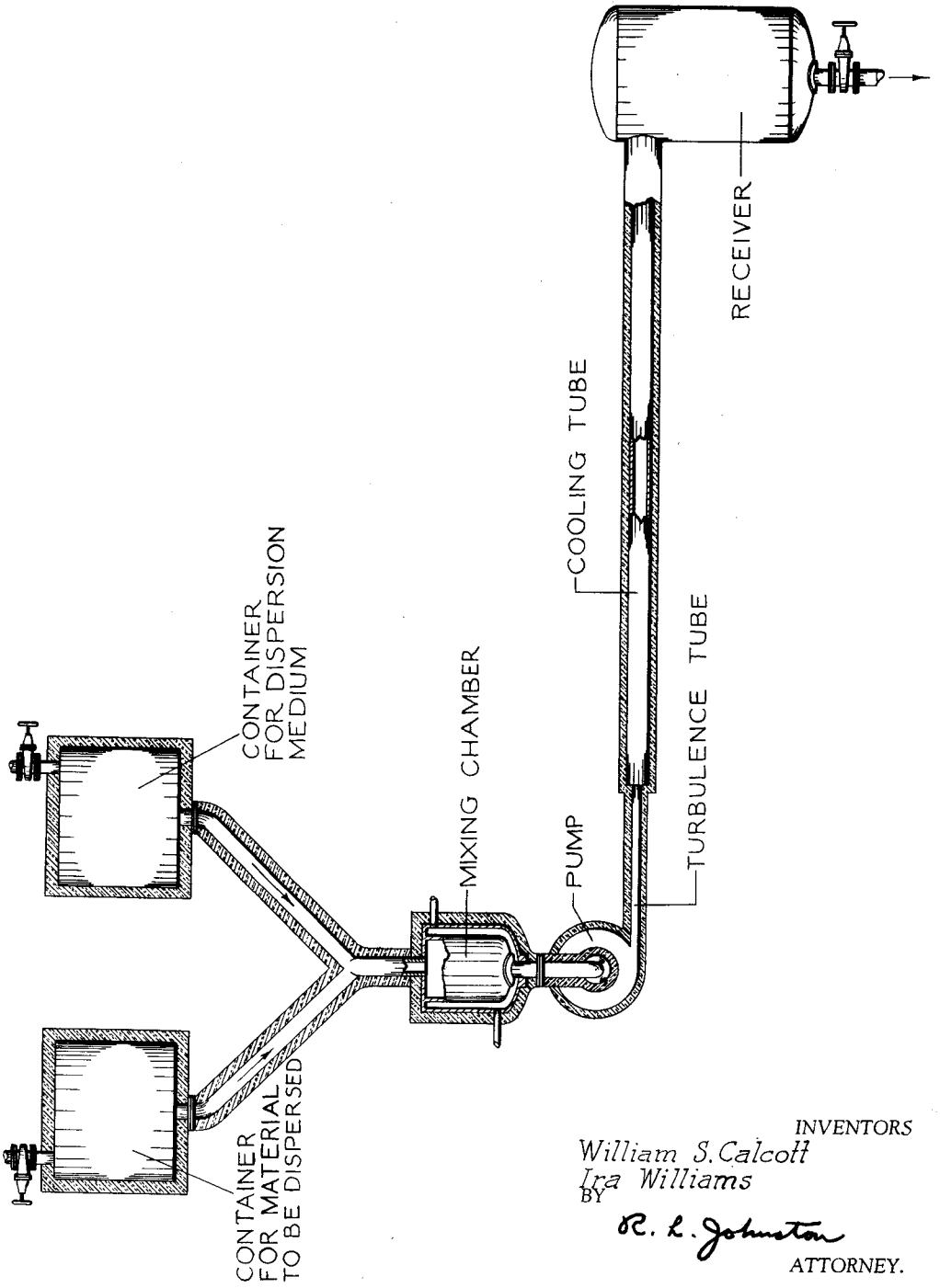
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PRODUCTION OF DISPERSIONS

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PRODUCTION OF DISPERSIONS

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This invention relates to finely divided substances, more particularly solid and liquid colloidal particles in dispersion media, and a process for the production thereof.

