Pulping with Organic Solvents other than Alcohols

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Pasteado con disolventes orgánicos distintos a los alcoholes

Pastament amb dissolvents orgànics diferents als alcohols

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RESUMEN

Se hace una extensa revisión bibliográfica, con 149 citas, que refleja el estado actual del pasteado con disolventes orgánicos distintos a los alcoholes (glicoles, fenoles ésteres, ácidos orgánicos, acetona y aminas).

Palabras clave: **Glicoles. Fenoles. Ésteres. Ácidos orgánicos. Acetona. Aminas. Pasta. Papel.**

SUMMARY

A comprehensive literature review encompassing 149 references that reflect the state of the art in organosolv pulping processes involving organic solvents other than alcohols (viz. glycols, phenols, esters, organic acids, acetone and amines) is presented.

Key words: **Glycols. Phenols. Esters. Organic acids. Acetone. Amines. Pulp. Paper.**

RESUM

Es realitza una extensa revisió bibliogràfica, amb 149 cites, que reflexa l'estat actual del pastament amb dissolvents orgànics diferents als alcohols (glicols, fenols èsters, àcids orgànics, acetona i amines).

Mots clau: **Glicols. Fenols. Èsters. Àcids orgànics. Acetona. Amines. Pasta. Paper.**

1. INTRODUCTION

Traditional chemical and semi-chemical pulping processes produce large amounts of highly polluting waste water particularly those using sulphur compounds. There is an increasing demand for paper and cardboard pulp that must be reconciled with a growing environmental concern. This requires focusing research on both the use of new, unconventional raw materials (*e.g.* previously unused plant species, whether annual or perennial, and agricultural and forest residues) and the development of more effective pulping processes using less polluting, more easily recovered reagents such as organic solvents. Such «organosolv» processes can be implemented at small industrial plants requiring only modest investments with a view to obtaining of quality products with low production costs and efficient transformation of the raw materials into high-yield pulp and interesting by-products.

The fact that alcohols are the most widely used solvents in pulping processes even at pilot plants and some industrial ones led us to compile the following review of processes using organic solvents other than alcohols (*viz.* glycols, phenols, esters, organic acids, acetone, ammonia and amines), which have been investigated to a much lesser extent.

2. BACKGROUND

The earliest references to the use of organic solvents for delignification date from 1893, when Kason used ethanol and hydrochloric acid for this purpose. In the 1930s, Aronovsky and Grotner, and Kleinert and Tayenthal, conducted interesting research in this area that was expanded by Brounstein in the 1950s and Kleinert(1, 2) in the 1970s. Until then, the prevalence of traditional chemical pulping processes remained unchallenged.

The 1970s saw the emergence of initiatives towards circumventing some of the typical shortcomings of the classical pulping processes (*e.g.* unpleasant odours, low yields, high pollution, poorly bleachable pulp, the need for large investments, and a high consumption of water, energy, reagents and raw materials). These problems were addressed by altering existing processes first and developing new ones using no sulphur-containing compounds later. However, the new processes raised other problems such as the difficulty of recovering the reagents and their polluting potential(3).

In the 1980s, a number of pulping processes involving organic solvents were revisited as alternatives to the classical processes. The principal advantage of so-called organosolv processes was their highly efficient use of the raw materials. Many such processes were employed to obtain hydrolysable cellulose, phenol polymers of lignin and sugars rather than pulp proper(4-6).

In the 1990s, the kraft process faced no competition among chemical pulping processes; however, the environmental problems it raised and the large investments involved in its implementation fostered a search for still new pulping processes such a those using organic solvents, which adhere to the currently widespread principle that the raw materials should be used to obtain not only cellulose fibres, but also any other product it may give. This has been called «wood refining» by analogy with the fractionation of oil crude(7).

Since the mid-1990s, a number of processes involving organic solvents have been developed that allow the efficient delignification of both conventional and alternative raw materials(4, 5, 8-12). Current organosolv pulping processes are intended to provide not only pulp, but also lignin, sugars and other by-products in order to exploit the full potential of the raw materials (4, 5, 9, 13-17).

