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# Kraft Green Liquor Pretreatment of Softwood Chips. 1. Chemical Sorption Profiles

Weiping Ban and Lucian A. Lucia\*

*Institute of Paper Science and Technology, Georgia Institute of Technology, 500 10th Street, NW, Atlanta, Georgia 30318-5794*

The chemical sorption characteristics of typical kraft mill green liquor (GL) chemicals into loblolly pine softwood chips were analyzed as part of a larger study investigating new, low-capital kraft pulping modifications. Specifically, the sorption profiles of hydroxide (alkali), hydrosulfide, and carbonate ions from industrially supplied GL into wood chips were compared and contrasted. The influence of pretreatment conditions, such as GL charge, pretreatment temperature, and time, were the main variables controlled to assess their influence on chemical sorption profiles. The results indicated that significant differences existed for the sorption characteristics of each chemical component. Alkali and sulfide showed more affinity for wood components than carbonate during the pretreatment. Thus, the order of decreasing sorption rates was as follows: alkali and sulfide, followed by carbonate. In general, it was found that pretreatment of wood chips with GL under a moderate GL charge and higher temperatures provides the maximum sulfide concentration in wood chips, although extended times are not necessary.

## Introduction

Environmental and societal pressures have been important drivers for the U.S. pulp and paper industry to seek modified pulping and bleaching technologies to decrease the total amount of effluent produced in their operations. In addition, global competition has forced the industry to improve its overall performance in order to maintain its operations. Viable solutions to address environmental and competitive pressures are to maximize the productivity of the pulping operations by decreasing the total chemical consumption during pulping and bleaching and to improve yields or final pulp properties. Extended delignification for pulping has been rigorously investigated as one of the main ways to produce lower lignin pulp, minimize the demand of bleaching chemicals, and provide improved pulp properties and yields.<sup>1-3</sup>

Many pulping operations in North America have promoted the extended pulping concept through modified kraft pulping technologies such as rapid displacement heating and modified continuous cook. The success of these latter technologies resides in their ability to address both environmental compatibility and capital efficiency while simultaneously maintaining delignification selectivity. Pulp yield selectivity requires that pulping be performed with increased lignin removal without incurring significant carbohydrate loss. Satisfaction of the extended pulping concepts has received considerable justification by the modifications introduced into the kraft pulping operations during the past several decades.<sup>4</sup> The basic principles that remain in effect today, as suggested by Hartler and Teder to improve kraft cooking, are as follows:<sup>5-8</sup>

(i) The concentration of hydroxide ion should be low at the beginning of the initial cooking stage and level out during the cooking period.

(ii) The concentration of sulfide should be as high as possible initially and at the start of bulk delignification.

(iii) The concentration of dissolved lignin and sodium should be low, especially during the residual delignification phase.

(iv) The temperature should be kept low, especially toward the beginning and end of bulk delignification.

The high concentration of sulfide ion in the initial cooking stage is critical to improving the overall efficiency of delignification. Our work in this area provides evidence that pretreatment of wood chips with a high sulfide-containing liquor such as mill green liquor (GL) is a highly effective method to ensure a required high sulfidity before the beginning of bulk delignification. GL is an extremely rich source of sulfide in kraft pulp mills, possessing a sulfide ion to hydroxide ion ratio of nearly 2:1.

Fundamental research in this area has demonstrated that modified kraft pulping through the use of GL pretreatments is beneficial for desirable pulp properties and qualities such as higher pulp yields, lower EA consumption in the digester, increased delignification, and improved pulp strength properties.<sup>9-16</sup> Klevinska and Treimanis<sup>10,11</sup> investigated the influence of variable liquor-to-wood ratios on the efficiency of pulping as a function of GL pretreatment and discovered that higher pulping selectivity and kinetics of delignification were achievable. Svedman and Tikka<sup>12</sup> have demonstrated that the use of GL or mother liquor (supernatant) from crystallized GL allows for a decrease in the consumption of pulping white liquor and bleaching chemicals and a concomitant increase in fiber strength. The influence of pretreatment conditions, pulping additives, and total alkalinity during the application of GL for the impregnation of wood chips has received attention recently by various researchers.<sup>9-12</sup>

The general uptake and retention process of a component by a solid can be referred to as sorption, a mass-transfer phenomenon which can be characterized as either (or both) physical entrapment (absorption) or (and) chemical bonding (adsorption) of the component in the solid. Throughout this research, the generalized

