

Jan. 7, 1936.

W. W. CARTER

2,027,090

MEANS AND METHOD FOR DISPERSING MATTER IN MANUFACTURED MATERIAL

Original Filed June 30, 1930

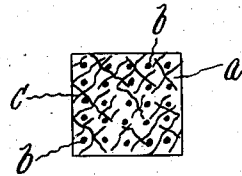


Fig. 1.

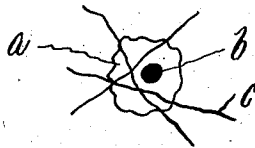


Fig. 2.

Inventor:
William W. Carter,
by Wright, Brown, Quincy & May
Attys.

UNITED STATES PATENT OFFICE

2,027,090

MEANS AND METHOD FOR DISPERSING
MATTER IN MANUFACTURED MATERIALWilliam W. Carter, Needham, Mass., assignor to
Brayton Morton, Fall River, Mass., trusteeApplication June 30, 1930, Serial No. 464,824
Renewed March 5, 1934

18 Claims. (Cl. 92—21)

The present invention is concerned with methods and means for effecting dispersion and retention of certain matters, substances or particles throughout and in the fabric or body of manufactured articles or goods of various descriptions; and with the products of such methods, consisting of articles and manufactures containing other matter in dispersed condition. It has two principal objects; first, that of incorporating in dispersed condition in a manufactured product a substance which, by reason of its adhesiveness or similar qualities, would be liable to adhere with detrimental effect to parts of the machinery used in making the product, and, second, that of distributing through and retaining in, a product which is made by assemblage of particles suspended in a liquid vehicle, particles or elements of different specific gravity which would, without assistance, normally tend to concentrate in a stratum instead of being distributed throughout the mass of the product, or would otherwise tend to leave the product with the liquid vehicle when the latter is drained off. The general objects are to prevent clogging and other objectionable effects on the manufacturing apparatus by the dispersed matter when such matter is of an adhesive nature; to insure effective distribution of the dispersed matter through the product being manufactured; and to avoid waste and loss of this material in the course of the manufacturing process.

The invention is a generic one applicable in the manufacture of many various products for the incorporation therein of various dispersoids. One important field of use is in connection with the manufacture of paper and similar products for incorporating therein substances to give desired qualities to the paper, such as impermeability by water or other fluids, enhanced insulating qualities, greater mechanical strength or stiffness, electrical conductivity, abrasive quality, desired color effects, etc. I can best explain the nature of the invention by describing its application to a particular illustrative use, and for that purpose in the present specification I will choose its application to paper making, particularly paper impregnated with phenolic resin, and compressed dielectric panels made of such paper for radio apparatus. This, however, is by way of illustration and not limitation.

In the paper manufacturing industry it has long been recognized as desirable to incorporate into the fundamental structure of the fabric, by introduction into the beater or Hollander, wherein the paper stock is prepared, large quantities of materials of a gummy or sticky nature, such as

latex, tars, resins, soaps, glues, casein, and the artificial or synthetic substitutes for these natural products. Many and various processes for doing so have been suggested, or both suggested and patented, and to some extent successfully used. These processes generally consist in introducing into the beater the desired material in the form of an emulsion produced by mechanical means, or by physico-chemical means with the aid of suitable emulsifying substances, and then beating the pulp and emulsion together and sometimes breaking up the emulsion or precipitating its suspensoid by suitable electrolytic agents, and passing the resulting mixture to the paper forming machines.

These processes have serious objections. One is the adhesion of the sticky added material to the surfaces of piping, pumps and containers and to the screens, felts and rolls of the paper making machinery, with the result of clogging the screens and seriously impeding or stopping the paper making process or otherwise spoiling the product. Other objections are that the losses of added material with the waste water draining out of the paper are usually very large, the cost of chemical reagents is high, and in some cases the coagulating or precipitating electrolyte used injuriously affects the product, especially if the product is made for electrical insulation.