3. ORGANIC SOLVENTS AND CATALYSTS

Organosolv pulping processes use a wide variety of organic solvents (*e.g.* methanol, ethanol, propanol, butanol, isobutanol, benzyl alcohol, glycerol, glycol, ethyleneglycol, triethyleneglycol, phenol, acetone, formic acid, acetic acid, propionic acid, dioxane, various amines, ester, esters, formaldehyde, chloroethanol), whether pure or in aqueous solutions, and in the presence or absence of acids, bases or salts as catalysts(1, 2, 4, 5, 8-11, 18-45).

Low-boiling organic solvents (*e.g.* methanol, ethanol, acetone, ethyl acetate) can be easily recovered by distillation; by contrast, high-boiling solvents (*e.g.* ethyleneglycol, triethyleneglycol) enable operation at low pressures, but are difficult to recover(22, 46).

No individual solvent can by itself delignify all types of classical wood (hardwood and softwood) without altering cellulose(47).

The organic solvents most widely used for pulping are by far alcohols [particularly ethanol, which combines a high delignification rate under favourable operating conditions with an easy recovery(5, 48). Primary alcohols are more selective towards delignification than are secondary and tertiary alcohols. So far, the best results have been provided by ethanol. On the other hand, methanol causes smaller hemicellulose losses than does butanol. In uncatalysed processes, alcohols are used as mere solvents and only poplar and aspen wood can be efficiently processed(47).

The catalysts used in organosolv processes include mineral acids (hydrochloric, sulphuric, hydrobromic acid), organic acids (formic, acetic, propionic, oxalic, malic, salicylic, succinic, nicotinic, benzoic, citric, phthalic), salts (aluminium chloride, boron fluoride, calcium and magnesium chlorides, sulphates and nitrates, sulphites, sulphides) and various others (alkalis, ammonia, anthraquinone)(8, 22, 24, 25, 37, 43, 47, 49, 50).

Organic acids are more effective than mineral acids with softwood. Even so, dilute sulphuric acid (< 0.01 M) allows the alcohol cooking temperature for cotton stalks to be reduced from 200 to 170 ºC(4). The optimum concentration of this acid is 0.01-0.02 M. At lower concentrations, the acid is neutralized by ash and other alkaline components; at higher ones, it results in condensation of released lignin, hydrolysis of hemicelluloses, degradation of cellulose and formation of condensation products insoluble in the cooking liquor. Removing condensed lignin on cellulose fibres requires dissolving a large amount of hemicelluloses in order to open up fibre pores; however, this degrades cellulose and considerably reduces pulp viscosity. Condensed lignin is insoluble in alcohol-water mixtures and requires a more effective solvent such as acetone, tetrahydrofuran, dimethyl sulphoxide or 3-5% NaOH(5, 22, 47).

Sulphur dioxide dissolved in a cooking liquor is a weak acid; by reaction with lignin, however, it forms a sulphonic acid that is as strong as some mineral acids(2, 22).

The use of alkaline-earth salts in alcohol-water processes is the sole variant capable of thoroughly removing fibres from some plant species and providing pulp yields as high as 60% and also high viscosity values(47).

Base-catalysed processes provide good results with softwood; the resulting pulp yields and properties are occasionally better than those obtained in sulphite and kraft processes(22, 47).

4. DELIGNIFICATION MECHANISMS

Delignification by organic solvents has been ascribed to the hydrolysis of α-aryl-ether and lignin-cellulose bonds, and to conform to a *pseudo* first-order kinetics like yield losses(47). Also, the dissolution of lignin has been ascribed to the cleavage of α-aryl and aryl-glycerol-β-aryl-ether bonds in its molecule(22).

In acid-catalysed processes, the delignification rate is controlled by the hydrolysis of α-ether bonds in lignin. Such processes additionally involve complex reactions such as condensation, partial hydrolysis of β-aryl bonds, release of formaldehyde and recombination of free radicals(22).

In an acid medium, delignification is preceded by the cleavage of α-aryl-ether and β-aryl-ether bonds by H^+ ions. The H^+ concentration increases with decreasing ethanol concentration and liquid/solid ratio, and increasing operating time(51). Although the cleavage of α-aryl-ether bonds is crucial, that of β-aryl-ether bonds also plays a prominent role (particularly with hardwood). In the delignification of aspen wood with methanol, the hydrolysis of β-aryl-ether bonds in addition to α-aryl-ether bonds has a direct influence on the initial delignification rate. Moreover, the delignification process is affected by lignin condensation reactions(52).

