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Verreyne et al.

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[54] **DELIGNIFICATION AND BLEACHING OF CELLULOSE PULP WITH OXYGEN GAS**

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Related U.S. Application Data

[62] Division of Ser. No. 840,639, July 19, 1969, Pat. No. 3,660,225.

[30] **Foreign Application Priority Data**

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Oct. 14, 1968	South Africa	68/6629

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[58] **Field of Search** ..... 68/5 R, 5 C, 5 D, 68/5 E, 181 R; 259/7, 8

[56] **References Cited**

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[57] **ABSTRACT**

Apparatus for dividing a stream of pulp into a series of layers containing discrete batches and progressively transferring the batches from layer to layer in controlled fashion so that the height of each layer does not exceed a maximum value at which the pulp at the bottom of a layer has a predetermined minimum gaseous content. The pulp is contacted with oxygen gas under pressure while progressively transferring pulp from one layer to the next in a series. The apparatus comprises a cylindrical pressure vessel including axially spaced floors which define chambers between them, the chambers being subdivided axially into compartments, each floor having an aperture and the compartments and floors being relatively movable to allow pulp in the compartments sequentially to be transferred from one chamber to the next as relative movement occurs.

8 Claims, 6 Drawing Figures

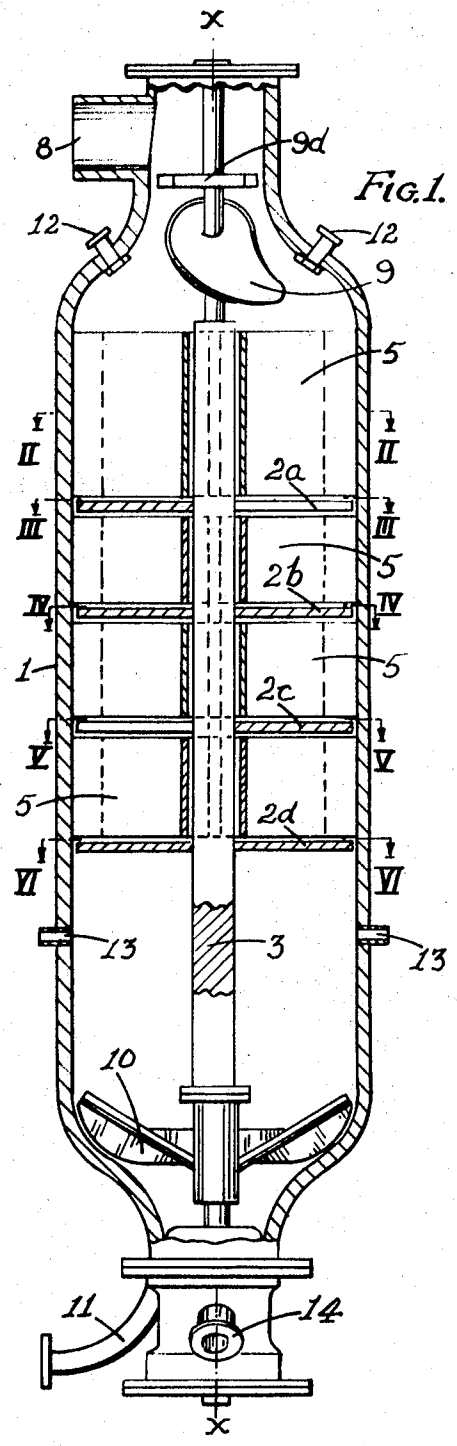


Fig. 1.

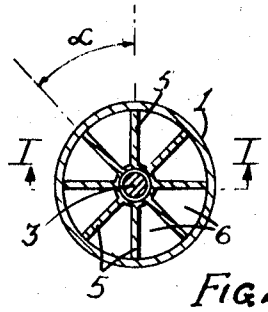


Fig. 2.

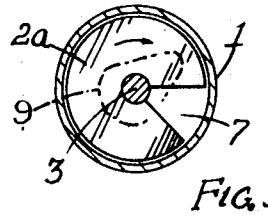


Fig. 3.

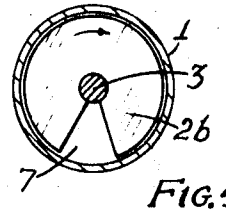


Fig. 4.

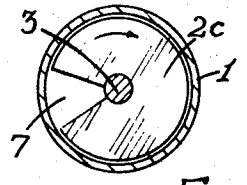


Fig. 5.

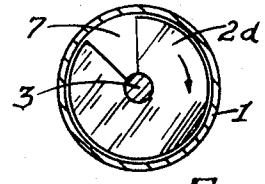


Fig. 6.