The object and accomplishment of my invention includes the avoidance and overcoming of these objections. This is accomplished by the production of a colloidal dispersion in which the substance desired to be added constitutes the dispersed phase, and the continuous phase is a colloid capable of coagulating to a firm gel enveloping the dispersoid. The gel is subdivided into particles small enough to give the proper distribution throughout the paper stock when added thereto in the beater. Fibers are also bound into the gel in such manner as to protrude from the particles into which the gel is divided and form tentacles capable of becoming entangled and of felting together with the fibers of the paper stock, and of preventing the particles of dispersoid, which are enveloped in the gel, from being carried away by the waste water and lost. Such tentacle fibers are introduced into and distributed throughout the liquid used to make the colloid dispersion which becomes the continuous phase, before coagulation of the colloid. They may with equal effect be introduced into the material constituting the dispersed phase, but practical and mechanical considerations make it

preferable in most cases to introduce them independently into the liquid as first above stated.

The fibers become locked in the gel when it coagulates; and when the gel is subdivided they do not break, but, owing to their great strength relatively to the strength of the gel, they pull out of the particles in which they are but slightly encased and remain attached to those particles in which they are more firmly or more deeply embedded. The result is that most, if not all, of the small separated gel particles carry each one or more fibers projecting at one or both ends beyond its boundaries in condition to be caught by the wire of the paper machine, or to become entangled with the fibers of the main material in the mixture to which these prepared particles are added, so that loss of the particles by passing through the screens or otherwise being carried off with the waste water is prevented.

The drawing herewith provided shows in Fig. 1 a section of coagulated gel greatly magnified;

Fig. 2 shows a separated gel particle still further magnified.

In both figures, *a* represents the colloid, the black dots designated *b* represent particles of the dispersed phase scattered through the colloid; and the lines *c* represent the fibers. Fig. 2 shows in principle how after subdivision of the gel the particles thereof contain bits of the dispersoid and projecting fiber tentacles. It is not asserted that every gel particle will contain a nucleus of the dispersoid and fiber tentacles exactly as here depicted. Some particles may divide from the mass without containing either, and others may have a greater or less number of particles of dispersoid and a greater or less number of encased and projecting fibers; but generally the particles will contain one or more globules or fragments of dispersoid and one or more fibers substantially as shown.

In making the specific illustration of the invention previously mentioned, bakelite paper for the production of radio panels, the following procedure, using materials and proportions as herein-after stated, is suitable.

100 pounds of dry alpha cellulose is treated with caustic soda and carbon bisulphid according to the well known method of making viscose. The resulting sodium thio-carbonate of cellulose is converted into a smooth paste by the addition of sufficient water to bring the cellulose concentration to about 15%. Into this solution is run slowly and stirred rapidly about 800 pounds of the "A" form of the phenol-formaldehyde condensation product. This is the viscid solution which settles from the liquid in the wet process of making bakelite. When the dispersion of the phenolic resin is substantially complete and uniform, water containing 2% of well beaten sulphite wood pulp fibers in suspension is added, slowly and with rapid stirring to the amount of about 1200 pounds. This mixture is then allowed to gel naturally, or gelling may be hastened by careful heating to a temperature of about 70° C. or slightly more. The resulting gel is aged until it begins to throw out water and is firm enough to hold together when dumped from the container. In this condition the gel is a solid, containing water, but not soluble in water or subject to disintegration by the action of water alone, and it is not sticky. But it is attached or bonded with considerable firmness to the fibers contained within it; and forms an envelope for the sticky dispersoid which prevents the latter from adhering to anything. This product constitutes an embodiment of the col-

loidal dispersion, containing a dispersoid and scattered fibers, of the preceding general description.

The gel is now introduced into a beater containing 500 pounds of paper stock already well beaten and in which the beater roll is adjusted so as just to brush the fibers. The gel is added slowly while the beater continuously operates. In a period ranging from half an hour to one hour the gel becomes divided into fine particles which are dispersed substantially uniformly through the pulp mixture, these particles consisting of the viscose colloid enveloping, or encysting, globules of phenol resin and carrying protruding fibers.