Many methods of producing colloidal materials have previously been used, such as, for example, the production of colloidal metals by means of an electric discharge, the precipitation of colloidal material by drowning a solution in a non-solvent, the precipitation of finely divided substances by chemical action and the reduction of particle size by grinding either in the presence or absence of other solid or liquid material. The production of emulsions of oils or low melting waxes in water may be accomplished by mechanical means, such as, for example, shaking or agitating with a mechanical stirrer, but in general, the method has been limited to those materials which are liquid, or which can easily be brought into a liquid condition. Furthermore, the various mechanical means for the production of emulsions and suspensions are generally attended by certain disadvantages, such as, for example, operation on a batch scale rather than continuously and the necessity for the use of power for operating the mechanical agitating means, which adds to the cost of operation. In many cases, moreover, rapid agitation by mechanical methods gives rise to excessive foaming.

It is an object of the present invention to provide a new and improved process for the production of solids and liquids in a fine state of subdivision. A further object is to provide a convenient method for the production of colloidal dispersions in a continuous manner. A still further object is to provide a method which facilitates the reduction to colloidal dimensions of substances which grind with difficulty, or which can be produced only in low concentration by solvent or chemical means. An additional object is the production of solid and liquid colloidal particles in dispersion media by a process characterized by simplicity and ease of operation. Another object is the provision of a method for the production of solid and liquid particles in disperson media by a process which proceeds smoothly without excessive foaming. Other objects will appear hereinafter.

These objects are accomplished by simultaneously passing materials to be dispersed and a dispersing medium, in liquid condition, through a tube at a velocity sufficiently great to produce turbulence, that is, at a velocity in excess of the so-called critical velocity.

While the invention is susceptible of considerable variation and modification in the manner of its practical application, particularly as regards the nature and proportions of the materials treated, the temperature and pressure of the treatment, the size of the turbulence tube and the exact method of procedure, the following examples will illustrate how the invention may be practiced.

Example I

Two gallons of light engine oil and two gallons of a 5% solution of sodium oleate in water were roughly mixed together in a 5 gallon closed container. Connected to the bottom of this container was a turbulence tube consisting of 40 feet of pipe ⅛ inch in inside diameter. Three hundred pounds of air pressure were immediately applied to the top of the liquid in the container. The 4 gallons of material were discharged in two and one-half minutes. The resulting product was a pure white colloidal suspension of oil in which the particles of oil when examined under the microscope were found to be in Brownian motion.

Example II

Two gallons of paraffin were melted in a closed container of 5 gallons capacity while 2 gallons of a 12% solution of sodium caseinate in water were heated in a similar container. A half-inch pipe issuing from the bottom of each container led into a small chamber of 50 cc. capacity which served as a mixing chamber. The outlet of the mixing chamber was connected to a turbulence tube consisting of 20 feet of iron pipe .2 inch in inside diameter. When both liquids were at a temperature of 70° C., a pressure of 800 pounds was applied to each container by means of a cylinder of nitrogen. The contents of the two containers were discharged in 4 minutes. On cooling to room temperature the resulting product was a thick paste which readily dispersed in water. On examining this diluted sample the particles of paraffin were found to be in Brownian motion.

Example III

One and one-half gallons of melted sulfur were placed in a steam jacketed container. In a similar container 3 gallons of a 15% solution of ammonium caseinate were placed. The bottom of each container was connected by means of steam jacketed pipes to a small jacketed mixing chamber. The outlet of the mixing chamber was closely connected to a jacketed pump which was capable of supplying any pressure below 1000

pounds per square inch. This pump was connected to a jacketed turbulence tube 0.2 of an inch in inside diameter and 3.0 feet long. The turbulence tube delivered into a larger jacketed pipe which served for cooling. This pipe was about ⅜ inch in diameter and 40 feet long. The sulfur and dispersion medium were forced through the turbulence tube at a rate of 1½ gallons per minute at a temperature of 125° C. Before issuing from the cooling tube the temperature was reduced below the boiling point of water. The cooled product was a white paste which readily dispersed in water to produce particles showing Brownian motion.

The invention is generally applicable to the treatment of any substances which are suited for the production of colloidal suspensions or emulsions. The dispersion medium may be any liquid which will not dissolve the dispersoid to any appreciable extent. If desired, the dispersion medium may contain substances which serve to increase its boiling point, e. g., a dispersion medium such as water may contain inorganic salts or sugar. The invention is particularly advantageous in the treatment of materials, as for example sulfur, which are extremely hard to grind in the solid state. As further and very desirable specific applications of the invention may be mentioned the production of colloidal water suspensions of ethyl-b-naphthylamine, chloro-2-butadiene-1:3, phenyl-b-naphthyl-amine and butyraldehyde-aniline condensation products, emulsions and suspensions of various mercury compounds (e. g., ethyl mercury oleate) or copper compounds in oil, sulfur in oil or glycerine and many other combinations to be used as fungicides, insecticides, preservatives, combined fungicides and insecticides, delustrants and pigments.

