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[54] **OXYGEN BLEACHING**

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[30] **Foreign Application Priority Data**

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[52] **U.S. Cl.** .... **68/15,** 68/181 R, 68/183, 68/207, 259/4, 259/6

[51] **Int. Cl.** **D21c 9/10**

[58] **Field of Search** .... 68/15, 5 C, 181 R, 68/183, 184, 207

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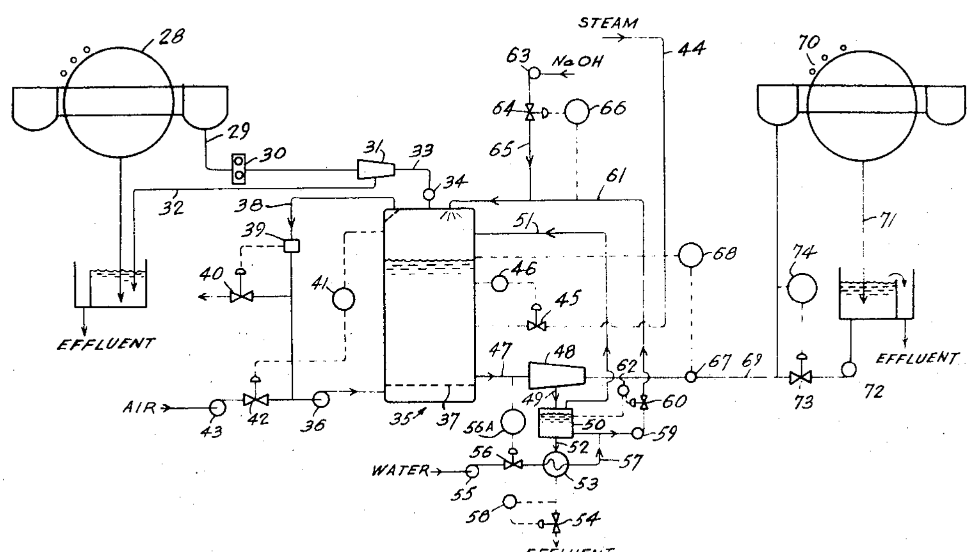
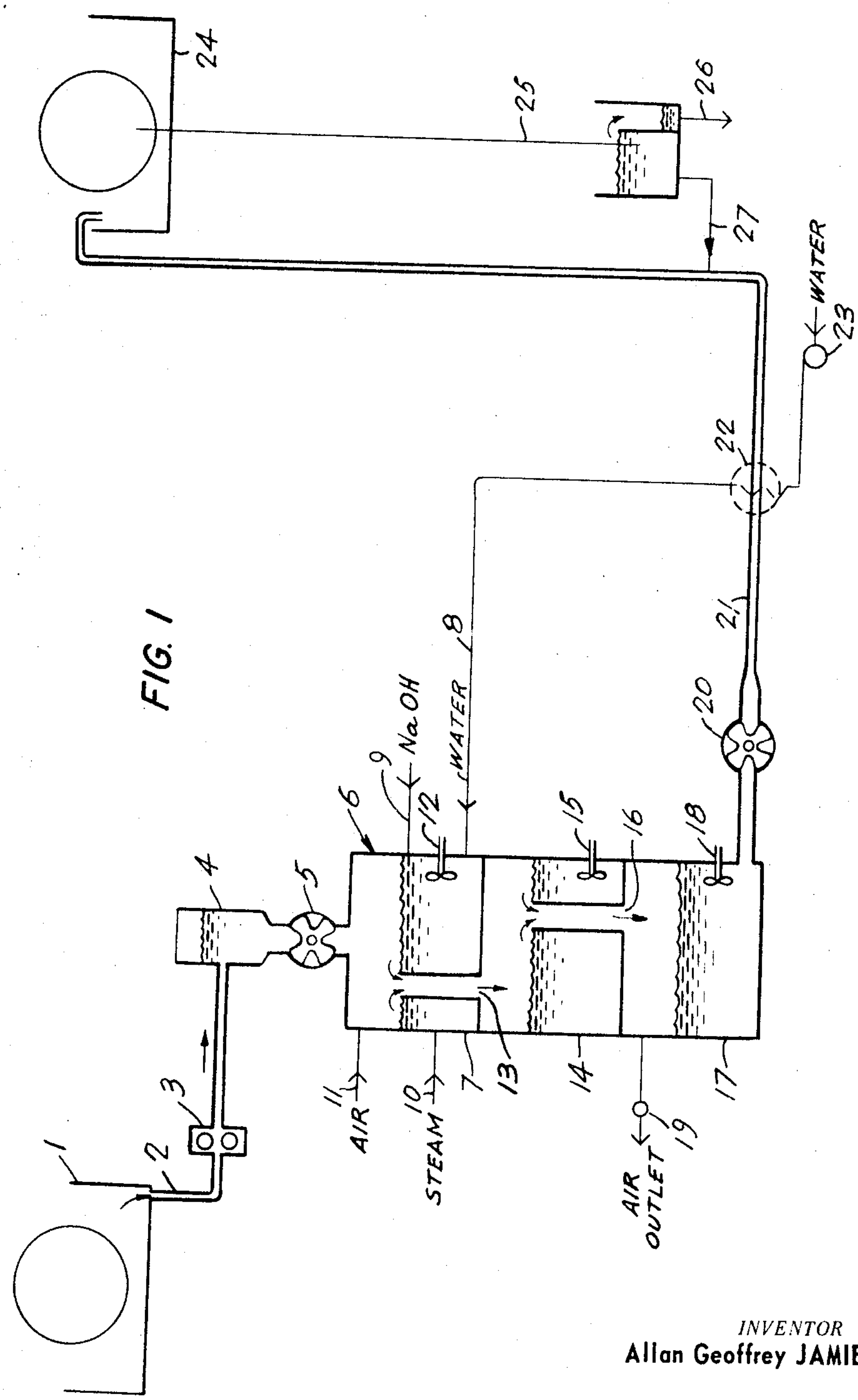
[57] **ABSTRACT**

