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APPARATUS FOR SHOCK GELLING NITROCELLULOSE

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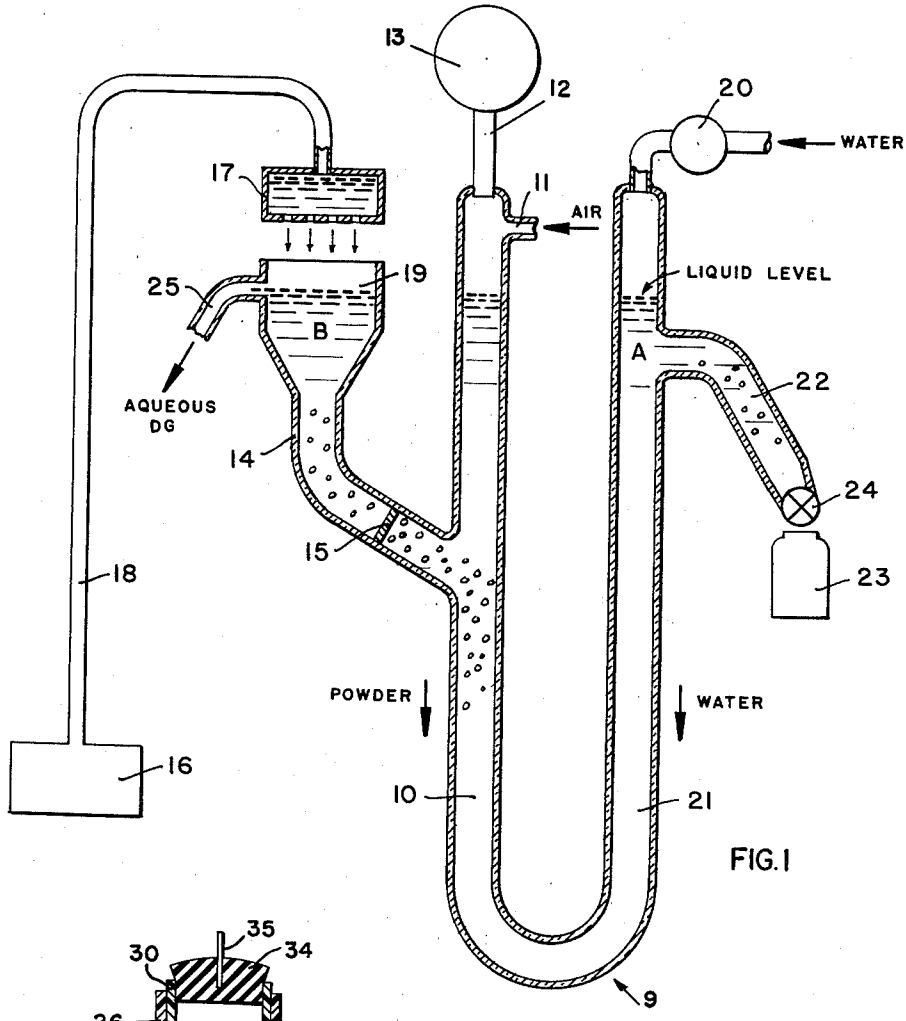

FIG. 1

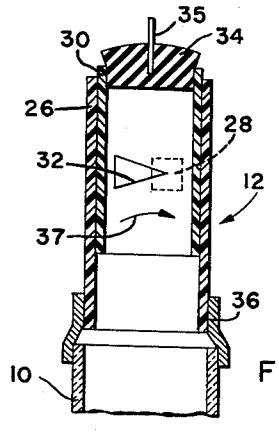

FIG. 2

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APPARATUS FOR SHOCK GELLING NITROCELLULOSE

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The present invention relates to an apparatus for leaching the alkoxy alcohol solvent from shock-gelled nitrocellulose casting powder.

In order for the shock-gel process for production of casting powder on a continuous basis to be economically feasible, the diglycol used as a solvent in the operation must be recovered. The extracting apparatuses known and available are both cumbersome and expensive and fail to produce completely leached shock-gelled casting powder. As the potentiality of shock-gelled casting powder grew, the need for efficient, continuously operable leaching equipment became apparent.

The problems confronting designers of a suitable apparatus which would function satisfactorily under all the difficult conditions encountered in effectively leaching shock-gelled nitrocellulose have been overcome by the present invention.

The primary purpose of this invention is to provide a continuous counter current apparatus for complete extraction of diglycol from the nitrocellulose phase and for the concentration of diglycol in the initial leaching phase. The proper removal of diglycol is necessary in order to produce a uniform product.

