will be dyed and subsequent separation and striations are avoided.

In the introduction of either powdered or liquid stabi-

lizers, or other additives the same method would apply as for pigmentation.

Referring to Figure 1, apparatus is shown which is suitable for carrying out the process described. The main hopper **10** is supported on a framework **11** at a suitable height from the ground. The upper end of the hopper is provided with a hinged cover **12** having a loading hatch **12'** through which the solid granular material is loaded into the hopper. The lower portion **13** of the hopper which is of frusto-conical shape is provided with a jacket **13'** which surrounds the hopper. The bottom of the hopper is provided with an opening **14** which communicates with a horizontal sleeve **15** near an end thereof that is closed. A vibrator **16** is attached to the outer side of the portion **13** of hopper **10** to prevent the particles from adhering to the hopper walls. A feed metering screw **17** operating in the sleeve **15** is driven by a belt **18** connected to a variable speed drive illustrated at **19**.

Near the open discharge end of the sleeve **15** is an opening **20** in the lower surface of the sleeve closed by a sliding valve **21**. Aligned with the opening **20** is a discharge spout **22** for removing the contents of the hopper when the operation has been completed. The open end of the sleeve **15** connects with a tube **23** which curves upwardly and discharges into the top of the hopper **10** against a baffle **23'**. The tube **23** is supported by supports **24** and **25** which are attached to the framework **11**.

A liquid tank **26** is supported by the framework **11** below the hopper **15**. The tank **26** is connected to a fluid metering pump **27** by a conduit **28**. A conduit **29** connects the pump outlet with a nozzle **30** which is located within the tube **23**. The pump pressure is controlled by means of a bypass **31** connecting the outlet side of the pump with the intake side. A gage **32** and valve **33** are provided in the bypass line **31**. The pump **27** is driven by a motor **34**. Conduit **29** enters the conveying tube **23** through an elbow **35** and extends concentrically with the tube **23** to the nozzle **30**.

Extending into the hopper **10** supported at the top is a vertical sleeve **40** open at the bottom and provided with a plurality of openings **41** at the upper end thereof. Immediately below the openings **41**, a conical baffle **42** is secured to the outer wall of the sleeve **40** tapering outwardly at the lower end thereof. A screw conveyor **43** is located within the sleeve **40**, and is driven by a variable speed drive **44** mounted above the hopper **10**. The tube **23** empties into the upper end of the hopper **10** at a point directly above the baffle **42** adjacent the openings **41** in the sleeve **40**.

An upwardly inclined outlet **45** for the conveying gas from the tube **23** is provided at the upper end of the hopper **10** at the opposite side from the point at which the tube **23** enters the hopper. An upwardly inclined filter housing **46** is connected to the outlet **45** and surrounds a filter bag **47**. A vibrator **47'** attached to the housing **46** vibrates the filter bag and thus returns the particles of solid material collecting therein to the hopper **10**. A conduit **48** connects the filter housing with a blower **49**, and conduit **50** connects the outlet from blower **49** with heat exchanger **51**. Conduit **52** connects the heat exchanger outlet with the horizontal sleeve **15** near the outlet thereof at point **55** to supply the gas at the proper temperature and velocity in which the solid particles are suspended.

The tube **23** is preferably made of stainless steel with a portion made of glass for purposes of visibility although any suitable material may be used.

When the apparatus is utilized for the absorption of a plasticizer by a thermoplastic material, it has been found essential to maintain an elevated temperature, the temperature range depending upon the thermoplastic material and its thermal stability as well as the boiling point of the plasticizer. In general, a temperature range of approximately 160°–250° F. is satisfactory. Further, it is necessary that this temperature be accurately controlled. To accomplish this purpose, the present invention includes the jacket **13'** surrounding the hopper **13**. A heat transfer fluid is circulated through the jacket **13'** entering at inlet **55** and leaving at outlet **56**. The fluid may be hot water or any other suitable medium for quickly bringing the particles in the hopper to the proper temperature. The heat exchanger **51** is utilized to control the temperature of the conveying gas. At the beginning of the operation, the gas is heated to the desired temperature. However, after the gas has passed through the blower several times, the temperature of the gas may increase to the point that the heat exchanger **51** must be used to cool the gas down to the desired temperature. The control of the heating medium and the cooling medium to the heat exchanger can be accomplished by use of a three-way modulating or proportioning valve.

The material in the liquid tank **26** is constantly agitated by means of a mixer **57** driven by a motor **58**. The use of the mixer **57** prevents the precipitation of any solids incorporated in the liquid in the tank **26**. A mixer which has been found particularly suitable for this purpose is the Eppenbach Homo-Mixer, described in United States Patent No. 2,393,360.

The gas used as the conveying gas should preferably be inert. Nitrogen has been found to be particularly suitable. One advantage of the use of an inert gas is to exclude oxygen, thereby preventing oxidation of the polymer and thus removing the combustion hazard.