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uptake and retention process will be simply referred to as sorption. The sorption of pulping chemicals such as sulfide-containing impregnating liquors into the wood chip is a critical issue for clarification to facilitate improvements in kraft pulping. Olm et al.<sup>17–19</sup> have indicated that the highest delignifying selectivity was obtained at a concentration ratio of sulfide ion to hydroxide ion that was in excess of 6 while achieving high sulfide sorption in the wood chips. They also found that higher temperatures in conjunction with lower hydroxide ion concentrations favored pulping selectivity enhancements. A difference between alkali and sulfide sorption in wood was found to exist, especially after impregnation. It was found that the alkali concentration inside the wood chips was higher than that observed in the impregnating liquor; however, no significant difference between the sulfide concentration inside wood chips and that in the liquor was found after impregnation.

The objective of the present study was to investigate and quantify the sorption characteristics of individual chemicals (hydroxide, sulfide, and carbonate) from GL into wood during pretreatment to provide a basis for continued efforts in this area. Furthermore, the effects of pretreatment conditions on chemical sorption were investigated and are presented. A complete assessment of the sorption profiles of the GL chemical constituents was the final outcome of these studies.

Experimental Section

**Wood.** Loblolly pine chips were obtained from the industrial member of IPST. 2–3 × 20 × 25–30 mm were the chip dimensions that were used for all experiments. The moisture content of the wood chips was 45.25%. All bark and knot wood chip samples were removed before pulping was begun.

**GL.** The GL used in all of the described experiments was provided from the same industrial sponsor. It was titrated and found to contain 19.18 g/L of NaOH, 31.70 g/L of Na<sub>2</sub>S, and 107.89 g/L of Na<sub>2</sub>CO<sub>3</sub> (all concentrations are expressed relative to Na<sub>2</sub>O), which was analyzed by the ABC titration method as modified in-house (see TAPPI Method T 626 cm-85).

**Pretreatment.** Pretreatments were carried out in a multiunit stainless steel bomb system that was home-built at the pulping and bleaching labs of IPST. Eight different samples were able to be pulped simultaneously. The pretreatment temperatures were controlled by the computer-programmed oil bath, and temperature ramps were on the order of 1.2 °C/min. Each bomb could accommodate a volume of 500 mL having approximately 50 mL of headspace and containing 50 g of wood chips (oven-dried weight). Pretreatment conditions are as follows: the ratio of pretreatment liquor to wood chips is 4:1, the GL charge on wood chips varied from 0.5 to 2.0 L/kg, the GL concentration varied from 12.5 to 50%, pretreatment temperatures were 90 and 120 °C, and the impregnation times were 60 and 120 min. Each sample was duplicated with selected triplicate runs, and the average of the data was used in the results.

**Analysis.** The sulfide alkali content in the pretreatment liquors was measured by the ABC titration method. The chemical sorption was determined by the difference of chemical content in the pretreatment liquor before and after pretreatment. All chemicals that were absorbed into wood or present in the pretreatment

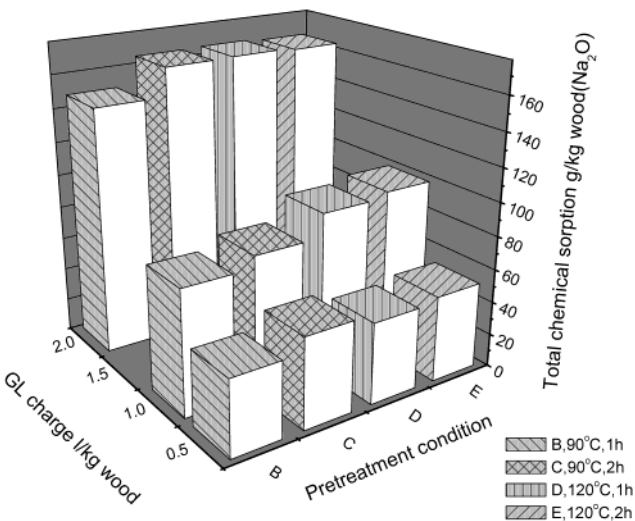


Figure 1. Total concentration of chemicals sorbed into wood chips under variable GL charges.

