

NEW PROCESS PRODUCES HIGHLY CONCENTRATED POLYSULFIDE
LIQUOR BY ELECTROLYSIS OF WHITE LIQUOR

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ABSTRACT

A new electrolytic oxidation process to produce highly concentrated polysulfide (PS) liquor from white liquor has been developed by the cooperative work of three Japanese companies. Advantages of this process have been verified through long-term operation of a bench-scale cell. Results obtained with the specially designed electrolytic cell demonstrate that the system is suitable for actual mill operation. The white liquor is oxidized electrically to produce PS liquor in the anode compartment, while sodium hydroxide is simultaneously formed in the cathode compartment. Because thiosulfate formation is suppressed in the electrochemical process, it was possible to produce highly concentrated PS liquor with efficient use of electrical current. Consequently, this process concentrates the sodium sulfide in the white liquor while simultaneously producing highly concentrated PS liquor and purified sodium hydroxide, thus maintaining the sulfur balance in the kraft pulping process. PS cooking of hardwoods was evaluated in a laboratory digester using the oxidized PS liquor produced in a bench-scale cell. Large increases of pulp yields and reductions in pulp kappa number were observed. These results suggest that the proposed process has the potential to improve pulp yield while reducing AOX (adsorbable organic halide). The application of this process to the modified kraft cooking process is also discussed.

INTRODUCTION

Polysulfide (PS) cooking is a well-known method of increasing pulp yield (1, 2, 3, 4). However, PS cooking has not been as widely adopted as many other methods, such as anthraquinone (AQ), because of the following two problems:

1. Low PS concentration in liquor: There is an upper limit of PS concentration because air oxidation promotes formation of thiosulfate.
2. Loss of effectiveness in modified cooking: Because PS easily decomposes at high temperature (5, 6, 7), applying PS to the high-temperature zone in the digester of modified cooking would be less effective.

PS liquors are produced commercially using air-oxidation methods, such as the MOXY (Mead oxidation) system and others, in which sodium sulfide (Na_2S) in white liquor is converted to PS by air oxidation with a special oxidation catalyst (5, 8). However, in the air-oxidation process, there is some limitation of PS concentration because of the formation of thiosulfate as a useless by-product. Consequently, the concentration of PS obtained by the air-oxidation method from ordinary white liquors is typically around 5 g/L at a selectivity of about 60% and a conversion of 60% based on the sulfide ions in the processes.

The thermal instability of PS is not a problem for conventional digesters impregnating cooking liquor at low temperature. However, thermal instability is a factor when PS liquor is used in the modified cooking process, where the cooking liquor is split and charged into the impregnation stage and also charged in additional stages at high temperature.

In order to solve the above two problems and to obtain the maximum effect of PS, the sulfur component in the cooking liquor added to the high-temperature zone should be removed and charged instead into the impregnation stage as additional PS. In other words, there is a need for a new process capable of concentrating sulfur content and thus producing a high-concentration PS from the white liquor.

We have investigated alternative systems for producing PS by oxidation without oxygen. One of the candidates for the new process is electrochemical oxidation of white liquor (9, 10). We have also developed a new electrolytic process to produce highly concentrated PS liquor from white liquor. The electrolytic cell designed for this purpose comprises an anode compartment and a cathode compartment separated by a cation-selective membrane. After removal of the suspended solids, the white liquor is fed into the anode compartment, where it is electrically oxidized to produce a PS liquor. Meanwhile, Na^+ ions flow through the membrane to form sodium hydroxide (NaOH) in the cathode compartment. Consequently, this process efficiently concentrates sulfur content in the white liquor while simultaneously producing highly concentrated PS liquor and maintaining the sulfur balance in the kraft pulping process. The electrolytic process seems well suited for use in modified cooking.

This paper compares the effects of electrically and air-oxidized PS liquors in conventional and modified kraft cooking of hardwoods. The NaOH produced in the cathode compartment is pure enough to be used either in an oxygen delignification stage or in a subsequent bleaching stage. A method of using this process for a closed system of bleached kraft pulp production is also proposed.