The dispersion may be effected in the presence of a protective material. In general, by the expression "protective material" is meant any dispersing agent, wetting agent, or protective colloid. As specific examples of such compositions may be mentioned glyptal resins, gum tragacanth, alkali metal salts of the higher fatty acids, gall acids and their derivatives, agar-agar, glue, methyl cellulose, sulfite cellulose lyes, sodium cellulose phthalate, calcium saccharate, sodium cellulose glycollate, gelatin, derivatives of cholesterine, phosphatides, gelloses, natural and artificial waxes, such as wool waxes, alkylolamine salts, quaternary ammonium compounds, clay, sulfonated tallow, sulfonated oils, alkyl sulfuric esters, and alkali metal caseinates.

The materials to be dispersed and the dispersing medium may be introduced into the turbulence tube in any suitable manner and the proportions of the different components may be varied as desired. In carrying out the process of the invention, the materials to be dispersed and the dispersing medium are preferably brought together in the liquid condition in the desired proportion either by roughly mixing the materials in a single container or by flowing the materials from separate containers into a mixing tube or chamber. The mixed materials are then forced through the turbulence tube by any suitable method, e. g., by means of a pump or gas pressure.

While, in accordance with the invention, two liquids can be emulsified in this manner at ordinary temperatures and pressures, in order to emulsify or produce colloidal suspensions of materials which are solids under normal conditions of temperature and pressure, it is desirable to maintain the temperature of the turbulence tube sufficiently high to preserve such materials in a liquid condition. In the production of dispersions of solids which melt substantially above the boiling point of the liquid dispersion medium, the pressure of operation should preferably be sufficiently high to prevent boiling of any of the components. This may vary considerably, depending largely upon the materials treated. In the application of the process to the production of colloidal sulfur, the melted sulfur to be dispersed should preferably have a temperature less than about 130° C., but, if desired, higher temperatures may be employed. The selection of such higher temperatures is limited by the physical characteristics of the sulfur and the particular conditions under which the dispersion is effected.

It is also possible to reduce the melting point of the material to be dispersed. Thus, the melting point of sulfur may be reduced by dissolving it in a second material, such as naphthalene or a di-chloro-benzene. The resultant dispersion system may be a combined emulsion and suspension depending largely upon the particular materials treated.

The speed of flow of the material to be dispersed and the dispersing medium through the turbulence tube may be any velocity greater than the critical velocity sufficient to reduce the material to the desired particle size. Generally speaking, the desired velocity is determined by empirical methods. It will depend upon such factors as the nature of the material flowing and on the size, surface and shape of the conducting vessel or tube. Flow at a speed about the critical velocity is accompanied by turbulence with the resulting intimate mixing of the fluids. This was observed, for example, by passage of a liquid mixture of kerosene and sodium oleate through a glass tube 0.15 inch in diameter. At a rate of about 15 gallons per hour emulsification began about 4 inches from the entrance of the tube.

The size of the turbulence tube may be varied within relatively wide limits to suit the requirements in regard to degree of dispersion, and capacity of the equipment. The turbulence tube may also be of any desired shape in cross section, such as round, square, oval or of irregular shape and may be either straight, coiled or bent into any other desired position. As a general rule, other things being equal, the smaller the diameter of the turbulence tube the shorter it may be in order to secure a desired result. However, if the diameter of the tube is very small, exceedingly high pressures may be required in order to force the liquid material through it at a speed greater than the critical velocity. On the other hand, the longer the tube the greater are the pressures required. In general, it is preferable to employ turbulence tubes having a length of about 3 inches to 6 feet and a diameter of about .06 inch to .375 inch.

The colloidal dispersion produced is preferably cooled rapidly. In practice, we have found that rapid cooling has the advantage that it tends to prevent precipitation or flocculation of the colloidal particles. Cooling may be effected in any suitable manner, for example, by blowing the colloidal dispersion on a cooled surface or by passing it through a cooling tube, preferably larger than the turbulence tube. This latter method is especially desirable in operating under superatmospheric pressures where the temperature of the turbulence tube is greater than the boiling

point of one of the substances employed. The size and amount of cooling in the second tube may be so regulated that the colloidal dispersion is cooled below the boiling point of all of its components before the material is discharged at atmospheric pressure. This tube not only cools the dispersed material, but serves to reduce the velocity of linear flow so that the issuing product can be more easily collected.