DELIGNIFICATION AND BLEACHING OF CELLULOSE PULP WITH OXYGEN GAS

CROSS RELATED APPLICATION

This Application is a division of application, Ser. No. 840,639 filed July 10, 1969 and issued as U.S. Pat. No. 3,660,225 on May 2, 1972.

This invention relates to the delignification and bleaching of cellulose pulps with oxygen gas under pressure.

For the purpose of this specification the term "oxygen gas" includes any gas containing free oxygen, such as air.

It is known that a pulp can be delignified and bleached by subjecting it in an alkaline medium to the action of oxygen gas. French patent specification No. 1,387,853 discloses the treatment of a chemical pulp with oxygen gas under pressure in an alkaline medium in the presence of a catalyst or protector acting to preserve the physical and mechanical strength properties of the pulp. South African patent specification No. 67/3680 discloses a method of delignifying and bleaching an alkaline pulp by pretreating the pulp with an acidic medium and thereafter subjecting the pulp in an alkaline medium to the action of oxygen gas under pressure.

Delignification and bleaching agents other than oxygen gas are known and normally such other agents react with lignins in an aqueous medium. Generally the correct amount of these other agents can be dissolved in the aqueous phase of the wet pulp. Oxygen gas treatment must, however, be conducted under pressure and even then only small amounts of oxygen can be dissolved in water at such high oxygen partial pressures. As a result, it is necessary for the oxygen gas to be applied directly to the pulp mass in sufficiently large quantity if a satisfactory reaction is to be obtained.

It has been found that the amount of oxygen required for a satisfactory reaction can be accommodated by the pulp only if the bulk density of the pulp is sufficiently low and if substantially no free liquid drains from the pulp. The presence of drained liquid is detrimental in that it accumulates in certain areas where it occupies space that ought to be occupied by oxygen and excludes further oxygen from re-plenishing that which has been dissolved and then consumed. Drainage of liquid also results in unequal distribution of the alkali normally added and corresponding variation in the final bleaching of the pulp.

It has further been found that the design of conventional pulp bleaching towers are not satisfactory for the treatment of pulps with oxygen gas. Normally, such towers operate at relatively low consistencies with no free gas occluded in the pulp. Tests have shown that if such towers are operated at higher consistencies the pulp compresses under its own weight and liquid is inclined to collect at the bottom of such conventional towers so that the oxygen content of the pulp towards the bottom of such towers is too low for satisfactory reaction.

Although methods of repeated exposure of pulp to a gas are known such methods generally suffer the disadvantage of complex mechanisms with consequent excessive wastage of space between stages and difficulty of sealing against over pressure as well as high equipment costs.

Methods using a series of trays provided with rake arms causing transfer alternatively at the periphery and the centre of the trays are known. These suffer the disadvantage that energy is used wastefully due to roll-back of free flowing materials while sequential flow and control of residence time is poor as a consequence. The applicants have found by experiment that this type of apparatus is incapable of handling beds of pulp deeper than 0.4 metres in a manner capable of achieving good results by the oxygen bleaching process. As a result of this height limitation either the number of plough systems must be increased or the diameter of the pressure vessel must be increased. Both of these actions cause a high cost penalty.

It is accordingly an object of the present invention to provide improved treatment of cellulose pulp with oxygen gas under pressure with which the above disadvantages are at least minimized.

Our co-pending U.S. Pat. application, Ser. No. 840,639, now U.S. Pat. No. 3,660,225, discloses a continuous method of treating a cellulose pulp with oxygen gas under pressure comprising the steps of continuously introducing pulp at a consistency of 16 percent to 67 percent into the upper region of a vertically elongated pressure vessel; heating the pulp; distributing the heated pulp in well divided form in a series of separate layers individually supported within the pressure vessel at different levels along the length of the pressure vessel; progressively transferring pulp downwardly from one layer to the next in the series after a residence period in each layer; contacting the pulp in each layer with oxygen gas at a partial pressure in excess of 3 bars absolute; restricting the height of at least certain of the layers to a maximum value at which the compaction of pulp under its own weight at the bottom of the layer is limited to a maximum value at which the pulp at the botton of the layer has at least a predetermined minimum oxygen gas content; and continuously withdrawing treated pulp from the lower region of the pressure vessel.

The minimum amount of oxygen required at the bottom of a layer will depend on the number of layers in the series and the period of time the pulp is retained in each layer and may also depend on the temperature and pressure.

