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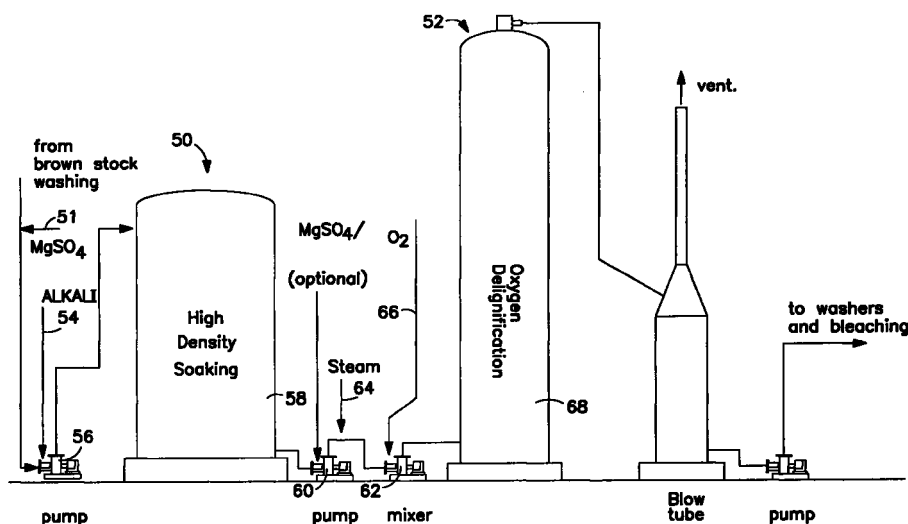
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(54) Oxygen delignification of lignocellulosic material

(57) The invention configures a process for delignifying pulp wherein one stage of a two stage oxygen delignification plant is obviated. In particular, a pulp soak stage enables a pulp stream to be mixed with an alkali feed and held in residence for a sufficient period of time to allow an alkali hydrolysis leaching of colored materials from the pulp and a swelling of the pulp fibers. Next,

the pulp stream is combined with an oxygen feed and is processed through a mixer. The output of the mixer is fed to a pressurized reactor where oxygen reacts with lignin of the pulp fibers. The swelled pulp fibers facilitate the oxygen reaction with lignin and removal of additional colored materials.

**FIG. 3****EP 1 061 173 A1**

DescriptionFIELD OF THE INVENTION

- 5 **[0001]** This invention relates to oxygen delignification of pulp and, more particularly, to an improved oxygen delignification process that requires reduced amounts of capital equipment.

BACKGROUND OF THE INVENTION

- 10 **[0002]** Pulp producers worldwide are striving to reduce water consumption in order to attain a minimum impact mill (MIM). Conceptually, a minimum impact mill is one that generates minimum water and air emissions without negatively affecting wood and energy consumption or product quality. The achievement of a MIM is a slow, stepwise process that requires many modifications and adjustments of current mill practices. These include: (1) minimization of spills; (2) closed water loops in the wood yard; (3) closed screen rooms; (4) efficient brown stock washing; (5) high yield and low energy intensive pulping processes; (6) extended oxygen delignification; and (7) partially closed bleach plants through reuse of some bleaching filtrate streams.

- 15 **[0003]** Lignocellulosic pulp originated from virgin or recycled fiber contains color-causing compounds, which must be removed during the bleaching operation in order to produce a bright and high quality pulp. The removal of such compounds from virgin or recycled pulp fibers is usually done in a single stage but more commonly in a sequence of chemical treatments that may include two or more of the following chemicals: oxygen, ozone, chlorine, hypochlorite, chlorine dioxide, hydrogen peroxide, peracids, chelants, alkali, enzymes, etc. An oxygen treatment is usually applied to the pulp in the beginning of the bleaching process to remove the bulk of the pulp colored materials such as lignin, extractives, dye, pigments, inks, etc.

- 20 **[0004]** Despite some concerns about the impact of oxygen delignification on pulp quality and bleachability with chlorine dioxide, the use of oxygen delignification is spreading worldwide. The most recent trend has been towards the so-called extended oxygen delignification approach. Of these, the most prominent technique is the double stage process. It is purportedly more efficient and selective than the conventional single stage process. Extended oxygen delignification is attractive to the MIM, because it leaves less lignin to be removed in the bleach plant and allows for terminating pulping at a higher kappa number. The kappa number test is used to determine the amount of lignin remaining in pulp after cooking. The kappa number is defined as the number of milliliters of 0.1N potassium permanganate solution consumed by one gram of pulp and corrected for 50% consumption of the potassium permanganate initially added. The higher the kappa number, the more lignin is present in the pulp and vice-versa.

- 25 **[0005]** Since oxygen delignification is more selective than pulping, it is wise to stop the cooking at a higher kappa number and remove as much as possible of the lignin by oxygen delignification. This way, process yield and mill throughput are increased; wood consumption is decreased; causticizing and recovery loads are decreased; and pulp quality is maintained.

- 30 **[0006]** The efficiency and selectivity improvements of a double stage oxygen delignification process in relation to the single-stage process are clear. However, it is difficult to state precisely the quantitative values because they are site specific. Mill experience with eucalyptus kraft pulp of kappa 16-18 has indicated an increase of 5-10% in kappa drop across the process when switching from single to double stage oxygen delignification. This gain has been achieved purportedly without any significant pulp viscosity penalty.

- 35 **[0007]** Ideally, a bleach plant should produce low volume effluent, containing low concentrations of metals, chlorides and organic matter. Thus, the initial stages of the bleaching sequence should generate filtrates, which are easily cycled back to the recovery system, i.e. they should contain low chloride and ideally be of an alkaline nature. This concept gave rise to the so-called elemental chlorine free (ECF) light bleaching processes, which necessarily require some form of extended oxygen delignification.

- 40 **[0008]** Double stage oxygen delignification, as presently practiced, requires high capital investment. Thus, there is a need for extended oxygen delignification which achieves the goal of ECF light bleaching, but at significantly decreased capital cost.

- 45 **[0009]** Double stage oxygen delignification can be practiced at medium consistency in a number of ways. Despite the high capital investment required to install this technology, most are suitable for combination with subsequent ECF light bleaching since they result in delignification rates in the range of 40-50% for hardwoods and 50-60% for softwoods. Among these include: (1) two pressurized stages at high pressure, with and without intermediate washing; (2) two pressurized stages, the first being at high pressure and the second at low pressure, with and without intermediate washing; (3) two pressurized stages, the first being at low pressure and the second at high pressure, with and without intermediate washing; (4) two pressurized stages as in cases 1-3, but with a metals removal step in between, through chelation; (5) two pressurized stages as in cases 1-3 (with intermediate washing) with addition of hydrogen peroxide in the second stage; (6) two pressurized stages as in cases 1-3 with oxidative lignin activation in between; and (7) two pressurized

stages as in cases 1-3 with mild acid hydrolysis in between to remove metals and hexene uronic acids, etc.

[0010] Approaches 1-6 have been commercially implemented and approach 7 is still at the bench scale. All these techniques are described in a paper by Barna, J., Salles, D.V.C, Salvador, E. and Colodette, J.L. (O Papel 58(8):57-66.1997), titled "The Effect of Hydrogen Peroxide Addition in the Second Stage of a Double Stage Oxygen Delignification Process".

[0011] The double stage process is better fitted to oxygen delignification kinetics. It is well known that oxygen delignification reactions occur in two phases. A first phase is rapid and is controlled by diffusion. Most of the reaction occurs rapidly via electrophilic attack of the oxygen and other free radicals on residual lignin structures containing free phenolic units. The second phase is slow and is controlled by chemical reactions of types which include not only electrophilic attack of oxygen and other intermediate species to the lignin, but also nucleophilic attack of peroxides (organic and inorganic), which are produced in the first phase of the reaction. Thus, in the first phase of reaction, there occurs significant delignification and almost no pulp brightening, whereas both delignification and brightening occur in the second phase.

[0012] The percent delignification that occurs in the first and second phases is a debated matter and it seems to depend upon the type of pulp and method of running the oxygen delignification. However, a significant number of researchers believe that the major part of the delignification happens actually in the first phase whereas most of the brightening occurs in the second phase.

[0013] Considering that the process occurs in two phases, it makes sense to design the process so as to take advantage of such reaction kinetics. Thus, the oxygen delignification process is more suitable to the kinetics if performed as a double stage process. Since the first phase or reaction is fast, the first stage of the double stage process can be run for a shorter period of time than the second, and the remaining reaction takes place in the second stage reactor which is run for a longer period of time.

[0014] Fig. 1 shows a schematic of a prior art double stage oxygen delignification process, without inter-stage washing. There it is seen that two high shear mixers 10 and 12 are required, one prior to first reactor 14; and the second prior to second reactor 16. Oxygen and oxidized white liquor (OWL), a highly alkaline liquid, and/or caustic are added prior to first reactor 14; but provisions can be made to add these chemicals between reactors 14 and 16, if required. A washing step between the two reactors is optional and is usually not required since its beneficial effects are questionable.

[0015] A layout of the system containing intermediate washing stage 18 is shown in Fig. 2. However, since it is eventually needed to add hydrogen peroxide in a second reactor 16, the presence of intermediate washing stage 18 is desirable to avoid peroxide losses in reactions with partially oxidized organic carryover.