An apparatus for treating lignocellulosic material such as wood pulp with oxygen under pressure as a step in an oxygen bleaching process. The apparatus introduces the lignocellulosic material, oxygen and other reagents into the pressurized vessel continuously, and continuously withdraws the treated product and spent reagents. The apparatus permits the carrying out of a continuous oxygen bleaching process.

13 Claims, 9 Drawing Figures





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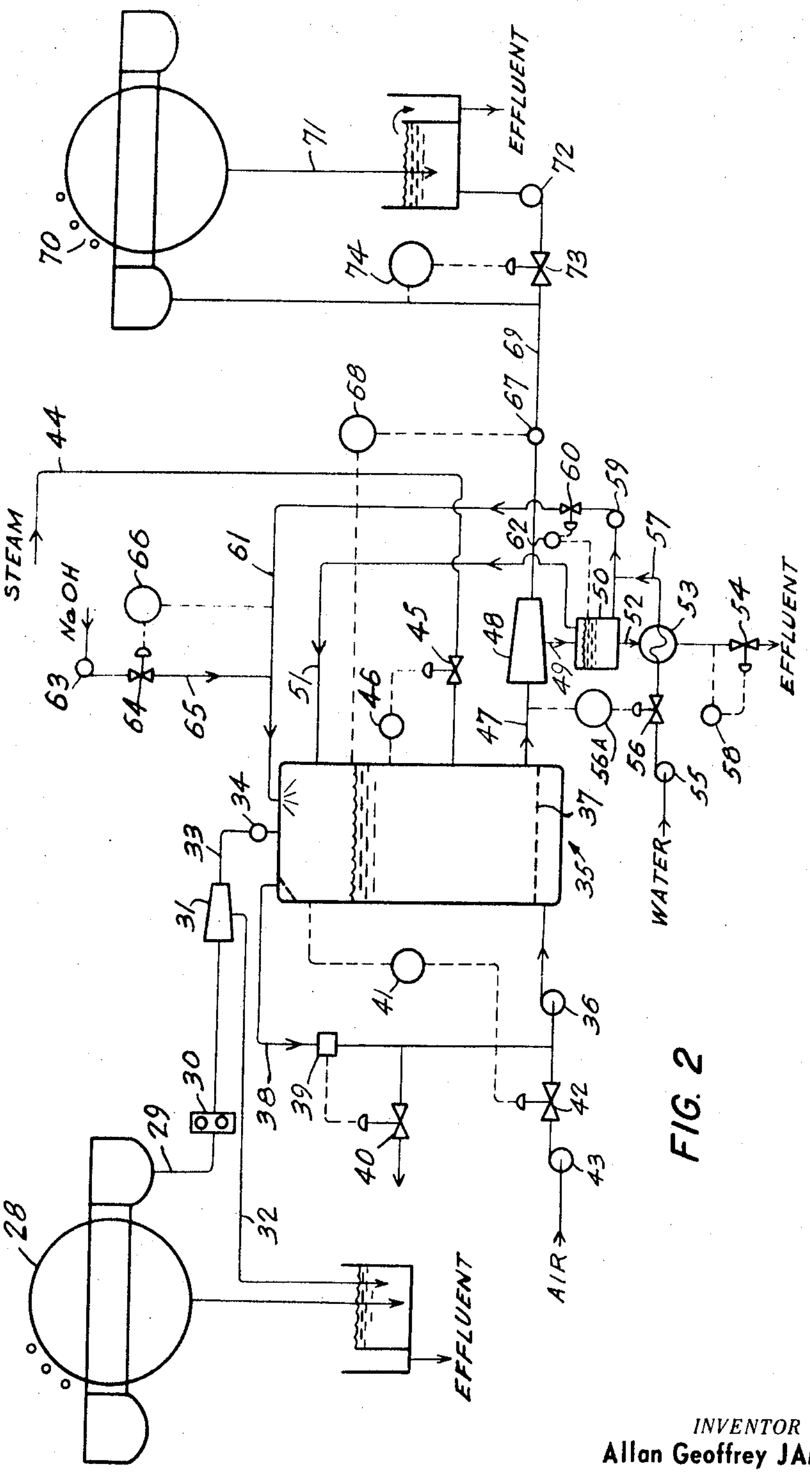