EXPERIMENTAL

Liquor preparation and chemical analysis

Model white liquors and model PS liquors

The model white liquors (WL) were prepared from NaOH, Na₂S solutions, and deionized water. Synthetic PS liquors were prepared by dissolving solid sulfur into the Na₂S solution at 90°C, and then model PS cooking liquors were prepared from the synthetic PS liquor and other components. Composition of the model PS liquor was determined by assuming the oxidation of the typical WL (100 g/L active alkali (AA) as Na₂O and 30% sulfidity). The conversion ratio of Na₂S was assumed to be 60% in all cases. The assumed selectivities (defined as the percentage of PS formed over Na₂S consumed) for air oxidation and electrochemical oxidation were 50% and 100%, respectively.

Mill white liquors (WL) and mill PS liquors (OL)

The mill WL (107 g/L AA and 32% sulfidity) was obtained from the Iwakuni mill in Japan. The mill PS liquor (5.5 g/L as S) produced by air oxidation was obtained directly from the MOXY plant on the same day. The air-oxidized liquor is designated as OL (orange liquor) in this paper because of the PS liquor's orange color. OL was stocked with oilslick coverage to avoid oxidation by the atmosphere.

Electrolytic PS liquors (EOL)

The EOL (electrolytic orange liquor) was electrochemically produced from the mill WL with a specially designed cell comprising an anode compartment and a cathode compartment separated by a cation-selective membrane. This cell was developed by the cooperative work of three Japanese companies. The mill WL was fed into the anode compartment, where the PS liquor was produced with high selectivity by electrochemical oxidation. The NaOH was simultaneously generated in the cathode compartment.

Chemical analysis

PS concentration in the cooking liquors (OL, EOL) was determined by using a high-performance liquid chromatography (HPLC) method (11). PS concentration was calculated from the difference of thiosulfate concentrations before and after reduction treatment with sodium sulfite. Compositions of model cooking liquors and mill cooking liquors used in this study are listed in **Table I**.

Liquor	AA, g/L (as Na ₂ O)	NaOH, g/L (as Na ₂ O)	Na ₂ S, g/L (as Na ₂ O)	PS, g/L (as S)	Sulfidity, %	Applicable Fig. Nos.
Model Liquors						
Model WL	100.0	70.0	30.0	0	30.0	2, 3
Model OL	95.5	83.5	12.0	4.6	12.6	3
Model EOL (5 g/L as S)	90.3	70.0	20.3	5.0	22.5	2
Model EOL (8 g/L as S)	84.5	70.0	14.5	8.0	17.2	2
Mill Liquors*						
Mill WL	110.0	74.4	35.6	0	32.4	7, 8
Mill OL	109.4	93.2	16.2	4.0	14.8	7, 8
Mill EOL	92.9	76.2	16.7	10.7	18.0	7, 8
* Mill liquors were sampled at different times. Concentration of mill NaOH from cathode compartment was ca. 10% (as NaOH)						

Table I. Concentrations of active alkali (AA), sodium hydroxide (NaOH), sodium sulfide (Na₂S), polysulfide (PS), and sulfidity of the model and mill liquors used in this study.

Laboratory cooking

Chips

The hardwood chips obtained from the mill were screened individually on a laboratory-scale vibration screen. The fraction passing through a 20-mm but maintained on a 9-mm square mesh was collected and then mixed in a fixed proportion of chip species. (Mixed chips consisted of *Eucalyptus sp.* and *Acacia sp.*)

Conventional cooking (CC)

All cooking experiments were performed in an air-heated rotational digester, which has a series of six stainless steel laboratory bombs. These are rotated within the air-heated chamber, thus enabling the chips to be cooked using a relatively low liquor-to-wood ratio. Each bomb contained 300 g of oven-dry (o.d.) wood chips. The liquor-to-wood ratio, the maximum cooking temperature, and H-factor were 2.5 L/kg, 160°C, and 500 respectively. The total charges of AA on oven-dry wood were varied from 14% to 16% to cover a wide range of kappa numbers. To compare KP and PS cooks at the same WL usage, AA charges for the PS cooks were adjusted to match the AA charges for the KP cooks. In all experiments involving modified cooking, SAQ (disodium salt of 1,4-dihydro-9, 10-dihydroxy anthracene) was added at a level of 0.03% on o.d. wood.

