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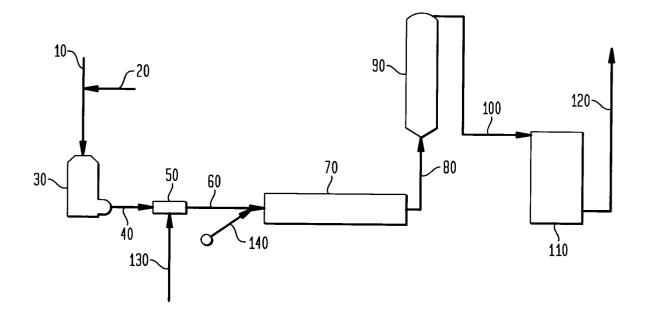
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## (54) Low consistency oxygen delignification process

(57) A method for delignifying softwood or hardwood pulp taken from a fiber line of a pulp and papermaking system comprises adding oxygen to the pulp slurry and mixing the slurry under conditions of temperature and pressure whereby the oxygen reacts with lignin present in the pulp. The delignification is performed on pulp having a low consistency, i.e. containing less than 8% by weight of cellulosic material. The pulp for delignification is preferably taken from a point upstream of knotters in the papermaking system but downstream of brown stock washers so as to make effective use the residual alkali and heat content in the incoming black liquor.

## FIG. 1



#### Description

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#### BACKGROUND OF THE INVENTION

**[0001]** The present invention relates to a method for delignifying softwood or hardwood pulp in a slurry at low consistency using a reaction device and oxygen.

**[0002]** Oxygen delignification of cellulosic material is generally practised at a region of medium consistency (8 to 16%). In such operations partially delignified pulp is reacted with oxygen in the presence of alkali (pH > 11) using high shear mixers and pressurized upflow reactors with retention times of 20 to 60 minutes at pressure in excess of 90 psi (6 bar) and temperatures as high as 110° C. The high shear mixers facilitate the contacting of the pulp slurry with the oxygen containing gas in a very turbulent state lasting a few seconds prior to entering the pressurized vessels.

**[0003]** Using such equipment in single and two-stage configurations have resulted in reported delignification of up to 45%. However, many of such systems perform below 40% kappa reduction due to less than optimal contacting of oxygen with the pulp. In addition, implementation of medium consistency oxygen delignification require high capital expenditures for high shear mixers, medium consistency pumps and large contact towers and have thus far excluded many mills from economically modifying their processes and taking advantage of this technology. Further, oxygen delignification systems have traditionally been located after the brownstock washers on a fiber line where the slurry is relatively cold and have been stripped of all caustic thus requiring the addition of the full complement of steam and caustic.

#### SUMMARY OF THE INVENTION

**[0004]** According to the present invention there is provided a method of delignifying pulp in a slurry from a fiber line of a papermaking system comprising drawing said slurry from said papermaking system, adding oxygen to said slurry and mixing said slurry under conditions of temperature and pressure whereby said oxygen reacts with the lignin present in said pulp, characterised in that the pulp is delignified at a consistency of less than 8% by weight of cellulosic material. The pulp may be a softwood or hard wood pulp

**[0005]** Typically oxygen is added to the pulp slurry and the resulting pulp slurry reaction mixture is introduced into a reaction system comprising an efficient mixer and contact tank. Preferably the pulp is removed from the brownstock processing section between the blow tank and the bleach section of the papermaking system. The reaction system is preferably physically located after (downstream of) the knotters and before (upstream of) the brown stock washers to make effective use of the residual alkali and heat content in the incoming black liquor. The physical location of the reaction system can be at a convenient position along the papermaking system.

**[0006]** By conducting the delignification at low consistency good contact between the oxygen and the lignin is facilitated. Further, the location from which the low consistency pulp is taken for delignification may be one at which the pulp has an elevated temperature and an alkaline pH, thereby enabling the consumption of external energy and alkali to be kept down.

**[0007]** For purposes of the present invention, "low consistency" is defined as that pulp containing less than 8% by weight of cellulosic material.

**[0008]** In practising the method according to the present invention, the reaction system may also be physically located and the pulp slurry drawn from the section of the fiber line after (downstream of) the digester and before (upstream of) the brown stock washers, or in between any two stages of a multi-stage brownstock washing step.

**[0009]** The hot pulp slurry may be taken from the knotters with black liquor still present. In a sulfate or kraft paper-making process the black liquor results from cooking pulpwood in an aqueous solution. Steam may be added to heat the slurry as well as caustic which can be used to adjust the pH.

In a typical example of the method according to the invention, oxygen is injected into the mixture which is then sent to the gas-liquid mixer. The pulp slurry containing the oxygen is mixed at conditions of temperature and pressure whereby the oxygen begins to react with the lignin to effect delignification. This reaction mixture is then sent from the mixer to a retention tank which is also capable of being kept at pressure, where the delignification reaction will continue further. The reaction products are then sent into a blow tank or a gas-liquid separator where reaction gases and unused oxygen gas is disengaged or removed from the liquid product and scrubbed and either vented to the atmosphere or recycled. [0010] The method according to the invention may be implemented as a retrofit in existing mills as well as new ones at relatively low capital cost. The method according to the invention can take advantage of the residual caustic and heat that will be present in the slurry as it leaves the knotters. Furthermore a low consistency oxygen delignification reaction is easier to control compared to operations at medium and high consistencies of pulp. Accordingly low consistency oxygen delignification before the brown stock washers of a fiber line can be achieved.