FIG. 2

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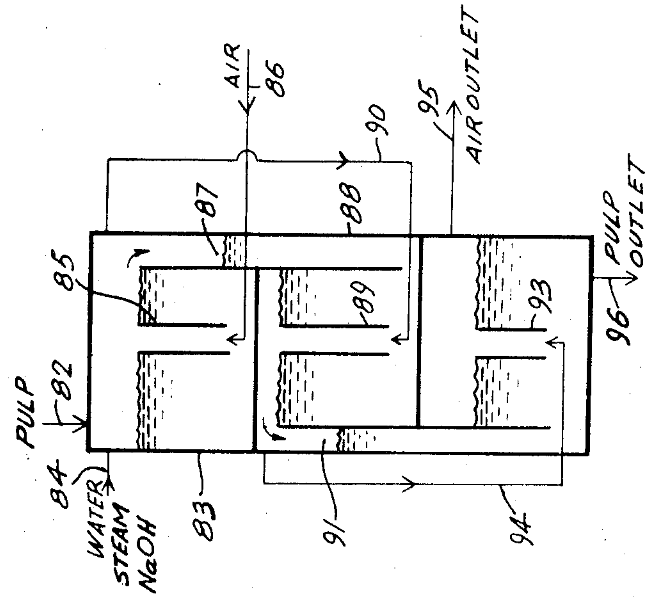

