March 20, 1945. F. W. WANDERER 2,371,722

PROCESS FOR WASHING AND DRYING COAGULA OF RUBBER AND SIMILAR MATERIALS

Filed Oct. 24, 1940

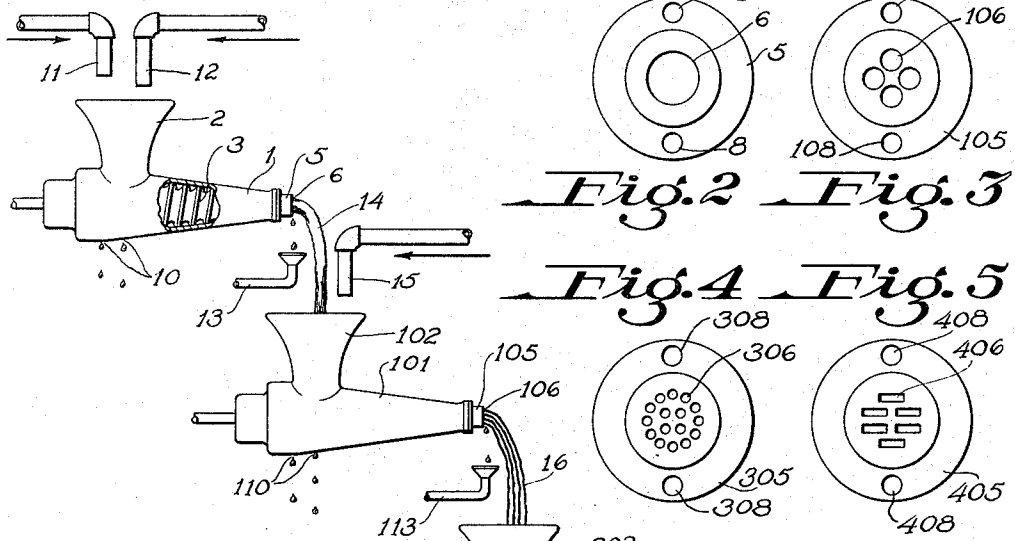
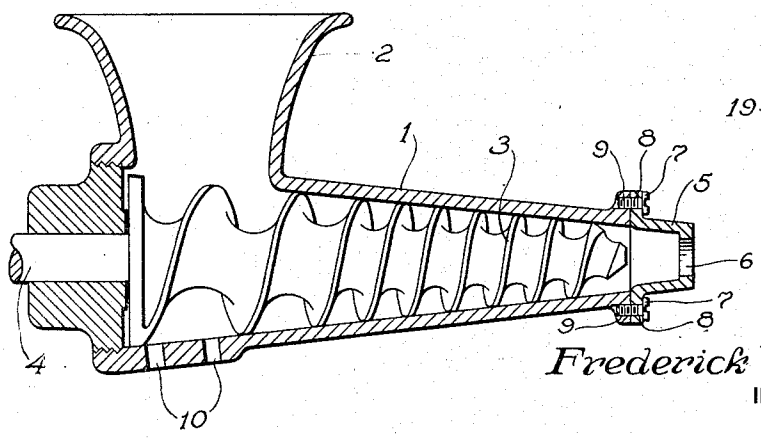


Fig. 6

Fig. 2 Fig. 3

Fig. 4 Fig. 5

Fig. 1



Frederick W. Wanderer
INVENTOR

BY Hugh P. K. Clark
ATTORNEY

Patented Mar. 20, 1945 2,371,722

UNITED STATES PATENT OFFICE

2,371,722

PROCESS FOR WASHING AND DRYING COAGULA OF RUBBER AND SIMILAR MATERIALS

Frederick W. Wanderer, Penns Grove, N. J., assignor to E. I. du Pont de Nemours & Company, Wilmington, Del., a corporation of Delaware

Application October 24, 1940, Serial No. 362,557

3 Claims. (Cl. 18—48)

This invention relates to the treatment of rubber-like materials, and more particularly, to a process for coagulating, washing, and drying aqueous dispersions of rubber-like materials comprising chloroprene.