The reactions involved in the soda-methanol process are possibly similar to those of the soda process with the exception that methanol dissolves some lignin and reduces the formation of condensation products(22). In alkaline media, the cleavage of β-aryl-ether bonds is more influential than that of α-aryl-ether bonds(53).

Phenol as a solvent reacts with lignin and increases its solubility; however, it causes the subsequent partial degradation of carbohydrates and lignin through mild hydrolysis(54).

Lignin-carbohydrate complexes and benzyl ethers react in the presence of butanediol, the addition of acetic acid favouring formation of the ethers(55).

The delignification rate in the pulping of bagasse with butanol conforms to a first-order kinetic law; in the presence of soda, however, it obeys a second-order law(56).

In the pulping of eucalyptus wood with acetic and hydrochloric acids, lignin removal results from the hydrolysis of α-aryl-ether bonds in two parallel first-order reactions, the first (faster) involving the hydrolysis of hemicellulose and the second (slower) that of cellulose. Under mild conditions (*viz.* a low temperature and HCl concentration), the second reaction does not occur and some cellulose remains unhydrolysed as a result(57, 58).

The acetosolv pulping of pine wood also involves two parallel first-order reactions, one being faster than the other(59, 60).

Delignification in the pulping of spruce wood involves two first-order steps; the first (faster) eliminates 70% of the lignin and the second (slower) the remaining 30%. Complete release of the fibres is accomplished at a yield of 57% and a kappa number of 21 (10% lignin). Delignification occurs mainly at outer walls and the central lamella, and only slightly at secondary walls. Although all lignin in the central lamella is removed at a yield of 60%, there is still some residual lignin in outer and secondary walls. Various techniques have revealed that the ease of delignification in the central lamella decreases in the following sequence of processes: catalysed organosolv > acid chlorite > neutral sulphite > acid sulphite > kraft. The sequence for secondary walls is as follows: kraft > acid sulphite > catalysed organosolv > neutral sulphite > acid chlorite. The delignification rate is controlled by diffusion in secondary walls and by physico-chemical differences between lignin in various morphological regions of plant tissues in outer walls(61).

Overall, hardwood is easier to delignify than other types of wood by virtue of its decreased lignin content, increased concentration of α-aryl-ether bonds which are easier to hydrolyse, reduced amenability to lignin condensation reactions and increased reactivity of β-aryl-ether bonds(51).

Brogdon and Dimmel(62-65) have conducted some interesting research into the different types of reactions involved in quinone pulping processes.

5. PULPING WITH ORGANIC SOLVENTS

5.1. Glycols

Nakanun and Takanti, in 1941, were the first to use ethyleneglycol as an organic pulping solvent; they employed it to obtain pulp from spruce wood. In the late 1970s, Unger found ethyleneglycol, propyleneglycol, butyleneglycol and other higher glycols to be useful for pulping purposes, especially if the raw material was previously soaked in sulphuric acid(4).

In the 1980s, Gast *et al.*(66-69) found the efficiency of ethyleneglycol to be improved by the addition of aluminium sulphate or chloride as catalyst and studied the kinetics of birch wood pulping with this solvent.

In the 1990s, Thring *et al.* studied the recovery of lignin by-products in ethyleneglycol pulping processes and the fractionation of poplar wood in a two-step process leading to the obtainment of cellulose, hemicellulose and lignin(4).

Ethyleneglycol has been used to pulp aspen, birch, beech, vine shoots, olive prunings, pine wood, forest residues and bagasse(27, 28, 31, 46, 69-74); also, ethyleneglycol-diethyleneglycol and ethyleneglycol-diethyleneglycol-soda mixtures have been used to obtain rice straw pulp(75).

Using glycol-acetic acid-water mixtures to pulp trembling poplar and pine wood revealed that this system constitutes an effective variant of glycol pulping as it allows the operating temperature and time to be reduced(72, 76).

Ethyleneglycol losses during pulping processes involving this solvent have been examined(77).