The general purpose of this invention is to design an apparatus which in operation utilizes a U-shaped column in which freshly gelled casting powder spheroids move in one direction by pneumatic pulsation while fresh water flows in the other direction. Solvent-free spheroids or balls of water-logged casting powder are removed at one end, and concentrated aqueous diglycol solution is withdrawn at the other end.

An object of the present invention is to provide an automatic counter current extractor with which completely leached shock-gelled casting powder is produced for use in rocket propellant grains.

Another object of the present invention is to provide an apparatus for recovery of diglycol from the nitrocellulose phase which may be re-used.

The exact nature of this invention as well as other objects and advantages will be readily appreciated as the same become better understood by reference to the following detailed description as illustrated in the accompanying sheet of drawing in which:

FIGURE 1 is a vertical view, partly diagrammatic in character, of an apparatus embodying the present invention; and

FIGURE 2 is a larger scale longitudinal section of the pressure valve shown diagrammatically in FIG. 1.

The leaching apparatus shown in FIG. 1 comprises a pump means, diagrammatically illustrated and generally indicated by numeral 16, a dropping pan 17 provided with holes in its bottom, said pan and pump being connected by a flexible conduit 18 made up of ½ inch I.D. Tygon tubing, the pan being positioned above the pump which is

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adapted to push liquid into the pan by peristaltic action without actual contact between the liquid and the pump; a U-tube 9 constructed of four inch I.D. Pyrex pipe with aluminum fittings and having a left and a right column designated by numerals 10 and 21 respectively, column 10 being about 86 inches in height and column 21 being about 88 inches in height, a leaching container 19 connected to the midportion of left column 10 of U-tube 9 by a tubular elbow 14 with a constricted passage 15, and an exit orifice 25 near its top through which concentrated diglycol solution is withdrawn; an inlet 11 through which air at constant pressure is introduced into column 10; a valve 12 mounted on column 10 for controlling the air pressure on the contents of the column and consisting of two pieces of concentric cellulose acetate tubing, the outer piece being stationary and the inner piece being movable relative thereto and driven by a constant speed motor 13, the pieces of tubing having openings therein such that with movement of the inner piece the pressure is first increased rapidly and then gradually released, thereby pulsating the contents of column 10 for pushing the contents forward rapidly and then allowing them to settle slowly; a flowmeter attached to the top of column 21 for introducing and regulating a countercurrent of water into column 21; an exit tube 22 about midway on column 21 for the exit of the spheroids; a gate valve 24 at the end of tube 22 and a portable container 23. FIG. 2 shows a longitudinal section of valve 12 mounted on column 10 by a metal pipe coupling 36 for controlling the air pressure on the contents of said column. Valve 12 consists of two pieces of concentric cellulose acetate tubing, the outer tubing 26 having an opening 28 approximately ¾ x ¾ inch square in size and the inner tubing 30, being movable relative thereto and having a triangular slit opening 32, approximately ¾ x 2.5 x 2.5 inches in size and being attached to a rubber stopper 34 which supports a steel rod 35 connected to speed motor 13 for rotating inner tubing 30 in the direction of the arrow 37. Air pressure is constantly supplied through an opening 11 below valve 12.

The preferred pump means 16 used in the present invention is a peristaltic type known as a "Sigmamotor" pump, which is designed to handle liquids, slurries or gases. It operates by means of a series of cam operated fingers which pass against flexible tubing 18 in sequence and impart a positive unidirectional movement to fluid (nitrocellulose solution) in the tube. The material being pumped does not come in contact with the pump parts so no contamination results and no cleaning of the pump is necessary. The pump is designed to use tygon, gum rubber, or neoprene tubes of varying internal diameters. Various pump combinations are available including motors and belt drives and variable speed units. The pumping combination utilized in the present invention is composed of a Sigmamotor pump, a ¾ H.P. hydraulic transmission and a ½ H.P. electric motor mounted on a base. Other suitable means may be used to introduce the nitrocellulose solution to the dropping pan; however, this preferred means eliminates the hazard that is associated with pumping nitrocellulose solution since the fluid being pumped does not come into direct contact with the pump parts.