Modified cooking (MC)

Experimental procedures of the modified cooking were similar to those used in the conventional cooks. The total charges of AA on oven-dry wood were adjusted to match the level used in the conventional cooks. However, the total volume of cooking liquor was split into two charges, with 70% of the liquor added in the initial stage. Once the cooking temperature of the bombs reached the maximum temperature, the other 30% of the cooking liquor, which had been heated to the same temperature, was squeezed into the bomb with nitrogen pressure. To avoid experimental error by the squeezing operation, blank operations of squeezing were also carried out in the conventional cooks.

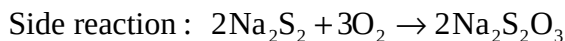
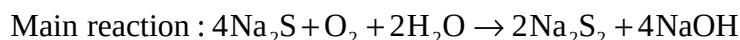
In the experiments of modified cooking using the electrolytic PS liquor, the PS liquor produced from the anode compartment was added to the initial stage. Moreover, the NaOH liquor produced from the cathode compartment was added to the additional stages after being diluted with deionized water to keep the splitting ratio of the cooking liquor volume at 7:3. Therefore, the actual splitting ratio of AA charge for the electrolytic PS was around 85:15, because of the difference in alkali makeup between PS and NaOH liquor from the cell. The final liquor-to-wood ratio was adjusted to 2.5 L/kg in all the experiments.

RESULTS AND DISCUSSION

Limitation of PS concentration with the air-oxidation method

We measured the efficiency of PS liquor production by air oxidation. **Figure 1** shows a typical result of PS generation by air oxidation with carbon catalyst (activated carbon coated with polytetrafluoroethylene) in a laboratory batch reactor. Air oxidation of sodium sulfide produces PS and NaOH, but the PS can also react with oxygen to form undesirable sodium thiosulfate, as shown in the following reactions:

Air oxidation :



Both are parallel reactions, and even at low conversion ratios of Na_2S , a relatively large amount of sodium thiosulfate was observed in the oxidized WL (so-called orange liquor). As PS concentration increases, the occurrence of the undesirable side reaction also increases. Indeed, Fig. 1 shows that the thiosulfate formation increases exponentially with the conversion ratio of Na_2S . Thus there is an upper limit of PS concentration in the air-oxidized method. As seen in Fig. 1, the concentration of PS liquors produced by air oxidation leveled off at about 6 g/L.

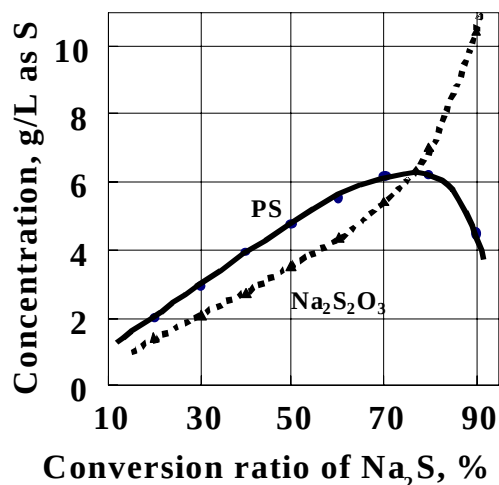


Figure 1. Concentrations of PS and $\text{Na}_2\text{S}_2\text{O}_3$ vs. conversion ratio of Na_2S in WL for PS liquors produced by air oxidation.

