Oct. 18, 1955 R. H. GREENWELL 2,720,981

CENTRIFUGE

Filed Nov. 27, 1951 2 Sheets-Sheet 1

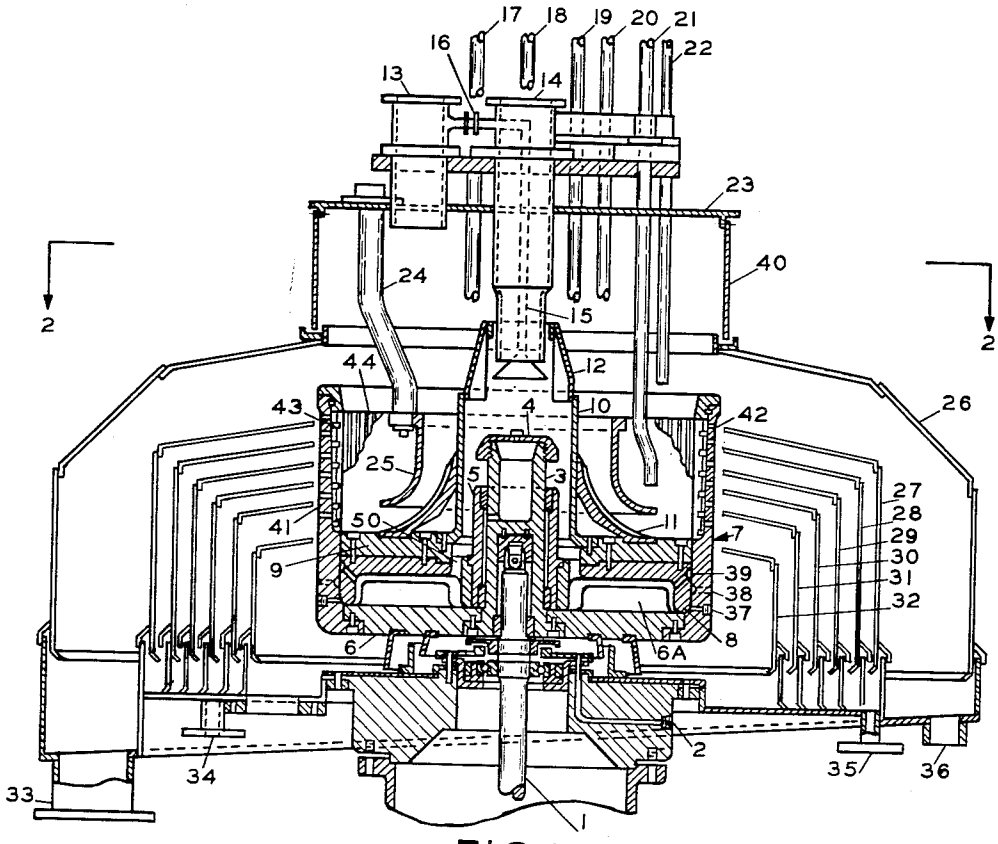


FIG. 1

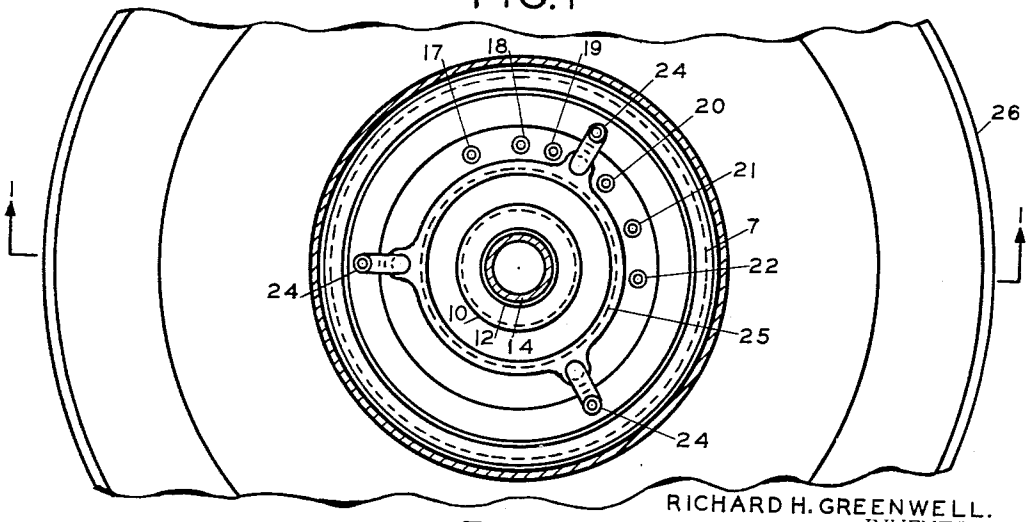


FIG. 2

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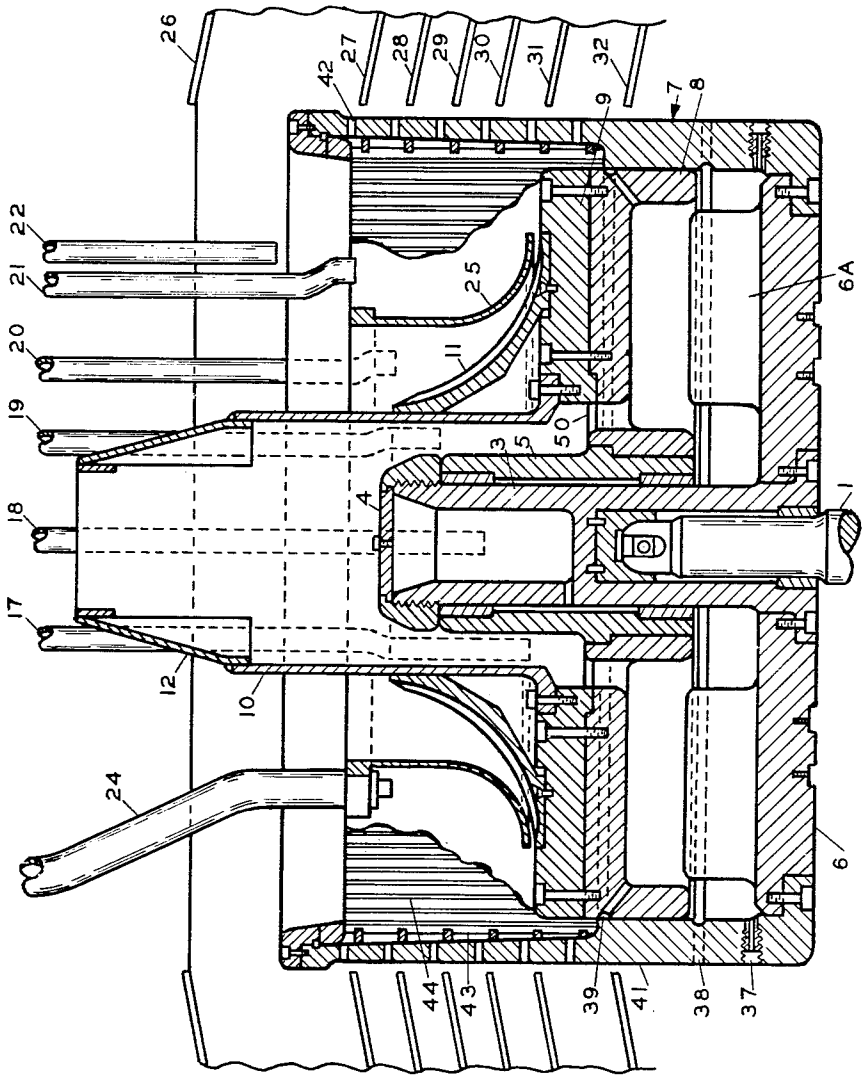


FIG. 3

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2,720,981

CENTRIFUGE

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Application November 27, 1951, Serial No. 258,364

1 Claim. (Cl. 210—68)

This invention relates to an apparatus and process for separating liquids from solid material. In a specific aspect this invention relates to an apparatus and process for separating spent nitrating acid from nitrocellulose and then washing the separated nitrocellulose. In a more specific aspect this invention relates to an improved apparatus for introducing a feed slurry of liquid and solid material to be resolved to a vertically disposed centrifuge.