FIG. 4

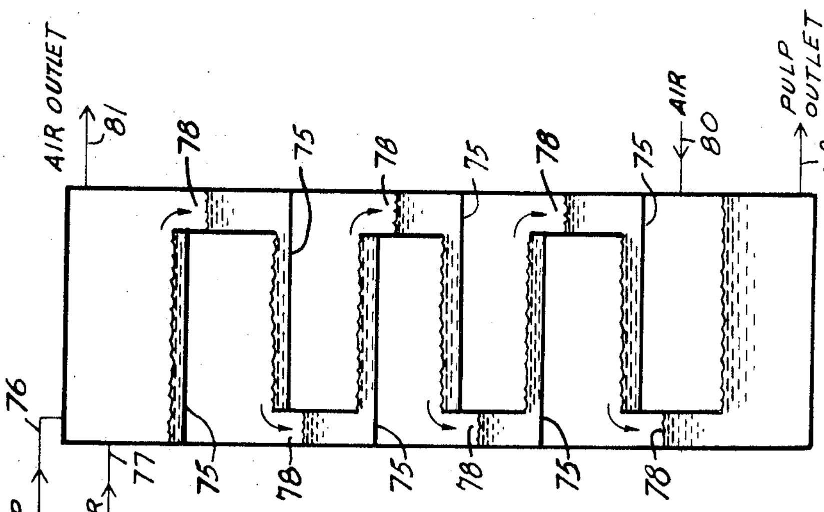

FIG. 3

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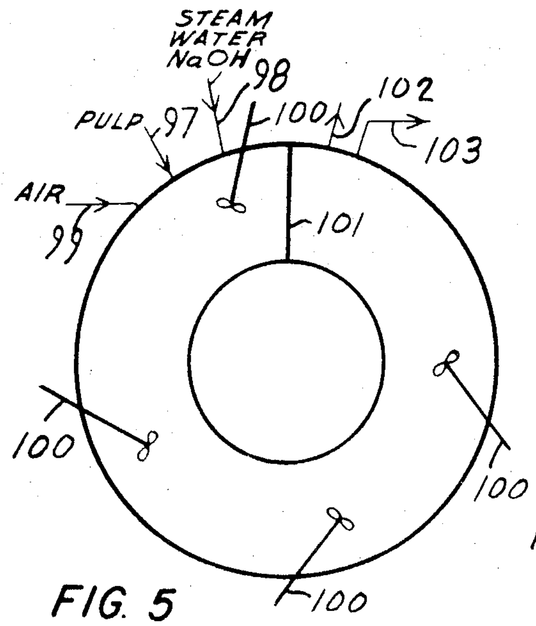

FIG. 5

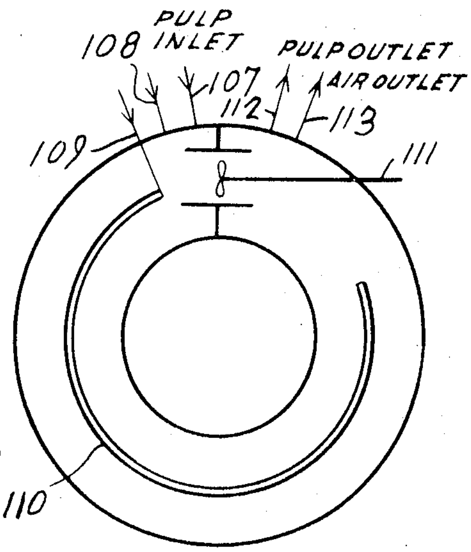

FIG. 8

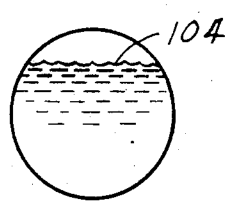

FIG. 6

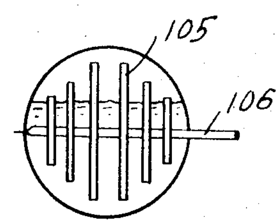

FIG. 7

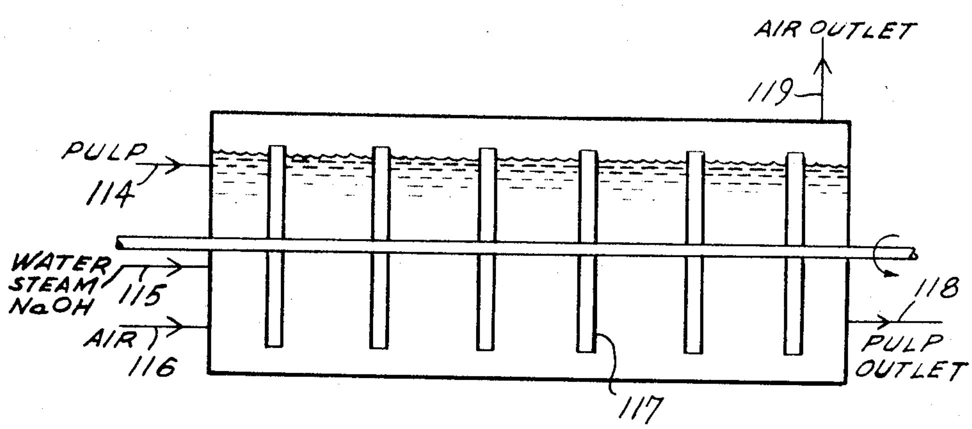

FIG. 9

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OXYGEN BLEACHING

This invention relates to a process for bleaching lignocellulosic material with oxygen and to an apparatus for carrying out the process.