Higher PS concentrations would provide higher pulp yields, as seen in **Figure 2**, which shows results obtained using model EOL and model WL on *Eucalyptus sp.* However, because thiosulfate does not contribute to delignification, the thiosulfate generated at high concentrations of PS represents a loss in white-liquor active alkali. In order to improve the efficiency of PS generation, air-blowing volume is usually controlled to keep PS concentration as high as possible. However, excessive air blowing beyond the capacity of the oxidation reactor and the catalyst leads to an increase in thiosulfate formation, which significantly reduces the PS concentration and thus the active alkali.

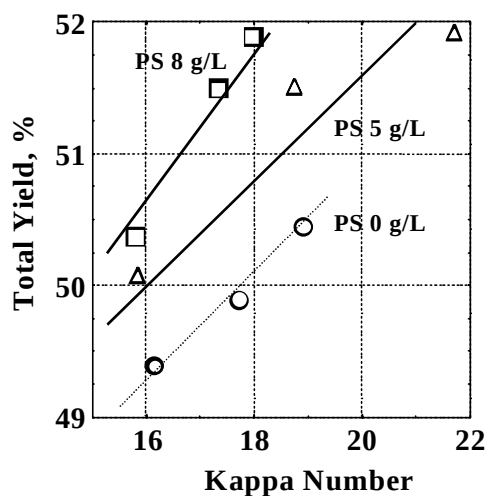


Figure 2. Total yield vs. kappa number for pulps treated with various concentrations of PS liquor.

Newer processes for producing highly concentrated PS liquors by using lime mud and oxidized manganese as oxidation catalysts have been reported (12, 13). However, even in these processes, the PS-degrading side reaction remains a problem, since they are still principally based on oxidation with oxygen. To improve the efficiency of PS production, a new method that can

suppress the side reaction and produce a higher concentration of PS liquor needs to be developed.

Loss of effectiveness of air-oxidized PS applied to the modified cooking process

The application of air-oxidized PS to the modified cooking process was investigated in laboratory cooks using the model OL and model WL. The total yield vs. kappa number for the conventional kraft cook, WL(CC), and the conventional and modified PS cooks, OL(CC) and OL(MC), respectively, is given in **Figure 3**. The total yield of OL(MC) (modified cooking with air-oxidized PS liquor) was higher than that of WL(CC) (conventional kraft cooking with WL), but lower than that of OL(CC) (conventional cooking with air-oxidized PS liquor). This indicates that applying the PS to the modified cooking reduces the effect of the PS liquor if this is the only alkali source added to the digester. It is likely that the PS liquor added to the high-temperature zone was thermally decomposed before sufficiently penetrating into the wood chips. If the untreated WL instead of the PS liquor is added to the high-temperature zone, it may be possible to avoid the thermal decomposition of PS, but the Na_2S in the WL added is still wasted. To solve such problems, a method that produces two streams of high sulfidity and low sulfidity from a WL should be developed.

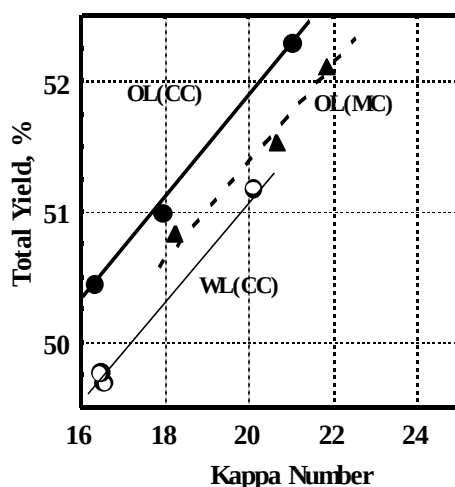


Figure 3. Total yield vs. kappa number for pulps from different processes.