The contents of the beater are now delivered to a paper machine and made into paper, which is formed and dried in the usual way. In passing through the beater and the paper machine, the particles of sticky resin are protected by the envelope of colloidal gel from contacting with the surfaces of the machinery and sticking to the wire and clogging it. The fibers protruding from the gel particles retain them in the web of paper being made.

After the paper has been dried it is cut to the desired size and a number of sheets are stacked upon one another sufficient to make a finished panel of the required thickness. This stack is molded under heat and hydraulic pressure in a mold. This treatment causes the particles of resin to expand and their colloidal envelopes to be crushed; actions which together liberate the fluid resin, which then spreads and permeates the entire fibrous mass. Panels so made are at least equal, and generally are superior, to those made by previous processes and their cost is substantially less.

It will be understood that the foregoing specific example is not in any sense a limitation to quantities nor even to exact proportions, but is an illustration of the general principle by a concrete example. Even in the making of this particular type of paper, the process may be carried out continuously, with continuous introduction of the colloidal dispersion into the beater and continuous delivery of the entire mixture to the paper machine.

I have successfully made paper in accordance with the foregoing example, with complete absence of trouble of any kind due to the sticky phenolic resin. The resin did not stick to the beater or its rolls, or to the associated piping and pumps; did not clog the wire of the paper machine, and did not adhere to the felts or rolls of the paper machine or drier, or interfere in any way with the successful formation of the paper web and its passage between the felts and press rolls and through the drier. There was no appreciable loss of the expensive artificial resin in the waste water. The molding of the stack of sheets under hydraulic pressure and heat was successfully accomplished and produced a mechanically strong and electrically excellent dielectric of low power factor.

For general use in paper making, as well as in the particular example given, a viscose cellulose is practical and satisfactory for the colloidal dispersion, as it is inexpensive, gels naturally, and on regeneration reverts to a cellulose of the same nature as the paper itself and has sufficient strength for the purpose. However, I am not limited in this particular, for other compounds and substances may be used for the continuous phase in the making of paper and other products according to this invention. For instance,

the continuous phase may be other cellulose products than viscose, or other organic compounds such as albumen, glue, casein, starch, etc. The coagulation of the gel may be allowed to occur naturally or may be produced or hastened artificially by application of heat or use of a special agent, as formaldehyde or an acid, the particular agent being determined for the particular material used in the continuous phase by the known requirements under physico-chemical laws for coagulation of the colloid to the gel form. It is not essential that the gel be introduced into the beater immediately after coagulation and while still holding a large content of water. Its characteristics are such that it does not deteriorate by aging, or suffer any change except loss of water through drying; and after drying it will take up water again.

The fibers dispersed through the continuous phase for anchoring the gel particles may be of widely various characters. In paper making they are generally and preferably fibers of the same pulp stock of which the paper is made, but not necessarily so. They may be any fibers of vegetable, animal or mineral origin, and of natural or artificial structure, as wood, cotton, linen, wool, silk, hair, rayon, asbestos, metallic wire, etc. Artificial filaments are thus included within the category of fibers for the purposes and within the scope of this invention. Fibers of specific materials are selected, and may be colored, for ornamental effect, strength, or other desirable effects. Metal fibers may be used when the electrical conductivity or the color appearance, strength, hardness or other qualities characteristic of metals are desired.

For some purposes the combination of fibers and gel particles alone may be used without any nucleus of dispersoid in the gel particles, as in cases where the fibers themselves are the desired added material. In such cases the gel particles attached to the fibers act as buoys or floats when the fibers are of high specific gravity, preventing them from settling out of the main mixture in a concentrated layer.