The operation of the system in general is as folows: The system is first purged of air by flowing an inert gas thereinto. The powdered or granular thermoplastic material is loaded into the hopper. Preferably the hopper is not loaded to more than 50% of total hopper volume for best results. The heating medium is passed through the hopper jacket **13'**. The two screw conveyors **17** and **43** and the mixer **57** are started in operation as well as the blower **49** and the pump **27**. The gas is compressed in the blower **49**, passed through heat exchanger **51** and thence is injected into the particles of thermoplastic material being discharged from the hopper **10** through the sleeve **15** at point **55** by means of the screw conveyor **17**. The gas which has been heated to the desired temperature in the heat exchanger **51** and injected into the outlet of sleeve **15** picks up the solid particles likewise at the desired temperature and conveys them in fluid suspension into the tube **23**. Liquid from the tank **26** is pumped at the desired rate and discharged through the spray nozzle **30** into the moving gas suspension whereupon the liquid contacts the individual particles and is absorbed by them. The gas suspension is conducted via tube **23** to the upper end of the hopper **10** and contacts baffle **23'** where, due to decreasing velocities, the solid particles drop out of gas suspension. The gas, which probably contains some volatile material as well as some of the solid material, is recycled through the filter bag **47**, blower **49** and heat exchanger **51**. The material in the hopper **10** is continuously intermixed by means of the screw conveyor **43** which picks up material from an intermediate portion of the hopper and discharges it through the openings **41** at the top of the vertical sleeve **40**. The treated material returning from tube **23** is brought into contact with the untreated material flowing through the openings **41**.

The rates of feed of the liquid and the solid particles are adjusted so that the solid particles will have been recycled at least two times before the total amount of liquid has been introduced. After the liquid has all been added and sufficient recycling has been carried on to insure complete absorption of the liquid by the particles, the treated solid particles are then discharged by opening the slide valve **21** and conveying the particles through opening **20** and discharge spout **22**.

The particles suspended in the gas are caused to move at high velocities, preferably from 1500 to 4000 feet per minute. The bend in the tube **23** produces extreme turbu-

lence which causes any formed agglomerates to break up and further disperse.

The following specific examples are intended to illustrate the present invention but are not intended to limit the same:

Example I

After the air has been purged from the apparatus by flowing nitrogen through the system, 1000 pounds of powdered vinylidene chloride-vinyl chloride co-polymer is added to the hopper. The particles of the material are of such a size that 2% or less by weight are left on a twenty mesh screen. The resin is heated by the hopper heating jacket **13'** to approximately 90° C. The nitrogen gas is likewise heated in the heat exchanger **51** to approximately 100–110° C. The liquid in the tank **26** comprises a combination of a plasticizer, stabilizer and a pigment. The pigment particles have an average particle size of one micron. As a plasticizer, any one or combination of the following can be used, dioctyl phthalate, dimethyl phthalate, diethyl phthalate, diallyl phthalate, dioctyl adipate, dioctyl sebacate, methyl phthalyl ethyl glycollate, triphenyl phosphate or poylmeric polyester types of plasticizers. As a stabilizer any suitable organic or inorganic stabilizer may be used. In the present example, dibasic lead sulphate is used. Any suitable pigment can be utilized depending on the color desired, as for example, blue-copper phthalocyanine, white-titanium dioxide, and black-carbon black. The following proportions of the additives are used:

Plasticizer	8–10% of weight of resin.
Stabilizer	0.5–3% of weight of resin.
Pigment	Up to 1% of weight of product.

The liquid is pumped in at such a rate that the resin is recycled twice while the liquid is being sprayed in. The treated resin is recycled once more after completion of the addition of liquid. The gas with the particles suspended therein is moved through the system at a velocity of about 4000 feet per minute.

Example II

The hopper is loaded with cellulose acetate flake until aproximately one-half full. The tank **26** is filled with a liquid plasticizer comprising equal parts diethyl phthalate and dimethyl phthalate. The relative quantities of resin and plasticizer are in the ratio of 70 parts resin to 30 parts plasticizer. Nitrogen is utilized as the inert gas and the process described in Example I is utilized.

Example III

The hopper is loaded with a copolymer of vinyl-chloride-vinyl acetate in finely divided form. The tank **26** is filled with a mixture of dioctyl phthalate, lead stearate and dibasic lead phosphate. The dioctyl phthalate is a plasticizer, whereas the lead stearate serves as a lubricant and the dibasic lead phosphate as a stabilizer. The relative proportions utilized are:

	Parts
Vinyl chloride-vinyl acetate copolymer	100
Dioctyl phthalate	30
Lead stearate	0.3
Dibasic lead phosphate	1.5

The process of Example I is utilized, and the resultant product is a finely divided, uniformly plasticized and stabilized vinyl chloride-vinyl acetate copolymer.

The method described is essentially a dry method which maintains the particle mass in free-flowing condition during the process. To this end, the rate of introduction of the liquid into the tube **23** must be controlled as to rate and manner of introduction so as to avoid appreciable wetting of the wall of the tube since if that occurs agglomeration or lumping of the particle material ensues.