Natural rubber is usually converted into the solid form in which it appears in commerce by coagulating the latex in the form of sheets or slabs, passing these between rollers, sometimes in the presence of water, to assist in the removal of soluble material, and finally drying the sheets or slabs thus obtained. Rubber-like materials synthetically prepared in aqueous dispersion cannot, in general, be obtained in a satisfactory solid form by the methods used for natural rubber, since usually the properties of the rubber-like material are adversely affected if any substantial quantity of the dispersing agent is allowed to remain and since the methods used by working up natural rubber remove only a part of the water-soluble material. In the processes now used for obtaining the synthetic rubber-like materials from their aqueous dispersions, they must, accordingly, be thoroughly washed with water, alcohol, or other solvent either by kneading them in apparatus in which fresh surfaces are continually exposed to the action of the solvent or by converting them into a form such as thin sheets or small crumbs which present a large surface relative to the volume. In either case, the material must finally be dried and converted into a form suitable for storage and shipment. It is obvious that such processes for converting the coagulum into the final dry form require elaborate equipment and considerable attention from the operating force. Both these factors add considerably to the cost of production of the synthetic rubber and form one of the main reasons why none of these synthetic products is yet as cheap as natural rubber. Furthermore, it would be noted that, although the present processes for working up coagulated natural rubber are considered satisfactory, they, too, are in need of further improvement.

It is an object of this invention to provide a simple and efficient method of removing the water phase from coagula of rubber-like materials, especially neoprene (polymeric chloro-2-butadiene-1,3). Another object is to coagulate such dispersions, wash the coagulum, dry the coagulum, and convert it into a form suitable for further processing, storage, or shipment. Other objects will appear hereinafter.

It has been discovered that the first of these objects can be accomplished by applying pressure to a coagulum by means of a rotating worm in a suitable housing and forcing it through small openings. The other objects can be accomplished by passing a dispersion and a coagulating agent into a housing containing a worm and forcing the coagulum through an opening or openings under pressure, and subsequently passing the coagulum through one or more similar steps. In its preferred form, the invention is accomplished by passing a dispersion of rubber-like material or natural rubber latex into a hopper, simultaneously passing a coagulating agent into the hopper, forcing the coagulum through a truncated cone from the larger end to the smaller end by means of a rotating worm and out of the cone under pressure through a perforated plate, passing the coagulum through a similar operation with a washing fluid, and then passing the coagulum through one or more similar operations, without additional fluid, to dry the said coagulum.

In the drawing:

Figure 1 is a vertical section through a suitable coagulating, washing, or drying apparatus;

Figures 2 to 5 are suitable perforated plates for such apparatus; and

Figure 6 is an elevation of an apparatus for the preferred continuous process.

Referring now more particularly to the drawing, Figure 1 is a vertical section through a suitable coagulating, washing, or drying apparatus. In the figure, housing 1 has a hopper 2 and a revolving worm 3 which is driven by a shaft 4 by any suitable means (not shown). The coagulum enters hopper 2 and is squeezed and compressed by worm 3 and is extruded, under pressure, through extruding head 5 having therein orifice 6. Head 5 is attached to housing 1 by screws 7, passing through suitable openings 8 and into threaded openings 9 in the housing 1. The water which is thereby removed from the coagulum leaves housing 1 through openings 10, a small amount also escaping through orifice 6.

Figure 2 is a planned view of extruding head 5 having extruding opening 6 and holes 8 for screws.

Figure 3 is another type of extruding head 105 having extruding openings 106 and screw holes 108.

Figure 4 is a third type of extruding head 305 having extruding openings 306 and screw holes 308.

Figure 5 is another type of extruding head 405 having extruding openings 406 and screw holes 408.

In order that the invention may be more fully understood, it will be described with reference to a dispersion of neoprene, but it is to be understood that the invention is not limited thereto as will become more apparent hereinafter.

