Innovative Approaches in Pulping of Lignocelluloses - A Review

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ABSTRACT

Modified conventional pulping methods, oxygen delignification, pulping with peroxyacids, organosolv pulping, hydrogen peroxide alkaline pulping, high-yield pulping, biopulping and some other minor methods for pulping are reviewed.

INTRODUCTION

Cellulose is one of the oldest known natural polymers. It is renewable, biodegradable, and can be derivatives to yield various useful products. Its main source, however, is wood, a natural composite, where cellulose is contained in combination with lignin in a texture. This natural polymer composite must be destroyed for the isolation of cellulose (pulping). In the very old technology of papermaking, little real progress is to be expected and problems tend to be focused around optimization and ecological issues. However, innovative approaches for pulping and bleaching of lignocellulosic materials can allow for the isolation of cellulose in a soft and ecologically benign manner and generally produces less hazard wastes (1).

Modified conventional pulping methods

It still seems that kraft that the modified kraft process will maintain its position as the most beneficial way to produce the majority of the global paper industry fibre made by chemical pulping (2). Kraft process already accounts for greater than 90% delignification of wood into bleachable grade pulp (3).

Conventional pulping drawback could be minimized by simple modifications (4). Extended delignification refers to modification of pulping processes, partially the kraft process, by prolonging or extending the cooking to lower pulp lignin content. The amount of lignin remaining in unbleached kraft pulp is reflected in the amounts of organochlorine and oxygen-consuming compounds, subsequently discharged in bleach plant effluents. The process is applied to bleachable grade pulp with main objectives of reducing the impurity load (mainly organics) in the bleach plant effluent and to decrease the chemical consumption during bleaching (5, 6). Extended delignification could be achieved by different techniques as cold blow cooking, modified continuous cooking (MCC) (6,7), rapid displacement heating (RDH) (8), and modified continuous cooking (9). Super batch cooking allows reaching very low kappa numbers, suitable for TCF bleaching, while maintaining excellent pulp quality (10). Rapid soda pulping of wheat straw by the impregnation rapid-steam-pulping process produced chemical pulp of low residual lignin (11).

The use of chemical additives as anthraquinone (AQ) and polysulphide (PS) is a low capital investment option for decreasing the kappa number and minimizing the impact on the recovery boiler load (12). Chemical additives accelerate the delignification. Additionally, a higher bleached pulp yield is possible (13). The reaction mechanism, advantages and combination of AQ with different pulping methods have been reviewed (14). Anthraquinone improves the delignification selectivity of kraft pulping (15). AQ addition of 0.1% significantly increased the rate of delignification and enhanced the screened yield. Cooking could be extended down to 13.5 kappa number without severe yield and viscosity loss (16). During sulphite pulping, AQ causes an enhancement of aryl ether-cleavage

reactions, reduction in refining energy and enhancement in the sheet strength (17). AQ has been added also to a fast soda/AQ process; this process used high pulping temperature and short residence time to stabilize the carbohydrates (18). Soda-anthraquinone process has been used for pulping of different agricultural residues as sunflower stalks (19), reed canary grass (20) and wheat straw (21). AQ enhances the sulphonation reaction that was shown to have a direct impact on the sulphite delignification process (22). Neutral sulphite anthraquinone seems to be an encouraging pulping process that produces kraft-like pulp with higher yield (23).

Extended kraft delignification could be achieved by addition of polysulphide or PS/AQ during pulping. Electrolytic oxidation process has been developed to produce highly concentrated polysulphide (PS) liquor from white liquor (24). The prototype mill-scale installation is a new, low-cost process for the production of polysulphide (PS) liquor by using two different impellers to convert a portion of the sodium sulphide in white liquor to PS (25, 26). Synergistic effect of both AQ and PS has been investigated (27). With 1.3% PS and 0.1% AQ kappa number is decreased from 30 to 20 compared to the kraft process under the same pulping conditions while pulp yield and strength are comparable (28). Cooking to a kappa number lower than 15 could be achieved with no detrimental effect on pulp properties (19,30). PS/AQ pulping consumed 7 and 18% less chemical during pulping and bleaching, respectively. It also lowers the black liquor solids and the heat load to the recovery boiler (31).