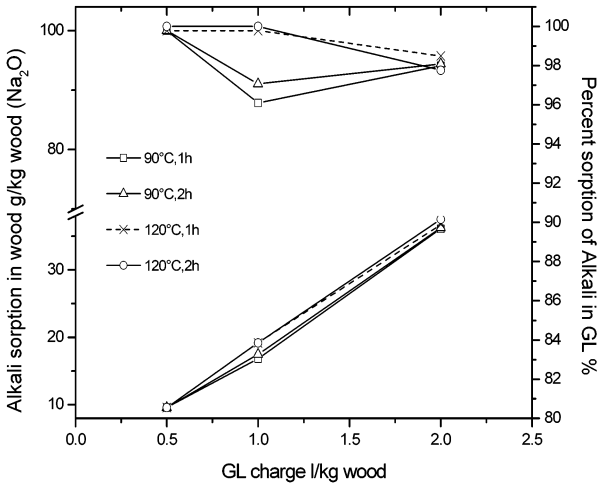


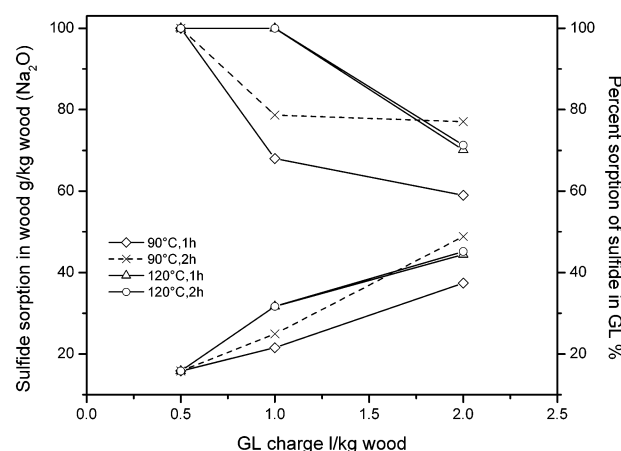
Figure 2. Effect of the GL charge on alkali sorption into wood chips.

liquor were expressed according to the Na<sub>2</sub>O convention by conversion of OH<sup>−</sup>, S<sup>2−</sup>, and CO<sub>3</sub><sup>2−</sup> species for convenience of comparison.

Results and Discussion

**Influence of the Total GL Charge on Chemical Sorption.** The main inorganic components in GL, viz., the sulfide, hydroxide (alkali), and carbonate anions, are sorbed into wood chips when treated. Figure 1 shows the total anion sorption profile of the inorganic chemicals in the wood chips as a function of the pretreatment time, temperature, and GL charge. In general, the chemical sorption increased as the GL charge increased on wood chips. Additionally, the higher temperature provided a linear sorption profile in the wood for all of the chemicals. Two important facts are illustrated: (1) a similar chemical sorption trend is shown by pretreatment at the same temperature, and (2) when the minimum (0.5 L/kg) and maximum (2.0 L/kg of wood) GL charges were applied in pretreatment, similar chemical sorption trends were obtained under different pretreatment temperatures and times.

In Figure 2, the relationship of the GL charge during pretreatment on the alkali sorption is illustrated. In all pretreatment cases, the sorption of alkali increased

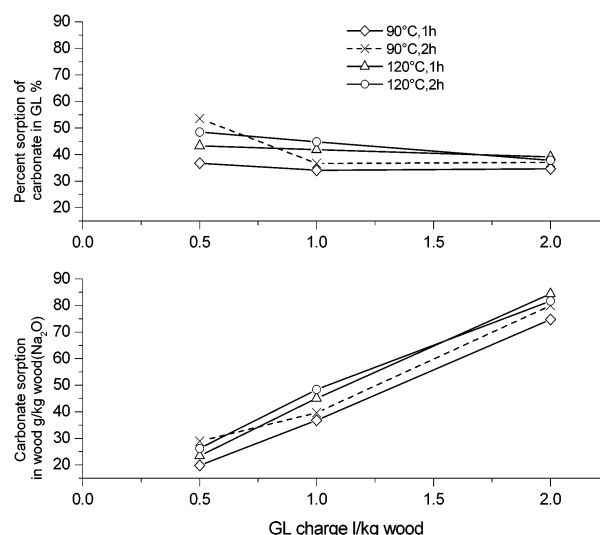


**Figure 3.** Effect of the GL charge on sulfide sorption into wood chips.

linearly with increasing GL charge. The higher temperature was much more influential for approaching the limit of the wood chip alkali sorption as a function of the total GL charge (19.18 g/L of GL). Clearly, however, the maximum alkali uptake was reached at the lower pretreatment time of 1 h at 120 °C, indicating that longer pretreatment times are not necessary for increases in alkali consumption. When the GL charge varied from 0.5 to 2.0 L/kg at 90 °C, the ratio of alkali sorption from the pretreatment liquor decreased from 100% to 75%, whereas at the higher temperature of 120 °C, it was completely or nearly completely sorbed from the pretreatment liquor. These results are in line with what was observed for the alkali uptake profiles in the wood chips.