WL(CC):	Conventional Cooking with the kraft cooking liquor
OL(CC):	Conventional Cooking with the air-oxidized polysulfide cooking liquor
EOL(CC):	Conventional Cooking with the electrically oxidized polysulfide cooking liquor
EOL(MC):	Modified Cooking with the electrically oxidized polysulfide cooking liquor

The modified cooking enhances the selectivity in the digester by removing additional lignin without decreasing yield. The four basic principles of extended delignification proposed by Teder *et al.* (14) are:

1. Level out the alkali concentration
2. Keep the HS^- concentration high, especially at the start of the cook.
3. Keep the dissolved lignin and Na^+ concentration low toward the end of the cook.
4. Keep the temperature low, especially at the beginning and end of the cook.

There is a recent market system satisfying three of these four basic principles (Nos. 1, 3, and 4). In an effort to satisfy No. 2, a black-liquor impregnation method and a variety of methods for concentrating sulfur content in the WL have been proposed (15, 16, 17, 18). It has been also reported that PS liquor would be more effective in increasing pulp yield if the PS was produced from a high-sulfidity cooking liquor (3). However, no one has yet developed a practical method of concentrating the sulfur content. If PS liquors are to be applied effectively in a modified cooking process, these methods are absolutely necessary. Development of such systems would satisfy the four basic principles of an ideal modified cooking process.

Electrolytic production of PS liquor from white liquor

We have investigated some efficient processes for producing PS that are not principally based on air oxidation. Methods to dissolve separated sulfur or hydrogen sulfide into the WL may be alternatives for high-efficiency production of PS. Nevertheless, these methods seem to be very complicated to remove sulfur from WL while maintaining the sulfur balance in the kraft process. Consequently, we have developed an efficient electrochemical method of producing highly concentrated PS liquors.

Figure 4 illustrates the electrolytic process. The cell, which is suitable for actual mill operation, consists of an anode compartment, a cathode compartment, and a cation-selective membrane. The WL is fed into the anode compartment and electrically oxidized, while the NaOH is simultaneously generated in the cathode compartment. The S^{2-} in Na_2S is electrolytically oxidized to form PS (S_2^{2-} , S_3^{2-} , S_4^{2-}) according to the following equation:

Electrolytic oxidation :



Side reaction : Insignificant

The above anode reaction can be controlled electrochemically by applying electric current, and the PS concentration rises with an increase of the current. The Na^+ transports through the membrane and forms the NaOH in the cathode compartment. The NaOH produced in the cathode compartment is pure enough to be used in subsequent bleaching steps.

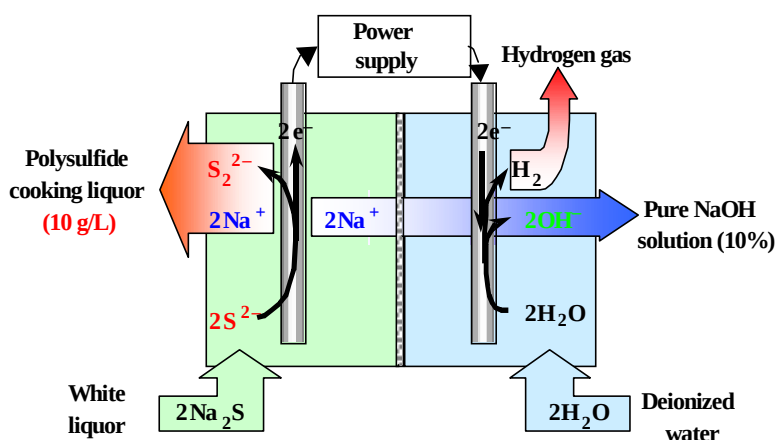


Figure 4. Schematic of the electrolytic process.

Figure 5 shows PS concentration as a function of conversion ratio of Na_2S for electrolytically oxidized PS liquors. The PS concentration increased linearly with the conversion ratio and then dropped significantly. This behavior contrasts with that observed in Fig. 1 for air-oxidized PS, where the increase in PS was nonlinear and peaked at a concentration of 6 g/L. It was possible to produce high-concentration PS liquor (>10 g/L) with efficient use of electrical current while maintaining normal sulfidity.