Preferably each quantity of pulp to be transferred through subsequent layers is determined towards the top of the pressure vessel. The pulp may be fluffed at the top of the vessel before it is distributed in layers. The pulp is transferred from one layer to the next in the series and away from the last layer in the series under the influence of gravity. The transfer is effected in stepwise fashion.

An advantage of such stepwise transfer of pulp is that the pulp may repeatedly be transferred through an atmosphere of oxygen Moreover the pulp is subjected to a rearrangement which is favorable for an even treatment thereof. gas, thereby to re-introduces further oxygen.

The pulp in each layer is subdivided into a plurality of discrete batches which are transferred sequentially in stepwise fashion from one layer to the next in the series and away from the last layer in the series substantially without intermixing with other batches.

The support for each layer of pulp is removed sequentially from underneath successive batches of pulp in the layer to permit the sequential transfer of succes-

sive batches of pulp from one layer to the next under the influence of gravity, the transfer of pulp from one layer to the next being controlled for a batch of pulp transferred from one layer to the next to occupy free space in said next layer which has previously been vacated by a batch of pulp transferred away from said next layer.

The pulp in each layer is collected in a plurality of separate compartments as discrete batches which are transferred sequentially in stepwise fashion from one layer to the next in the series and away from the last layer in the series substantially without intermixing with other batches, new batches of pulp being collected sequentially in successive compartments in the first layer in the series to replace batches which are transferred from the first to the second layer in the series.

The applicants have found that for satisfactory reaction between the pulp and the oxygen gas, the following are important,

a. The pulp should be at a consistency which is sufficiently high for the pulp to contain enough oxygen and so that there is no substantial drainage of liquid from the pulp;

b. The pulp is in a well divided form, such as in the form of noodles, or preferably in fluffed form since the liquid retention of fluffed pulp is better than that of noodle pulp and the bulk density of the former is lower than the latter;

c. The height of any pulp layer is not too great otherwise the pulp compacts under its own weight with a resultant expulsion of liquid and/or oxygen at the bottom of the layer.

The applicant's tests have indicated the following:

a. That water begins to drain from softwood noodle pulp at a consistency of 15 percent under zero pressure and at a consistency of 18 percent under a pressure of a 2.40 metre high pulp layer.

b. The water begins to drain from hardwood noodle pulp at a consistency of 13 percent under zero pressure and at a consistency of 16 percent under the pressure of 2.4 metre high pulp layer.

c. That fluffed pulp is less inclined to drain water.

d. That for satisfactory reaction between pulp and oxygen gas, the pulp should contain at least 1 percent oxygen by weight on a dry pulp basis at the bottom of a layer of pulp. This condition is attained with different pulps with the following maximum layer heights under a selected typical reaction condition of 170 p.s.i.g. and 130°C and using pure oxygen:

Softwood noodle pulp at 20% consistency	3.00 metre.
Softwood fluffed pulp at 18% consistency	3.00 metre.
Hardwood noodle pulp at 18% consistency	3.00 metre.
Hardwood fluffed pulp at 18% consistency	3.00 metre.

The heights of these layers can be increased if the sidewalls exert any friction. However, the object is not to unnecessarily increase the friction.

In the light of the above results, the method of the present invention is preferably carried out with pulp at a consistency of not less than 18 percent in the case of hardwood noodle and fluffed pulps and of softwood fluffed pulp and with a pulp consistency of not less than 20 percent in the case of softwood noodle pulp. Fluffed pulp is to be preferred.

Furthermore, the heights of the various layers may be arranged so as not to exceed 3.00 metres and preferably not to exceed 1.20 metres, especially if lower operating pressures are used.

According to the present invention, apparatus suitable for carrying out the above method comprises a vertical pressure vessel; spaced decks located transversely within the pressure vessel and defining a series of pulp trapping chambers in the vessel; an outlet port from each chamber located in the deck thereof and communicating with an adjacent chamber so as to provide a path for pulp to move gravitationally from chamber to chamber, each oulet port having a radial dimension substantially equal to the effective radial dimension of the chamber; dividing walls projecting upwardly transverse to the floor of each chamber to divide the chamber into a plurality of compartments; means for contacting the pulp with oxygen gas during passage along the pathway; said dividing walls being stationary; and means for moving the decks of the chambers relative to the dividing walls to permit the deck of each chamber to sweep past the lower edge of its associated dividing wall so that the outlet port in the deck passes sequentially underneath successive compartments in the chamber.

Preferably, the decks are spaced so that the layers of pulp supported thereby do not exceed a maximum value at which the pulp at the bottom of a layer contains a predetermined minimum gaseous content as outlined above. The decks may be essentially horizontal but may be somewhat inclined towards the center.