In general, sulfide sorption into the chips increased with increasing GL charge, but the sorption differences are prominent based on the pretreatment conditions, as shown in Figure 3. With increasing GL charge, the sorption of sulfide at the lower temperature (90 °C) exhibited a similar increase but ramped up slowly with increasing time. At the higher temperature (120 °C), the sulfide sorption efficiency into the wood chips was greater than 50% of what was observed at the lower temperature. In fact, unlike the lower temperature cases, the uptake of sulfide was observed to be the same for the two different treatment times. This latter result clearly demonstrated that longer pretreatment times are not necessary for the maximum extent of sulfide sorption, a result not unlike that observed by Olm et al. (vide infra). Likewise, as indicate previously, this result applies equally well to the extent of alkali sorption. A limiting time for alkali and sulfide uptake appears to be 1 h while maintaining the pretreatment temperature at 120 °C.

As shown in Figure 3, the ratio of sulfide sorption sharply decreased using GL charges from 0.5 to 1.0 L/kg under low temperature and short time and further decreased at a 2.0 L/kg charge. At a 1.0 L/kg GL charge under higher temperature (120 °C), the sulfide was fully sorbed from the pretreatment liquor, but only 70% from the pretreatment liquor was sorbed at a GL charge of 2.0 L/kg. Not surprisingly, these results indicate that virtually the same sorption of sulfide was found at 120 °C for both 1 and 2 h of pretreatment time. This implies that the sorption of sulfide approached a saturation point in the wood for pretreatment at higher temperatures but diminished at higher GL charges. The rationale for the upper ceiling of sulfide sorption in wood



**Figure 4.** Effect of the GL pretreatment parameters on carbonate sorption into wood chips.

chips is presently unclear and is currently under investigation.

Figure 4 illustrates carbonate sorption during pretreatment. A linear increase of carbonate sorption with the GL charge increase was observed, and also the pretreatment temperature and time showed a limited influence; i.e., higher temperatures and longer times increased the carbonate sorption to a small degree. However, an interesting phenomenon is that the sorption ratio of carbonate remained unchanged with the GL charge increase under various pretreatment conditions. This result demonstrates that carbonate displays a distinctively different behavior relative to alkali and sulfide.