Example I

Chloroprene (100 parts) containing 0.6 part of sulfur and 4 parts of rosin, is emulsified by mechanical agitation in 233 parts of water, containing 0.5 part of ammonium persulfate, 0.8 part of sodium hydroxide and 0.5 part of the sodium salts of the dinaphthyl methane sulfonic acid prepared according to U. S. Patent 1,336,759. The rosin is neutralized by the sodium hydroxide forming sodium abietate which, along with the sulfonic acids mentioned above, acts as the emulsifying and dispersing agent. The dispersion is kept at 40° C. in a suitable vessel, which can be externally cooled or heated, until the polymerization is substantially complete, as shown by the density of the dispersion which is approximately a linear function of the proportion of chloroprene which is polymerized. The dispersion is then treated with 2.5 parts of tetra ethyl thiuram disulfide dispersed in 10 parts of the above emulsifying solution. The entire process up to this point may conveniently be carried out in a continuous manner as described in the application of Calcott and Starkweather, Serial No. 308,386, filed December 9, 1939.

The dispersion so prepared, which contains about 30 per cent of neoprene, is processed in an apparatus illustrated in Figure 6. This figure shows an apparatus consisting of 4 successive units. In the figure, similar parts in the various units are indicated by numerals which differ from each other by 100, and any part not specifically described hereinafter by number will be understood to be the same as the corresponding part of the first unit of the apparatus. Referring more particularly to Figure 6, the dispersion of neoprene enters housing 1 through hopper 2 from conduit 11. Simultaneously, ⅓ of its weight of a 3 per cent aqueous solution of aluminum sulfate is passed into hopper 2 from conduit 12. Coagulation occurs in the hopper. The coagulum descends upon revolving worm 3 which forces it through housing 1 and out of extruding head 5. The size of the opening 6 in extrusion head 5 is of such size that the revolving worm builds up pressure within the housing and the coagulum is thus subjected to a combined pressure and tearing action which removes much of the aqueous phase and water-soluble materials. This aqueous phase escapes through perforations 10 and also, to a small extent, through opening 6 through extrusion head 5, the water escaping through opening 6 being carried off by drain 13. The extruded coagulum 14 containing about 50 per cent water passes into second hopper 102. Simultaneously, a stream of fresh water enters through this hopper through conduit 15. The coagulum is thus washed as it passes through hopper 102 and into housing 101. The coagulum is propelled through housing 101 by a revolving worm which subjects it to pressure and tearing action and the coagulum comes out of extrusion head 105 purified and containing less than 20 per cent water. Extrusion head 105 has therein several openings 106 and the coagulum in the form of several strings 16 passes into hopper 202 of housing 201 where it is subjected to the action of a revolving worm as before. The coagulum is extruded from this unit through extrusion head 205 having an increased number of openings 206. The coagulum, in the form of more numerous and smaller strings 17, then passes into hopper 302 of housing 301 where it is again subjected to the action of a revolving worm and is extruded through head 305 through still smaller openings 306. The strings of coagulum 18 contain less than 5 per cent water and is very porous in structure. The remaining water quickly evaporates on air-drying and the coagulum is collected in container 19.

Although the process has been described in terms of the treatment of a dispersion of polymerized chloroprene in apparatus of specific form, the invention is, in fact, very much broader. Thus, it may be applied to the treatment of rubber or any rubber-like material in the form of a wet coagulum. It is clear from the above disclosure that the invention has to do with unvulcanized rubber and rubber-like materials. These may be generically called vulcanizable plastic materials. In addition to natural rubber and rubber-like polymers of chloroprene, it may be applied to other types of so-called synthetic rubbers such as the polymers of butadiene and homologous dienes prepared with or without the use of other polymerizable substances, such as styrene, methyl methacrylate, and acrylic nitrile. It may also be applied to the products obtained from the reaction of organic dichlorides, such as ethylene dichloride, and beta, beta-dichloro-diethyl ether with metallic polysulfides. The type of dispersing agent used in the preparation of the dispersions of these rubber-like materials is not critical to the present invention. The coagulum used in the invention may be prepared by any of the appropriate methods known in the art such as the addition of salts, such, for example, as aluminum sulfate, magnesium sulfate, etc., acids, alcohols, and ketones, or by freezing as described in U. S. Patent No. 2,187,146. The coagulum may be introduced into the apparatus in the form of separate pieces or in the form of a continuous sheet or strip such as, for example, that formed by the operation of the preferred form of the above patent. It is often advantageous to carry out the coagulation within the hopper of the extrusion apparatus as in the above specific example.