[0016] It has been shown that the selectivity of the first oxygen delignification phase is not very sensitive to the alkali charge whereas the efficiency is. On the other hand, the selectivity is sensitive to the alkali charge in the second phase whereas the efficiency is not. Taking into account these facts, it is preferred that the first oxygen reactor 14 be operated at high alkalinity to maximize efficiency. On the other hand, the second reactor 16 should operate at low alkalinity to maintain process selectivity. It has also been shown that selectivity is negatively affected by oxygen pressure in the first phase of the reaction, whereas efficiency is affected positively. On the other hand, both efficiency and selectivity are positively affected by oxygen pressure in the second phase. Thus, first oxygen reactor 14 must operate at low oxygen pressure and second oxygen reactor 16 at high oxygen pressure in order to maintain process selectivity. In other words, the alkali charge must be kept high in first reactor 14 and low in second reactor 16, and the opposite is valid for the oxygen charge (partial pressure). Thus it makes sense to add the total amount of alkali to the pulp in first reactor 14 and use only the residual alkali in second reactor 16. The oxygen, on the other hand, should be split between the two reactors, with the larger fraction being applied in second reactor 16.

[0017] The function of mixer 12 located between the two stages is the mixing of the oxygen added in the second stage and re-mixing the residual oxygen bubbles that eventually coalesced in the first reactor. Since the alkali readily mixes with the pulp, there is no need to re-mix it.

[0018] It has been also shown that the selectivity is negatively affected by temperature in the first phase of the reaction, whereas efficiency is affected positively. On the other hand, temperature positively affects efficiency and has no effect on selectivity in the second phase. It would thus be preferred to run the process such that the temperature in the first reactor is lower than in the second reactor.

[0019] In the process of the prior art, the desired rates of delignification are achieved using forms of the double stage oxygen delignification process depicted in Figs. 1 and 2. As compared to conventional, single stage oxygen delignification, the double stage processes are more expensive to install since they require heavy equipment. In the case of Fig. 1, an additional reactor and mixer are required. In the case of Fig. 2, an additional reactor, mixer and washer are required. Thus, the benefit of having a double stage oxygen delignification is hampered by the substantial capital investment required for its installation.

SUMMARY OF THE INVENTION

[0020] The invention configures a process for delignifying pulp wherein one stage of a two stage oxygen delignification plant is obviated. In particular, a pulp soak stage enables a pulp stream to be mixed with an alkali feed and held in residence for a sufficient period of time to allow an alkali hydrolysis leaching of colored materials from the pulp and a swelling of the pulp fibers. Next, the pulp stream is combined with an oxygen feed and is processed through a mixer. The output of the mixer is fed to a pressurized reactor where oxygen reacts with lignin of the pulp fibers. The swelled pulp fibers facilitate the oxygen reaction with lignin and removal of additional colored materials.

[0021] In a first preferred mode of running the process, no inter-stage washing step is required. In a second embodiment, a washing step is introduced between the soaking and the oxygen reaction stage to increase process efficiency, but at added capital cost.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022]

Fig. 1 is a schematic of a prior art double stage oxygen delignification plant without inter-stage washing of the pulp stream.

Fig. 2 is a schematic of a prior art double stage oxygen delignification plant with inter-stage washing of the pulp stream.

Fig. 3 is a schematic of an oxygen delignification plant that incorporates the invention, without inter-stage washing of the pulp stream.

Fig. 4 is a schematic of an oxygen delignification plant that incorporates the invention, with inter-stage washing of the pulp stream.

Fig. 5 is a schematic of an oxygen delignification plant that incorporates the invention, with inter-stage press washing of the pulp stream.

Fig. 6 is a schematic of another embodiment of an oxygen delignification plant that incorporates the invention.

Fig. 7 is a schematic of still another embodiment of an oxygen delignification plant that incorporates the invention.

DETAILED DESCRIPTION

[0023] The present invention is directed towards an improvement in the process of removing colored compounds from lignocellulosic material using alkali and oxygen. In this process, the pulp is first treated with alkali and then further treated with oxygen. A key feature of the present invention is the treatment of the pulp with alkali, prior to oxygen delignification. The alkali soaking of the pulp fibers results in an alkali hydrolysis/leaching of easily accessible colored materials and also in the swelling of the pulp fibers, facilitating removal of the colored materials in the subsequent oxygen delignification stage. As a result, a 5-10% improvement in the rate of removal of colored materials is achieved as compared to conventional oxygen delignification processes. The invention allows for an achievement of the same rate of delignification as the prior art double stage oxygen delignification process (which requires much more capital investment).

[0024] Further, the process of the invention is particularly efficient to delignify lignocellulosic material containing lignin contents higher than usual. For example, it is preferred that pulps having the following kappa numbers be subjected to the process of the present invention, that is: 30-40 for softwood kraft pulps, 17-25 for hardwood kraft pulps and 15-30 for recycled fibers. This specification expresses all compositions in weight percent, unless specifically expressed otherwise.

[0025] The present invention comprises the treatment of the lignocellulosic pulp with alkali in a vessel at atmospheric pressure. The treatment, hereafter called pulp soaking, is carried out at a pH in the range about 9-14, advantageously about 11-12. Preferred conditions for the pulp soaking are as follows: temperature 70-90°C, consistency 10-12%, retention time 0.5 to 1 hour and alkali dosage of 1-2% based on pulp dry weight. The alkali dosage may vary substantially, depending upon the type of lignocellulosic material being treated. The preferred way to control the pulp soaking is through monitoring of the pH of the slurry rather than the alkali dosage. The lignin removal during the soaking, as measured by kappa number, is a function of the pulp's initial lignin content. The higher the initial pulp kappa number, the more efficient is the lignin removal during the soaking process, particularly when the pulp is washed or pressed after soaking.

[0026] Following the alkali soak, the pulp may go directly to a subsequent oxygen delignification stage or be washed in conventional washers or thickened to a consistency of about 30-35% in a wash press.

[0027] The soaked pulp is then treated with oxygen in a medium consistency oxygen delignification stage, hereafter designated as the O-stage. This O-stage operates at 10-12% consistency, 11-12 pH, 60-95°C, 60-90 min reaction time,

400-600 kPa pressure and at a 1.2-2.5% oxygen dose. A magnesium salt, magnesium sulfate or the like, at a dosage of 0.01-0.1 wt% Mg (based on dry pulp) and most advantageously about 0.02-0.03 of Mg, may be added to protect the pulp carbohydrates against alkali-induced degradation.

[0028] The magnesium salt should be added before the alkali addition in order to facilitate mixing of the salt with the pulp. Note that the dosage of alkali and of a magnesium salt will depend on whether or not there is a washing or pressing step in between pulp soaking and the O-stage.

[0029] The process of the invention can be more clearly understood by reference to Figs. 3-7. Fig. 3 depicts the process of the invention wherein washing between pulp soaking and the O-stage is omitted. The process comprises two separate stages, a pulp soak stage 50 and an O-stage 52, with no washing or pressing between them. The pulp incoming from a brown stock washing operation receives both MgSO_4 via feed 51, and alkali (e.g., NaOH with oxidized white liquor or unoxidized white liquor) from a source 54 through pump 56. The pulp/alkali slurry then goes into a high density soak vessel 58 at atmospheric pressure, where it remains for a desired residence time.

[0030] The pulp slurry is then sent to O-stage 52 where additional alkali and a magnesium salt may be added (if necessary) in the suction of a pump 60. Thereafter, the slurry goes to a high shear mixer 62 where medium pressure steam and oxygen are added via feeds 64 and 66, respectively, to the pulp slurry. The pulp slurry is then pumped to pressurized reactor 68 where the slurry is maintained for a desired reaction time. After the reaction completes, the pulp is discharged into a blow tube and is pumped to post O-stage washers (not shown).

[0031] Fig. 4 shows a second embodiment of the invention wherein the pulp slurry is washed between pulp soak stage 50 and O-stage 52. In this case, after the soaking, the pulp slurry is pumped by pump 70 to a washer 72 and is then conveyed to conventional O-stage 52, as previously described. Fig. 5 shows a third embodiment wherein the pulp, after soaking in soak stage 50, is pumped to a wash press 80 instead of a regular washer. In this case, the slurry is dewatered to a consistency between 35-40% and is then conveyed to a standpipe 82 where it is diluted to a consistency between 10-14% with filtrate from the subsequent O-stage. At standpipe, the pulp receives additional alkali and a magnesium salt via feed 84, and then goes to O-stage 52 for processing as above described.

[0032] Further embodiments of the invention are shown in Figs. 6 and 7. In Fig. 6, a pulp processing sequence is shown that comprises two separate stages, an alkaline soak stage 100 first and then an O-stage 102, with no washing or pressing between them. The embodiment shown in Fig. 7 is similar except for the fact that pulp is washed or pressed between the alkaline soak and oxygen delignification stages. The major difference between these processes, as compared to those described above with respect to Figs. 3-5 is that the oxygen treatment is effected at a lower reaction pressure (hydrostatic pressure), i.e., a mini-O. This stage is carried out at 10-12% consistency, 11-12 pH, 70-80°C, 60-120 min reaction time, 150-300 kPa pressure (overpressure or head pressure), 0.5-1.05% oxygen dose. A magnesium salt, magnesium sulfate or the like (dosage of 0.02-0.03% Mg) may be added to protect the pulp carbohydrates against alkali induced degradation. The magnesium salt must be added before the alkali addition in order to facilitate mixing of the salt with the pulp. Note that the dosage of alkali and of a magnesium salt depends on whether or not there is a washing or pressing step between the pulp soaking and the O-stage.

[0033] In the case where the pulp is not washed (i.e., Fig. 6), the alkali requirement is minimum, only about 10-20% of the total amount required. This additional alkali is necessary to replenish the fraction consumed during the soaking stage. On the other hand, if the pulp is pressed or washed after soaking (i.e., Fig. 7), additional alkali is required. An alkali dose of 1-1.5% should be added. Note that the alkali requirement varies substantially depending upon the type of lignocellulosic material.