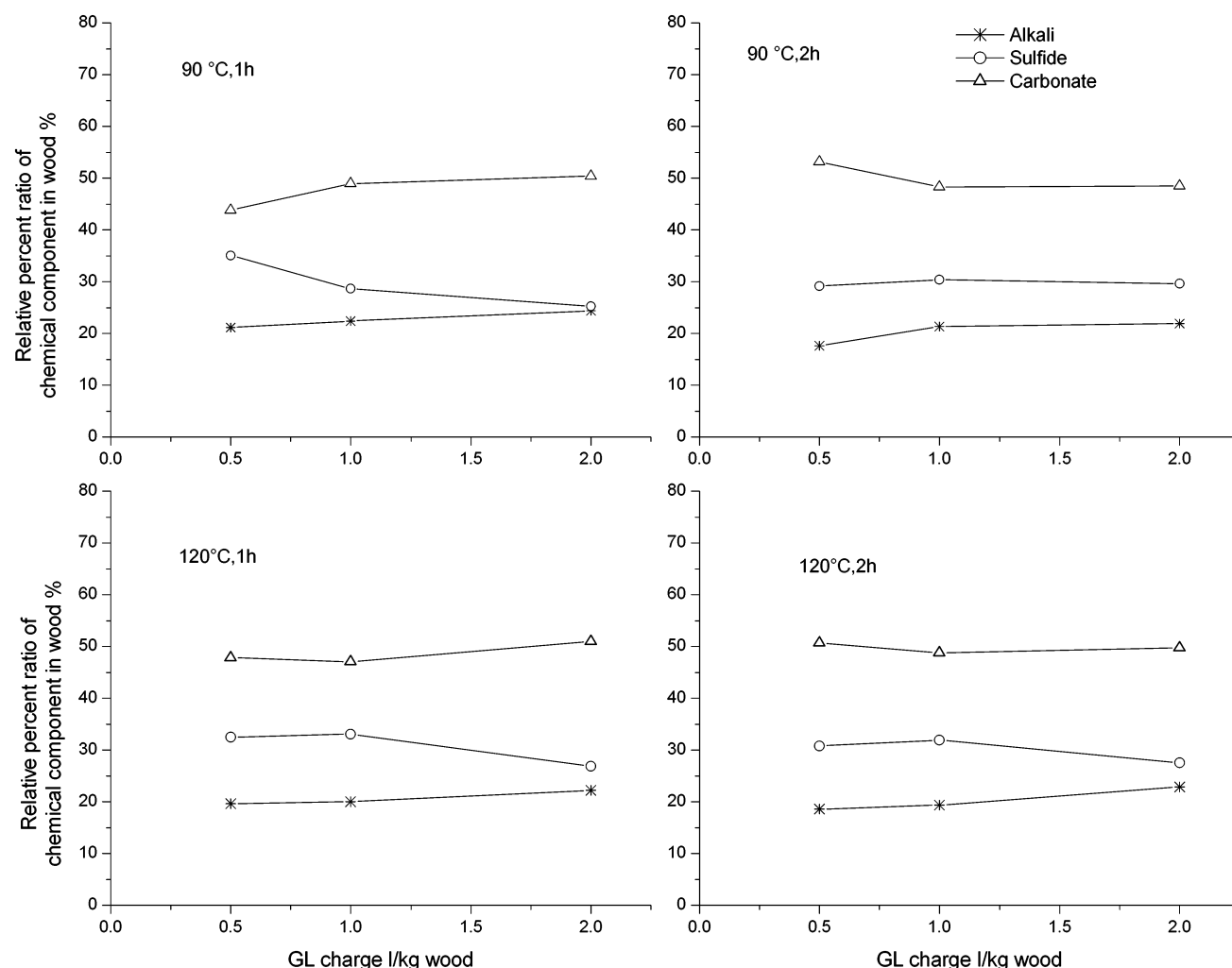
**Chemical Sorption Characteristics and Compositional Changes during GL Pretreatment.** The process of pretreating wood chips is characterized by liquor penetration, chemical component diffusion, and chemical reactions. After pretreatment, the chemicals in the GL begin to diffuse into the wood chips along with a substantial amount of water. Although the total chemical sorption profiles are not notably different as shown in Figure 1, the individual chemical components have been shown to display variable sorption characteristics under differing pretreatment conditions. Figure 5 displays the relative percent contents of chemical components absorbed into wood chips as a function of the GL charge on wood chips during a pretreatment process that employed two different times and temperatures.

In general, after GL pretreatment, the relative sorption profile of chemicals in wood chips was as follows: 50% carbonate, 30% sulfide, and 20% alkali (based on Na<sub>2</sub>O) as compared to the composition in the original GL, which was 68% carbonate, 20% sulfide, and 12% alkali. The higher alkali concentration is probably due to the rapid uptake of alkali in a neutralization reaction with the sugar acids in wood. However, the increase in the sulfide concentration from the pretreatment suggests that it has a stronger affinity to the wood components.

The pretreatment conditions also impact the sorption phenomena to some extent. When the pretreatments are conducted at lower temperature and shorter time, the relative percent composition of sulfide in the wood chips decreases, whereas the percent composition of hydroxide and carbonate increases as a function of increasing GL



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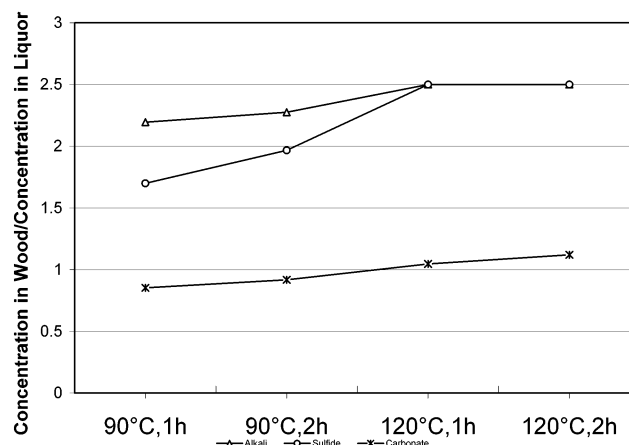


**Figure 5.** Changes of the relative contents of alkali, sulfide, and carbonate absorbed by wood chips under variable GL charges.

charge. This indicates that a slower sorption of sulfide at lower temperature and shorter time results in a decrease in the sulfide content, as shown in Figure 3. Extending the pretreatment time under the lower temperature increases the sulfide ratio in wood, but beyond a 1.0 L/kg GL charge, little change in the chemical sorption profile was observed.