As will be apparent from the foregoing description, the invention in its several aspects has a wide field of utility applicable to many different specific materials and combinations thereof to produce many different kinds of desired end products.

It will likewise be obvious to those skilled in the art that various changes may be made without departing from the spirit of the invention and therefore the invention is not limited to what is shown in the drawings and described in the specification but only as indicated in the appended claims.

I claim:

1. The method of plasticizing a mass of finely divided thermoplastic material which includes the steps of continuously removing particles of the thermoplastic material from the mass, suspending the particles in a rapidly moving inert gaseous medium, gradually spraying the plasticizer into the suspended particles of thermoplastic material, separating the treated particles from the gaseous medium and returning the treated particles to the mass.

2. The method in accordance with claim 1, wherein the plasticizer has incorporated therewith a stabilizer and a pigment.

3. The method of plasticizing a mass of thermoplastic material in particle form which includes the steps of continuously removing particles of the thermoplastic material from the mass, suspending the particles in a rapidly moving inert gaseous medium, spraying the plasticizer into the suspended particles, separating the treated particles from the gaseous medium and returning the treated particles to the mass for recycling, recycling the treated particles at least two times before the total amount of plasticizer has been added to the suspended particles.

4. The method in accordance with claim 1 wherein the temperature of the mass of particles and of the gaseous medium is maintained relatively constant during the process.

5. The method in accordance with claim 1 wherein the inert gaseous medium used is nitrogen.

6. The method in accordance with claim 1 wherein particles of thermoplastic material are continuously removed from the mass and brought into contact with the treated particles being returned to the mass.

7. The method of plasticizing a mass of thermoplastic material in particle form which includes the steps of continuously removing particles from the bottom portion of the mass, suspending the particles in a rapidly moving inert gaseous medium, gradually spraying the plasticizer into the suspended particles, separating the treated particles from the gaseous medium, returning the treated particles to the upper portion of the mass, continuously removing particles from an intermediate portion of the mass, and bringing the particles from the intermediate portion of the mass into contact with the treated particles being returned to the upper portion of the mass.

8. The method in accordance with claim 7, wherein the temperature of the mass of particles and of the gaseous medium is maintained relatively constant during the process.

9. The method in accordance with claim 7, wherein the gaseous medium separated from the treated particles is filtered, pumped, passed in heat exchange relation and thence recycled to the system.

10. The method of plasticizing a mass of thermoplastic material in particle form which includes the steps of continuously removing particles from the bottom portion of the mass, suspending the particles in a rapidly moving inert gaseous medium, gradually spraying the plasticizer into the suspended particles, separating the treated particles from the gaseous medium, returning the treated particles to the upper portion of the mass for recycling, continuously removing particles from an intermediate portion of the mass, and bringing the particles from the intermediate portion of the mass into contact with the treated particles being returned to the upper portion of the mass for recycling, recycling the treated particles at

least two times before the total amount of plasticizer has been added to the suspended particles.

11. The method in accordance with claim 1, wherein the thermoplastic material is a vinylidene chloride-vinyl chloride copolymer.

12. The method in accordance with claim 1, wherein the thermoplastic material is cellulose acetate and the plasticizer is a mixture of equal parts diethyl phthalate and dimethyl phthalate.

13. The method in accordance with claim 1, wherein the thermoplastic material is a vinyl chloride-vinyl acetate copolymer and the plasticizer has a stabilizer incorporated therein.

14. Apparatus for treating a mass of material in particle form with a fluid treating agent comprising a hopper for the mass of particles, a heating jacket surrounding said hopper, said hopper having an opening at the lower end thereof and an opening near the upper end thereof, a sleeve communicating with said lower opening, a screw conveyor operating within said sleeve for conveying particles out of the said hopper, a tube connecting the said sleeve with the opening at the upper end of the hopper, means for injecting a rapidly moving gaseous conveying medium into the said tube near the connection between the tube and the sleeve, a nozzle in the said tube slightly upstream from the point of injection of the gaseous medium for spraying the fluid treating agent into the said tube, a source of supply for the fluid treating agent connected to the said nozzle, a vertical sleeve extending into the hopper open at the lower end and having openings at the upper end,

a baffle surrounding the sleeve located below the said openings, a screw conveyor operating within the vertical sleeve for conveying particles upwardly from the mass of material and discharging said particles through the said openings against the said baffle, said upper opening in the said hopper located adjacent the said openings in the vertical sleeve to intermix the particles being discharged through both the hopper opening and the sleeve openings.

15. Apparatus in accordance with claim 14, wherein a second opening is provided near the top of the hopper for discharge of the said gaseous conveying medium, an upwardly extending housing connected to said second opening, a filtering means located within the said housing, and conduit means connecting the said housing with the said tube for recycling the gaseous conveying medium.

16. Apparatus in accordance with claim 15, wherein a heat exchanger for the gaseous conveying medium is interposed between the filtering means and the said tube.

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