

UNITED STATES PATENT OFFICE

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METHOD OF PREPARING DISPERSIONS

No Drawing.

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This invention relates to an improved method of preparing colloidal suspensions, emulsions and the like.

Hitherto it has been proposed to colloidize substances, generally, by a variety of methods, the more permanent results of which involve, in the case of solid materials, trituration in the presence of neutral solvent, or formation of the desired product by chemical precipitation.

These methods have not met with any material success for reasons which are not as yet clearly known.

In colloidizing it is generally understood that the question of size of the particles, when dealing with solid materials, is a controlling factor, and inability to attain a desired range of particle size has militated against the effectiveness of purely mechanical preparations. To overcome this difficulty it has been proposed to subject solid particles, however prepared, to the action of various organic substances known as "stabilizers" or "peptizing" agents. The preferred stabilizers cover a rather wide range of organic materials, usually of starchy or feculous nature, used alone, or in conjunction with gums, resins, and like materials. A common characteristic of colloidizing agents, so called, is the necessity of having them substantially alkaline, in order to exert a desired protective or colloidizing action. The best known of the agents to be used has been ammonium tannate, which is a reaction product of tannic acid and ammonia set forth in various disclosures.

It is an object of the present invention to provide an improved method of preparing stable colloidal suspensions and emulsions.

It is a further object of this invention to provide a novel method which is adapted to improve the colloidizing of materials generally, including mineral and vegetable oils and the like.

Yet another object of this invention is the provision of the use of an improved colloidizing agent of a chemical nature used with any suitable mechanical means.

Yet another object of this invention is the combination of the improved colloidizing

agent with improved mechanical means.

These and other desirable objects and advantages of the present invention will be described in the following specification, preferred embodiments being given by way of illustration only, for, since the underlying principles may be incorporated in other specific methods and devices, it is not intended to be limited to the ones herein disclosed, except as such limitations are clearly imposed by the appended claims.

In the practice of the present invention a suitable material to be colloidized, as for example, a pigment, is ground or triturated in a suitable vehicle, such as varmospirits or cellosolve. By subjecting such material to a very high degree of mechanical disintegration, such as may be obtained by the types of disintegrators generally known as mills, sometimes referred to as "colloid mills", it is possible to secure a relatively stable suspension of particles of the pigment in the vehicle. The particle size ordinarily obtainable by mechanical means ranges, generally, from .5 to 2.0 micra. To secure a suspension of substantially permanent stability, it is necessary to attain a particle size of about .5 micra or less. As will be readily appreciated the mechanical difficulties involved in the preparation of particles of this size is very great, due to the fact that in the region between the microscopic and ultramicroscopic, the particles have a tendency to flow in the direction of the rotating element of the disintegrator, thereby preventing any reaction upon themselves by the opposed grinding or crushing surfaces. Such a condition is remedied, to a considerable extent, by the use of a mill of the type described in my co-pending application, Number 301,804, in which the attrition or disintegration of the particles is effected by forcing them under positive pressure between two flexibly supported counter-revolving opposed discs. In such a type of instrument there are set up opposed forces, which, acting at high speed, exert a positive disruptive effect upon any particles positioned between the counter-revolving discs. Such a

device gives an improved result, and with this type of machine the particles retain what may be defined as a relatively permanent stability of solution.

5 While starchy materials and the like have been proposed as adjuvants in the practice of such processes, their use involves considerable trouble, both in time and labor involved in preparation, as well as the inability to secure
10 compositions of known physical and mechanical characteristics whereby uniform results could be obtained at will. A further marked disadvantage of the stabilizers noted, is the high cost on the market. Tannic acid, the
15 best-known material used in this connection, commands a price of substantially \$1.35 a pound. Such prices are prohibitive and preclude the use of these materials to a great extent, or, in a great variety of processes
20 which could be benefited to a marked degree by the application of the principles of colloid chemistry in their manufacture.

As a result of numerous experiments with a wide variety of materials which could possibly be used as adjuvants to the mill disclosed
25 in the application above referred to, it has been found that excellent results can be secured by the use of organic wood derivatives, or the like, the preferred composition being
30 that known in the art as "sulphite waste liquor". This liquor is, as its name implies, a waste product from the process of making paper pulp. The tonnage of this product is enormous and pulp mills generally, are now
35 after many years of experimenting, endeavoring to find a method of disposing of this material and look to finding a substantial market for it.

Various methods have been adopted to make this so-called waste material available
40 for many uses, its main use, heretofore, being restricted to its application as a substitute for pitch, or the like as a cementing or binding agent for artificial fuels, roadways, etc.
45 This sulphite material is further treated by desiccation and the powdered product used for the purposes above enumerated.

In the practice of the present invention the inclusion of a moderate amount of sulphite
50 liquor in the aqueous vehicle above considered, when subjected to the action of the improved mill heretofore referred to, produces a remarkable increase in efficiency of the colloidizing process, and in fact, gives a
55 result that materially surpasses any result heretofore obtained by any agent used alone.

One great advantage of the agent under discussion is its conjoined action with the mill referred to, besides the fact that it is neutral,
60 as for example, inks prepared from pigments in a suitable mill, of the type described, in the presence of sulphite liquor, showed no deposition of particles over a considerable period of time, and it is further to be noted
65 that the compositions under test contained

neither glue, gelatine, tannic acid, dextrine, or feculous material of the types normally considered necessary for stability.