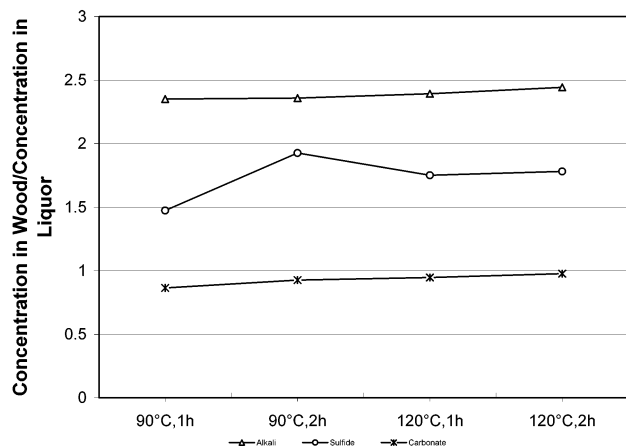
Under the high pretreatment temperature (120 °C), as opposed to low temperature, the chemical sorption was influenced only at high GL charge. The sulfide percent composition decreased as the GL charge increased from 1.0 to 2.0 L/kg. This result can be ascribed to the gradual sulfide saturation in the wood chips followed by a concomitant decrease in its activity. In the meantime, a decrease in sulfide resulted in higher relative ratios of alkali and carbonate.

Figures 6 and 7 illustrate the comparison of the chemical concentrations between liquor in wood and in the original pretreatment liquor, expressed as the ratio of the concentration of the chemical component in liquor in wood chips to that in the original pretreatment liquor. Changes in each chemical component are easily gleaned from the ratio of the chemical concentrations in both liquors. The higher ratios for alkali and sulfide suggest that chemical reactions are occurring during the pretreatment. This receives support from the different sorption characteristics of each chemical component in GL. When alkali sorbed into wood chips, it underwent a neutralization process with sugar acids; thus, it was

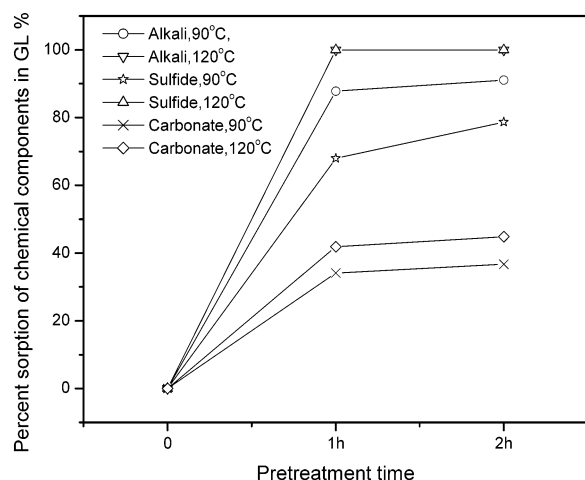


**Figure 6.** Change in the ratios of chemical component concentration of liquor reserved in wood chips to the original pretreatment liquor under various pretreatment conditions (1.0 L/kg GL charge).

continuously consumed during the pretreatment and resulted in a higher sorption. The behavior of sulfide in the pretreatment is more complex; the high difference of the sulfide concentration between wood chips and the original pretreatment liquor confirms that sulfide displays a more pronounced sorption. Unlike alkali, sulfide should not display much chemical reactivity with the chemical components in wood at the conditions employed during GL pretreatment. The likely rationale for its behavior can be explained by its affinity for wood



**Figure 7.** Change in the ratios of chemical component concentration of liquor in wood chips to the original pretreatment liquor under various pretreatment conditions (2.0 L/kg GL charge).

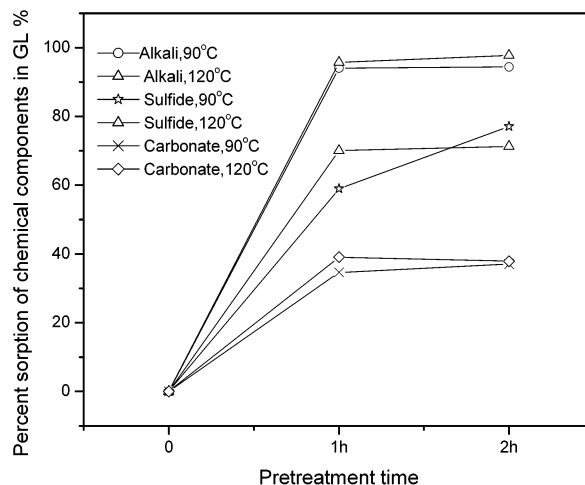


**Figure 8.** Sorption rate of chemical components (1.0 L/kg GL).

components. Werthemann<sup>20</sup> suggested the concept of “xylophilicity” to elucidate the effect of sulfide in the kraft cook, but it has not received wide acceptance. The mechanism of sulfide reactivity during the GL pretreatment still requires further investigation.