[0034] After soaking, the pulp slurry is conveyed directly to the O-stage where additional alkali and a magnesium salt are added in the suction of the pump; following the slurry goes to a high shear or static mixer where medium pressure steam and oxygen are added to the pulp. The pulp is then pumped to a preretention tube 104 followed by a down-flow tower 106 (or directly to an up-flow tower) where it is maintained for the desired reaction time. Preretention tube 104 is usually pressurized up to 200 kPa and down-flow tower 106 operates atmospherically. The up-flow tower operates atmospherically but the head pressure of the column; the pressure in the bottom of the tower where the oxygen is injected depends upon the tower height. After the reaction completion, the pulp is pumped to post stage washers (Fig. 6). Fig. 7 illustrates the presence of an inter-stage washing system.

[0035] The major advantages of the techniques described in regards to Figs. 3-7 are: (1) they are less capital intensive than double stage processes since they require one less pulp washer, high shear mixer and pressurized reactor, while maintaining pulp quality and process efficiency; (2) they are easily retrofitted to single stage oxygen delignification installations without major investment, with the advantage of enhancing delignification performance by 5-10%; and (3) they can also be applied to mini-O processes (low pressure oxygen delignification) enhancing rates also by 5-10% with minimal capital investment.

[0036] Thus, the difference between the process of the invention in relation to the prior art is a reduction in overall capital cost investment when compared to double stage oxygen delignification processes. Also, they can enhance delignification by 5-10% when applied in connection with both conventional single stage and mini-O oxygen delignification processes.

[0037] In summary, the invention treats lignocellulosic pulp with alkali at atmospheric conditions followed by a high or low-pressure exposure of the pulp to oxygen in pressurized vessels. The invention is applicable to all kinds of fibrous raw material including pulps manufactured by processes such as kraft, soda, sulfite, magnesite, cold soda, NSSC and the like. These fibers may be obtained from hardwood, softwood, bamboo, bagasse, straw and other non-wood fiber supplies. The process is also applied to de-inked recycled fibers and certain grades of brown recycled fibers.

[0038] Below are presented a range of alternative ways and conditions in which the various stages of the process of this invention can be practiced and also the preferred way of practicing them.

[0039] The pulp alkali treatment can be performed in atmospheric and pressurized vessels, preferably in atmospheric ones. The alkali used can be of several origins including plain NaOH, oxidized white liquor (OWL), unoxidized white liquor (WL) or the like. Most advantageously, the reaction occurs with 0.5-5 weight percent NaOH. The vessel may be a dedicated one or the high-density tower already existing in most pulp mills. The treatment called of pulp soaking (S) is carried out with a pulp consistency of about 6-14 weight percent. A pH of at least 11 facilitates the alkali soaking. Most advantageously, the soak occurs at a pH between about 11 and 12. Elevating the soaking temperature to between 40 and 95°C accelerates swelling of the lignocellulosic pulp. Most advantageously, the soaking occurs at a temperature between 70 and 95°C to accelerate the reaction. A soaking time of 15 to 240 minutes advantageously swells the fibers to facilitate the reaction between the oxygen and the lignin. Most advantageously, a soaking time between about 20 or 30 minutes and 60 minutes accomplishes the soaking. Advantageously the alkali dose consists about 0.5-5% NaOH, most advantageously, about 1-2% NaOH. The alkali dosage however may vary substantially, depending upon the type of lignocellulosic material being used.

[0040] Following the alkali soaking, the pulp may go directly to a subsequent oxygen treatment or be washed in conventional washers or thickened to a consistency of about 30-35% in a wash press. The equipment required to wash or thicken the pulp is standard and available in the market. This operation affects the overall direct and indirect costs of this invention. When the pulp is washed after soaking, provisions can be made for additional pulp treatments such as chelation to remove metals and mild acid hydrolysis to remove hexene uronic acids and metals prior to the subsequent oxygen treatment.

[0041] The soaked pulp is then treated with oxygen three different ways: (1) at low pressure (mini-O), in a process hereafter designated as (EO); (2) in a conventional MC (medium consistency) single stage oxygen delignification process, hereafter designated as O; and (3) in a MC double stage oxygen process, hereafter designated as O/O or OO, depending on whether the pulp is washed between stages or not. The oxygen used in this treatment may be of purity varying from 80-100%, preferably 90-100%.

[0042] The low pressure oxygen treatment or mini-O (EO) may be carried out at a consistency of from 8-14 weight percent, preferably from 10-12 weight percent. Preferred conditions for other parameters are: pH of from 10-14, preferably 11-12, alkali dose of 1-4% preferably 1-2%, temperature of 50-120°C, preferably 70-90°C, reaction time of from 30-180 min, preferably 60-90 min, reaction pressure of 100-600 kPa, preferably 150-300 kPa and oxygen dose of 0.2-2%, preferably 0.5-1%. At this stage a magnesium salt, magnesium sulfate or the like, may be added to protect the pulp carbohydrates against alkali induced degradation. The magnesium salt can be added before or together with the alkali, but preferably before the alkali in order to facilitate mixing of the salt with the pulp. The dose of the magnesium salt may be in the range of about 0.01-0.1% (as Mg) based on the pulp dry weight, preferably about 0.02-0.03%. Hydrogen peroxide may also be added in the (EO) stage to boost delignification in the dose of 0.2-4%, preferably 0.5-1%. The addition of peroxide is feasible only when the pulp is washed after soaking.

[0043] The conventional single stage oxygen treatment, O, may be carried out at a consistency of from 8-14%, preferably from 10-12%. Preferred conditions for other parameters are: pH of from 10-14, preferably 11-12, alkali dose of 1-4% preferably 1-2%, temperature of 50-140°C, preferably 80-100°C, reaction time of from 30-180 min, preferably 60-90 min, reaction pressure of 100-800 kPa, preferably 400-600 kPa and oxygen dose of 0.5-4%, preferably 1-2%. At this stage a magnesium salt, magnesium sulfate or the like, may be added to protect the pulp carbohydrates against alkali induced degradation. The magnesium salt can be added before or together with the alkali but preferably before the alkali in order to facilitate mixing of the salt with the pulp. The dose of the magnesium salt may be in the range of 0.01-0.1% (as Mg) based on the pulp dry weight, preferably 0.02-0.03%. Hydrogen peroxide may also be added in the (O) stage to boost delignification in the dose of 0.2-4%, preferably 0.5-1%; the addition of peroxide is feasible only when the pulp is washed after soaking.

[0044] The double stage oxygen treatment, O/O or OO, may be carried out at a consistency of from 8-14%, preferably from 10-12%. Preferred conditions for other parameters are: pH of from 10-14, preferably 11-12, alkali dose of 1-4% preferably (1.5/0.5%), temperature of 50-140°C, preferably (85/95)°C, reaction time of from 30-180 min, preferably (30/60) min, reaction pressure of 100-800 kPa, preferably 400-600 kPa and oxygen dose of 0.5-4%, preferably (1.5/0.5%). At this stage a magnesium salt, magnesium sulfate or the like, may be added to protect the pulp carbohydrates against alkali induced degradation. The magnesium salt can be added before or together with the alkali but preferably before the alkali in order to facilitate mixing of the salt with the pulp. The dose of the magnesium salt may be in the range of 0.01-0.1% (as Mg) based on the pulp dry weight, preferably (0.02/0.0%). Hydrogen peroxide may also be

added in the double stage oxygen treatment to boost delignification in the dose of 0.2-4%, preferably (0.0/0.5%); the addition of peroxide is feasible only when the pulp is washed after soaking.

[0045] For purposes of this specification, the "/" divides the first stage addition or condition from the second stage addition or condition. For example, (0.03/0% Mg) indicates a 0.03% Mg addition to the first stage and a 0% Mg addition to the second stage.

EXPERIMENTAL

[0046] For the confirmation of the present invention and a best understanding thereof, different types of lignocellulosic material were subjected to the steps described above. The comparisons between the process of the present invention and those of the prior art are based on results of kappa drop, viscosity drop and brightness gain across the various processes. The kappa drop, viscosity drop and brightness gains were calculated with the following equations: $\text{kappa drop} = (\text{kappa in} - \text{kappa out}) / \text{kappa in}$; $\text{viscosity drop} = (\text{viscosity in} - \text{viscosity out}) / \text{viscosity in}$; $\text{brightness gain} = (\text{brightness in} - \text{brightness out})$. The values of kappa number, viscosity, and brightness were measured according to the Technical Association of the Pulp and Paper Industry (Tappi) standard procedures. All experiments described were carried with two repetitions, being the results presented average values.

[0047] Except when specified, the procedures and operating conditions used for the various processes discussed are the ones described below:

[0048] Pulp Alkali Soaking (S): was effected at 12% consistency, 85°C, 30 min, with 1.5% alkali. The reaction was carried out in a high shear mixer/reactor made of hasteloy having temperature and pressure controllers and devices for injection and relief of gases. The mixing was done intermittently every 1-min at 2000 rpm for 4 seconds. Variations in alkali dose, temperature and reaction time were practiced but they are described at the proper examples.

[0049] Single O-stage (O): was carried out at 10% consistency, 95°C, 60 min, 600 kPa pressure with 1.5% oxygen, 1.5% NaOH and 0.03% magnesium as such. The reaction was carried out in the same equipment and settings above described in the item alkali soaking.

[0050] Double O-stage, (O/O and OO): The first O-stage was carried out at 10% consistency, 85°C, 60 min, 600 kPa pressure with 1.5% oxygen, 1.5% NaOH and 0.03% magnesium as such. The second stage was carried out 10% consistency, 95°C, 60 min, 600 kPa, 1.5% NaOH and 0.5% oxygen. When washing was carried out between the two stages, this was done through press washing as described below. Double O-stage without intermediate washing is designated as O/O-stage, and with intermediate washing as OO-stage. The reaction was carried out in the same reactor/mixer and settings above described in the item alkali soaking.