Centrifuges of various types have been employed heretofore to separate liquids from solid material. In many instances the centrifuges employed have been of the batch type, and in order to decrease the operating costs of the apparatus, efforts have been made to improve upon the batch-type centrifuges and to construct a continuous type of centrifuge. One continuous type has employed a scraping or peeling knife within the separating drum in order to provide for continuous removal of separated material from the drum. Another type of continuous centrifuge has achieved continuous operation by providing means for axially moving the peripheral wall of the separating drum over an annular wall or walls in order to obtain automatic discharge of the separated solid material from the centrifuge. The various centrifuges employed heretofore have had obvious disadvantages, particularly when employed in such operations as the nitration of cellulose where the nitrated cellulose is separated from spent nitrating acid by centrifuging and then washed free of remaining spent nitrating acid by a series of water washes.

Conventional practice in the manufacture of nitrocellulose involves treating, for example, cotton with nitrating acid for a desired period of time by a batch procedure. Upon completion of the nitration, the discharge valve of the nitrator is opened and the reaction mixture is rapidly metered into a centrifuge which is brought up to the necessary speed for removal of acid from the nitrated cellulose. Spent acid is run by gravity from the outer jacket of the centrifuge to suitable recovery equipment. The centrifuge is stopped and the nitrocellulose is manually forked out through the bottom of the centrifuge into a water immersion basin below. The manual handling of the nitrocellulose and the delay incident to discontinuous operation are objectionable, and the stopping of the centrifuge to fork out the nitrocellulose requires excessive time and causes too much delay for highest quality of product.

In the copending application of Stuart, Serial No. 258,392, filed November 27, 1951, a novel apparatus and procedure are described for separating solids from liquids, for example, nitrocellulose from spent nitrating acids. In some instances, such as in the separation of nitrocellulose from spent nitrating acids, it was found that, when employing a slurry distributor and a splash sleeve, such as those described in Fig. 12 of the above copending application, a portion of the feed adhered to the inner side of the splash sleeve and thus prevented efficient operation of the apparatus. It is one of the primary objects of this invention to provide the separating apparatus

with a splash sleeve and slurry distributor designed to eliminate the adherence of feed thereto.

It is another object of this invention to provide a novel apparatus and method for separating solids from liquids.

It is another object of this invention to provide a novel apparatus and method for separating cellulose derivatives from liquid medium employed in the preparation of said cellulose derivatives.

It is a further object of this invention to provide a novel apparatus and method for separating nitrocellulose from spent nitrating acid obtained during the preparation of said nitrocellulose and for washing said nitrocellulose.

Further and additional objects and advantages of this invention will be apparent from the detailed disclosure hereinbelow.

The objects and advantages of this invention will be clearly apparent from the detailed description of the apparatus shown on the accompanying drawings and the operations thereof with reference symbols referring to like parts wherever they occur.

Apparatus

Fig. 1 is an elevational view of the apparatus with the reciprocating piston in its inactivated position.

Fig. 2 is a plan view of the apparatus through section 2—2.

Fig. 3 is an enlarged elevational view of the centrifuge basket and accompanying equipment with the reciprocating piston in its activated position.

Referring now to the accompanying drawings, Fig. 1 is an elevational view of a centrifuge that can be used to effect the desired separation of liquids and solids. The centrifuge shown in Fig. 1 contains a foraminous or perforate basket supported and driven by rotor shaft **1**. Rotor shaft **1** is attached to and operated by a driving mechanism not shown, and the lower portion of rotor shaft **1**, as shown on Fig. 1, is combined with the necessary bearings, packings, snubbers, snubber rings, oil and grease lines, and the like for the operation of rotor shaft **1**. Since this equipment is conventional and since it does not form a part of the invention, a detailed description thereof is not necessary for an understanding of the invention. However, this conventional equipment is maintained free of corrosive fluids such as acid fumes by air line **2**. Air under pressure is continually passed through line **2** and in this manner the entrance of corrosive fluids to the critical mechanism surrounding shaft **1** is prevented.