Introduction of polysulphide (PS) in kraft pulping reduces the amount of toxic sulphur compounds and reduces odour emission (32). Polysulphide or polythionate can serve as odour abatement, it absorbs mercaptans and reduces the odour problem (33). The reliability and performance of the new thermal oxidizer in community odour decreasing has been excellent (34).

The addition of suffactant-based additives can reduce surface tension between the liquor and chip and allows a through wetting of the chip surface. This facilitates rapid penetration and diffusion and can result in faster delignification of chips, improved pulp properties, reduced kappa number and screen rejects and overall improvements in pulp quality (35, 36). A conversion of some rejects into accepts increases pulp yield without sacrificing pulp quality (37). A surfactant-based digester additive has helped to decrease the active alkali charge in the soda pulping as alternative process to kraft pulping (38).

Addition of methanol to a kraft cook increases the delignification rate and improves the selectivity, measured as viscosity at a given kappa number (39). Kraft pulping, modified by anthraquinone and/or methanol addition, gives lower lignin content and higher pulp yield at comparable pulp strength (40, 41). 20% from sodium sulphide used in kraft pulping could be successfully replaced with sodium sulphite to lower sulphur contamination. Pulp yield was higher and anthraquinone and/or methanol addition compensates the lower strength and higher rejects (40). High initial hydrosulphide concentration improves conventional and modified kraft cooking by producing two or more white liquors of chemical concentrations optimized for the different phases of the cook (42). Sulphide pretreatment in kraft pulping increases the delignification rate and carbohydrate protection and improves the pulp properties and bleachability (43, 45). Best physical properties of sulphite pulps using low charges of chemical were obatined by optimization of neutral sulphite semi-chemical (NSSC) pulping process (46, 47).

Oxygen delignification

Oxygen delignification is an extension of the delignification process initiated in the cooking operation and as such provides the bleach plant with a pulp of considerably reduced lignin content (48, 50). The main obstacle for oxygen delignification was poor oxygen selectivity towards lignin (51, 53). Oxygen tends to degrade cellulose under caustic conditions (54). It is generally considered that the limit for the use of oxygen for delignification purposes is at approximately 50% delignification, and that any further delignification using oxygen and alkali will result in severe carbohydrate degradation leading to fibre strength decrease and yield losses (55, 57). The use of oxygen has increased to the development of medium consistency oxygen delignification technology (58). Two-stage extended oxygen delignification has been developed for pulp yield improvements (59, 61), Pulps showed slight losses in viscosity and fibre strength, but brightness increased significantly and chemical demand during bleaching was reduced (62). AQ addition during oxygen delignification increases the yield and improves viscosity and fibre strength (56, 62). Delignification using sodium carbonate and oxygen has the purpose to minimize effects of low oxygen solubility and oxygen transport from the gas into the solid phase (63). Brownstock washing plays a major role on selectivity of oxygen delignification. High solids and black liquor carryover improves the delignification selectivity (64, 65).

Peracetic acid pretreatment was found to increase the efficiency of the oxygen delignification stage with no loss of pulp viscosity (67, 68). Oxygen delignification is more selective than the kraft cook process and potentially an increased final product yield can be realized (69, 71). The higher pulp yield can translate into a lower organic load to the recovery boiler (68, 72). During oxygen delignification the guaiacyl phenolic units of softwood residual kraft lignin eliminate with the concomitant formation of carboxylic acids. The rate for the elimination of phenols and the formation of acids were found to significantly increase when the reaction temperature was above 100°C (73).

It is well known that the presence of certain transition metal ions in pulps can have a detrimental influence during oxygen bleaching (74, 75). Iron, copper and manganese are thought to play a major role in this particular problem, because these metals are present in the wood or bagasse itself and can also accumulate from source including process equipment. Several inorganic species are known to have stabilizing influences during bleaching, sodium silicate and magnesium salts are used commonly during industrial processing (74). Magnesium compounds were used to reduce the cellulose degradation at the oxygen delignification stage. Addition of catalytic quantity of sodium borohydride to the magnesium-based oxygen delignification of sulphite pulps improves pulp yield, viscosity, and strength properties. Treatment of pulp with sodium borohydride reduces the carbonyl groups to hydroxyl groups, thus stabilizing the cellulose against degradation during the oxygen delignification stage (76).

