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METHOD OF CONVERTING NONAQUEOUS PULPS

Emile C. De Stubner, Charleston, W. Va.

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The present invention relates to a process for incorporating finely divided solids into vehicles which may form a part of ultimate consumer products. The process achieves a fine subdivision of the finely divided solids and their substantially uniform and complete dispersion into the vehicle, while eliminating the customary grinding of such solids into the vehicle.

Finely divided solids dispersed in a dispersion medium to form a pulp have an individual particle size and a completeness of dispersion which is very desirable to maintain in the consumer product. The dispersion mediums which act best as pulping mediums for finely divided solids are mobile liquids. The formation of pulps of finely divided solids dispersed in a mobile pulping medium of an aqueous liquid has heretofore been suggested. The use of such aqueous pulps and their incorporation directly into the commonly utilized vehicles has not been satisfactory because such vehicles usually are less mobile than such pulping mediums and are immiscible therewith. The presence of the aqueous liquid in the pulp thus tends to impair the quality and completeness of the dispersion and is not practical from the commercial standpoint because of the difficulties inherent in removing the water from the completed dispersion. I have found that mobile liquids of a non-aqueous nature may be used in forming the initial pulp. Such pulps may be formed from pulps in which the solid particles may be pulped directly into the non-aqueous mobile liquid as the pulping medium.

In the present process, therefore, I propose to utilize the desirable properties of the pulp form of finely divided solids in a mobile non-aqueous pulping medium which is relatively more volatile than the vehicle into which the solids are to be incorporated in the process. By so doing it is possible to secure dispersion of the finely divided solids directly into the vehicle without grinding the solids in the vehicle. The pulping mediums which may be used satisfactorily in the present process are chiefly mobile liquids of a non-aqueous nature which are miscible in or compatible with the selected final dispersion mediums and are relatively more volatile than such dispersion mediums. In each instance such pulping medium should be selected which has a surface tension lower than the surface tension of the final dispersion medium and one which has a preferential affinity for the finely divided solid particles, so that the pulping medium acts as an efficient wetting agent for the finely divided solids. Among such pulping mediums are liquids in the field of organic solvents, such, for example, as toluene, benzene, acetone, turpentine, and the like. The use of such pulping medium will be found to preserve the beneficial features of the pulp formed of finely divided solids in a mobile pulping medium in such a manner as actually to facilitate the transfer of the finely divided solids to the final dispersion medium which may be the vehicle of a consumer product.

The present application is a continuation in part of my co-pending application Serial 579,662, entitled Method and process of converting non-aqueous pulps.

Thus one of the objects of the present invention is to utilize the non-aqueous pulp form of finely divided solids in the art of compounding. It is well known that finely divided solids are now combined in a direct manner with other elements in which combination these finely divided solids are the necessary and desired constituents. A great number of various types of mechanical devices are employed by chemical manufacturers in the preparation of such combinations, and it is my purpose to disclose a process and method whereby these finely divided solids in pulp form may be more readily and advantageously combined with such products, in order thereby to simplify such processes, while at the same time producing superior products inasmuch as these finely divided solids will be more uniformly distributed in such products by the present process than by previously known processes.

It is a further object of the present invention to provide a method for converting non-aqueous pulps of finely divided solids and to displace or replace either wholly or partially the non-aqueous pulping medium of such pulps by other constituents or vehicles of the desired product. In other words, to disclose a process adapted to the incorporation of finely divided solids such as abrasives, fillers, colors, and the like, into products useful as and/or in the manufacture of adhesives, insecticides, leather, leather finishes, artificial leather, printing ink, soap, candles, cosmetics, lubricants, rubber goods, tires, paint, paint enamels, matt varnishes, varnish stains, oil stains, putty, wood finishes, metal finishes, flatting compounds, polishes, floor wax, sealing wax, coating compositions, thermo-plastics, molding compounds, plastics, Celluloid, lacquer, oilcloth, linoleum, etc.

In a preferred embodiment of the present invention, a further object is to disclose a method of replacing the volatile pulping medium of the finely divided solids in pulp form in its vapor phase

under atmospheric pressure, increased pressure, or diminished pressure (vacuum), by a non-volatile constituent, the latter constituent being a component also of the final and/or consumer product. Such preferred embodiment consists in general in the transformation of the solids of non-aqueous pulps having a volatile pulping medium into non-volatile constituents, vehicles, or composites.

Further and additional objects of the present invention will more readily appear in the perusal of my specification and examples.

While the present process possesses economical advantages over present methods, the product made in accordance with my teachings is of excellent quality and derives a distinctive and pleasing appearance and/or superior effectiveness from the uniformity and evenness of size and dispersion of the finely divided solids, whether abrasive, color or filler, from the nature and characteristics of the parent pulps having a non-aqueous volatile pulping medium. This mark of uniformity of appearance and superior effectiveness which distinguishes the individual batch or run may be found in any number of successive batches or runs. Thus variations of or in manufacture may be substantially eliminated by the present process which excludes to a large extent the human element and substitutes therefor a control of the process based on the physical constants of the volatile pulping medium as well as the relatively non-volatile replacing or displacing constituents.