Rotor shaft **1** is attached to cylindrical casing **3** which is capped by stop nut **4**. Surrounding casing **3** and contiguous thereto is a second cylindrical casing **5** which is rigidly attached to and moves in conjunction with a rotor piston which is inside the basket of the centrifuge and which will be described in detail infra.

Rotor bottom **6** which forms the bottom of the centrifuge basket is rigidly attached to casing **3** at its inner extremity, and at its outer extremity it is rigidly attached to the upwardly or vertically disposed side wall **7** of the centrifuge basket. The upper portion of side wall **7** is perforated to permit the passage of separated liquids therethrough while retaining separated solids on the inner surface of the vertical side wall. Disposed above rotor bottom **6** is rotor piston **8** which on its lower or bottom side contains a plurality of indentations which surround and thus mesh with elevated and upwardly extending sections **6A** of rotor bottom **6**. Attached to the upper surface of rotor piston **8** is rotor piston face **9** which moves in conjunction with piston **8**, but, if desired, piston face **9** and piston **8** can be combined and made integral to form the rotor piston for the apparatus.

The upper portion of the centrifuge side wall **7** is perforated in order to permit the passage of liquid through the side wall. The actual construction of the side wall

will depend upon such variables as the ultimate use of the centrifuge and the liquids and solids to be separated. A suitable perforated side wall for the separation of spent nitrating acid from a slurry thereof with nitrocellulose has been found to include an outer casing containing a plurality of small openings. Within the outer casing and attached thereto is a screen formed by placing a plurality of vertically disposed metallic bars at regularly spaced distances around the inner surface of the outer casing. This preferred foraminous side wall is described in the copending application referred to above.

Surrounding cylindrical casing **5** and forming an annular space therewith is a third cylindrical casing **10** which is rigidly attached to rotor piston face **9**. Attached to both casing **10** and rotor piston face **9** is slurry distributor **11** which is concavely shaped and which directs the flow of the incoming slurry feed toward the side wall of the rotor basket when the centrifuge is in operation. Disposed above casing **10** and attached to the upper extremity thereof is a frusto-conical casing **12** surrounding the lower portion of the activating liquid tube to be described infra. Since both casing **10** and distributor **11** are attached to piston face **9**, these members, as well as casing **12**, move in conjunction with piston face **9**.

The upper portion of the centrifuge is provided with fume tube **13** for removing undesirable fumes from the centrifuge and with actuating liquid tube **14** for introduction of the liquid necessary for the hydraulic operation of piston **8** and movement of suitably attached members. Tube **14** extends downwardly into casing **12** and it is provided with a second tube **15** of smaller diameter for additional removal of air and fumes displaced from the nitrocellulose. Tube **15** communicates with tube **13** through connecting line **16**. Extending downwardly into the centrifuge and with their lower extremities within the cylindrical side wall of the centrifuge are a plurality of tubes **17**, **18**, **19**, **20**, **21** and **22** for the introduction of wash liquids to the centrifuge. The lower extremities of these wash tubes are at varying levels and spaced so that the separated solids in the form of a layer or mat on the inner periphery of the side wall of the centrifuge can be subjected to a plurality of washings. The centrifuge is provided with cover **23** to which are attached a plurality of splash sleeve hangers **24**. Hangers **24** extend downwardly into the centrifuge, and at their lower extremities they are attached to splash sleeve **25** which forms an annular space with slurry distributor **11** for passage of the feed slurry to the centrifuge. Perforate blow out panel **40** is disposed between cover **23** and curb **26**.

Disposed outside the centrifuge basket are a plurality of curbs **26**, **27**, **28**, **29**, **30**, **31** and **32** for removal of separated solid material, separated liquids and wash liquids. Separated solids are removed via outlet **33** and the separated liquid and wash liquids are removed by suitable outlets provided between the lower extremities of the curbs. Typical curb outlets for liquid removal are shown on the drawing as outlets **34** and **35**. Water or other suitable inert liquid is introduced to the base of the centrifuge via inlet **36**.

The lower portion of side wall **7** is provided with a plurality of thief valves **37** and a second plurality of openings **38**. The importance of and the necessity for these openings in side wall **7** will be more fully explained in the subsequent discussion of the operation of the centrifuge. Opening **39** in rotor piston **8** permits the lubrication of the adjoining surfaces of side wall **7** and piston **8** and piston face **9** by the actuating liquid.