The current research also demonstrates that, despite varying the prevailing pretreatment conditions, the carbonate concentration in the wood chips is virtually unchanged compared to its original concentration in the pretreatment liquor. *The unchanged concentration gradient of carbonate (as opposed to alkali and sulfide) indicates that carbonate is an inactive component and displays no obvious chemical or physical effect during the process of pretreatment.*

**Behavior of Inorganic Components in GL during the Pretreatment.** Based on the time changes in the concentration of each chemical component in the pretreatment liquor, the rate of component sorption during pretreatment was compared and is shown in Figures 8 and 9. Similar rates of change were observed by using GL charges of 1.0 or 2.0 L/kg. Overall, these experimental results demonstrate general regularities in the sorption rates of GL chemicals. First, maximum sorption for all of the GL chemicals was completed within 1 h, whereas longer pretreatment times did not substantially contribute to an increase in chemical sorption, even at higher pretreatment liquor concentrations. Furthermore, each chemical component displayed



**Figure 9.** Sorption rate of chemical components (2.0 L/kg GL).

a different sorption rate, with the highest to lowest being hydroxide, sulfide, and carbonate, respectively.

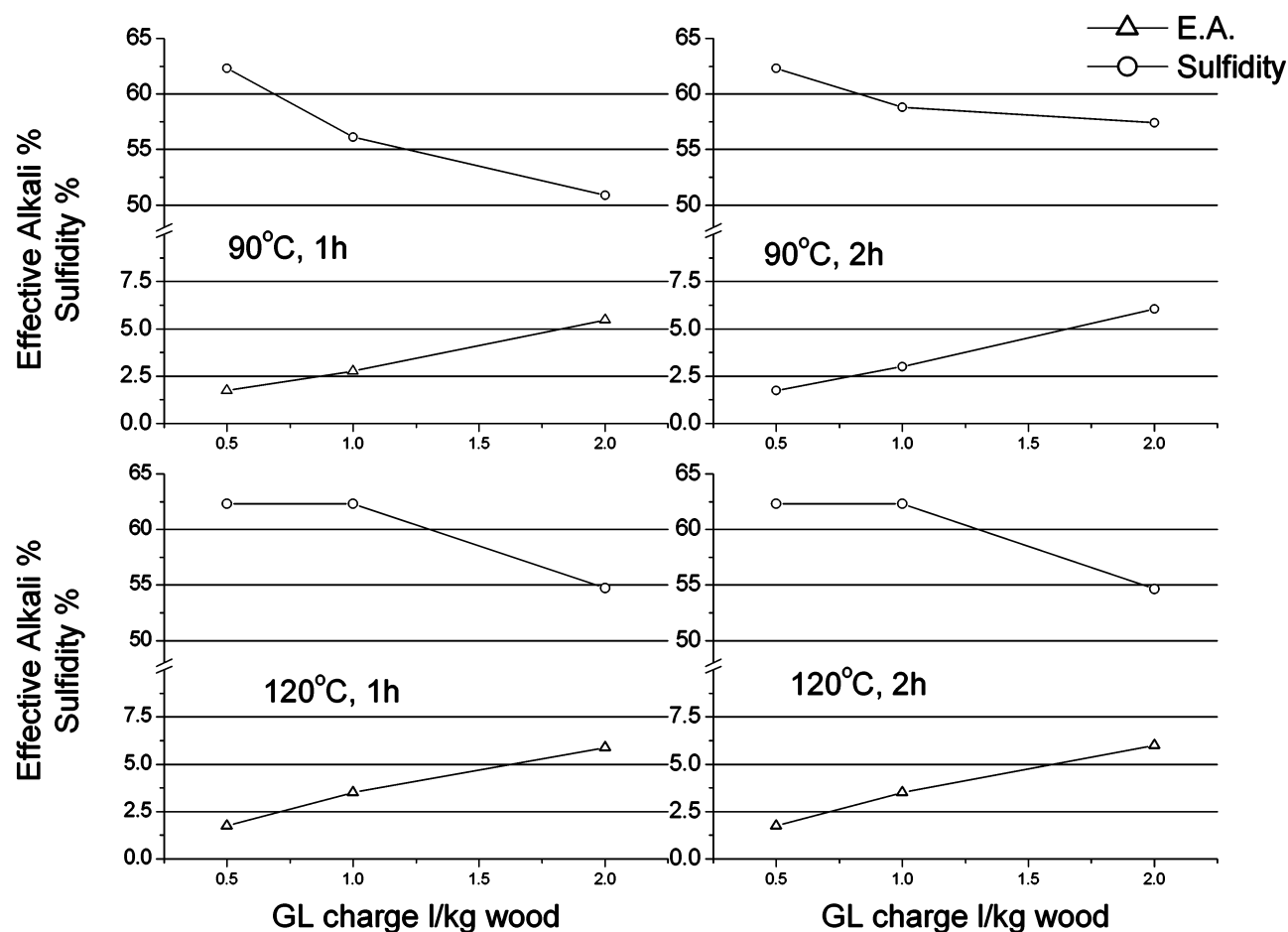
On the basis of a comparison of the concentration of each chemical component sorbed by the chips with what is remaining in the pretreatment liquor, hydroxide, sulfide, and carbonate clearly exhibited different sorption properties. The chemical that provided the most efficient uptake by softwood chips was hydroxide, closely followed by sulfide, and ultimately ending with carbonate.