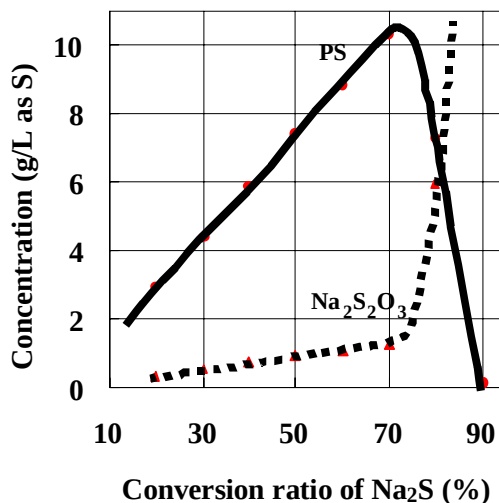


Figure 5. Concentration of PS and $\text{Na}_2\text{S}_2\text{O}_3$ vs. conversion ratio of Na_2S in WL for PS liquors produced by electrolysis.

Figure 5 also shows the formation of undesirable sodium thiosulfate. The sodium thiosulfate concentration was very low compared with the result obtained for the air oxidation in Fig. 1. These figures indicate that the electrolytic method is highly selective and produces a liquor with a higher concentration of PS (about two times greater than that for air oxidation). In the electrolytic process, the electric current is controlled to suppress formation of by-products, and thiosulfate formation is kept to a minimum. The results showed that this process produced highly concentrated PS while maintaining the sulfur balance in the kraft pulping process.

The bench-scale cell installed in the Iwakuni mill has been operating for long terms, and we are planning a pilot test with full-scale cells.

PS cooking by using the two liquors from the electrolytic cell

The proposed electrolytic process can produce high-concentration streams of PS liquor (EOL) and NaOH simultaneously. Since the EOL has high sulfur content and high PS concentration, it may be useful to increase concentrations of HS^- ion and PS in the initial cooking stage. On the other hand, the NaOH may be useful to maintain Na^+ concentration constant through the whole cooking zone, even though it is at high temperature there. **Figure 6** is a schematic figure depicting how this process could be applied a modified kraft cook.

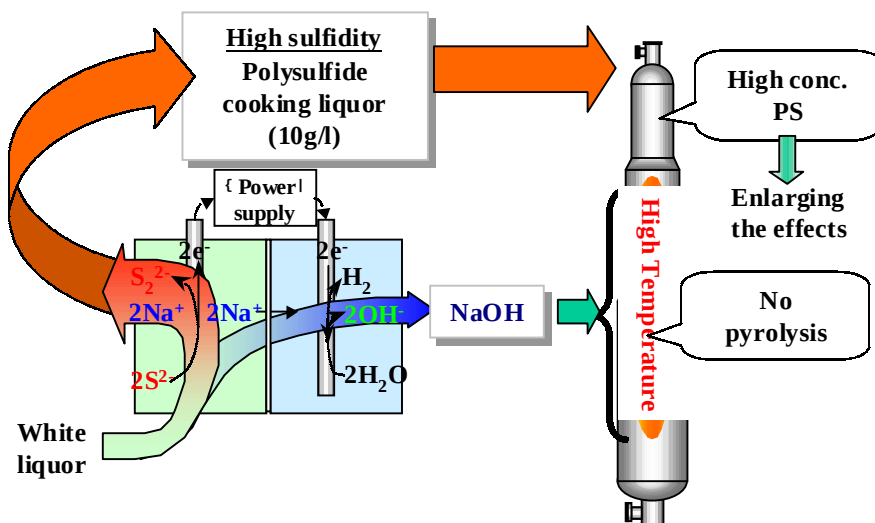


Figure 6. Schematic showing the application of two liquor streams for modified cooking.