Fig. 2 is a plan view through section **2—2**. This figure shows the relative locations of wash pipes **17**, **18**, **19**, **20**, **21** and **22**. Fig. 2 also shows the location of splash sleeve **25** which is cylindrically shaped and flared at its lower extremity. Splash sleeve **25** is connected to cover **23** by three vertically disposed splash sleeve hangers **24** extending downwardly into the centrifuge. Splash sleeve **25** is maintained motionless by its attachment to hangers **24**. Sleeve **25** does not revolve when the centrifuge is in operation and it does not move in conjunction with the hydraulic activation and deactivation of rotor piston **8**.

Fig. 3 is an enlarged elevational view of the centrifuge showing rotor piston **8** in its activated position. Rotor piston **8** is activated by the introduction of activating liquid through activating liquid tube **14** and thence via the annular space between casing **5** and casing **10**. The rotation and acceleration of the mass of activating liquid hydraulically elevate rotor piston **8** and attached rotor piston face **9** to the point where cylindrical casing **5** approaches but preferably avoids actually contacting stop nut **4** and to the point where the lower extremity of rotor piston **8** at least partially uncovers opening **38**. Since activating liquid is removed via opening **38** to halt the upward movement of cylindrical casing **5**, the position of rotor piston **8**, as shown in Fig. 3, is one of complete activation. In this position of complete activation rotor piston **8** has been elevated substantially above the elevated sections **6A** of rotor bottom **6**, but the indentations in rotor piston **8** still partially mesh with the elevated portions **6A** of rotor bottom **6**, i. e., the lower extremity of rotor piston **8** is slightly below the upper extremity of the elevated sections **6A** of rotor bottom **6**. Fig. 3 also shows the relative position of slurry distributor **11** and splash sleeve **25** when rotor piston **8** and rotor piston face **9** are in a position of complete activation. Additionally, Fig. 3 depicts the relative positions of the lower extremities of the six wash pipes. These wash pipes are positioned so as to wash the separated solid material adjacent to the side wall of the centrifuge at various levels as the separated solid material is pushed upwardly along the centrifuge side wall in the form of a mat or layer thereon by the operation of rotor piston **8**.

It will be understood that the apparatus described above in detail has wide application for the separation of a slurry of liquid and solid material. One application for which the apparatus is particularly suitable is for the separation of nitrocellulose from spent nitrating acids that are obtained during the nitration of cellulosic material. It is a preferred application of this apparatus to effect such a separation of nitrocellulose from spent nitrating acid, and the actual operation of the apparatus will be described in detail employing a feed slurry of nitrocellulose and spent nitrating acids from a cellulose nitration process.

Operation

To initiate the separation, the operation of the centrifuge is begun by the driving mechanism (not shown) operating on shaft **1** which in turn effects the revolution of the centrifuge basket and accompanying members. It will be understood that the centrifuge can be operated at a speed suitable for effecting the necessary separation. With a feed slurry of nitrocellulose and spent nitrating acids and with a centrifuge basket having a diameter of about 23 inches a minimum speed of 700–800 R. P. M. can be employed, and the maximum speed has been found to be about 1700 R. P. M. With the centrifuge at the desired speed and with the rotor piston in the inactivated position as shown in Fig. 1, a measured quantity of feed slurry is introduced to the apparatus passing through the annular space between splash sleeve **25** and distributor **11**. Since the centrifuge is revolving rapidly, the feed slurry is accelerated and transferred toward bar screen **43** along the inner periphery of side wall **7**. For effective separation of a slurry of nitrocellulose and spent nitrating acid it is preferred that the maximum space between the metallic bars **44** of bar screen **43** be no greater than 0.01 inch. The operation of the centrifuge is continued and spent nitrating acid is forced out of the nitrocellulose while the latter is retained in a mat on the inner surface of the bar screen. The spent nitrating acid passes through the bar screen and through openings **42** in perforated casing **41** surrounding the bar screen, and after passage