In kraft pulping, effective alkali (EA) or active alkali (AA) and sulfidity are commonly used to describe the concentration profiles of alkali and sulfide. Thus, to evaluate the impact of GL pretreatments on the subsequent kraft cook, Figure 10 illustrates the changes in EA and sulfidity under various conditions. The AA linearly increased with increasing GL charge at all pretreatment conditions because the wood chips, as described previously, have a strong capacity to sorb alkali, whereas the pretreatment temperature and time had no obvious impact on the EA change. Within the range of GL usage (0.5–2.0 L/kg), it was found that after the pretreatment 2.0–6.25% EA was sorbed into the wood chips, which is directly proportional to the GL charge. However, the GL usage showed a negative correlation to the change in sulfidity. It was found that higher GL charges did not result in higher sulfidity; in fact, when the GL charge was varied from 1.0 to 2.0 L/kg, the sulfidity decreased from 62.5% to 55% at 120 °C. *Thus, the concept of high sulfide content in wood before a kraft cook should be distinguished as (1) high sulfide content and (2) high sulfidity. Both of these terms may or may not be identical depending on the chemical composition of the pretreatment liquor and the pretreatment conditions.* When GL was used for pretreatment, moderate application (1.0 L/kg) ensured both high sulfide sorption and high sulfidity in the wood chips before cooking.

## Conclusion

Increasing the pretreatment temperature and time, as well as the GL concentration, can increase the total amount of chemicals sorbed by softwood chips during GL pretreatment. Approximately a level of 45–55 g (Na<sub>2</sub>O)/kg (wood) of chemicals could be sorbed at a 12.5% concentration of pretreatment liquor (corresponding to a 0.5 L/kg GL charge on wood), 75–100 g (Na<sub>2</sub>O)/

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**Figure 10.** Changes of EA and sulfidity during the pretreatment.

kg at a 25% concentration of pretreatment liquor (1.0 L/kg GL charge on wood), and 150–165 g (Na<sub>2</sub>O)/kg at 50% (2.0 L/kg GL charge on wood). In most cases, when the pretreatment was conducted using GL at a level of 1.0 L/kg, both the alkali and the sulfide were completely absorbed, whereas at 2.0 L/kg GL, the sorption of alkali was nearly complete, but only about 70% of sulfide was absorbed. The GL usage and pretreatment conditions have important effects on chemical sorption, especially on sulfide sorption; for example, pretreatment conducted under higher temperature with a moderate GL charge benefits higher sulfide sorption. Long pretreatment times are not necessary to improve chemical sorption.

In general, after pretreatment with GL, the chemical composition in wood chips relative to the GL is approximately 50% carbonate, 30% sulfide, and 20% alkali, which varied as a function of pretreatment conditions, especially GL charge and pretreatment temperature. However, on the basis of the comparison with the original concentration in GL, sulfide showed a higher affinity for wood, but carbonate appeared to be an inactive component.

On the basis of the concentration of each chemical in the original pretreatment liquor, they can be ordered according to their sorption rate as follows: sodium hydroxide, the strongest, sulfide, slightly weaker, and sodium carbonate, the weakest.

During the pretreatment, the high sulfide sorption does not always correlate with high sulfidity. Nonetheless, a high sulfidity can be achieved using a lower GL charge (0.5–1.0 L/kg). However, AA linearly increased with increasing GL charge, but the final effect on the

postcook from both AA and sulfidity in the pretreatment requires further investigation.

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