Pulping with peroxyacids

The pulp and paper industry is under great pressure to bleach kraft pulps without chlorine bleaching chemicals. The peracids are being investigated as replacement oxidants. Preparation of peroxy-carboxylic acids, its use in pulping, lignin characterization, washing, paper properties and bleachability of pulp have been previously reviewed with 56 references (77). European mills are now using the peracids during TCF bleaching (78).

Treatment in formic acid media has been reported to be suitable for pulping softwoods, hardwoods and nonwoody plants (79, 80). Beech wood samples were subjected to two-stage formic acid-peroxyformic acid treatments (82, 83). Pulp yield of46.6% with 86.3% cellulose, 5.5% lignin and 4.3% xylan were obtained. After TCF the pulp shows a potential use as feedstock for dissolving pulp production (83). Chemical pulps from Eucalyptus grandis wood chips and sugar cane bagasse is produced by an improvement in the peroxyformic acid process (84). The pulping was carried out in a single stage at 75°C (3 h). Efficient distillation of the spent liquor allowed recovery of a large quantity of formic acid and phenolic lignin. The pulping process was completed with a 0.25 M NaOH extraction of the pulp at 60°C to dissolve more lignin. Good quality unbleached pulps were obtained. Kappa numbers of 14 and 13, and intrinsic viscosities η of 1130 and 980 $dm^3 kg^{-1}$ were obtained, respectively, for E. grandis and sugar cane bagasse. The lignins were found to undergo demethylation, condensation, ring opening, formulation of hydroxyl groups, and arly-alkyl ether cleavage during pulping (84, 85).

Peracetic acid could be used in a combined delignification stage with ozone (86), with alkaline peroxide (83), or with oxygen-delignification (67) to obtain pulp of low kappa number. Kinetics of delignification of spruce wood in the system H_2O_2-H_2O-AcOH-AcOOH-catalyst was considered in the context of the general theory of homogeneous catalysis (87). The role of transition metal species in delignification with distilled peracetic acid was investigated (78).

Peroxy mineral acids were used successfully for delignification. Peroxymonosulphate treatments (Caroate delignification) were performed to improve the overall selectivity of two-stage oxygen delignification. Dimethyldioxirane (DMD), a selective pulp delignifier, is formed in the presence of acetone and peroxymonosulphate (88). Cobalt and copper cations catalyzed the delignification by Caroate process (89). The kinetics of the ketone-catalysed decomposition of peroxymonosulfuric acid (Caro's acid) has been ivestigated (90). Treating finely divided aspen wood with pernitric acid and extracting it with dilute alkali readily delignifies the wood (91). Peroxymonophosphoric produce a highly delgnified, bright pulp more rapidly and selectivity than peroxymonosulphuric acid and pernitric acid (92).

Organosolv pulping

For organosolv pulping economically attractive mills can be built at about one fourth the size of a modern kraft mill (93). Different approaches to solvent pulping have been proposed and many authors have reviewed the progress in organosolv delignification and their advantages over conventional chemical pulping (94, 96).

Organosolv pulping by alcohols have been

extensively studied in our laboratory to delignify bagasse, cotton stalks and wheat straw (96, 102). Uncatalyzed ethanol and methanol pulping of sugar cane bagasse was conducted (96). The study indicated that alcohol pulping of sugar cane bagasse has to befollowed by a proper washing precedure in order to reduce the final lignin content of the pulp to levels suitable for a subsequent bleaching process. Alkali addition to ethanol pulping is recommended to attain easily bleachable pulp of lower chlorine number at a suitable cooking temperature. Bagasse pulping with butanol shows a comparable results (103). Stem pretreatment before ethanol pulping was investigated (97).

Cotton stalks could be partially pulped by the aqueous ethanol process without catalysts. While the alkaline process would enable higher potential for strength and hence constitute possibilities for chemical pulp qualities. Screened yield of 41%, rejects content of 1.5% and chlorine number of 4.0 could be obtained by pulping with 18% NaOH, ethanol concentration 40%, liquor ratio 5:1, maximum temperature 180°C and time at maximum temperature 1 h (98).