In order to more effectively introduce oxygen at each transfer level and in order to minimise any tendency for compaction, the chamber between the top two decks may be somewhat less in height than all subsequent chambers;

The chamber between the two top floors may thus be not more than 3.00 metres and preferably not more than 1.20 metres high, although for apparatus handling pulps well fluffed and not below 20 percent consistency this restriction need not apply.

Prefeably each stationary dividing wall extends over substantially the entire height of its chamber. Thus the spaces above each deck are divided into a plurality of separate upright pulp holding apartments, each deck being arranged for pulp to be transferred sequentially from different compartments above it to the space below it. The pulp from a compartment above a deck may be transferred to a compartment below the deck and preferably, the pulp is transferred in a uniform manner without any hang-ups.

The means for moving the decks comprises a shaft which extends axially in the pressure vessel, the decks being mounted on the shaft for rotation therewith.

Each stationary dividing wall has a lower edge so located relative to its associated movable deck to retain the pulp in each compartment as a discrete batch substantially without intermixing with other batches during movement along the pathway.

The outlet ports of adjacent movable decks are angularly off-set from one another with respect to a central axis of the vessel, so that substantially all the pulp passing into a compartment is retained in that compartment for a controlled residence period.

Means may also be provided for fluffing pulp on introducing it to said pathway. Preferably a rotary feed-

ing device is provided for distribution of pulp above the upper floor.

The pressure vessel may be provided with a pulp inlet towards its upper end and with a pulp outlet towards its lower end. The vessel may further be provided with one or more steam and oxygen gas inlets to maintain the temperature and pressure at a selected value such as 130°C and 140 p.s.i.g.

The pressure vessel towards its outlet may be provided with means to re-unite the pulp into a single column which may be washed, diluted, cooled and discharged from the vessel in known manner.

An advantage of the apparatus is that the shaft may enter the vessel at the bottom below the level of the diluted pulp in this way no glands operate in the gaseous phase thus simplifying the shaft sealing problem.

An added advantage is that the compartments are run substantially full which is an important factor in equipment operating under pressure.

For a clear understanding of the invention a preferred embodiment will now be described by way of example with reference to the accompanying drawings in which:

FIG. **1** is a vertical sectional view of one embodiment of a pulp treating apparatus according to the invention,

FIG. **2** is a cross-section on the line II—II in FIG. **1**,

FIG. **3** is a cross-section on the line III—III in FIG. **1**;

FIG. **4** is a cross-section on the line IV—IV in FIG. **1**;

FIG. **5** is a cross-section on the line V—V in FIG. **1**; and

FIG. **6** is a cross-section on the line VI—VI in FIG. **1**.

Referring to FIGS. **1** to **6**, reactor **1** comprises a cylindrical pressure vessel which is provided with a plurality of vertically spaced pulp support decks **2***a*, **2***b*, **2***c* and **2***d* in the form of spaced discs, mounted on shaft **3** which is located co-axially within reaction vessel **1** and which is adapted to be rotatably driven by a motor (not shown). The number of decks may be increased to any convenient number and are not limited to **4** as in this example.

A plurality of vertically extending, radially disposed stationary dividing walls **5** are located above each of the decks **2***a*, **2***b*, **2***c* and **2***d* to divide the chamber above each deck into a plurality of upright compartments **6** disposed radially about shaft **3**. The dividing walls **5** at the different levels are vertically aligned to provide corresponding compartments **6** at the different levels which are vertically aligned. The dividing walls **5** above the deck **2***b* are about 1.20 metres high and the dividing walls **5** above the decks **2***c* and **2***d* are about 1.30 metres high. The height of the chamber above deck **2***a* is more than 1.20 metres in order to be able to provide the pulp layer of 1.20 metres in the chamber above deck **2***b*.

As can be seen from FIGS. **2** to **6** of the drawings, each deck is provided with an aperture **7** which may for instance conform to the cross-sectional configuration of the compartments **6**, the apertures of the various decks being angularly off-set preferably by an angle greater than α but usually less than 2α where α corresponds to the angle subtended by one compartment **6** at the axis. The angle is so adjusted that all the pulp passing into the compartment is retained for the controlled residence period.

At its upper end, reaction vessel **1** is provided with a pulp inlet **8** and a rotary feeding device **9** which is mounted on shaft **3** and which is adapted to direct incoming pulp into the various compartments **6** above upper deck **2***a*. Device **9***d* is provided to fluff incoming pulp. At its lower end, reaction vessel **1** is provided with dilution nozzles **14** and with a scraper and extractor **10** for discharging treated pulp through outlet **11**. The reaction vessel **1** is further provided at its upper end with oxygen and steam inlets **12** and at its lower end with oxygen inlets **13**.