[0051] Low Pressure or mini-O-stage, (EO): This stage was carried out at 10% consistency, 85°C, 60 min, 200 kPa pressure with 1.0% oxygen, 1.5% NaOH and 0.03% magnesium as such. The pressure was dropped from 200 kPa to zero pressure during the 60-min reaction, simulating a hydrostatic tower. The reaction was carried out in the same reactor/mixer and settings above described in the item alkali soaking, except that the pressure was dropped manually from 200 kPa to zero at 5-min intervals.

[0052] Press Washing: Press washing between stages was effected by diluting the pulp after the stage to a consistency of 4% and then pressing it to a consistency of about 35%. This corresponds to a washing efficiency of about 80%, considering for example the pulp entering the washing stage at 10% consistency. Note that washing between stages has no representation whereas no washing is usually represented by a slash symbol (/).

[0053] The following examples are provided to illustrate the present invention:

Example 1: Optimization of Soaking Time and Temperature

[0054] The kraft pulp sample employed in this example was obtained in the laboratory from eucalyptus wood. After pulping, the brown pulp had a initial kappa number of 19.6, a viscosity of 58.7 mPa.s and a brightness of 28.9% ISO. The soaking was carried out at, 65, 75 and 85°C for periods of time of 15, 30, 60 and 180 min. Other conditions were maintained constant as described in previous sections. After soaking, the pulp was thoroughly washed and then analyzed for kappa number, viscosity and brightness. The results shown in Table 1 indicate that soaking efficiency as measured by kappa drop is positively influenced by both time and temperature. However, the benefits of the soaking somewhat decrease after 30-min reaction, particularly at the 85°C temperature. Increasing the time from 30 min to 240 min resulted in only a 1% increase in the kappa drop. Thus, the time of 30 min was considered sufficient at the 85°C temperature. Since the temperature had no significant impact on pulp viscosity, the 85°C value was considered the most adequate, since practically, the subsequent O-stage is usually carried out at temperatures equal or above this value. The brightness gain across the soaking stage was very low and did not follow the kappa drop. Likely, the pulp exposure to alkali at warm temperatures triggered lignin darkening reactions that overshadowed the expected brightness gains derived from partial lignin removal.

Table 1

Effect of the temperature and reaction time on overall performance of pulp soaking with alkali*				
Soaking Time, min	Soaking Temp., °C	Results		
		Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
15	55	3.3	1.7	0.1
	70	4.2	1.7	0.0
	85	5.3	2.2	0.2
30	55	5.5	2.0	0.4
	70	7.4	2.3	0.7
	85	8.9	2.4	0.8
60	55	5.9	3.2	0.8
	70	8.5	3.3	0.7
	85	9.3	3.8	0.9
240	55	6.5	3.8	1.0
	70	9.1	4.1	1.2
	85	9.9	4.5	1.2

*soaking: 12% consistency with 1.5% alkali; 12±0.2 initial pH (experimental).

Example 2: optimization of the soaking pH

[0055] The same kraft pulp sample employed in Example 1 was soaked with alkali doses of 0.5, 1.0, 1.5, 2.5 and 4% NaOH. Other soaking conditions were maintained constant as described above. After soaking, the pulp was thoroughly washed with distilled water and the values of kappa number, viscosity and brightness measured. The results in Table 2 denote that increasing soaking pH above 12 produces only slight benefits in terms of kappa drop but penalizes somewhat pulp viscosity. Thus the pH 12, which for this pulp sample is equivalent to an alkali charge of 1.5%, was considered to be the most satisfactory.

Table 2

Effect of the pH on overall performance of pulp soaking with alkali*				
Alkali Dose, %	Soaking pH	RESULTS		
		Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
0.5	11.2	1.3	0.7	0.2
1.0	11.5	4.8	1.4	0.3
1.5	12.0	8.9	2.4	0.8
2.5	12.7	9.3	4.8	1.3
4.0	13.4	9.9	7.7	0.3

*soaking: 2% consistency, 85°C and 30 min.

Example 3: effect of the pulp degree of delignification

[0056] The various hardwood kraft pulp samples employed in this Example were obtained from eucalyptus wood. Pulping conditions were adjusted in order to produce low, medium and high degree of delignification pulps, for hardwood standards. After pulping, the sample of low degree of delignification had 14.3 kappa, 35.7 mPa.s viscosity and

34.9% ISO brightness; the medium degree of delignification sample had 16.8 kappa, 47.5 mPa.s viscosity and 32.3% ISO brightness; the high degree of delignification sample had 19.6 kappa, 58.7 mPa.s viscosity and 28.9% ISO brightness. The soaking was carried out under the conditions previously described. After soaking, the pulp was thoroughly washed with distilled water and the values of kappa, viscosity and brightness measured.

[0057] The results shown in Table 3 reveal that the soaking is more effective to reduce kappa number when applied to the pulp of higher initial kappa number. This may be explained by the higher content of lignin potentially hydrolyzable/leachable with alkali in the higher kappa pulp. The effects on pulp viscosity and brightness gain were negligible and apparently independent of pulp initial kappa number.

Table 3

Effect of pulp degree of delignification on overall performance of pulp soaking with alkali			
	Results		
Pulp Initial Kappa Number	Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
14.3	5.9	2.2	0.6
16.8	7.8	2.5	0.7
19.6	8.9	2.4	0.8

*soaking: 12% consistency, 85°C, 30 min, 1.5% NaOH, 12±0.15 initial pH.

Example 4: effect of the type of lignocellulosic material

[0058] Four types of lignocellulosic material were compared. An industrial hardwood kraft pulp (HWD) of 17.1 kappa, 43.4 mPa.s viscosity and 36.8% ISO brightness; an industrial softwood kraft pulp of 32.2 kappa, 42.7 mPa.s viscosity and 26.4% ISO brightness (SWD); an industrial recycled fiber sample produced from de-inked low grade mixed office waste (MOW) of 14.4 kappa, 11.9 mPa.s viscosity and 55.1% ISO brightness; an industrial recycled fiber sample produced from deinked curbside material (RCM) of 69.8 kappa and 42.5% ISO brightness. The soaking was carried out at fixed conditions as described above. After soaking, the pulp was thoroughly washed with distilled water and the values of kappa number, viscosity and brightness measured. The results showed in Table 4 point out that the performance of the alkali soaking operation depends substantially on the type of lignocellulosic material. The highest kappa drop was achieved with the hardwood kraft pulp indicating that this material contains the largest quantities of alkali promoted lignin leachable/hydrolysable. Additionally, the viscosity of the hardwood pulp was not substantially changed during the soaking treatment. In spite of their higher lignin content both the softwood kraft and the RCM pulps were not very susceptible to the soaking treatment. These pulps resulted in lower kappa drops across the soaking as compared to the HWD sample. Besides, the SWD experienced larger viscosity loss than its HWD counterpart. Both the RCM and MOW samples lost brightness across the soaking indicating the occurrence of alkali promoted darkening reactions.

[0059] In the case of the MOW sample, there was very little effect of the soaking on kappa drop; it is possible that the colored materials present in the MOW sample are mostly of a non-lignin nature and resistant to alkali hydrolysis/leaching.

Table 4

Effect of the type of lignocellulosic material on overall performance of pulp soaking with alkali*				
		Results		
Pulp Type	Initial Kappa	Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
HWD kraft	17.1	7.5	2.2	0.9
SWD kraft	32.2	6.7	5.9	0.4
MOW recycled	14.4	3.4	1.2	-1.3
RCM recycled	69.8	5.7	-	-8.7

soaking: 12% consistency, 85°C, 30 min, 1.5% NaOH, 12±0.35 initial pH.

[0060] The following examples show the effects of the pulp alkali soaking (S) on the overall performance of several types of subsequent oxygen bleaching stages, with and without intermediate washing.

Example 5: Effect of pulp soaking on overall performance of various types of oxygen delignification of a hardwood kraft pulp

[0061] The hardwood kraft pulp sample employed in this example was the same described in Example 1. The soaking was carried out at fixed conditions as described in the above sections. The subsequent oxygen delignification treatments were carried out as described above, under the following conditions: O: 10% consistency, 95°C, 60 min, 600 kPa overpressure, 1.5% NaOH, 1.5% O₂, 0.03% Mg; (EO): 10% consistency, 85°C, 60 min, 200 kPa pressure, 1.5% NaOH, 0.8% O₂, (0.03 Mg); O/O: 10% consistency, (85/95°C), (30/60 min), 600 kPa pressure, (1.5/0% NaOH), (1.5/0.5% O₂), (0.03/0% Mg); OO: 10% consistency, (85/95°C), (30/60 min), 600 kPa pressure, (1.5/1.0% NaOH), (1.5/0.5% O₂) and (0.02/0.02% Mg).

[0062] The results in Table 5 indicate that the amount of lignin removal caused by the soaking is not additive to that obtained in the subsequent oxygen treatment, regardless of the oxygen application mode under consideration. However, more than half of the soaking benefit is transferred to the subsequent oxygen delignification stages. For example, the soaking by itself resulted in 8.9% kappa drop and the conventional O-stage in 37.5%. If the benefits were additive, a 46.4% delignification would be expected after the S/O treatment. Instead, a value of 43.7% was obtained experimentally. This very same trend was also seen for the S/(EO), S/(OO), S/O/O and S/OO treatments. This difference was initially attributed to the absence of a washing step between the soaking and the various oxygen treatments. However, the insertion of the washing step between these treatments, still was not enough to make additive the benefits of the treatments. Likely, the soaking treatment removes lignin fractions that would otherwise be removed in the subsequent oxygen treatments. Although additive benefits are not to be expected, there is still an advantage of applying the soaking since overall delignification can still be increased by up to 7.4% with this technique.