The quality of the above-enumerated products depends largely upon the fineness of their incorporated solids and to an even greater extent upon their even distribution in their vehicles. It is technically far easier to obtain pulps of these solids when the pulping medium is a volatile mobile liquid in which the solids possess a high degree of fineness, than in an oil or heavy viscous or plastic vehicle. Therefore, I have selected and desire to employ these solids in their pulp form, preferably utilizing non-aqueous pulping mediums of a relatively volatile nature. This is distinguishable over present manufacturing methods which utilize the finely divided particles in the dry state. I have discovered that such a pulping medium may be displaced or replaced by a non-volatile vehicle incorporating the solid constituents of such pulps by applying this principle of replacement or displacement in my novel process to obtain a most even and uniform distribution of said solids into the vehicle.

For the purpose of clarifying the terms used herein, I hereby define "pulps" as being any soft or semi-liquid mixtures of solid particles in liquids, the liquids being hereinafter referred to as the pulping mediums.

By the use of the term "non-aqueous" I intend to include substances chiefly of an organic nature which are substantially free of water or aqueous liquids and by the use of such term to exclude the substances which are chiefly of water or of an aqueous nature.

The term "dispersion medium" as used herein includes substances forming a component element of the end or consumer product and which may exist either as a liquid or a plastic under the conditions existing at the time of the volatilization of the non-aqueous pulping medium. Such dispersion medium may exist under normal atmospheric temperatures and pressures, either as a solid or plastic such as a resin, or as a liquid such as an oil.

In order that the present invention may be more clearly understood and the process of converting pulps of finely divided solids having a non-aqueous volatile pulping medium into compounds having non-volatile constituents, it is essential that the non-volatile constituent must displace or replace the volatile pulping medium and that this must be accomplished at the same time that the finely divided solids are uniformly distributed throughout the non-volatile constituent or product.

The following examples are illustrative of the operation of my invention and processes and are here included for such purposes and not for the purpose of limitation.

Example I

In order to make "Prussian Blue in Oil" I proceed as follows: 100 lbs. of toluene Prussian-blue pulp of 30% pigment content is mixed with 30 lbs. of linseed oil. This mixture is then subjected to distillation and agitation until the 70 lbs. of toluene of the Prussian-blue pulp are distilled off.

In practice I prefer to employ a kneading machine of the type of Werner & Pfleiderer or other suitable type which is provided with a jacket, hollow agitating blades, a tight cover, a still neck, an attached condenser, an inlet valve which permits feeding for continuous operation, and thermometer wells for measuring the temperatures of the machine contents as well as the vapor in the still neck. In such an apparatus distillation may proceed under atmospheric pressures or under either increased or diminished pressures. In such apparatus I place 1000 lbs. of Prussian-blue toluene pulp of 30% pigment content and 300 lbs. linseed oil. The blades revolve at a speed of 60 R. P. M. The cover is tightly closed and connected with the still neck and condenser, which in turn are tightly connected with a 300-gallon receiver and the latter joined with a vacuum creating source. The temperature of the mix is now raised to 75° C. by means of suitable means, either in the jacket or in the hollow blades. While this batch is agitated by the blades which exert a smearing or kneading or squeezing action, the vacuum line is opened and a vacuum of 25 to 27 inches of mercury is created inside the apparatus. Under these conditions the toluene is distilled off and there remains in the apparatus 600 lbs. of "Prussian Blue in Oil" (in this case linseed oil) which may be discharged through a valve in the bottom or by tilting or any other convenient means.

Example II

To incorporate carbon black into rubber as both filler and color, I place in the apparatus described under Example I, 1000 lbs. carbon black benzene pulp of 10% carbon black content and 300 lbs. of washed crepe rubber under a vacuum of 12 to 15 inches, a temperature of 60° C. and while the mixing blades are in motion at a rate of between 30 and 40 R. P. M., the benzene is volatilized and distilled off until there remains in the apparatus a compound consisting of 300 lbs. rubber and 100 lbs. carbon black. If zinc oxide is desired as a filler and/or color, then instead of carbon black pulp a zinc oxide pulp of 25% zinc oxide content is used, but otherwise the process proceeds the same as in the case of carbon black.

Example III

In order to make black printing ink base, I mix 60 lbs. of printing ink varnish with 200 lbs. of acetone carbon black pulp of 20% pigment

content and drive off the acetone by distillation until 100 lbs. printing ink base remain.

If desired, an apparatus similar to that disclosed in Example I may be used in which case I will use 1000 lbs. carbon black acetone pulp of 20% pigment content and 300 lbs. printing ink varnish. I raise the temperature to 60° C. and keep mixing blades revolving at about 60 R. P. M. I place the machine under vacuum of 20 inches and distill off the acetone until I have a batch of 200 lbs. carbon black and 300 lbs. printing ink varnish.