**[0011]** The reactor assembly typically consists of a controlled cavitation device or other efficient mixing device, coupled with a residence tank.

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**[0012]** The heat and alkali requirements for the delignification reaction are typically obtained primarily from the residual alkali and heat content in the incoming black liquor and oxygen containing gas.

**[0013]** An excess of oxygen over that required for delignification purposes can be supplied so as to react with some of the organic acids in the black liquor to reduce the heat value of the black liquor, and to react with sulfur compounds in the black liquor to reduce or eliminate the potential for total reduced sulfur (TRS) emissions.

**[0014]** A further advantage of the present invention is that un-oxidized white liquor can be employed as the source of needed alkali. Additionally the excess oxygen can be employed to oxidize some of the sulfur compounds in the white liquor.

[0015] The pulp slurry preferably has a consistency of between 2 to 8% by weight.

[0016] When necessary, a small amount of caustic is preferably added to bring the slurry pH to at least 11 to 12.

[0017] Where necessary, steam is preferably added to bring slurry temperature to between 85° C to 120° C.

[0018] The reaction mixture is preferably sent to a disengagement tank where the reaction gases are separated from the slurry and sent for treatment and disposal.

**[0019]** The delignified slurry is preferably sent to the next stage in the bleach line of the papermaking process, which could be the screens or brown stock washers.

#### BRIEF DESCRIPTION OF THE DRAWINGS

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**[0020]** The method according to the present invention will now be described by way of example with reference to the accompanying drawing which is a schematic representation of an oxygen delignification process according to the practice of the present invention.

### DETAILED DESCRIPTION OF THE INVENTION

[0021] In the embodiment shown in Figure 1, pulp slurry taken from the knotters (not shown) of a papermaking plant is contacted with oxygen in the presence of caustic, under conditions of temperature and residence time (normally short) in a retention tank that enable the oxygen to react with and delignify the pulp. The oxygen also reacts with the organic solid in the black liquor resulting in a reduction of the heat value of the black liquor going to the recovery boiler. Additional reactions take place between the oxygen and sulfur compounds in the liquor, thus reducing or eliminating potential sources of total reduced sulfur (TRS) emissions by the pulp mill.

**[0022]** Pulp slurry from the knotters (with the black liquor still present) at a consistency of between 2 to 8% is transferred by a pump 30 through pipelines 10 and 40 to a steam mixer 50. Caustic, if needed, may be added to the pulp slurry through side pipe 20 to maintain the pH of the pulp slurry between 11 and 12. At the steam mixer, steam is added to the pulp slurry through pipe line 130 to bring the temperature of the slurry to between 90 and 110° C before it flows into the efficient mixing device which can be a cavitation type reactor 70 through line 60. Oxygen containing gas is introduced through a side inlet 140 and enters the efficient mixing device with the pulp slurry stream.

**[0023]** The pulp slurry and the oxygen containing gas are brought into intimate contact by the shearing and cavitation and mechanical forces generated as the rotor rotates at a high speed in the mixing device 70. The contacting is performed under suitable conditions of temperature and pressure whereby the oxygen begins to react with the lignin under caustic conditions to effect delignification. The reaction mixture proceeds through pipe line 80 into a retention tank 90 where under conditions of temperature, pressure and time the delignification reaction is continued further.

**[0024]** In order for the reaction to proceed as mentioned above, an oxygen containing gas is used. Preferably oxygen with a minimum purity of 93% is employed. The preferred total reaction pressure should be not less than 80 psig and more preferably above 90 psig. The preferred minimum reaction temperature is  $90^{\circ}$  C but the temperature can typically be as high as  $110^{\circ}$  C.

**[0025]** The residence time of the reaction mixture in the retention tank is not less than 10 minutes but preferably up to 30 minutes. The reaction mixture is withdrawn through outlet 100 into a blow tank or gas-liquid separator 110 where reaction gases and unused oxygen gas disengages from the liquid product and is scrubbed and vented. The pulp slurry is withdrawn through line 120 and sent to the washing stage of the bleach line of the pulp and papermaking process.

**[0026]** By practising the methods of the present invention, bleaching can be done with much less capital, specifically by not having to build, own, operate and maintain separate pressure towers for traditional oxygen delignification. Unoxidized white liquor can be used as the source of caustic as the oxidation of the white liquor also takes place in the mixer. Also, the exothermic heat of reaction of oxidation of white liquor raises the temperature of the pulp resulting in steam savings for oxygen delignification and most of the oxygen delignification reactions can be done at the existing pulp temperature.

[0027] Oxygen delignification before the brown stock washers lowers the BTU value of the black liquor allowing for increased capacity of steam limited recovery boilers. Additionally, less oxidized white liquor will be needed in this sequence than traditional two stage oxygen delignification thus taking the load off the lime kiln and other white liquor

systems.