[0063] The soaking plus a single O-stage, S/O and SO, resulted in delignification rates similar to those obtained with double stage oxygen delignification, O/O and OO, without any significant penalty on pulp viscosity and brightness gain. It is worth noting that soaking plus single O-stage require much less capital cost to install than a double stage oxygen delignification process.

[0064] Another interesting aspect shown in Table 5 is with regard to the soaking plus low pressure (mini-O) oxygen delignification, S/(EO) and S(EO) processes. The soaking treatment adds an extra 5-7% kappa reduction to a (EO) stage. This benefit is rather significant given the fact that the EO (oxygen extraction) operation is designed to achieve lower delignification levels than conventional O-stages. A 5% improvement in the (EO)-stage, which gives 20-25% delignification rate, is more significant than a 8% improvement in the O-stage that gives 35-40% delignification rate. Note that this benefit is obtained with no penalty to pulp quality and with very low capital investment.

Table 5

Effect of the soaking* on subsequent oxygen delignification performance for HWD kraft pulp			
Treatment Type	Results		
	Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
S	8.9	2.4	0.8
O	37.5	35.4	12.8
(EO)	23.4	28.3	7.9
O/O	43.8	39.3	13.7
OO	44.6	37.9	14.1
S/O	43.7	41.8	13.4
S/(EO)	29.9	33.4	8.1
S/O/O	44.7	42.5	13.8
SO	44.9	41.2	14.3
S(EO)	31.0	32.1	9.0
S/OO	45.8	42.1	14.9

*soaking: 12% consistency, 85°C, 30 min, 1.5% NaOH, 12±0.05 initial pH.

Example 6: Effect of pulp soaking on overall performance of various types of oxygen delignification of a softwood kraft pulp

[0065] The softwood kraft pulp sample employed in this example was obtained from a Western North American pulp mill and was made from Spruce. After pulping, the brown pulp had a 32.2 kappa number, a 42.7 mPa.s viscosity and a 26.4% ISO brightness. The soaking was carried at fixed conditions as described in previous sections. The subsequent oxygen delignification treatments were carried out under the following conditions: O: 10% consistency, 95°C, 60 min, 600 kPa pressure, 2.0% NaOH, 2.0% O₂, 0.03% Mg; (EO): 10% consistency, 85°C, 60 min, 200 kPa pressure, 2.0% NaOH, 1.0% O₂, 0.03% Mg; O/O: 10% consistency, (85/95°C), (30/60 min), 600 kPa pressure, (2.0/0% NaOH), (1.5/0.5% O₂), (0.03/0% Mg); OO: 10% consistency, (85/95°C), (30/60 min), 600 kPa pressure, (1.5/1.0% NaOH), (1.5/0.5% O₂) and (0.02/0.02% Mg).

[0066] The results obtained in the various oxygen delignification treatments for the softwood kraft pulp sample followed the same trends observed for the hardwood treatments. However, the soaking benefits were less pronounced for the softwood sample as illustrated in Table 6. The benefits caused by the soaking were particularly slim when done prior to a low pressure oxygen stage, S/(EO) and S(EO) processes. Although the benefits of the soaking were lower for the softwood sample as compared to the hardwood sample, the performance of the oxygen delignification stages were much higher for the softwood sample. This fact is, however, quite well documented in the literature and in mill scale operations worldwide.

Table 6

Effect of the soaking* on subsequent oxygen delignification performance for SWD kraft pulp			
Treatment Type	Results		
	Kappa Drop, %	Viscosity Drop, %	Brightness Gain, %
S	6.7	5.9	0.4
O	46.5	37.9	5.8
(EO)	26.9	23.7	2.5
O/O	51.4	40.1	6.5
OO	51.7	38.8	7.4
S/O	51.3	41.1	6.3
S/(EO)	29.1	24.2	2.9
S/O/O	52.7	43.7	6.9
SO	52.5	40.8	6.4
S(EO)	31.2	23.4	3.1
S/OO	53.6	43.5	6.9

*soaking: 12% consistency, 85°C, 30 min, 1.5% NaOH, 12±0.35 initial pH.

Example 7. Impact of the soaking on overall pulp bleachability with the sequence D(EOP)D

[0067] The oxygen treated hardwood and softwood kraft pulp samples described in Examples 5 and 6, respectively, were further bleached by an ECF (elemental chlorine free) bleaching process with the sequence D(EOP)D. The brightness target was 90% ISO for the HWD sample and 89% for the SWD one. Following the recommendation of the Technical Association of Pulp and Paper Industry (Tappi), as detailed in the Tappi publication TIS 0606-21 entitled "recommended pulp bleaching stage designation method", the D(EOP)D designation represents a sequence which comprises three separate stages, the first D-stage, an (EOP) stage and then another D-stage, with a washing or pressing step between these stages. In the (EOP) stage, alkali, oxygen and hydrogen peroxide are injected in the same stage, apart from each other by fractions of minutes. The conditions used in the various bleaching stages were as follows: first D-stage: 10% consistency, 75°C, 60 min, 3.0 final pH and a kappa factor of 0.24; (EOP) : 10% consistency, 85°C, (15/75) min, 200 kPa pressure, 10.5 final pH, 1.4% NaOH, 0.5% O₂, 0.5% H₂O₂, 0.03% Mg; second D-stage: 10% consistency, 75°C, 240 min, 3.8 final pH and variable amounts of ClO₂ depending upon pulp previous treatment and type. The control of pH in the first and second D-stages was achieved through small additions of NaOH or H₂SO₄.

in the stages as required.

[0068] The results presented in Table 7 for the hardwood pulp sample point out that the combination of soaking plus conventional oxygen delignification, S/O or SO processes, results in chlorine dioxide savings (shaded column) of the same magnitude as the double stage oxygen delignification, O/O or OO processes. The ClO₂ savings are calculated by the difference between a certain treatment and the reference. If compared with the single O-stage (O), the S/O and SO processes saved 1.1-1.3 kg of ClO₂/odt of pulp. On the other hand, when compared to the low pressure oxygen delignification, (EO), the S(EO) and S(EO) processes also resulted in chlorine dioxide savings in the order of 1.2-1.4 kg ClO₂/odt of pulp. For the softwood pulp sample (Table 7), similar trends were observed, but absolute values of chlorine dioxide savings were not the same.

[0069] Taking into account the aforementioned considerations, it may be concluded that soaking plus conventional oxygen delignification produce results that are similar to double stage oxygen delignification. On the other hand, if applied before conventional or low pressure oxygen delignification, such as in the processes S/O, SO, S/(EO) or S(EO), the soaking allows for chlorine dioxide savings in the order of 1-1.5 kg ClO₂ per over dried ton of fully bleached pulp. The washing stage between soaking and the high or low-pressure oxygen stage shows benefits; but they seem to slim to justify the installation of an expensive washing system. In those cases where the washing installation already exists, washing between stages is recommended.

Table 7

Effect of various pulp treatments on overall pulp bleachability with the sequence D(EOP)D, as measured by total chlorine dioxide consumption.								
		ClO ₂ Composition %			ClO ₂ Savings	Brightness	Viscosity	Reversion
Pulp Type	Pulp Treatment	D ₀	D ₁	Total	kg/odt pulp	ISO	mPa.s	%
HWD	None	1.79	1.43	3.22	Ref.	90.1	22.8	2.34
	S	1.63	1.43	3.06	1.6	90.0	21.7	2.28
	O	1.12	0.97	2.09	11.3	90.1	15.4	2.24
	(EO)	1.37	1.18	2.55	6.7	90.0	18.4	2.28
	S/O	1.01	0.97	1.98	12.4	90.1	14.1	2.25
	SO	0.98	0.97	1.95	12.7	89.9	14.5	2.18
	S/(EO)	1.25	1.18	2.43	7.9	90.2	17.0	2.30
	S(EO)	1.23	1.18	2.41	8.1	90.1	17.6	2.25
	O/O	1.00	0.97	1.97	12.5	89.9	17.4	2.38
	OO	0.99	0.97	1.96	12.6	90.2	18.0	2.31
SWD	None	2.94	1.65	4.59	Ref.	89.1	17.5	2.45
	S	2.74	1.65	4.39	2.0	89.0	16.8	2.43
	O	1.57	1.19	2.76	18.3	89.2	14.3	2.29
	(EO)	2.20	1.33	3.53	10.6	89.0	15.9	2.33
	S/O	1.43	1.19	2.62	19.7	89.1	13.9	2.27
	SO	1.39	1.19	2.58	20.1	88.9	14.2	2.25
	S/(EO)	2.08	1.33	3.41	11.8	88.8	15.1	2.33
	S(EO)	2.02	1.33	3.35	12.4	88.9	15.8	2.35
	O/O	1.43	1.19	2.62	19.7	89.0	13.2	2.38
	OO	1.42	1.19	2.61	19.8	88.9	13.7	2.32

Example 8. Impact of pulp soaking on the strength properties of the bleached pulp

[0070] As measured by viscosity and brightness reversion, no significant impact of the soaking on pulp quality is observed in Table 7. To further confirm this, the hardwood sample described and treated as shown in Example 5 and bleached as described in Example 7, was submitted to beatability and strength property tests. A beating curve was developed for the never dried pulp samples using the PFI mill at 0, 1500, 3000, 4500 and 6000 revolutions. The strength properties of the bleached pulps were measured using Tappi standard procedures. The values reported in Table 8 are at 40°SR.

[0071] Because the alkali soaking is carried out at hot temperature and under alkaline conditions, it could be speculated that the pulp strength properties would be somewhat impaired. The results in Table 8 indicate, however, that neither the strength properties nor the pulp beatability were changed by the soaking treatment. Furthermore, the oxygen treatments when carried out alone or in combination with the soaking had no negative effect on pulp strength properties and beatability. Thus, it is concluded that soaking, when carried out under well optimized conditions, does not negatively impact pulp strength.