Example IV

In order to make yellow printing ink, I place in the apparatus of Example I, 1000 lbs. of neutral lead chromate acetone pulp of 30% pigment content and 400 lbs. printing ink varnish. I raise the temperature of the contents to about 60° C., having the blades revolve at from 60 to 80 R. P. M., and apply a vacuum of 20 inches which are favorable conditions for distilling off the acetone. During the distillation, I feed from the pigment pulp tank through an inlet valve an amount of pulp equal in volume to the acetone distilled off and collected in the receiver, until the composition in the machine consists of 600 lbs. neutral lead chromate and 400 lbs. of printing ink varnish.

Example V

For making "Chrome Orange in Japan", I place in the apparatus of Example I, 1000 lbs. of 50% basic lead chromate acetone pulp and 500 lbs. of "Grinding Japan" and distill off the acetone.

Example VI

For making a red lacquer base for coloring lacquer of cellulosic base, I proceed thus: 1000 lbs. of toluidine red alcohol pulp of 16% pigment content and 300 lbs. of a compound consisting of two parts of ester gum and two parts of dibutyl phthalate, and one part of castor oil are placed in the apparatus and at a temperature of 65° to 80° C. and under a vacuum of 20 to 25 inches with the blades revolving at 60 R. P. M., the alcohol is distilled off and the resulting red colored composition of 160 lbs. toluidine red and 300 lbs. compound forms the base for coloring clear lacquer.

Example VII

To make base white for coating compositions for artificial leather, I place in the apparatus similar to that of Example I, 1000 lbs. of titanium dioxide alcohol pulp of 25% pigment content and 500 lbs. of castor oil and distill off the alcohol under conditions as in Example VI, and add another 1000 lbs. of pulp as in Example IV, (continuous feed) to finally obtain a white paste of 500 lbs. of titanium dioxide and 500 lbs. of castor oil which may be used to color clear pyroxylin for white coating compositions.

Example VIII

In order to make red sealing wax, I place in the apparatus of the type disclosed in Example I, 1000 lbs. of vermilion alcohol pulp of 10% pigment content and 900 lbs. of shellac or the uncolored sealing wax compound and distil off the alcohol as under Example VI.

I may in a similar way, proceed to convert the fillers or pigments or abrasives necessary in t production of any of the products outlined abc or other similar consumer products or interme ate products.

In all of the foregoing examples of proces embodying the present invention, it will be not that in each instance a pulp of the finely divid solids in a non-aqueous pulping medium, whi is relatively more volatile than the dispersi medium, is admixed with the dispersion mediu and is then volatilized and dissociated from t mixture, whereupon the finely divided solids a transferred to and suspended in the dispersi medium. In each instance the pulping mediu is compatible with or miscible in the dispersi medium prior to its volatilization and remov from the admixture.

While I have named particular substances f particular purposes in the foregoing examples, do not wish to be limited thereby to the utiliz tion of such substances or such purposes as t present process is adaptable to many other pu poses, using many substances other than tho particularly specified.

I claim:

1. A method of dispersing finely divided soli in a selected organic dispersion vehicle in a sub stantially water free condition which consists i forming a preliminary non-aqueous pulp of fine divided solids in a substantially water free or ganic liquid pulping medium for said solids, sai pulping medium being selected particularly fc its compatibility with the selected dispersion ve hicle and chosen from the group of such pulpin mediums which have a greater wetting action o the solids than does said selected dispersion ve hicle, and which are directly miscible with th said selected dispersion vehicle and which ar relatively more volatile and have a relativel lower surface tension than said dispersion vehicl then adding said preliminary suspension directl to the selected dispersion vehicle and thereafte simultaneously distilling off the pulping mediur and kneading the mass whereby the dispersio vehicle replaces the pulping medium and th finely divided solids are uniformly distribute throughout said vehicle.

2. A method of dispersing finely divided solid in a selected organic dispersion vehicle in a sub stantially free water condition which consists i forming a preliminary non-aqueous pulp of finel divided solids in a substantially water free or ganic liquid pulping medium for said solids, sai pulping medium being selected particularly fc its compatibility with the selected dispersion ve hicle and chosen from the group of such pulpin mediums which have a greater wetting action o the solids than does said selected dispersion vehi cle, and which are directly miscible with the sai selected dispersion vehicle and which are rela tively more volatile and have a relatively lowe surface tension than said dispersion vehicle, the adding said preliminary suspension directly t the selected dispersion vehicle and thereafte simultaneously distilling off the pulping mediur and kneading the mass under a vacuum whereb the dispersion vehicle replaces the pulping me dium and the finely divided solids are uniformly distributed throughout the said vehicle.

EMILE C. DE STUBNER.