In operation, nitrocellulose casting powder-diglycol solution is forced by Sigmamotor pump means 16 through tubing 18 to a dropping pan 17. Dropping pan 17 is pro-

vided with numerous holes through which the solution passes thereby forming drops. These drops of nitrocellulose-diglycol are shock-gelled in the form of casting powder spheroids in the initial leaching bath **19**. The shock-gelled particles fall through constriction **15** to the bottom of the U-tube **9**. The pulsating movement imparted to the water and the weight of the additional powder causes the spheroids to be gradually forced up column **21**. The level of the spheroids in column **10** of U-tube **9** remain essentially constant except for an oscillating motion. The contents of U-tube **9** are oscillated by fluctuation of the air pressure controlled by the operation of valve **12** in which the inner tubing **30** rotates in a direction such that the point of slit **32** first crosses the first vertical edge of the square opening **28** in the outer tube **26**. Continued rotation allows a gradually increasing opening in the inner tubing **30** to be opposite that of the square opening **28** in the outer tubing **26** until the rear edge of the slit **32** coincides with the first vertical edge of the square opening. Then the opening through the tubes decreases until the rear edge of slit **32** passes the second vertical edge of the square opening **28**. At this point pressure builds up rapidly and is abruptly applied on the water in column **10** which causes the water and pellets to be forced upward in column **21**. To keep the force diverted in a downward direction in column **10**, tubular extension **14** of the leaching bath **19** is constricted at **15** by means of a ⅛ inch rubber sheeting containing an opening 1⅜16 inches in diameter. When the inner tubing **30** of valve **12** has rotated so that the slit again allows pressure to be decreased slowly by allowing the air to be bled off through the opening, the water in column **21** recedes slowly and the spheroids or pellets tend to stay at the height to which they were forced. The combination of newly formed pellets in the bottom of column **10** and each additional pulsation tends to force the pellets higher in column **21** until they overflow into the side arm tube **22**. As the spheroids are forced up column **21**, a countercurrent flow of water is introduced through a flowmeter **20**. Air-free water was found to be the most satisfactory, since tap water caused bubbles to collect on the spheroids which produced a buoyant effect. The diglycol is continuously removed from the freshly gelled spheroids until complete leaching has occurred with the aqueous phase becoming gradually enriched with diglycol. The concentrated aqueous diglycol solution is exhausted through exit **25**. When equilibrium is established a gradient of concentration of diglycol ranging from pure water at point A of exit **22** to 25–45% aqueous diglycol at point B of exit **25** exists in the aqueous phase. The solution inside the shock-gelled powder ranged from being diglycol-free at the point of exit **22** for said spheroids to being concentrated in diglycol at the bottom of column **10**. When the leached spheroids are forced toward the top of column **21**, the spheroids fall into tube **22** from which they are removed by means of a two inch gate valve **24**. This valve is opened to permit the spheroids to fall into container **23**. The removal of the leached powder is the only phase of this process which requires manual operation.

Data from three runs are summarized below. The time in minutes was measured from initial dropping of nitrocellulose casting powder-diglycol solution. The concentration of diglycol was determined by refractive index measurements. The accuracy of this method is limited to 0.2 to 0.4% diglycol which is in the range found in the leached balls in Tables II and III. The weights of the wet powder and consequently the dry powder were approximated.

Conditions for the three runs were essentially the same except for the rate of addition of water to the column. A rate of 200 ml./min. was not fast enough in the first run as the refractive index measurements indicated a fairly large amount of diglycol remained in the shock-gelled nitrocellulose. A rate of 400 ml./min. was too fast in the second run as the effluent was of a low concentration.

The rate of 270 ml./min. in the final run gave spheroids that were essentially diglycol free and a 29% concentration of diglycol in the effluent.

Table I

Time (min.)	Wt. Wet Powder (g.)	Percent DG in Wet Powder	Percent DG in Effluent	
105	1,700	0.1	30.5	Water rate, 200 ml./min. to 198 min. and 220 ml./min. to 318 min.
120	1,700		36.0	
135	1,700		39.5	
150	1,700	0.9	40.7	
162	1,700		42.0	
177	1,700		42.6	
198	1,700	1.0	41.7	
214	1,700		41.8	
234	1,700	0.7	41.3	
250	1,700		41.0	
269	1,700	0.5	40.2	
295	1,700		38.3	
318	1,700	0.5	37.0	
Total 213	20,400	0.82 lb./hr. (dry weight)		

Table II

Time (min.)	Wt. Wet Powder (g.)	Percent DG in Wet Powder	Percent DG in Effluent	
80	300	0.1		Water, 410 ml./min. to 150 min. and 300 ml./min. to 280 min.
90	900		16.0	
95	300	0.2	16.2	
125	2,800		16.2	
133	2,800		16.2	
137	510	0.2		
150	1,770			
175	1,650	0.2	18.9	
203	3,390			
224	3,390	0.2	25.0	
236	3,520			
251	3,520	0.4	23.5	
270	3,010			
280	3,010	0.3	27.2	
Total 200	17,900	0.78 lb./hr. (dry weight)		