Table 8

Impact of several pulp treatments on overall strength properties and beatability of the final bleached pulp			
Pulp Treatment	Tear Index at 40 °SR mN.m ² /g	Tensile Index at 40 °SR, N.m/g	PFI Revolutions to Reach 40 °SR
None	9.95	100.5	2739
S	9.81	98.7	2715
O	9.83	98.6	2878
(EO)	9.78	99.1	2796
S/O	10.0	99.7	2802
SO	9.98	97.3	2789
S/(EO)	10.05	98.2	2785
S(EO)	10.03	100.6	2805
O/O	9.65	96.8	2773
OO	9.67	95.4	2716

[0072] It should be understood that the foregoing description is only illustrative of the invention. Various alternatives and modifications can be devised by those skilled in the art without departing from the invention. Accordingly, the present invention is intended to embrace all such alternatives, modifications and variances which fall within the scope of the appended claims.

Claims

1. A method for treating lignocellulosic pulp, comprising the steps of:

- a) mixing, in a pulp soaking stage, said lignocellulosic pulp with an alkali to create a mixture and allowing said mixture to remain in residence therein for a determined period of time to enable an alkali hydrolysis leaching of colored materials from the mixture and a swelling of pulp fibers, said lignocellulosic pulp containing lignin bonded to cellulose fibers;
- b) mixing a feed of said mixture from said pulp soaking stage with both oxygen and steam to form a combined stream, and
- c) feeding said combined stream to a pressurized oxygen delignification stage to enable a reaction between said oxygen and said lignin of said lignocellulosic pulp, said swelled fibers facilitating said reaction between said oxygen and said lignin of said lignocellulosic pulp and further removal of colored materials from said lignocellulosic pulp.

2. The method as recited in claim 1, wherein said pulp soaking stage is carried out as a pH of at least about 11.

3. The method as recited in claim 1, wherein said alkali added to said pulp soaking stage is at least one of: NaOH, oxidized white liquor and unoxidized white liquor.
- 5 4. The method as recited in claim 1, wherein said pulp soaking stage is carried out within a temperature range of from about 40-95°C.
5. The method as recited in claim 1, wherein said pulp soaking stage is carried out at a pulp consistency of from about 6-14 weight percent.
- 10 6. The method as recited in claim 1, wherein said pulp soaking stage is carried out for a retention time of about 15-240 minutes.
7. The method as recited in claim 1, wherein step b) further includes the action of washing said output of said pulp soak stage.
- 15 8. The method as recited in claim 1, wherein step a) further includes the action of adding magnesium sulfate prior to said alkali.
- 20 9. The method as recited in claim 8, wherein said magnesium sulfate is added in a dose amount in a range of about 0.01-0.1 weight percent (as Mg) based on pulp dry weight in said mixture.
10. The method of claim 9 wherein said soaking occurs at a temperature between 70 and 95°C.

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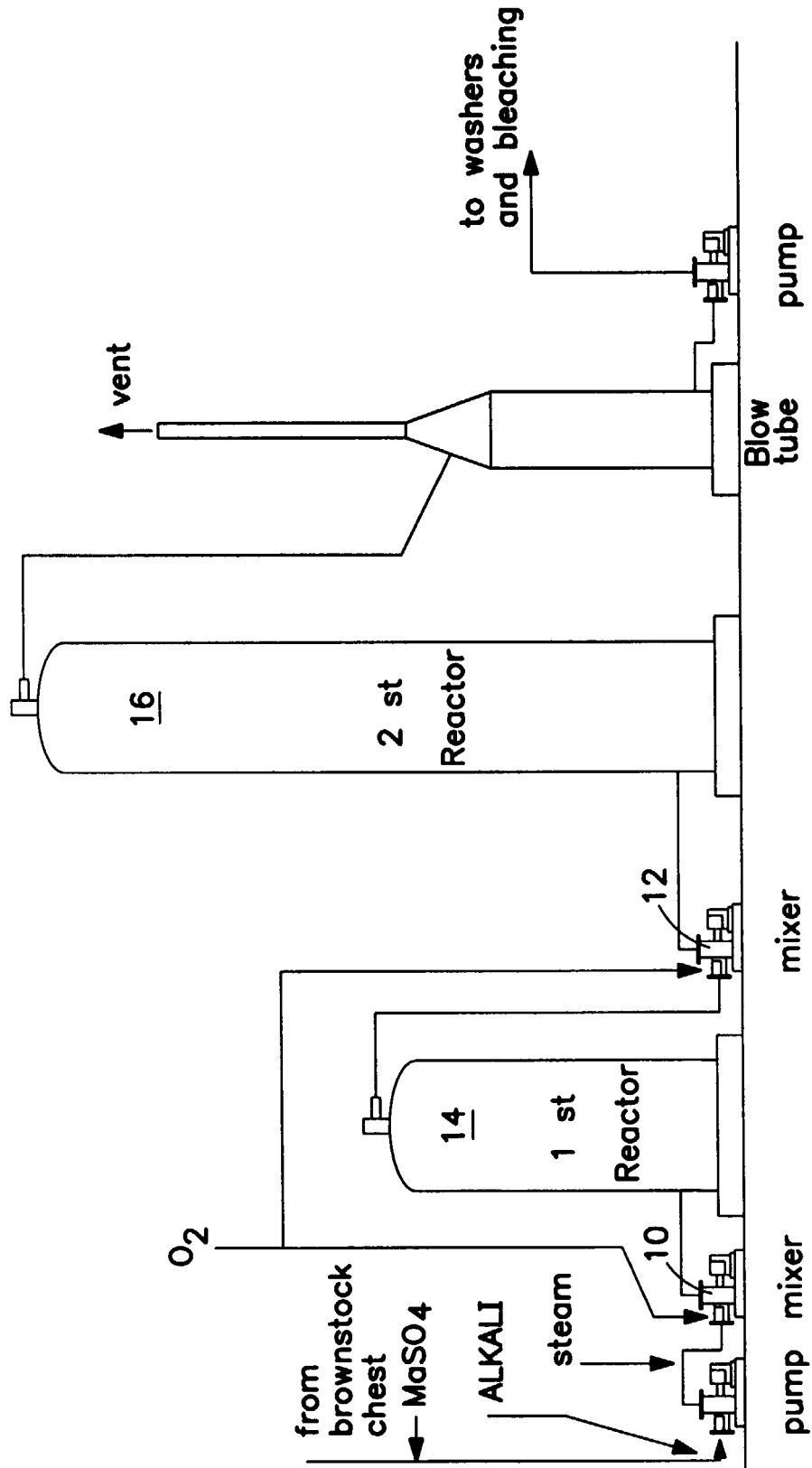