The dispersoid may be any liquid, or solid granular or powdered, material which for any reason it may be desired to combine in dispersed condition with any manufactured product. In the class of liquids and semi-liquids are included natural or artificial substances as tars, bitumens, gums, waxes, resins, oils, soaps, latex or other rubber dispersions, etc. Solid dispersoids include such matters as insoluble pigments and dyes, abrasives, mineral fillers, etc. For instance, an abrasive paper superior to sandpaper, emery cloth and the like may be made by adding to the paper stock grit in the nature of sand, emery, carborundum or the like dispersed and incorporated as taught by this invention.

I have endeavored by the last preceding paragraphs to indicate the wide scope of the invention without attempting or intending to give an exhaustive list of the manufactures which may be made and the materials which can be dispersed through them, or the colloidal substances and fibers which may be used to effect dispersion and retention of the added matters in the products, according to this invention. In general any manufacture which at an unfinished stage is in a liquid state, or in a condition of liquid suspension, capable of having added materials disseminated through its mass is within the range of products producible by the invention. Any material, capable of being dispersed through the

liquid which is to become a gel, may be combined in the product as the dispersed added material. Any gel material capable of receiving a dispersoid before coagulation may be used, with this proviso, that the material must produce a gel of such strength and nature that it will persist as a gel when incorporated in the main mixture. Of course neither the gel material nor the added material should be of a nature to injure the main mixture.

The gel associated with either a nucleus of dispersoid or with anchor fibers, or with both, constitutes a mechanical operator or tool by means of which the added matter is brought into the substance of the main product. Combined with a nucleus, the gel particle is a mechanical operator which brings a chemical substance into the main product and protects or insulates such substance from the machinery and vice versa. In association with fibers, the gel particles are in some cases buoys or floats for the fibers and in other cases the fibers are anchors which retain the gel particles in the unfinished main product.

The gel particles are usable in three forms, as (a) the complete combination of the gel particle with its enclosed nucleus of dispersoid and attached fibers; (b) the gel and nucleus; or (c) the gel particle and attached fiber. These combinations I desire to protect broadly and specifically in all the applications and associations where they are useful. From one point of view, the gel particle in its association with a nucleus and a fiber constitutes a binder attaching the nucleus to the fiber, thereby performing a function additional to or independent of that of insulating the nucleus against possibility of adhering to the surfaces of apparatus.

What I claim and desire to secure by Letters Patent is:

1. The method of incorporating added material in dispersed condition throughout a fibrous product of manufacture which consists in first attaching separated particles of the added material with fibers, so that ends of fibers project from such particles, mixing said fibers and attached particles of added material with the fibers of the main manufacture in liquid suspension and separating the liquid vehicle from the mass of mixed fibers so that the fibers which carry attached particles become felted with the fibers of the main mixture.

2. The method of distributing fibers throughout the mass of a product in course of manufacture which consists in first distributing such fibers throughout a body of gel forming liquid, breaking up the gel resulting from coagulation of such liquid into particles smaller than the lengths of such fibers, and mixing the particles of gel with attached fibers throughout the main material of the manufacture in liquid suspension, and subsequently separating the liquid vehicle from the resulting mixture.

3. The method of incorporating an added material in dispersed condition in paper which consists in first attaching encysted particles of the dispersoid to fibers of a length sufficient to interlace with the fibers of paper pulp, apart from the after-mentioned furnish, then mixing the fibers and their attached particles of dispersoid with a paper pulp furnish, and forming the furnish with such admixture into paper.

4. The method of incorporating in distributed condition in paper, fibers of a different character from the paper fibers, which consists in attaching to such added fibers, apart from the

after-mentioned furnish a colloid which is assimilable with the stock of which such paper is made, separating such colloid into particles with attached fibers and mixing the same with a paper pulp furnish, and forming the furnish mixture into paper.

5 The method of incorporating an added material in paper which consists in dispersing the material to be added through a body of liquid cellulose viscose, coagulating the viscose into a gel, subdividing the gel into particles containing nuclei of the dispersoid, mixing the gel particles with a paper pulp furnish, and forming the furnish mixture into paper.