Organosolv pulping of wheat straw has been studied using aqueous ethanol and alkali ethanol processes. Optimum results for the aqueous ethanol process were obtained by using 1:1 aqueous ethanol (v/v) and 7:1 liquor ratio at 175°C for 2 hours. The pulp yield was 56% and chlorine number 15. In this process more than one half of the residual lignin of the pulp could be diminished by a proper washing procedure. Best results obtained in the alkaline ethanol process gave a pulp yield of 56.5% and chlorine number 3 by using 0.3 mol NaOH/1 (8.4% on raw material), 0.05% anthraquinone, liquor ratio 7:1 and ethanol concentration 50% at 150°C for 90 minutes (99).

Kinetic studies on wheat straw pulping by the alkali ethanol process shows that the reaction rate is in direct increase with the temperature, and it doubles if the alkali concentration is doubled. The Arrhenius equation is applicable in describing the delignification. The activation energy, in the bulk phase, is 16 kJ/mol. This pulping process exhibits fast and selective delignification under mild conditions even at longer cooking times. Wheat straw exhibits facile delignification ability at lower energy consumption and shorter pulping time (100).

The strength properties, for all raw material studied, of NaOH/EtOH pulps were comparable with those of the NaOH pulp prepared under the same conditions, but with higher alkali charge. Bleaching of NaOH/EtOH pulps gave higher brightness than the NaOH pulps irrespective of the method used for comparison (10).

Pulping of bagasse according to the IDE process (Impregnation, Depolymerization, Extraction) under the following conditions for impregnation: 0.5 mol Na_2CO_3/1 and 1 h at 100°C, for depolymerization: 50% EtOH at 180°C, for 1.5 h; and for the two stage extraction: 50% EtOH, 1 h each, decreasing temperature from 180 to 150°C; resulted in 58.7% screened pulp yield and chlorine number 1.2. The results obtained with the IDE process for bagasse were comparable with those obtained in normal EtOH-NaOH-AQ pulping. The benefit of the replacement of most or all NaOH for Na_2CO_3 is evident for the chemical recovery and the environment (102).

Remarkable improvement of the pulp properties was achieved by pre-hydrolysis in methanol/water followed by alkaline soda/AQ/methanol pulping processing (104). The pulp can be TCF bleached to 85% ISO brightness with corresponding strength to that of TCF softwood kraft pulps. Methanol based pulping of Eucalyptus globules (105, 106) and wheat straw (107), provides pulp with low kappa number and acceptable viscosity with a high yield. Alkali sulphite anthraquinone methanol (ASAM) pulping process has been developed and accomplished for the pilot plant application (108).

Autocatalyzed ethanol-water pulping (93, 109, 110), were extensively studied. Alcohol-water pulping employed to low lignin content pulp, high yield of by-products were obtained (111). Kinetic parameters were determined (109, 112) it was found that with increasing mole fraction of ethanol in the cooking liquor there was an increase in the reaction order and a decrease in the activation energy for the delignification process (112). Ethanol-kraft pulping proves a higher yield and superior properties compared with kraft process (112-114).

Fungally- pretreated wood samples were submitted to organosolv delignification. The cooking liquor used was methanol/water (78:2 v/v) containing $CaCl_2$ and $MgSO_4$ each at a concentration of 25 mmol dm^{-3}. The cooking process was performed at 180°C for reaction times varying from 5 to 100 min. The highest delignification and xylan removal rate constants were observed in the sample decayed by T versicolor for 2.5 months (17% weight loss) and also for sample decayed by Punctularia artropurpurascens for only 0.5 months (1.2% weight loss) (115).

Organosolv pulping of wheat straw could be

achieved by use of phenol (116) or acetone-water mixtures (117, 118). Pulping of whole jute plant by soda-amine liquor (119), and aqueous glycerol delignification of wood chips and ground wood (120) has been investigated.