FIG. 1

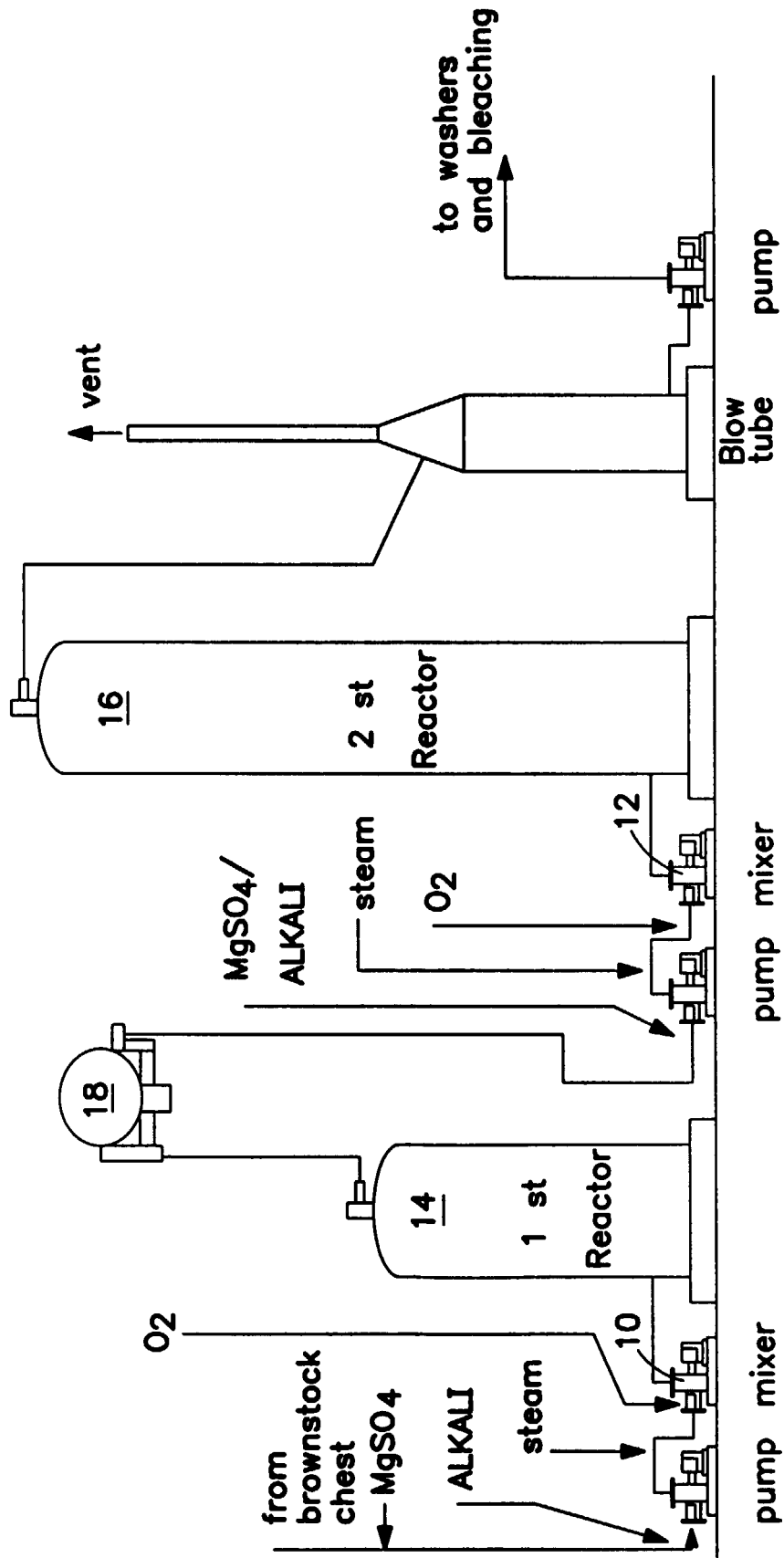


FIG. 2

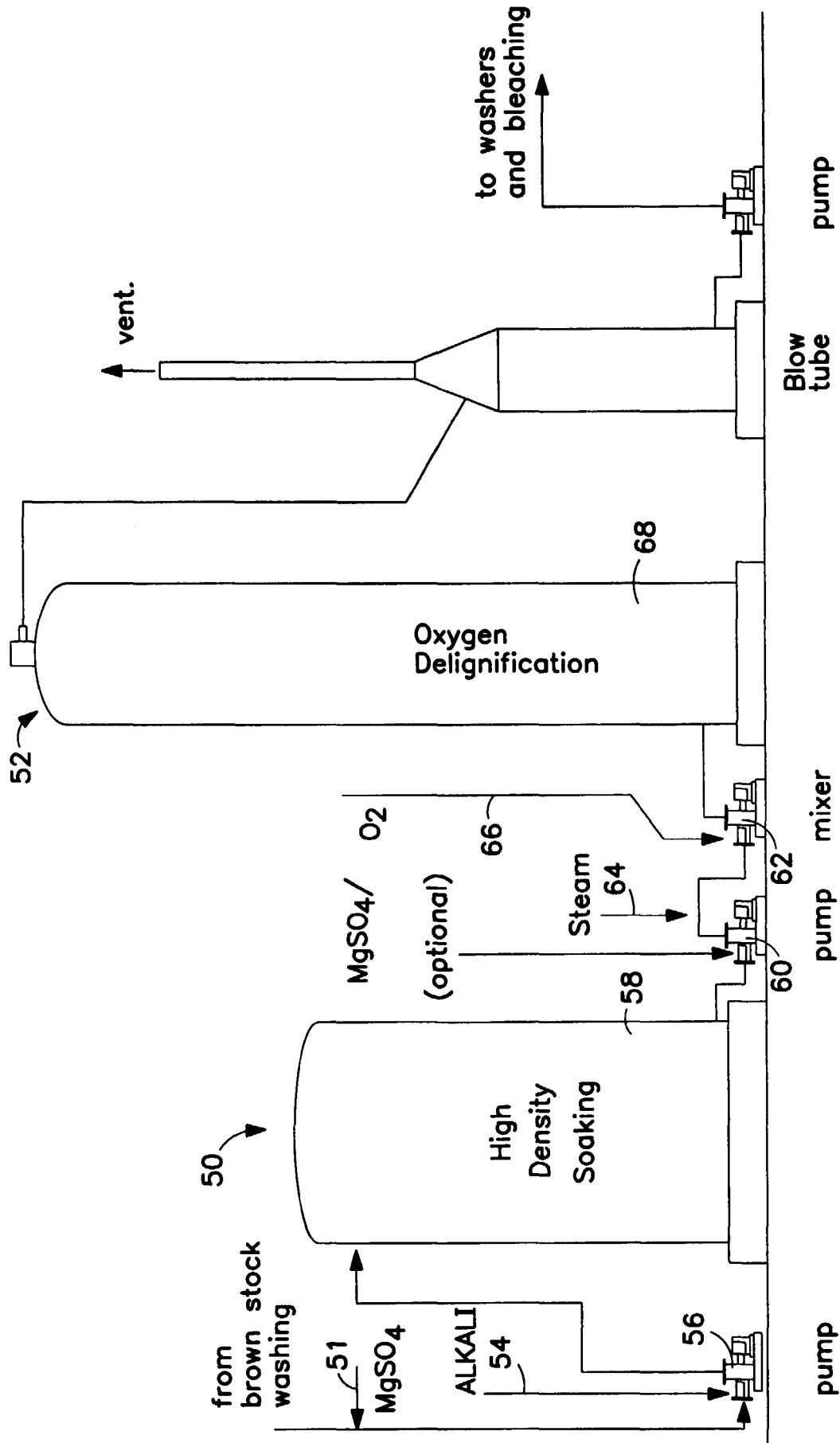


FIG. 3

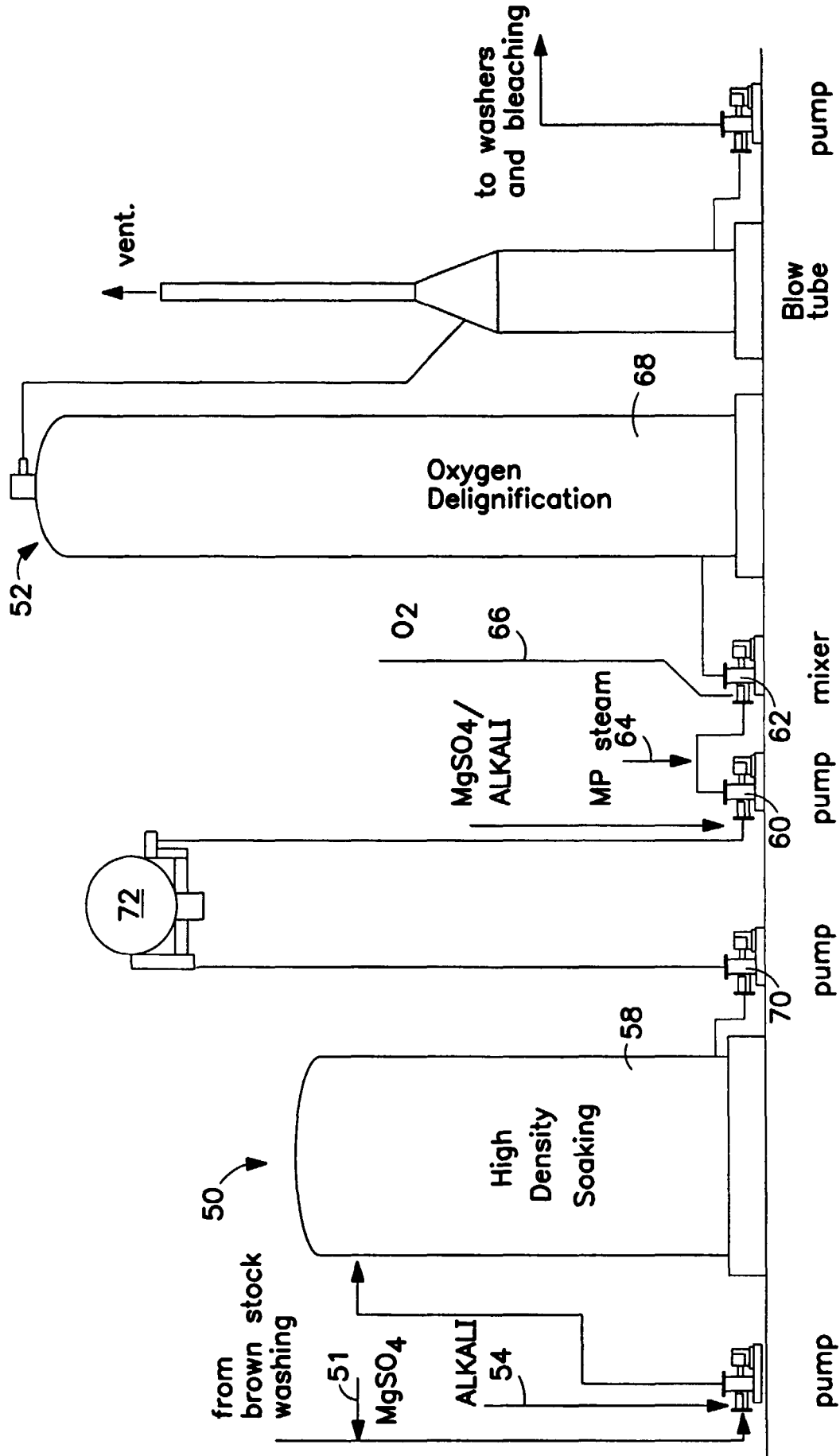


FIG. 4

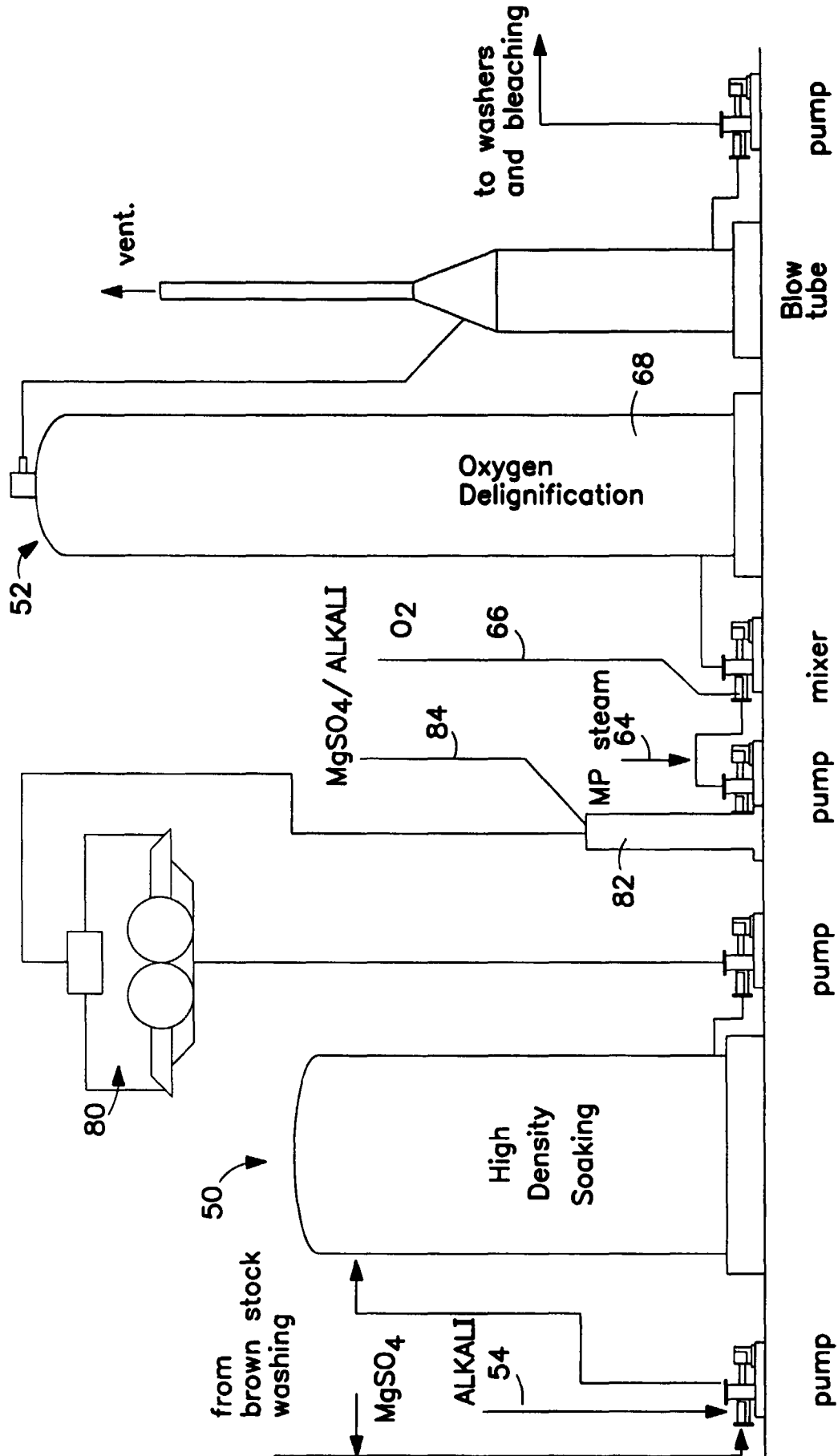


FIG. 5

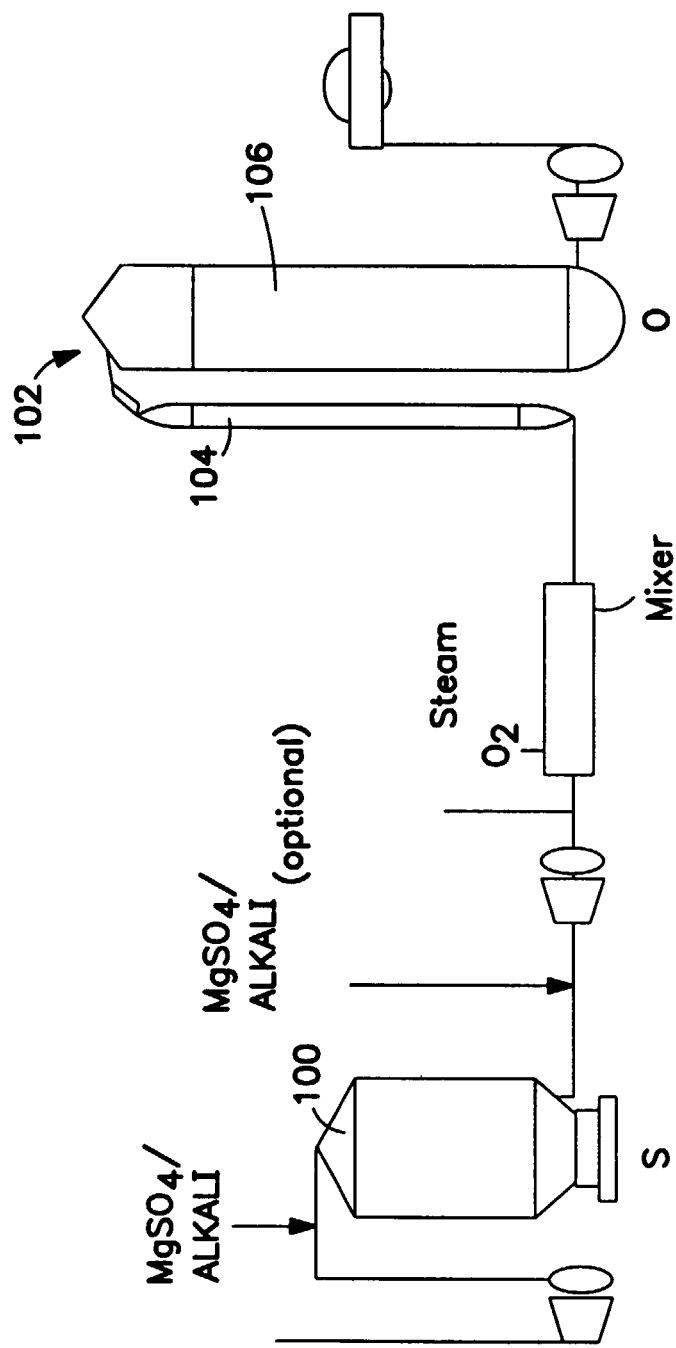


FIG. 6

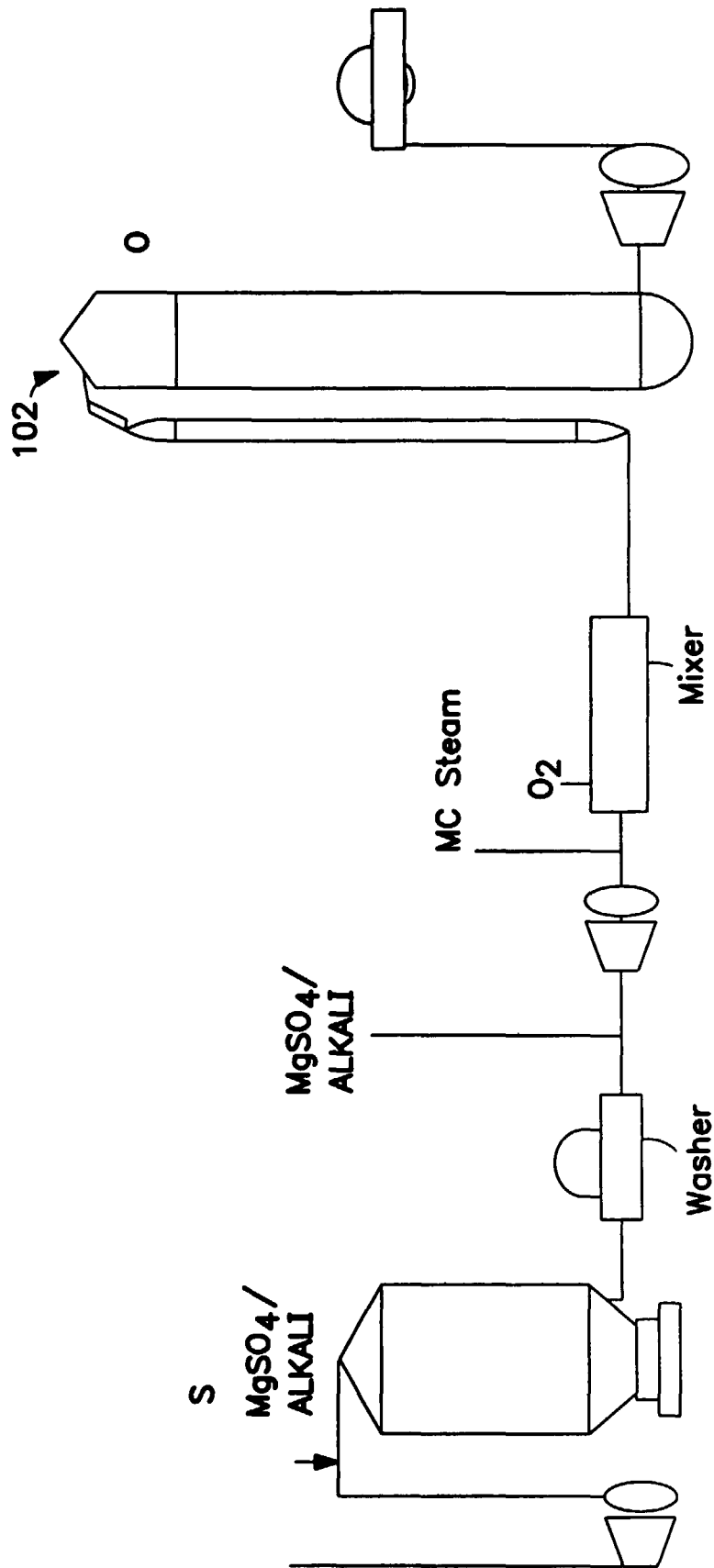


FIG. 7



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Application Number
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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 1 September 2000	Examiner Bernardo Noriega, F
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