Lignocellulosic material is used widely for making paper and related products. The raw material usually is wood and must be treated to remove the lignin that bonds the cellulose fibres together. Normally the initial part of this treatment takes place in a pulp digester. In some cases the pulp from the digester is suitable for paper making without further purification. For many applications, however, the pulp is too dark in colour and must be bleached. Various bleaching procedures are known, the most common employing chlorine or chlorine containing compounds. Recently oxygen under pressure in alkaline medium has come into use as a means for solubilizing the residual lignin of lignocellulosic material. The efficient commercial utilization of an oxygen bleaching process requires means for contacting large amounts of pulp in alkaline medium with oxygen under pressure. Although batch processes are possible there is advantage in employing a continuous process. A continuous process, however, involves the provision of means for the continuous introduction of pulp and reagents into a pressurized reactor and means for the continuous removal of the treated pulp and spent reagents from the pressurized reactor. The apparatus of the present invention provides means for carrying out such a continuous process.

It is thus a primary object of the invention to provide means for the continuous oxygen treatment of lignocellulosic material. Additional objects will appear hereinafter.

The apparatus of this invention comprises a reaction vessel adapted to operate at supra atmospheric pressure, means for supplying oxygen or an oxygen-containing gas to the vessel under supra atmospheric pressure, means for introducing continuously lignocellulosic pulp in aqueous suspension into the pressurized reaction vessel, means for supplying alkaline reagent to the reaction vessel, means for heating the contents of the reaction vessel, means for renewing continuously the interface between the lignocellulosic pulp and the gas, means for controlling the residence time of the lignocellulosic pulp in the reaction vessel and means for withdrawing continuously oxygen treated lignocellulosic pulp from the pressurized reaction vessel.

Optionally means may be provided for controlling the partial pressure of the oxygen in the vessel and for recirculating the gas and/or the aqueous solution through the reaction vessel.

The process of treating lignocellulosic material employing the apparatus of this invention comprises the steps of

1. introducing an aqueous lignocellulosic pulp of 3 percent to 30 percent consistency into a reaction vessel in a continuous stream,
2. admixing with the pulp 1 to 10 percent by weight of alkaline material,
3. contacting the alkaline pulp in the reaction vessel with oxygen or an oxygen-containing gas at a pressure in the range 40–250 p.s.i.g., and 100°C. to 150°C. for a period of 10 to 30 minutes, and
4. withdrawing the oxygen treated pulp continuously from the pressurized reaction vessel, the introduction and withdrawal of the pulp from the reaction vessel taking place continuously while the vessel is maintained under supra atmospheric pressure.

The oxygen-containing gas employed in the process commonly will be air. The use of air requires higher pressures to be used than with oxygen. For example air will be employed at 150 to 250 p.s.i.g. whereas oxygen is effective at pressures of 40 to 150 p.s.i.g. Normally the oxygen consumed will be equivalent to about 1 percent by weight of the dry pulp, but may be greater.

The lignocellulosic material may be unbleached or partially bleached material. For example, it may have undergone a prior treatment with chlorine.

The alkali reagent commonly employed is sodium hydroxide. Sodium carbonate and calcium hydroxide are alternative alkali reagents.

Apparatus suitable for carrying out the invention are illustrated in the accompanying drawings wherein

FIG. 1 is a diagrammatic sectional view of a reactor and associated equipment;

FIG. 2 is a diagrammatic sectional view of an air mixed reaction vessel and associated equipment;

FIG. 3 is a diagrammatic sectional view of a reactor employing perforated trays as a mixing device;

FIG. 4 is a diagrammatic sectional view of a reactor employing a draft tube air lift for mixing the reactants;

FIG. 5 is a diagrammatic plan view of a toroidal form of reaction vessel;

FIG. 6 is a sectional diagrammatic view along line A—A of FIG. 5;

FIG. 7 is a sectional diagrammatic view of an agitator suitable for use in the reaction vessel of FIG. 5;

FIG. 8 is a diagrammatic plan view of another toroidal form of reaction vessel, and

FIG. 9 is a sectional diagrammatic view of another type of reaction vessel.