The apparatus described above may also be varied, for example, as to the size, shape, and pitch of the worm, and the volume and shape of the chamber between it and the plate through which a material is extruded. It is preferred, however, that the chamber in which the worm rotates and the worm itself should be of conical rather than cylindrical form with the outlet at the narrow end. The number of holes through which material is extruded and also their cross-section may be varied. Thus, in the first stage, when the material has only slight plasticity, a large hole is used, while several smaller holes are used as the material becomes more plastic in the later stage. It is always preferred, however, that the size and number of the openings are small enough to cause the exertion of considerable pressure by the worm upon the material being processed. The form of the hole need not be circular, and, in fact, it is often desirable in the case of more plastic materials to extrude in the form of flat ribbons or other shapes offering a large surface for subsequent drying.

Instead of a short air drying followed by collecting the coagulum, as shown in Figure 6, the coagulum, as it comes from the last unit of the

drying apparatus in the form of porous strings having a small amount of moisture, may be passed upon a moving belt through a drying tunnel counter-current to a stream of warm air, and then collected. When so treated, less than 0.37 per cent of moisture remains in the coagulum.

The conditions under which the apparatus is operated may also be varied. These variables include the rate of rotation of the worm, the rate at which the material is supplied to the machine and the temperature. Because of the work done upon the material, the temperature of the extruded material is higher than that of the material introduced. This is generally advantageous where it is desired to remove part of the water content by evaporation, but, if the material be processed is sensitive to heat, this heating may be reduced by jacketing the apparatus with a cooling liquid or by operating at a slower speed.

The principle of the present invention is roughly similar to that by which natural latex coagulum is usually processed in that the coagulum is dehydrated by pressure. The process, however, differs by compressing the material by a worm rather than by rolls and by keeping it compressed for a much longer time with the result that the removal of the aqueous phase (and, hence, the removal of water-soluble materials) is much more efficient and complete. Moreover, the action of the worm breaks up the cells of the coagulum more completely, thus aiding in releasing imprisoned water. Another unexpected advantage is that the extruded product is porous in nature which gives it improved air drying for the small amount of residual moisture and also facilitates compounding operations. Furthermore, the present invention has the further advantages over the prior art that the apparatus required is much more compact and is much better adapted for continuous and automatic processing of the material.

The process is especially desirable with neoprene because the action of the revolving worms unexpectedly plasticizes this material to a considerable extent and reduces the amount of milling necessary in subsequent compounding operations.

It is apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, and, therefore, it is not intended to be limited except as indicated in the appended claims.

I claim:

1. The process which comprises passing an aqueous dispersion of a vulcanizable material comprised essentially of chloroprene polymer into a path of decreasing cross-section, simultaneously subjecting the dispersion to coagulation conditions, forcing the mass along said path of decreasing cross-section, subjecting the mass to tearing action, and withdrawing the aqueous phase therefrom.

2. The process of washing and drying a coagulum, obtained by coagulating an aqueous dispersion of a vulcanizable material comprised essentially of chloroprene polymer which comprises forcing the coagulum and fresh wash water to traverse a path of decreasing cross-section whereby the coagulum becomes more and more compact while simultaneously subjecting the coagulum to tearing action whereby the aqueous phase is released from the coagulum, simultaneously drawing off the wash water and exuded aqueous phase, and then forcing the coagulum simultaneously through a plurality of orifices, said orifices having a total cross-section area less than the cross-section area of the path just preceding them.

3. The process which comprises passing an aqueous dispersion of a vulcanizable material comprised essentially of chloroprene polymer into a path of decreasing cross-section, simultaneously subjecting the dispersion to coagulation conditions, forcing the mass along said path of decreasing cross-section, subjecting the mass to tearing action, and withdrawing the aqueous phase therefrom, then forcing the coagulum and fresh wash water to traverse a second path of decreasing cross-section whereby the coagulum becomes more and more compact while simultaneously subjecting the coagulum to tearing action whereby the aqueous phase is released from the coagulum, simultaneously drawing off the wash water and exuded aqueous phase, and then forcing the coagulum simultaneously through a plurality of orifices, said orifices having a total cross-section area less than the cross-section area of the path just preceding them.

FREDERICK W. WANDERER.