Formic acid/acetone mixture has been used for delignification of Pinus radiata and Eucalyptus globules (121). Fungally-pretreated Pinus radiata samples, with the white rot fungi Ceriporiopis subvermisopora and Punctularia artropurpurascens, were delignified by formic acid/acetone (7:3) at 150°C. Delignification rates and xylan solubilization rates were higher for the decayed samples than for the undecayed control. C subvermispora proved appropriate for treating the wood samples before organosolv pulping, since pretreatment with this fungus resulted in faster wood delignification and pulps with lower residual lignin. Increases in tensile index ranging from 3% to 22% were observed for most pulps prepared from biotreated samples. However, tear and burst indexes and brightness were lower than or similar to those of pulps prepared from the undecayed control (122).

The pulps from acidic (formic acid or acetic acid) process have high cellulose content as well as a notable chemical recovery (123). The high proportion of cellulose reduces the pulp losses by dissolution in white waters, while the high reactivity favours its application as dissolving pulp (124). Formic acid pulping of rice straw (125), wheat straw (126) and kenaf (127) produces pulp of higher yield and comparable properties compared with the conventional pulping processes. The acetosolv process only uses acetic acid as cooking solvent at temperature 180-190°C. This process can be applied successfully to different raw materials (128, 129). The pulps have kappa number between 10 and 25 and can be ECF and TCF bleached (130). In pulping with organic acids, they are usually combined with acidic or oxidant additives, as well with other solvent (131). The acidity is enhanced by mineral acids, such as HCl, which allows the increment of delignification (130, 132). Treating beech wood samples in HCl-catalyzed acetic acid solution, under selected condition, gave pulp with 5.8-7.5% lignin, 77-83% cellulose and 3.3-6.1% xylan at 46-50% pulp yield (132). Experiements on the Acetosolv pulping of Eucalyptus globulus wood have been carried out under a variety of operational conditions (133). In acidic medium the cleavage of lignin bonds starts with the protonation of α-hydroxil and α-ether groups to give the corresponding conjugate acids. The α-ether bond can be broken directly in the presence of acids with the formation of benzyl carbonation or a quinone method, it can lead to the cleavage of a β-ether linkage nearby (134).

Biopulping

Biotechnology is a new option in the field of pulp and paper industry. Enzymes from wood destroying fungi cab be used to destroy lignin selectively. This can allow for the isolation of cellulose in a "soft" and ecologically benign manner. The big disadvantage remains the low through put of these processes, which at present, rules them out on economic grounds. Therefore, though the biochemical processes operate at low temperature and normal pressure, they require much longer time and can, therefore, hardly compete with ordinary pulping processes. However, they pose no ecological problems and generally produce no wastes. But the use of enzymes in various technical reactions, including pulping and bleaching, is still growing (158).

It has been postulated that if the lignin macromolecule is partially depolymerized in an initial step, mild cooking conditions are feasible and carbohydrate degradation can be prevented. This pretreatment can be carried out by fungal degradation of lignocellulosic materials using selective white-rot fungi. Fungal pretreatment provides faster delignification rates. As a consequence, the same residual lignin content in the fungal pretreated samples are achieved at shorter reaction times, which means energy saving and pulp of increased strength properties. The effect of fungal pretreatment is usually greater if the cooking is carried out at low severity (159).

Energy consumption remains a key issue limiting the use of high-yield mechanical pulps. One possible strategy for breaking through this energy barrier is biological treatment of the raw materials prior to fibreization or refining. Biomechanical pulping with fungi is selectively degraded and modifies lingin without sacrificing yield or degrading wood carbohydrates, especially cellulose. Substantial energy savings (up to 20%-50%) and significant improvements in handsheet strength properties have been obtained with lignin-degrading fungi such as Phanerochaete chrysosporium and ceriporiopsis subvermispora (160).

Biological treatments using white-rot fungi may provide an alternative to chemical pretreatments in high-yield wood pulping (161). Fungal pretreatment of wood (162), bagasse (163), kenaf (164) and Agave sisalana (165) prior to mechanical pulping reduces the electrical energy requirements during refining, and

increased pulp strength. A production increase of 20% could be attained (166). The pretreatment of bagasse with the white rot fungi Ceriporiopsis subvermispora prior to mechanical refining resulted in better strength properties and reduced energy usage compared to pretreatment with Pleurotus ostreatus (163). Pretreatment with Phlebiopsis gigantea for biomechanical pulping of whole logs was investigated (162).