Referring to FIG. 1 a pulp washer is shown at **1**. Pulp from the washer passes by line **2** to high density pump **3** which pumps it to hopper **4**. Hopper **4** feeds the pulp to rotary pocket valve **5**. Pocket valve **5** introduces the pulp into pressurized reaction vessel **6**. Reaction vessel **6** has three sections. In upper section **7** the pulp is mixed with water introduced from line **8**, sodium hydroxide solution from line **9** and steam from line **10**. The steam serves to maintain the pulp at the desired temperature. Compressed air enters the upper section through line **11**. The alkaline pulp is agitated continuously by propeller mixer **12**. From the upper section of the reactor the pulp passes by downcomer **13** to the central section **14** of the reaction vessel. Here the pulp is agitated continuously by propeller mixer **15**. From the central section the pulp passes by downcomer **16** to lower section **17**. Here the pulp is agitated continuously by propeller mixer **18**. The compressed air employed in the pulp treatment is exhausted from the reaction vessel through valve **19**.

The treated pulp is withdrawn from the pressurized reaction vessel through rotary pocket valve **20** and passes by line **21** through heat exchanger **22** to washer **24**. Heat exchanger **22** serves to preheat water fed to the reaction vessel by pump **23** through line **8**. Wash water from washer **24** passes partly to waste through line **26** and partly by line **27** to dilute incoming pulp in line **21**.

Typical dimensions and conditions for operation of the reactor of FIG. 1 in a 500 ton per day bleach plant when following a first stage chlorination of the pulp are

Total height of vessel	37 feet

Total diameter of vessel	11 feet
Total mean retention of pulp	10 minutes
Air flow	1630 standard cu. ft./min.
Sodium hydroxide	3% based on oven dry pulp
Pulp input consistency	12%
Consistency in vessel	4%
Temperature	130°C.
Pressure	200 p.s.i.g.

If higher consistency pulp is employed, e.g. 7 percent consistency, the dimensions of the reaction vessel can be reduced while still retaining the pulp for an average of 10 minutes. It is possible to use alternative means for introducing the pulp into the pressurized reaction vessel. Instead of the rotary pocket valve a high density pump can be used. Also the rotary pocket valve at the outlet of the vessel can be replaced by other types of valve such as butterfly or gate.

In FIG. **2** the use of an air mixed reaction vessel is illustrated. Pulp from washer **28** passes by line **29** through high density pump **30** to screw press **31.** In screw press **31** the consistency of the pulp is increased, the excess water passing by line **32** to the effluent from washer **28.** The high consistency pulp next passes by line **33** to rotary pocket valve **34** by which it is introduced into pressurized reaction vessel **35.** The contents of reaction vessel **35** are mixed continuously by compressed air circulated by pump **36.** The air used for mixing can of course be replaced by another oxygen-containing gas or by oxygen alone. This air passes by diffusing grid **37** in the bottom of the vessel through the vessel contents and returns to pump **36** by line **38.** In line **38** is fitted oxygen analyzer **39** which monitors the oxygen content of the circulating air and when this reaches a predetermined minimum level operates automatic valve **40** to exhaust air from the system. Fitted to the reaction vessel is pressure gauge **41** which monitors the pressure in the vessel and controls valve **42** from air compressor **43** to replenish air exhausted from the vessel. The contents of the vessel are heated by steam introduced from line **44** through valve **45.** Valve **45** is controlled by temperature controller **46** which monitors the temperature of the contents of the vessel.