The dispersion system produced may vary widely in character depending largely upon the materials employed. Thus, they may consist of one or more different kinds of solid particles in a liquid suspending medium, both solid particles and liquid particles in a dispersion medium, or one or more different kinds of liquid particles in a dispersion medium. The dispersion medium may contain more than one component, as for example, solutions of glycerine and water (preferably 15–20% water) have been found to be very desirable dispersion media for sulfur.

While the invention is not limited to any theory, it is based upon the principle that stream line flow through a tube cannot be maintained above a certain critical velocity. By increasing the velocity sufficiently the shear due to turbulence becomes great enough to reduce the suspended liquid to colloidal dimensions. This type of mixing is exceedingly efficient because it takes place throughout the entire moving mass of the liquid while with ordinary stirring the actual mixing takes place principally in the liquid closely surrounding the stirrer.

A particular advantage of the invention is the elimination of foaming since no air or gases are in contact with the liquids during the period of mixing. Another advantage is that the invention permits the easy and rapid dispersion of liquids at any temperature. Furthermore, the dispersion is uniform and complete because all of the material which passes through the turbulence tube has been subjected to the same action whereas with the usual methods of stirring considerable material may remain at different positions in the vessel in an undispersed condition. The invention is further advantageous in that the process may be operated continuously. An additional advantage of the invention is its applicability to the production of both emulsions and suspensions with the same equipment since methods for producing emulsions do not necessarily give desirable results in the production of suspensions and vice versa.

The invention is distinguished from the so-called homogenizing process in which liquid materials such as milk are passed through holes or orifices in that the particle size obtained by such processes is largely determined by the size of the orifice whereas in the present process tubes of practically any size may be used as long as the velocity of the liquids passing through the tube is greater than the critical velocity. The causes of the reduction in particle size in processes of the homogenizing type and in the present process appear to be entirely different. In homogenizing the reduction in particle size apparently results from the liquid particles contacting the edge of the orifice. The time of contact must of necessity be relatively short. According to our process the dispersion results not only from the fact that the liquid particles are continuously brought into contact with the inner surface of the tube but also by reason of the fact that they are continuously brought into contact with each other by the flow of liquid past liquid. The duration of the mixing period in a turbulence tube as herein described is obviously much longer, relatively, and the number of contacts of a given particle much greater in our process than by the old method of homogenizing. Moreover, homogenizing processes usually require the passage of liquids through orifices of such small size that pressures in the neighborhood of 2500 to 3000 pounds per square inch are necessary, whereas according to our process much lower pressures may be employed.

The term "alkali metal" as herein used is intended to cover not only the alkali metals, for example, sodium, potassium, and the like (see Periodic Table, Handbook of Chemistry and Physics, 12th Ed. p. 474–5) but also the ammonium (NH_4) radical.

Throughout the specification and claims it will be understood that by an emulsion is meant a system of liquid particles in a suspending medium. By a suspension is meant a system of solid particles in a suspension medium. Emulsions and suspensions are known collectively as dispersions. The foregoing definitions are in accordance with those in the Chemical Age Dictionary (1924).

As many apparently and widely different embodiments of this invention may be made without departing from the spirit thereof, it is to be understood that we do not limit ourselves to the foregoing examples or description except as indicated in the following claims.

We claim:

1. In a process of producing finely divided substances, the step which comprises simultaneously passing materials to be dispersed and a dispersing medium, in liquid condition, through a tube at a velocity greater than the critical velocity and in unobstructed flow.

2. In a process of producing finely divided solid particles, the step which comprises simultaneously passing a material to be dispersed and a dispersing medium, in liquid condition, through a tube at a velocity greater than the critical velocity in unobstructed flow, said material to be dispersed being a solid under normal conditions and being insoluble in the dispersing medium.

3. In a process of producing finely divided solid particles, the step which comprises simultaneously passing a material to be dispersed and a dispersing medium, in liquid condition, through a tube at a velocity greater than the critical velocity in unobstructed flow, said material to be dispersed being a solid under normal conditions and being insoluble in the dispersing medium, and rapidly cooling the resultant dispersion.