The combination of enzyme treatment and fibre fractionation is to separate the pulp into fractions by fibre and treat only the long, coarse fibres with the enzyme. This combined approach increases the pulp yield and improved both the density and smoothness of handsheets made from the unrefined pulps. The tensile strength of handsheets was increased by as much as 10% over that of handsheets derived from control furnishes. With refining, fewer enzymes are needed to reach the same improvements in papermaking properties (167). Coarse thermomechanical pulps (TMP), rather than chips, receive a short pretreatment with cellulases or hemicellulases before secondary refining. Pulps were treated at 5% consistency and 45°C, and most pretreatments were for 2 hours. The enzyme treatment did not degrade pulp optical properties, and yield losses were insignificant (less than 0.3%) (160). Enzymatic treatment of mechanical pulp fibres before papermaking improves the strength properties (168).

The challenge today is to develop more efficient recycling processes and to reduce costs. High-temperature biotreatment is an attractive option for inserting a thermophilic biological treatment step to close the water circuit in TMP because it obviates the need to cool the process water. Moreover, the treated water can then be recycled in the mill without reheating. Thermophilic biotreatment, combined with membrane filtration, is a viable method of removing organic contaminants from recirculating process waters (169).

Biopulping is the fungal pretreatment of wood chips for production of mechanical or chemical pulps. The concept of biopulping is based on the ability of some white-rot fungi to colonize and degrade selectively the lignin in wood. The fungal pretreatment can offer some apparent benefits to the pulp and paper industry, these include the potential increase in pulp yield, higher brightness ceiling, reduced chemical loads, improved effluent quality and increased pulp efficiency and mill capacity (170).

Most studies on biopulping are focused on the biotreatment steps before the mechanical pulping processes. Biomechanical and biochemical pulping of sugarcane bagasse with Ceriporiopsis subvermispora fungal and xylanase pretreatments was reported (171). The literature on combined biopulping and chemical processes is mainly restricted to the use of conventional kraft pulping (172, 173). Sycamore chips were pretreated using a cellulase/hemicellulase mixture prior to kraft pulping (176). Biosulphite pulping with selected white-rot fungi was suggested for dissolving pulp production (177). Fungal pretreatment of Eucalyptus wood can strongly decrease the amount of lipophilic extractives during chlorine free kraft pulping (178). Inoculation, aeration and heat dissipation are the key parameters for maintaining fungal activity (179).

Fungal pretreatment of wood samples before organosolv delignification were extensively studied (115, 170, 180). Fungal pretreatment offers a higher delignification rate and possibility of dissolving pulp production.

Minor methods

Alternative methods for kraft pulping with lower impact on environment have been proposed. Manufacturing of sulphomethylated pulp from oak wood was attained by selective delignification caused by sulphonation and sulphomethylation reaction of lignins during cooking. This pulping process having higher pulp yield and higher brightness than kraft pulp (181). Using of teramenthylammonium hydroxide (TMAH) (Quatam process) as a new pulping chemical into pulping technology of different lignocelluloses were studied and reviewed. Quatam process has much advantage over sulphite and/or kraft process. It produces a high quality pulp without using compounds containing sulphur, it is efficient for different lignocellulosic materials, the cooking time be shorter and the pulps have a lower lignin content and it is easy to bleach (182).

A process for producing bleached pulp from a fibrous plant material has been developed (183).

An improved pulping process comprises contacting wood chips and the like with a liquid mixture comprised of pulping liquor and a compound of the aminoalkoxylsilanes having an organic moiety from 1 to 100 carbon atoms (184).

More and more karft pulp mills are using anthraquinone, but anthraquinone is not the only effective catalyst. Even more effective as pulping catalysts are dimethylated anthraquinone, or DiMAQ, and octahydro-DiMAQ, or ODiMA. As an example, a soda pulp produced from pine with 0.05% DiMAQ

added has the same kappa number, yield, viscosity, bleaching response, and strength as a soda pulp cooked with 0.10% AQ. Kraft and polysulphide pulping showed similar results. These DiMAQ pulps had about four times more residual dimethyl anthraquinone than the amount of anthraquinone left as residual in AQ pulps. Superior survival may account for the greater effectiveness of the new catalyst, since the concentration stays high during the cook (185).

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