The oxygen treated pulp is withdrawn from the reaction vessel through line **47** by which it passes to screw press **48.** Excess liquor is removed from the pulp in the screw press and passes mixed with air through line **49** to separator tank **50.** Here the air is separated and returned to the reaction vessel through line **51.** Part of the liquor in tank **50** passes to effluent by line **52** carrying the liquor through heat exchanger **53** and exhaust valve **54.** Fresh feed water from pump **55** passes through control valve **56,** through heat exchanger **53** and by line **57** joins liquor being returned to the reaction vessel. Valve **56** is controlled by consistency meter **56A.** Valve **54** controlled by flow controller **58,** governs the loss of liquor to effluent. Alternatively a pH controller could be employed to govern the effluent flow. Pump **59** returns liquor from tank **50** to the reaction vessel through valve **60** and line **61.**

Valve **60** is controlled by controller **62** which operates the valve to maintain a predetermined level of liquid in tank **50.** The alkalinity of the recirculated liquor is maintained at the desired level by caustic solution pumped in by pump **63** through valve **64** and line **65** to line **61.** Valve **64** is controlled by pH controller **66** which monitors the pH of the recirculated liquor passing through line **61.** The high consistency pulp issuing

from screw press **48** passes to rotary pocket valve **67** where it leaves the high pressure section of the system. Vlave **67** is controlled by level controller **68** which acts to maintain the level of the contents of the reaction vessel at a predetermined level. The pulp next passes by line **69** to washer **70.** The washings from this washer pass by line **71** partly to effluent and partly are recycled by pump **72** and valve **73** to the washer feed. Valve **73** is controlled by controller **74** which acts to maintain the consistency of the washer feed at the desired level.

In a process utilizing the apparatus of FIG. **2** to bleach 500 tons of pulp per day reaction vessel **35** can have dimensions of **13** feet diameter by **40** feet high permitting a **10** minute retention time. The pulp delivered by washer **28** will have a consistency of 12 percent which will be increased by screw press **31** to 25 percent consistency. The consistency of the pulp in the reaction vessel will be 2 to 6 percent. The caustic concentration will be about 3 percent and the air pressure 200 p.s.i.g. Air recirculation through line **38** will be at 2000 cu.ft./-min. Liquor circulation through line **61** will be at 1700 U.S. gals./min. if consistency of the pulp in the reactor is 4 percent. Steam for heating will be at 200 p.s.i. at the rate of 1100 lbs./hr.

In FIG. **3** is illustrated an alternative form of reaction vessel. This is fitted with a series of perforated trays **75** over which the alkaline pulp suspension flows and through which air is forced. The pulp enters the reaction vessel through line **76** and mixes over the first tray with water, sodium hydroxide and steam entering from line **77.** The alkaline pulp is contacted intimately with air rising through perforated tray **75** and then passes by downcomer **78** to the tray below. In this manner the pulp passes to the bottom of the reaction vessel and is withdrawn through line **79.** Air enters the reaction vessel through line **80** and after passing through the series of perforated trays leaves through line **81.**

In FIG. **4** is illustrated another form of reaction vessel in which a draft tube air lift mechanism is employed to mix the air with the pulp. The pulp enters the reaction vessel through line **82** and flows into upper compartment **83.** Here it is mixed with water, steam and sodium hydroxide introduced through line **84.** Centrally located in compartment **83** is draft tube **85.** Air is introduced into the bottom of tube **85** through air line **86** displacing the pulp upwards in the tube. From compartment **83** the pulp flows by downcomer **87** to central compartment **88** fitted with air lift draft tube **89.** Air is circulated from the upper compartment by line **90** to the bottom of draft tube **89.** The pulp from compartment **88** flows by downcomer **91** to lower compartment **92** fitted with air lift draft tube **93.** Air to operate air lift draft tube **93** is provided by line **94** which circulates air from the central compartment. Air is exhausted from the lower compartment through line **95.** The treated pulp passes from the reaction vessel by line **96.**

In FIG. **5** is shown a toroidal shaped reaction vessel. This vessel is installed horizontally so that the pulp with a pulp-gas interface can travel in a circular path. The pulp enters the reaction vessel by line **97.** Water, steam and sodium hydroxide enter by line **98** and air by line **99.** The vessel is provided with four propeller agitators **100** positioned so as to pump the pulp around the vessel. A segmented baffle **101** is positioned so as to prevent air from flowing freely around the vessel but permitting pulp to pass underneath without restriction. The pulp is withdrawn from the vessel through line **102,** and air through line **103**. The pumping action of the agitators is such that a pulp fibre will on average make more than one rotation of the vessel before passing through outlet **102**. The volume of the reaction vessel is designed to provide a mean residence of **10** minutes for the pulp phase while giving a gas phase volume sufficient to permit easy transfer of air around the vessel. A typical dimension for 500 ton per day using **4%** consistency pulp is a toroidal tube of 8 foot internal diameter and a centre locus of 22 feet diameter.