[0028] The black liquor produced will have a lower viscosity because of the reaction of the oxygen with the  $Na_2S$  and a portion of the dissolved lignin allowing for a higher percentage solids firing in the recovery boiler. Methanol and other alcohols present in the black liquor upstream of the brown stock washers will react to form a reducing environment in the pulp. With the availability of hydrogen limited, the side reactions that hurt pulp strength will also be limited. Indeed pulp strength may increase.

[0029] The following is an example of a practice of the invention in accordance with the embodiment illustrated in FIG 1. In this example a side stream of softwood slurry at a consistency of 2.5% by weight and containing residual black liquor was withdrawn with a Gould's pump 30 at a flow rate of 50 gallons per minute (gpm) through a side pipe 10 on the stock line going from the knotters to the first brown stock (BS) washers. The pH of the slurry was above 11.5 and so no additional caustic was added. Slurry temperature was 87° C. The slurry was heated to 90° C adding low-pressure steam through pipe 130 and mixed into the slurry with the steam mixer 50. Pure oxygen was added to the slurry through a side pipe 140 at a rate of 60 pounds per hour before it entered a high efficiency gas-liquid mixer, in this case a controlled cavitation reactor 70. The slurry and gas were mixed for a few seconds at about 90 pounds per square inch (psi) in the reactor 70, the stock was transferred into a retention tank 90 and kept for 10 minutes and was finally returned to the bleach line just before the first BS washer. The pulp Kappa # is the amount of lignin in the pulp sample. Samples were taken from the exit slurry stream for analysis. The following results were obtained:

Table 1

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	Inlet	Outlet	% Reduction
Pulp Kappa #	25.8	16.5	37
Na <sub>2</sub> S, mg/kg solids	21000	<200 ( non detect)	almost 100
Dissolved Lignin, As % solids	32.1	29.1	9.3

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**[0030]** Comparatively, for conventional single stage oxygen delignification systems operating under similar conditions of temperature and pressure on a 10% consistency pulp, residence times of 40 minutes and higher will be required to achieve the 37% delignification.

**[0031]** While this invention has been described with respect to particular embodiments thereof, it is apparent that numerous other forms and modifications of this invention will be obvious to those skilled in the art. The appended claims and this invention generally should be construed to cover all such obvious forms and modifications which are within the true spirit and scope of the present invention.

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#### **Claims**

- 1. A method of delignifying pulp in a slurry from a fiber line of a papermaking system comprising drawing said slurry from said papermaking system, adding oxygen to said slurry and mixing said slurry under conditions of temperature and pressure whereby said oxygen reacts with the lignin present in said pulp, **characterised in that** the pulp is delignified at a consistency of less than 8% by weight of cellulosic material.
- 2. A method as claimed in claim 1, characterised in that the temperature of said slurry is in the range of 85° C to 120° C.
- **3.** A method as claimed in claim 1 or claim 2, **characterised in that** the pressure under which the delignification is performed is in the range of 2.4 to 11.9 bar (20 to 160 psig).
- **4.** A method as claimed in any one of the preceding claims, chracterised in that the mixing is conducted for a period of up to 2 minutes in duration.
- **5.** A method as claimed in any one of the preceding claims, **characterised in that** said pulp slurry contains black liquor.
- 6. A method as claimed in any one of the preceding claims, **characterised in that** said oxygen reacts with lignin present in said black liquor.
  - 7. A method as claimed in any one of the preceding claims, characterised in that said mixing occurs in a gas-liquid

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mixer.

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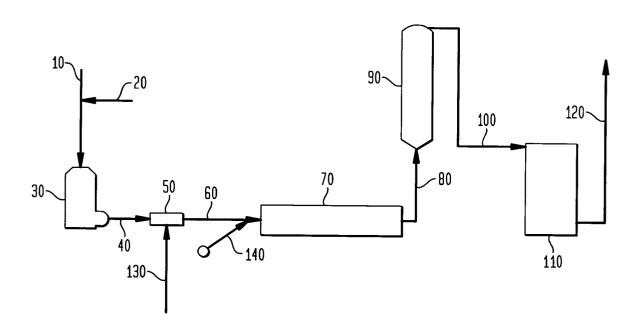
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- 8. A method as claimed in claim 7, characterised in that said mixer is a controlled cavitation device.
- 5 9. A method as claimed in claim 7 or claim 8, characterised in that the mixed pulp is held in a residence tank.
  - 10. A method as claimed in claim 9, characterised in that said pulp slurry is maintained at a pressure in the range of 2.4 to 11.9 bar (20 to 160 psig) for 5 to 30 minutes in said residence tank.
- 10 11. A method as claimed in any one of the preceding claims, characterised in that said treated slurry is returned to a bleach line of the papermaking system.
  - 12. A method as claimed in any one of the preceding claims, characterised in that said pulp slurry is drawn from said fiber line at a point selected from the group consisting of a point downstream of the knotters and upstream of the brown stock washers; a point downstream the digester and upstream of the brown stock washers; and a point intermediate any two stages of a multi-stage brownstock washing step.

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FIG. 1





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**Application Number** EP 04 25 6503

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