Glycerols have also been used to produce pulp(31, 78, 79). The results of glycerol pulping of wood from species such as *Ailanthus altissima* and *Spruce orientalis* can be improved by adding an alkali, which, however, causes more cellulose to be lost.

Butanediol and propyleneglycol have also be used as pulping agents(38, 46, 79), and so have ethanol-ethyleneglycol mixtures(28, 61).

5.2. Phenols

The Battelle-Geneve process uses phenol as solvent. This is the best-known process using a high-boiling solvent and hydrochloric acid as a catalyst, and is especially effective for hardwood, spruce and grassy plants; however, it usually requires a long time to process softwood and provides pulp of less quality than kraft processes. The Battelle-Geneve process has the advantage that it involves modest costs for industrial implementation and produces small amounts of waste water. However, it poses some problems associated with washing of the pulp, recovery of the reagents and detoxification of effluents containing small amounts of phenols or cresols(2, 21).

Phenol-ethanol mixtures have proved effective for pulping purposes(8). Also, some authors have used phenol cooking liquors to delignify wood chips(54, 80, 81), wheat straw(82) and *Ulex europea*(83). Phenols have been successfully used by Schweers *et al.*(35, 36) to produce pulp.

Finally, the use of cresols(84) at a 70% concentration in the presence of acetic acid at 180 °C for 2 h has been found to efficiently pulp fir and spruce wood.

5.3. Esters

The ester pulping process, which was patented by Young and Baierl, uses a mixture of water, acetic acid (the catalyst) and ethyl acetate (intended to remove lignin fragmented by hydrolysis) as cooking liquor. This process is suitable for poplar wood, but not for hardwood (eucalyptus, red oak) or softwood (pine, spruce) wood, nor when the pulp is to be subsequently bleached. The quality of poplar pulp thus obtained is in between that of sulphite pulp and kraft pulp(21). Some authors, however, have found esters to efficiently delignify both hardwood and sofwood, and to provide pulp with good mechanical properties(8).

5.4. Organic acids

The Acetocell process, which uses acetic acid and usually hydrochloric or sulphuric acid as a catalyst, has been applied to various raw materials including bagasse, birch and hardwood and softwood species, as *Eucalyptus globulus*, *Eucalyptus grandis* and *Pine pinaster*(24, 83-96). Also, it has been used at high temperatures in the absence of catalyst to obtain pulp from hardwood and softwood, and annual plants; application to spruce wood provided pulp with a high yield and a kappa index of 16-20 that was easily bleached with ozone and hydrogen peroxide, and exhibited acceptable mechanical properties(97). The kinetics of delignification of the wood was examined and kinetic parameters were correlated with the catalyst concentration(92). With acetic acid in the presence of hydrochloric acid, the delignification selectivity was found to be independent of the HCl concentration and temperature at yields above 50%(95). The delignification of red spruce with acetic acid-water, acetic acid-carbon dioxide and supercritical acetic acid-carbon dioxide mixtures was studied by examining the effect of the acetic acid concentration, pressure, temperature and time on the extent of delignification. The best results were obtained with the acetic acid-water system(98). Acetic acid has also been used in the pulping of birch and the process studied as regards the influence of a prior hydrolysis, hydrogen peroxide and other variables(99), and also in that of rice straw at atmospheric pressure(100). Recently, beech wood was pulped with acetic acid in the presence of HCl as catalyst(101).

Pulping with formic acid is a very old process. In fact, the acid was used as early as 1917 to delignify wood and cereal straw. This process has some advantages over others using organic solvents such as low operational and solvent costs, and the ability to use also low temperatures and pressures(102). It has been applied to wood from various species including *Eucalyptus globulus* and *Eucalyptus grandis*(103). The best operating conditions for E. grandis were found to be a formic acid concentration of 92%, an HCl concentration of 0.22%, a time of 90 min and a temperature of 90 °C especially with refluxing. The presence of water has been found to result in improved yields and fibre quality; the process results in the loss not only of lignin, but also of substantial amounts of hemicelluloses but little α-cellulose(102).