Any suitable quantity of sulphite liquor may be used for the purposes described, various materials requiring different amounts to impart the necessary stabilizing property of the product. Generally from 4 to 10 per cent of the adjuvant will be sufficient for light weight materials and liquids, while as much
75 as 20 to 40 per cent may be required for heavy materials. The specific amounts required will depend entirely upon the particular material being treated and will have to be determined by the user in a preliminary
80 test or series of tests.

While the sulphite liquor, or its equivalent in the desiccated condition, gives improved results in the ordinary methods of preparation, such as hand-grinding in a mortar, or grinding in a ball-mill or colloid mills of the fixed and rotating or nested cone types, the combination with a device of the counter-revolving opposed disc type noted above, gives very remarkable results. Whether
85 these exceptional results are due to the mere improvement in the mechanical destruction of the particles by the counter-revolving discs, or is to be ascribed to what may be termed the improved wetting power of the sulphite solution, is not known, but the fact of the improved results obtained by the use of such a combination of means, is herewith set forth for the purpose of fully illustrating the present invention, and for definitely
90 stating the remarkable results.

Material prepared in the above manner, examined under the ultra-microscope, shows a particle size substantially below .5 micra in diameter, and is further characterized by the fact that the particle size is substantially uniform throughout the whole mass, there being no aggregates or clumps of material such as is commonly found in the materials prepared by the ordinary colloidizing processes, where there is a distinct gradation in particle size. This uniform occurrence and distribution of particle size in the materials prepared according to the process of the present invention, markedly increases the reactive capacity of such materials and involves, as a consequence, a distinct increase in economy of preparation of any desired product, it being well known that the reaction rate is a direct function of the surfaces of the reacting materials exposed to one another.
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Another feature of considerable scientific and commercial importance, is that the use of the relatively cheap sulphite liquor does away with high-priced alkaline reagents such as ammonia, and the like, the neutral sulphite liquor requiring no care or calibration in its use. The neutrality of the material used is a highly desirable condition to be obtained, as it is well known that the maximum results,
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relative to stability of suspensions, emulsions, etc., are obtained at or near the neutral point, i. e., in a solution having a p_H of about 7.0 or slightly above.

The rapidity of action of the improved stabilizer of the present invention permits of great economies in point of time, the reacting materials merely requiring a single treatment in an approved grinding and disintegrating apparatus to assure complete reaction.

In the preparation of paints, inks, and other like pigment base material, it is possible to produce tons of a finished product in the time formerly required for as many ounces. In addition, the fineness of the resulting particles and their intimate association or dispersion in the carrying medium insures homogeneity of product, which feature makes for a marked economy, both in sale as well as in use.

While the sulphite liquor has been discussed with relation to the preparation of colloidal materials of the suspensoid type, it is equally efficient when used in conjunction with the mills of the type disclosed in my said application in the preparation of suspensions of liquid in liquid, of the mutually insoluble or immiscible types, which products are commonly known as emulsions. The desideratum in the preparation of emulsions is the dispersion of a body of liquid disintegrated into fine particles or globules, as a disperse phase in a continuous phase of the carrying liquid.

Emulsions hitherto have been stabilized by the addition of glues, gelatine, soaps, starches, and other stabilizing materials which have materially affected the quality of the emulsion and the cost of manufacture. While not markedly deleterious in their effect the ordinary stabilizing agents have not been particularly efficient, which fact may be attested by housewives, to whom the instability of the average prepared emulsion of edible oil in acidulated water, commonly known as "mayonnaise," is well known. This trouble is due to the tendency of the dispersed oil particles to coalesce into large globules, until they finally attain a sufficient size to separate out from the body of the emulsion. This separating out is a progressive action which is stimulated by the presence of any large globules, and is generally the outstanding difficulty met with in industries where one or more liquids are dispersed into a liquid vehicle. The ramifications of the problems involved in preparing suitable emulsions extend into the paint and varnish industry as well as foods and pharmaceuticals.

The combination of means set forth in the present invention enables the preparation of stable emulsions, generally, due to the fact that the globules or particles of liquid to be dispersed are literally torn apart after be-

ing greatly attenuated, and the protective action of the sulphite, or other adjuvant materials is instantly rendered available, thereby preventing coalescence of the dispersed particles and the consequent formation of globules.

It will now be seen that there has been provided an improved process for colloidizing solid and liquid materials, generally, which process permits the use of a relatively cheap waste product to secure highly efficient and economical results, as well as permitting the preparation of new combinations of materials which have hitherto not been available.

The simplicity of operation and freedom from involved processing steps is in marked contrast to the hitherto obtaining methods and permits increased efficiency in manufacture, distribution and sale of the resulting products.

What is claimed is:

1. The method of preparing colloidal suspensions comprising subjecting the material to be dispersed to the conjoint action of sulphite waste liquor and a grinding surface which is flexibly supported.

2. The method of preparing colloidal suspensions comprising subjecting the material to be dispersed to the conjoint action of sulphite waste liquor and counter-revolving flexibly supported opposed disintegrating surfaces.

3. The method of preparing colloidal suspensions comprising subjecting the material to be dispersed to a preliminary grinding in the presence of sulphite waste liquor, and subsequently subjecting the resulting product to the action of a plurality of flexibly supported counter-revolving opposed disintegrating surfaces.

4. The method of preparing materials from relatively immiscible fluids comprising subjecting the material to be dispersed to a preliminary grinding in the presence of sulphite waste liquor, and subsequently subjecting the resulting product to the action of a plurality of flexibly supported counter-revolving opposed disintegrating surfaces.

5. The product of the conjoint action of sulphite liquor and flexibly supported grinding surfaces characterized by a particle diameter of substantially less than .5 micra.

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