Figure 7 shows total yield vs. kappa number, and **Figure 8** shows kappa number vs. AA on o.d. wood for the different pulping processes. EOL(CC) in Figs. 7 and 8 indicates a conventional cook where the entire charge of electrolytically generated PS liquor was applied in the digester. EOL(MC) indicates modified cooks where the PS liquor was applied in two stages. Figures 7 and 8 also present the results for conventional kraft cooks using white liquor, WL(CC), and air-oxidized PS liquor, OL(CC). As seen in Fig. 7, the yield gain for the EOL(CC) pulps was twice as large as that obtained for the OL(CC) pulps. In Fig. 8, the kappa number for the EOL(CC) pulps was lower than that for both the OL(CC) and WL(CC) pulps. These results indicate that not only large increases of pulp yield but also large reductions of kappa number can be obtained with the electrolytic process. Clearly, this process demonstrates great potential for improving pulp yield and reducing AOX at the same time.

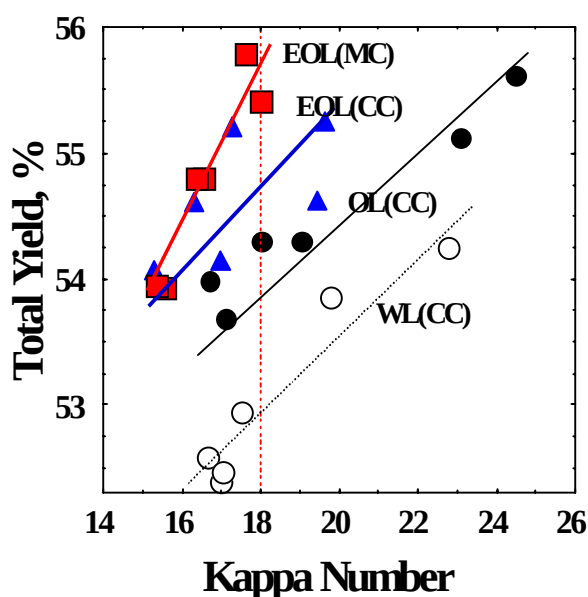


Figure 7. Total yield vs. kappa number for pulps from different processes.

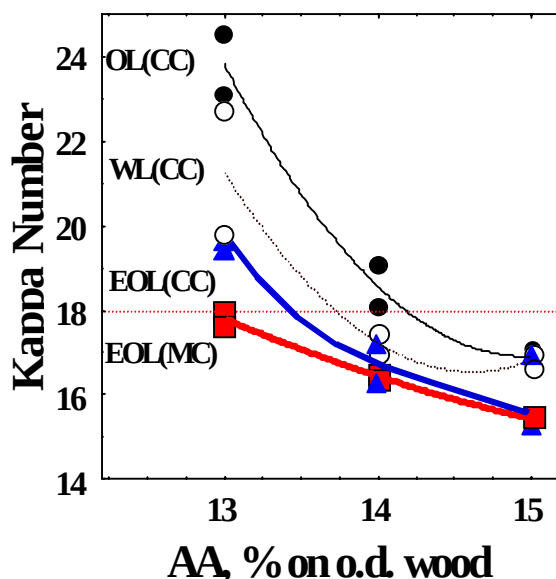


Figure 8. Kappa number vs. AA for pulps from different processes.

In the air-oxidation method, around 10 % of active alkali is lost during PS generation because of the formation of sodium thiosulfate, while in the electrolytic process the loss of active alkali does not occur normally. The reason for the significant reduction in kappa number for the electrolytic process is not clear and is being investigated.

In the case of combination cooking of the electrolytic process and the splitting charge of cooking liquors, EOL(MC), the yield gain was three times larger than that for the air-oxidized PS, OL(CC). The combination cooking also increased the reduction in kappa number. These results suggest that the electrolytic PS can be efficiently applied to the modified cooking process by utilizing both functions of PS production and sulfur concentration. In the electrolytic process, the cooking liquor from the anode compartment, with a calculated sulfidity 20% higher than that for the original WL, should be added in the first stage for the modified cooking, where high sulfidity is required. On the other hand, for the subsequent stages, where high sulfidity is not required, only NaOH should be added.

Electrolytic processing of WL has the following three features.