along curb 32 it is recovered for further use. After a sufficient period of time has elapsed for removal of spent nitrating acid from the solid nitrocellulose, activating liquid is introduced to the centrifuge via tube 14. This activating liquid may be any liquid, preferably compatible with the spent nitrating acid, that will provide suitable hydraulic operation of piston 8 and the actual liquid that is employed will vary with the separation that is being effected by the centrifuge. In some instances water will be found to be a suitable activating liquid. For the separation of nitrocellulose from spent nitrating acid a portion of the spent nitrating acid itself can be used as the activating liquid. Thus, in the preferred operation being described, a predetermined quantity of spent nitrating acid enters the centrifuge via tube 14 and it passes through the annular space between casing 5 and casing 10. Then it passes through openings 50 in piston 8 and it enters the space between the indentations in piston 8 and the elevated sections 6A of bottom plate 6. The activating liquid builds up sufficient pressure, due to the centrifugal action, to hydraulically elevate piston 8, and consequently the movement of piston 8 and attached piston face 9 effects an upward movement of the mat of nitrocellulose on the bar screen equivalent in distance to the length of stroke of piston 8. The activating liquid causes the movement of piston 8 sufficient to bring the lower extremity of that piston in such a position that openings 38 in side wall 7 are at least partially uncovered, and activating liquid then escapes through openings 38. Thus, the upward movement of rotor piston 8 is controlled by escape of the activating liquid and by the volume of activating liquid employed. Any number of openings in side wall 7 corresponding to opening 38 can be used to effect the desired removal of activating liquid. Actually, in operation twenty-four openings have been employed, but this number can be widely varied, and the number used will depend upon the actual size of the individual openings. During the activation of rotor piston 8, thief valves 37 in side wall 7 are open continuously, and consequently activating liquid is continuously escaping through these valves. The area of these valves is considerably less than the area of openings 38, and the area of valves 37 is adjusted to effect the desired lowering or inactivation of rotor piston 8 in a predetermined time interval. The upper limit of the stroke of piston 8 is determined by the position of openings 38, and the rate of release of activating liquid through valves 37 determines the speed at which rotor piston 8 is returned to its inactivated position. The number of thief valves that is necessary for the efficient operation of the centrifuge is variable over relatively wide limits. In operation eight thief valves corresponding to valve 37 have been employed in side wall 7, but it will be seen that this number can be varied while remaining within the scope of the invention.

As rotor piston 8 begins its descent from its activated position to the inactivated position, a second measured quantity of feed slurry is introduced to the centrifuge as previously described, and as spent nitrating acid is being removed from this second portion of feed slurry the first portion of nitrocellulose introduced to the centrifuge is washed with a sufficient quantity of spent nitrating acid diluted to about a 60% concentration introduced via line 17 to displace spent nitrating acid, which generally has a concentration in excess of 60%, remaining in the mat of nitrocellulose. The latter is recovered after passage along curb 31, and it can be subsequently used as an activating liquid or it can be subjected to suitable steps for the recovery of the acids. Upon completion of a predetermined time interval during which the first portion of nitrocellulose is washed with 60% acid and spent nitrating acid is centrifugally wrung out of the second feed portion, another predetermined amount of activating liquid is introduced to the centrifuge as previously described to effect the upward movement of rotor piston 8 and the mat of nitrocellulose being formed upon the inner walls of the bar screen.

Upon continued operation of the centrifuge as previously described, a continuous mat of nitrocellulose is formed upon the inner surface of the bar screen. As the nitrocellulose progresses upward along the bar screen, it is washed at various levels with varying concentrations of spent nitrating acid. As already pointed out, the first washing was effected with a 60% acid. The second washing can be effected with a 40% acid; the third washing with a 20% acid, and the final washing with water. These acid concentrations are given merely by way of example, and it will be understood that other concentrations can be used without departing from the scope of the invention. The acid which is displaced from the nitrocellulose during each of the washings is recovered after passage along the respective curbings, and the recovered acids can then by recycled and used to carry out additional washing of the nitrocellulose mat inside the centrifuge. The final washing of the nitrocellulose is effected with water and subsequently the washed nitrocellulose is effected with water and subsequently the washed nitrocellulose is sloughed over the top of the bar screen and side wall 7 with an additional amount of water. This nitrocellulose and water pass along curbing 27 to the base of the centrifuge. Additional amounts of water are introduced to the centrifuge via inlet 36 to wash the nitrocellulose along the sloped base of the centrifuge and to remove the nitrocellulose via outlet 33. The nitrocellulose thus removed can then be separated from accompanying water by any suitable process, and it is then ready for any subsequent steps in the nitrocellulose manufacturing process.