10 6. The method of making paper containing an added material in dispersed condition therein which consists in dispersing the added material throughout liquid viscose cellulose, distributing fibers of paper pulp throughout the viscose, coagulating the viscose, separating the coagulated gel into particles containing nuclei of the dispersoid and pulp fibers, distributing the particles throughout a paper pulp furnish, and forming the furnish mixture into paper.

20 7. The method of preparing matter for dispersion within a manufactured product which consists in mixing separated particles of such material throughout a body of coagulatable cellulose gel forming liquid so that such material becomes the dispersed phase of a colloidal dispersion in which the coagulated gel is the continuous phase, and permitting the gel to coagulate before incorporation in the product.

25 8. The method of preparing material for distribution within a manufactured product, which consists in mixing separated particles of such material throughout a body of liquid adapted to coagulate into a gel, also mixing through such liquid fibers of substantially greater length than the particles of the dispersoid, and permitting the gel to coagulate prior to incorporation in the product.

30 9. A colloidal dispersion for use in distributing a sticky material through a product in course of manufacture, comprising a colloidal gel as the continuous phase, adapted to be broken into discrete, non-unitable particles, separated particles of the material to be added constituting the dispersoid, and fibers of substantially greater length than the dimensions of the dispersoid distributed in non-felted separation from one another throughout the gel.

35 10. The method of manufacturing a product containing a different substance dispersed through its mass which consists in distributing the material to be added in dispersed condition throughout a gel forming liquid, subdividing the solid gel coagulation of such liquid into discrete particles containing particles of the dispersoid, and mixing the gel particles in liquid suspension with the material of the product in course of manufacture.

40 11. The method of incorporating an added material into a paper manufacture which consists in dispersing particles of such added material throughout a body of gel forming liquid, dividing

the solid gel resulting from coagulation of such liquid into discrete particles containing bits of the dispersoid, mixing such gel particles with paper stock in liquid suspension, and forming the resultant mixture into paper.

5 12. The method of incorporating added material in paper which consists in distributing the added material in small particles throughout a body of gel forming liquid, mixing fibers in dispersed condition through the liquid, allowing the liquid to coagulate into a gel and to age until it is firm enough to cohere when removed from its container, separating the coagulated gel into particles containing nuclei of the dispersed material and fibers protruding from said particles, mixing such particles with a paper pulp furnish, and forming the furnish mixture into paper.

10 13. The method of incorporating fibers or strands of other material than paper pulp fibers throughout paper, which consists in distributing such fibers in separated condition through a gel forming liquid which is assimilable with paper, coagulating such liquid into a coherent gel, subdividing the gel into particles smaller than the length of such fibers so that the fibers protrude from the particles, mixing the fibers with their attached gel particles throughout a paper furnish, and making the furnish mixture into paper.

15 14. The method of adding dispersed matter to a fibrous product which consists in first mixing fibers and dispersed added material with a gel forming liquid, allowing the liquid to coagulate into a gel, and then breaking up the coagulated gel into particles containing and enveloping the said added material, such particles being generally smaller in dimensions than the fibers so that ends of such fibers project from them, and mixing the broken particles with the stock of the main product in liquid suspension and forming the resulting stock mixture into the end product, with extraction of the liquid of suspension therefrom.

20 15. A paper manufacture comprising intermingled fibers substantially free from attached masses or films of added substances, and other fibers interlaced with the first named fibers and having in attached connection with them particles of added material encysted in colloidal envelopes.

25 16. A paper manufacture containing in distributed condition within its mass, particles of a different material bound into the paper by interfelting with the fibers thereof of other fibers to which the said different material is attached.

30 17. A manufactured product consisting of interlaced and felted fibers, discrete particles of extraneous matter encysted in colloidal gel bound to certain offset fibers, and other fibers being free of such attached particles.

35 18. A paper product comprising interfelted fibers and particles of gummy material encased in solid non-sticky colloidal envelopes and attached to scattered fibers of the mass in such manner as to leave the product substantially open and porous.

WILLIAM W. CARTER.