1. During the PS generation, active alkali and sulfur component are not wasted.
2. The efficiency of PS is improved by the modified cooking process, with NaOH added to the high-temperature zone and the thermally unstable PS liquor added to the low-temperature zone in the digester.
3. Increasing the sulfidity of the cooking liquor for the initial stage makes it possible to perform ideal modified cooking.

These three features completely satisfy the four basic principles of ideal modified cooking as outlined previously (14).

System development for a future closed system of bleached kraft pulp production using the electrolysis process

Since the only material introduced into the electrolytic cell is WL, which is recycled and reproduced in the kraft process, both the electrolytic PS liquor and NaOH can be recycled to the chemical recovery process without losing the balance of sodium and sulfur. NaOH produced in the cathode compartment is pure enough to be used not only in the additional charges in the digester but also in subsequent bleaching steps, such as oxygen delignification. In the prior art of oxygen delignification, Na_2S in WL is completely air oxidized to be a sodium thiosulfate, and this oxidized WL is used as alkali. However, this is a loss of alkali and a waste of Na_2S . If the NaOH produced with the electric cell is used as alkali for the oxygen delignification, the Na_2S in the WL can be used to concentrate the sulfidity in the initial stage. Moreover, the efficiency of the oxygen delignification is improved with the pure NaOH. Furthermore, if a TCF bleaching system — effluents of which are recyclable to the recovery boiler — is developed, the NaOH can be used for the other bleaching steps, such as peroxide bleaching and alkaline extraction. **Figure 9** shows how the electrolytic process could be used in a closed system of bleached kraft pulp production.

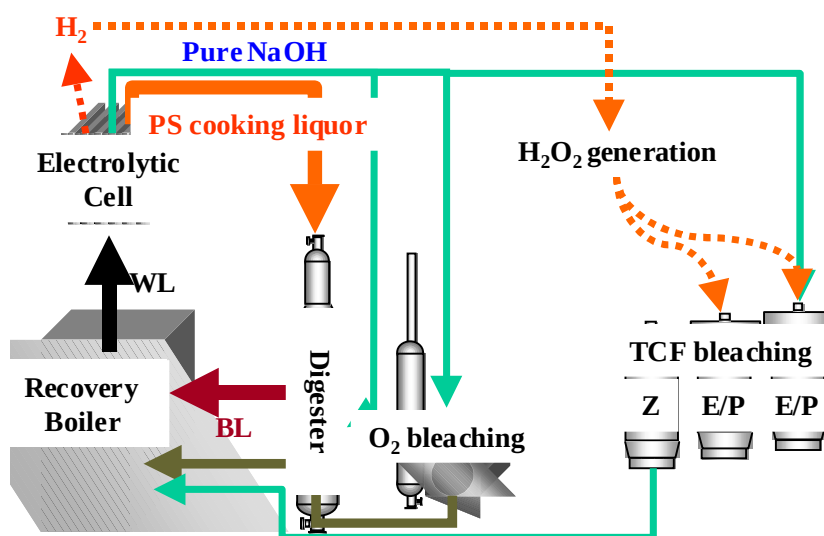


Figure 9. Schematic of a closed system for production of bleached kraft pulp with electrolytic cell.

CONCLUSION

A new electrolytic process can produce highly concentrated polysulfide (PS) liquor from kraft white liquor (WL). Because the electrolytic oxidation suppresses formation of by-product thiosulfate, it is possible to produce PS liquors with concentrations as high as 10 g/L (as S) while concentrating Na_2S in the WL and maintaining the sulfur balance in the kraft pulping process. This process simultaneously produces two streams of highly concentrated PS liquor and NaOH. The NaOH produced with the electrolytic cell was pure enough to be used either in an oxygen delignification stage or in a subsequent bleaching stage.

Laboratory cooking results showed that the electrolytic process significantly increased pulp yield and reduced kappa number. These effects were intensified when the PS liquor was used in a

modified cooking process with extended delignification. A method of using this process for a closed system of bleached kraft pulp production is also proposed.

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