The washing of the solid mat on the inner surface of the bar screen, as described above, is particularly useful in the separation of nitrocellulose from spent nitrating acid. In effecting other separations of solid material from accompanying liquids, such washing steps may or may not be necessary. In some instances a fewer or greater number of washing steps than those described above will be found useful, while in other instances no washing steps will be required. It will be understood that the number of washing steps that are employed can be varied without departing from the scope of the invention and that it is within the scope and spirit of the invention to operate the centrifuge without any washing steps.

The splash sleeve of this invention is preferably concentrically disposed and rigidly attached to the upper portion of the centrifuge, and it remains stationary when the centrifuge is in operation, i. e., the splash sleeve does not revolve in conjunction with the centrifuge basket. The splash sleeve extends downwardly into the centrifuge basket concentric with cylindrical casing 10 and flares or curves outwardly near its lower extremity with the lower segment of the splash sleeve being in a substantially horizontal plane parallel with piston face 9. Slurry distributor 11 is concavely shaped with its upper extremity attached to casing 10 and with its lower extremity attached to piston face 9 below the lower horizontal portion of splash sleeve 25. Slurry distributor 11 moves upwardly and downwardly with casing 10 and piston face 9 when the centrifuge is in operation. Slurry distributor 11 and splash sleeve 25 form an annular space for introduction of feed slurry to the centrifuge, and the distance between the two members is such that slurry distributor 11 approaches but does not touch splash sleeve 25 when piston 8 is in its activated position. When the centrifuge is in operation, slurry distributor 11 directs the flow of slurry feed outwardly toward the bar screen of the centrifuge side wall, and splash sleeve 25 directs the flow of feed slurry toward the lowermost section of the bar screen. The combination of the splash sleeve and slurry distributor orients the incoming feed slurry on the section of the bar screen that is cleared of solid material by the upward movement of piston 8 and piston face 9,

and the design of the splash sleeve and slurry distributor prevents the adherence of feed slurry to the inner surface of the splash sleeve.

Numerous other variations and modifications within the scope of the invention will be apparent to those skilled in the art from the detailed disclosure above.

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

In a centrifuge having a vertically disposed cylindrical drum with a solid bottom and a perforate side wall rotatably mounted on a vertical drive shaft and having a solid horizontally faced piston disposed within the drum coaxial therewith and rotatable with said drum, said piston being movable axially with a reciprocating stroke to displace solid substances upwardly in the form of a layer on said side wall along the inner periphery of said side wall, the improvement comprising in combination, a centrifugal feed distributor attached to the face of the piston to rotate therewith and having a concave feed distributing face, and a stationary sleeve disposed between the distributor and the side wall of the drum and concentrically spaced apart from said distributor, said sleeve being vertically cylindrical in its upper portion and curving outwardly near its lower extremity with the lowermost segment of said sleeve being in a substantially horizontal plane parallel with said horizontally faced piston, the curved portion of said sleeve being convexo-concave in cross-section with the convex face opposing the concave face of said distributor, the lower periphery of said stationary sleeve being in close proximity to but not in

contact with said distributor when said piston is at the upper limit of its upward stroke, said distributor and said stationary sleeve forming an annular space between them into which feed slurry is fed on each downward stroke of the piston for distribution onto the perforate side wall of the drum, the concave face of said distributor directing a substantial portion of the feed slurry to said perforate side wall of the drum against the convex face of said stationary sleeve, the combination of said distributor and said stationary sleeve orienting the feed slurry uniformly onto the portion of said perforatae side wall of the drum that is cleared of accumulated solid material by upward movement of the piston and preventing adherence of feed slurry to the convex face of said stationary sleeve.

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