In FIG. **6** is shown a cross section of the tube of FIG. **5** indicating the boundary **104** between the pulp and gas phases.

FIG. **7** illustrates an alternative form of agitator for the vessel of FIG. **5.** This is a finger type agitator having fingers **105** mounted on rotating shaft **106.**

FIG. **8** illustrates an alternative form of toroidal reaction vessel. Pulp is introduced through line **107.** Water, steam and sodium hydroxide enter the reaction vessel through line **108,** air is introduced by line **109** connected to sparger line **110** located centrally within the vessel. Agitation of the vessel contents is provided by shrouded propeller **111.** The pulp is withdrawn from the reaction vessel through line **112** and air is exhausted through line **113.**

In FIG. **9** is illustrated another alternative design of reaction vessel. The vessel is cylindrical and the direction of flow of pulp is from left to right. Pulp enters through line **114,** water, steam and sodium hydroxide through line **115** and air through line **116.** The vessel contents are agitated by finger type agitator **117.** Pulp is withdrawn from the vessel through line **118**; air is exhausted through line **119.**

The apparatus of this invention provides a means of bleaching lignocellulosic material with oxygen in a continuous process employing an aqueous pulp suspension which being fluid is readily transferred through the apparatus piping.

What we claim is:

1. An apparatus for the continuous treatment of lignocellulosic material with oxygen or an oxygen-containing gas which comprises

1. a reaction vessel adapted to operate under supra atmospheric pressure,
2. means for supplying oxygen or an oxygen-containing gas to the vessel under supra atmospheric pressure,
3. means for introducing continuously aqueous lignocellulosic pulp into the pressurized reaction vessel,
4. means for supplying alkaline reagent to the reaction vessel,
5. means for heating the contents of the reaction vessel,
6. means for mixing the lignocellulosic pulp with the oxygen or oxygen-containing gas so as to renew continuously the interface between the lignocellulosic material of the pulp and the gas,
7. means for controlling the level of the aqueous lignocellulosic pulp in the reaction vessel thus controlling the residence time of the lignocellulosic pulp in the vessel, and
8. means for withdrawing continuously oxygen treated aqueous alkaline lignocellulosic pulp from the pressurized reaction vessel.

2. An apparatus as claimed in claim **1** wherein the reaction vessel is a multi-compartment vessel provided with mechanical agitators in each compartment.

3. An apparatus as claimed in claim **1** wherein the reaction vessel is a single compartment vessel provided with a gas dispersing grid located at the bottom of the vessel, said grid being supplied by gas from the means for supplying oxygen or oxygen-containing gas, said grid serving to mix the contents of the vessel.

4. An apparatus as claimed in claim **3** wherein there is provided means for controlling the partial pressure of the oxygen in the reaction vessel.

5. An apparatus as claimed in claim **3** wherein the means for introducing pulp into the reaction vessel is a rotary pocket valve.

6. An apparatus as claimed in claim **3** wherein the means for withdrawing pulp from the reaction vessel is a rotary pocket valve.

7. An apparatus as claimed in claim **3** wherein the means for heating the contents of the reaction vessel is a source of steam feeding into the vessel.

8. An apparatus as claimed in claim **3** wherein there is provided means for recirculating the oxygen-containing gas through the pulp suspension.

9. An apparatus as claimed in claim **3** wherein there are provided means for separating alkaline aqueous liquor from the lignocellulosic pulp withdrawn from the reaction vessel, and means for recirculating this liquor to the reaction vessel.

10. An apparatus as claimed in claim **1** wherein the reaction vessel is a multi-compartment vessel having a plurality of perforated plate air dispersing members.

11. An apparatus as claimed in claim **1** wherein the reaction vessel is a multi-compartment vessel having draft tube air dispersing members.

12. An apparatus as claimed in claim **1** wherein the reaction vessel is toroidal in shape.

13. An apparatus as claimed in claim **1** wherein the reaction vessel is a single compartment tubular vessel provided with means for mechanical mixing of the vessel contents.

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