4. The process of producing dispersions which comprises simultaneously passing an aqueous dispersion medium and a water-insoluble liquid material in unobstructed flow through a tube at a velocity greater than the critical velocity.

5. The process of producing colloidal sulfur which comprises simultaneously passing a dispersing medium, sulfur maintained in molten state and a dispersing agent through a tube at a velocity greater than the critical velocity and in unobstructed flow.

6. In a process of producing finely divided substances, the step which comprises simultaneously passing sulfur and a dispersing medium, in liquid condition, through a tube at a velocity greater than the critical velocity and in unobstructed flow.

7. The process of producing colloidal sulfur which comprises simultaneously passing melted sulfur, a stable protective material and a dispersing medium in unobstructed flow through a

tube maintained at a temperature from about 125° C. to about 130° C., at a rate of flow greater than the critical velocity.

8. The process of producing colloidal sulfur which comprises simultaneously passing an aqueous dispersion medium, melted sulfur and a stable protective material in unobstructed flow through a tube maintained at a temperature from about 125° C. to about 130° C., at a rate of flow greater than the critical velocity.

9. The process of producing colloidal sulfur which comprises passing an aqueous dispersion medium, melted sulfur and an alkali metal caseinate in unobstructed flow through a tube maintained at a temperature from about 125° C. to about 130° C., at a rate of flow greater than the critical velocity.

10. The process of producing colloidal sulfur which comprises simultaneously passing an aqueous dispersion medium, melted sulfur and ammonium caseinate in unobstructed flow through a tube 0.2 of an inch in inside diameter and about 3.0 feet long, at a temperature of about 125° C., and at a rate of flow greater than the critical velocity.

11. The process of producing colloidal sulfur which comprises passing a mixture containing melted sulfur and a 15% aqueous solution of ammonium caseinate, in proportions corresponding to about 1½ gallons of melted sulfur and 3 gallons of ammonium caseinate solution, through a tube 0.2 of an inch in inside diameter and about 3.0 feet long, at a rate of about 1½ gallons of the mixture per minute, and passing the resultant product through a tube about ⅜ inch in inside diameter and about 40 feet long.

12. The process of producing colloidal sulfur which comprises passing melted sulfur, a dispersing agent and a dispersing medium consisting of glycerine containing about 15–20% water through a tube maintained at a temperature above the melting point of the sulfur but below about 130° C., at a rate of flow greater than the critical velocity, and rapidly cooling the resultant dispersion.

13. The process of producing emulsions which comprises simultaneously passing a material to be dispersed and a dispersion medium, in liquid condition, through a tube in unobstructed flow at a velocity greater than the critical velocity, said material to be dispersed being a liquid under normal conditions and being insoluble in the dispersion medium.

14. In a process of producing dispersions, the steps which comprise simultaneously passing materials to be dispersed and a dispersion medium, in liquid condition, through a tube of small diameter at a velocity above the critical velocity, and then through a tube of larger diameter at a decreasing velocity.

15. In a process of producing dispersions, the step which comprises simultaneously passing a liquid dispersion medium and a material to be dispersed which is a solid under normal conditions and insoluble in the dispersion medium through a tube heated sufficiently to maintain said solid in the molten state at a velocity above the critical velocity, and then through a tube cooled sufficiently to rapidly reduce the melted particles of the solid substance to the solid state.

16. In a process of producing dispersions, the step which comprises simultaneously passing a liquid dispersion medium and a material to be dispersed which is a solid under normal conditions and insoluble in the dispersion medium in unobstructed flow through a tube at a velocity above the critical velocity, and maintaining the temperature of the tube sufficiently high to keep said solid in the molten state.

17. The process of producing colloidal sulfur which comprises simultaneously passing melted sulfur and a dispersion medium maintained at a temperature from about 125° C. to about 130° C. in unobstructed flow through a tube at a velocity greater than the critical velocity.

18. The process of producing colloidal sulfur which comprises simultaneously passing melted sulfur and a water-glycerin mixture through a tube at a velocity above the critical velocity and at a temperature from about 125° C. to about 130° C.

19. The process of producing colloidal sulfur which comprises simultaneously passing a water-glycerin mixture, an alkali metal caseinate and molten sulfur maintained at a temperature from about 125° C. to about 130° C. through a tube at a rate of flow greater than the critical velocity.

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