One variant called the Milox process uses peroxyformic acid, which forms spontaneously on mixing 80% formic acid with hydrogen peroxide(2, 104-106). This process is especially effective for delignifying birch wood in three steps, namely: treatment with peroxyformic acid at 80 °C, treatment with formic acid at 100 °C and bleaching with hydrogen peroxide to obtain pulp with 90% ISO brightness. The mechanical properties of the resulting pulp are roughly the same as those of kraft pulp. Softwood, however, provides worse results with this process. The greatest shortcoming here is that distilling the solvent for reuse produces an azeotrope of formic acid and water the concentration of which, 78%, is inadequate for the process(2). The Milox process has also been applied to fir wood(107) and alternative raw materials(108, 109). The process was initially used with various materials(39, 41, 114-116) and, more recently, with spruce(49,110), eucalyptus(111, 120), pine(121), bagasse(111), rice straw(112, 113), cane(122) and banana stalks(123).

The Formacell process, which uses a mixture of acetic acid, water and formic acid, has provided pulp with a small kappa index from beech, pine and aspen wood. The pulping of trembling aspen and birch wood requires a low temperature and a high acetic acid concentration in order to avoid hemicellulose losses(117, 118). With wheat straw, the process gives pulp with a small kappa index and a high viscosity(124).

Acetic acid containing sulphuric acid and phenols also provides good results from birch wood(119).

Finally, supercritical acetic acidBcarbon dioxide-water mixtures have also provided good pulping results(136).

5.5. Acetone

Acetone has been used as a pulping solvent by Jiménez *et al*., both individually(125, 132, 134) and in mixtures with ethanol(126, 131). Other authors have used it in mixtures with formic acid(121, 127, 128), or applied it to raw materials previously treated with water vapour(133).

One other novel process uses oxygen with acetone-water mixtures and has been applied to cotton stalks, eucalyptus and poplar. The influence of the temperature, time and oxygen partial pressure on the delignification rate was examined and oxygen transfer found to be the rate-determining step(129, 130). This process has also been applied to wood from fir and other species(76).

5.6. Ammonia and amines

Ammonia and amine based process provide high pulp yields and facilitate retention of hemicelluloses. Worth special note is that based on 1,6-hexamethylenediamine (HMDA), which provides high yields with both hardwood and softwood; the pulp, however, is difficult to bleach. Poplar wood provides pulp with properties similar to those of kraft pulp, but pine wood gives poorer results. Pulping fir wood with ammonia and acetone (or methyl ethyl cetone) provides higher yields and better properties tear index excepted than does kraft pulping. The use of ammonium sulphide with ethanol-water mixtures decreases the operational time required relative to ammonia alone; also, it gives pulp with similar properties. The process has been applied to hardwood, cereal straw and bagasse, and provides high-yield, strong pulp containing little lignin relative to pulp from softwood. Ammonia and amine based processes are subject to some problems such as the difficulty of recovering the solvents (HMDA has a high boiling point), the tendency of lignin to condense or the presence of volatile contaminants resulting from the use of sulphides(2).

Treating a bed of poplar chips with a supercritical ammonia-water mixture revealed that the extraction of cellulose, hemicellulose and lignin was dependent on the time, pressure and, especially, solvent concentration and temperature(135).

The earliest ethanolamine pulping experiments date from the late 1970s, when Wallis(30) found monoethanolamine to be more effective than diethanolamine and triethanolamine in the pulping of pine and eucalyptus wood; he obtained yields of 11-16% which exceeded those of kraft pulping and pulp with strength-related properties similar to those of kraft pulp. Wallis(137) also studied the pulping of *Pinus elliottii* with ethanolamine and obtained pulp as strong as kraft pulp but in higher yields. Ethanolamines have also been used by Kubes *et al*.(29), who applied them to spruce wood, and by Guha *et al*.(45), who used it on rice straw. Adding ethanolamine to an alkaline cooking liquor raises the lignin degradation rate(139). Finally, amines have recently been used to pulp various raw materials including beech, spruce, olive prunings, jute and cotton(140-147).

5.7. Other solvents

Formamide and dimethylformamide have been used in the cooking of bagasse and provided pulp with a high α-cellulose content, and good physical and mechanical properties(148, 149).

Recently, eucalyptus wood has been cooked with dioxane in the presence of hydrochloric acid as catalyst(150). This solvent has also been applied to other materials(37, 151-153). Finally, sulphur dioxide-ethanol-water mixtures have been used in the pulping of leafy wood(154).

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