In operation, heated pulp plus chemicals and oxygen plus steam are introduced into reaction vessel **1** at its upper end through inlets **8** and **12** respectively. The oxygen is admixed with the pulp by the fluffing device and then directed into compartments **6** above upper deck **2***a* by feeding device **9**. The pulp may be premixed with oxygen before entering reaction vessel **1**, if necessary. By rotatably driving shaft **3**, the decks **2***a*, **2***b*, **2***c* and **2***d* are rotated so that the aperture **7** in each deck passes successively underneath the compartments **6** above such deck. It will be appreciated that as each deck rotates, a batch of pulp equal in quantity to the layer supported by deck **2***b* drops out of each successive compartment **6** above the deck into the corresponding compartment below the deck. Once the aperture **7** in a deck has passed a compartment **6** above the deck, the pulp in that compartment **6** above the deck remains substantially undisturbed until the aperture **7** reaches that compartment again during the next revolution of the deck. In this manner pulp is transferred compartment by compartment in stepwise fashion from the upper end of the pressure vessel **1** towards the bottom of the vessel from where treated pulp is discharged through outlet **11**. The pulp drops by gravity in a stepwise cascade so that the pulp is spread or loosened and oxygen in reaction vessel **1** has access and can penetrate evenly into the smaller spaces and pores of the pulp before the pulp forms a horizontal layer again. It will be appreciated that the pulp contained in reaction vessel **1** is divided into a plurality of batches, on each of the decks **2***a*, **2***b*, **2***c* and **2***d* by the dividing walls.

The lower region of each stationary dividing wall **5** is closely spaced from its movable floor or deck **2***a*, **2***b*, **2***c* and **2***d* so that the dividing wall **5** retains the pulp in each compartment **6** as a discrete batch substantially without intermixing with other batches during movement along the pathway.

The speed of rotation of shaft **3** and therefore, of the decks, is dictated by the total height of the superimposed layers of pulp in reaction vessel **1** and the required reaction time. It will be appreciated that in the above example during each revolution less an angle equal to the angle of off-set of the decks, the pulp moves down about 1.20 metres, i.e. the height of the pulp layer above deck **2***b*. Where, for example, the total height of pulp in the reaction vessel is 15 metres; and the required reaction time is 30 minutes, and the pulp is required to move downwardly at a speed of 0.5 metres per minute, the shaft speed would be $(0.5/1.2) = 0.41$ r.p.m.

It will be appreciated that the reaction time can be changed by changing the speed of rotation of the shaft and thus of the decks. In that event, it may also be necessary to change the rate of pulp introduction into and extraction from the reaction vessel for satisfactory continuous operation.

We claim:

1. Apparatus for treatment of cellulose pulp with oxygen comprising a vertical pressure vessel; spaced decks located transversely within the pressure vessel and defining a series of pulp trapping chambers in the vessel; an outlet port from each chamber located in the deck thereof and communicating with an adjacent chamber so as to provide a path for pulp to move gravitationally from chamber to chamber, each outlet port having a radial dimension substantially equal to the effective radial dimension of the chamber; dividing walls projecting upwardly transverse to the deck of each chamber to divide the chamber into a plurality of compartments; means for contacting the pulp with oxygen gas during passage along the pathway; said dividing walls being stationary; and means for moving the decks of the chambers relative to the dividing walls to permit the deck of each chamber to sweep past the lower edge of its associated dividing wall so that the outlet port in the deck passes sequentially underneath successive compartments in the chamber.

2. Apparatus as claimed in claim 1 wherein the chamber between the top two movable decks is less in height than all subsequent chambers.

3. Apparatus as claimed in claim 2 wherein the chamber between the top two movable floors is not more than 3 meters high.

4. Apparatus as claimed in claim 1 wherein each stationary dividing wall extends substantially the entire height of its associated chamber.

5. Apparatus as claimed in claim 1 wherein the means for moving the decks comprises a shaft which extends axially in the pressure vessel, the decks being mounted on the shaft for rotation therewith.

6. Apparatus as claimed in claim 1 wherein each stationary dividing wall has a lower edge so located relative to its associated movable deck to retain the pulp in each compartment as a discrete batch substantially without intermixing with other batches during movement along the path.

7. Apparatus as claimed in claim 1 wherein the outlet ports of adjacent movable decks are angularly off-set from one another with respect to a central axis of the vessel, so that substantially all the pulp passing into a compartment is retained in that compartment for a controlled residence period.

8. Apparatus as claimed in claim 1 comprising means for fluffing pulp on introducing the same to said path.

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