Table III

Time (min.)	Wt. Wet Powder (g.)	Percent DG in Wet Powder	Percent DG in Effluent	
110	500			Water rate 270 ml./min. Casting Powder DG rate 110 g./min.
148	2,130	0.1	25.1	
172	2,130	0.4	27.1	
196	2,130	0.2	28.7	
218	2,130	0.4	28.3	
240	2,130	0.3	28.6	
262	2,130	0.2	29.0	
290	2,130	0.4	28.6	
310	2,130	0.3	28.7	
Total 200	17,000	0.73 lb./hr. (dry weight)		

Various modifications are contemplated and may obviously be resorted to by those skilled in the art without departing from the spirit and scope of the invention, as hereinafter defined by the appended claims.

What is claimed is:

1. Apparatus for leaching granular nitrocellulose, said apparatus comprising a pump having extending therefrom a conduit, a dropping pan provided with holes in its bottom, said pan and pump being connected by said conduit and the pan positioned on a level above the pump, an initial leaching bath container provided with an exit orifice near its top and a tubular extension in communication with one column of a U-tube at a point lower than the container and about midway of said column of the U-tube, said dropping pan positioned adjacent said leaching bath container, a valve positioned at the top of said one column of the U-tube adapted to control the air pressure in said column, a flowmeter positioned at the top of the other column of said U-tube for providing constant countercurrent flow of water, an

exit tube positioned about midway on the said other column of said U-tube and a gate valve means at the end of said exit tube.

2. In an apparatus for shock-gelling and leaching granular nitrocellulose, the combination comprising a pump, a dropping pan provided with holes in its bottom; said pan and pump being connected by a flexible tubing, said pan being located above said pump, and said pump being adapted to pump liquid into said pan by peristaltic action without actual contact between the liquid and the pump; a U-tube having a right and a left column; a leaching bath container connected to said left column by a conduit having a constricted passage, said container having an exit orifice near its top; said U-tube being arranged with the leaching bath container directly underneath said dropping pan, whereby the solution to be leached flows through the holes in said pan thereby forming drops which fall into said leaching bath container to be shock-gelled into spheroids, said spheroids falling through said conduit and into said left column, said constricted passage acting as a check means to prevent reverse movement of said spheroids up the conduit; means attached to the top of said right column for introducing and regulating a countercurrent of water into said right column, said right column being provided with an exit tube below its top for the exit of the spheroids, and means connected to said left column for imparting a pulsating movement to the contents of the U-tube, said valve means comprising inner and outer tubings concentrically arranged, the outer tubing being attached to the top of the left column and having a square opening, the inner tubing having a triangular opening and moving relative to the outer tubing; whereby said spheroids are forced through the U-tube and into the exit tube and excess water is forced out of the exit orifice of the leaching bath container.

3. An apparatus for shock-gelling nitrocellulose comprising, a U-tube having a left and a right column, a leaching container connected to the midportion of said left column by a tubular elbow having a constricted passage and an exit orifices near its top; an air inlet positioned near the top of said left column through which air at constant pressure is introduced; a valve means mounted at the top of said left column through which air at constant pressure is introduced, said valve means comprising inner and outer tubings concentrically arranged, the outer tubing being attached to the top of the left column and having a square opening, the inner tubing having a triangular opening and rotatable relative to the outer tubing and driven by a constant speed means for the purpose of pulsating the contents of said left column; a flowmeter attached to the top of said right column for introducing and regulating a counter-current of water into said right column; an air exit elbow about midway on said right column having a gate valve at its end for exit of nitrocellulose; a dropping pan provided with holes in its bottom positioned above said leaching container and having a conduit extending therefrom connected to a means adapted for pushing nitrocellulose solution through said conduit into said pan.

References Cited in the file of this patent

UNITED STATES PATENTS

2,189,387	Wissler	Feb. 6, 1940
2,223,747	Suss	Dec. 3, 1940
2,332,157	Mapson	Oct. 19, 1943
2,381,965	Berry	Aug. 14, 1945
2,448,439	Kimberlin et al.	Aug. 31, 1948
2,744,066	Spiess et al.	May 1, 1956
2,923,033	Baldwin	Feb. 2, 1960

FOREIGN PATENTS

341.581	Great Britain	Jan. 22, 1931