

# Steam Explosion Pulping of Aspen Pretreated with Alcohol Mixed with A Non-Sulfuric Chemical

Aziz Ahmed, Bohuslav B. Kokta and Felix Carrasco\*

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**ABSTRACT:** - A process based on alcohol to produce ultra-high-yield pulp as an alternative to conventional processes has been studied in our laboratory at the University of Quebec at Trois-rivieres. Aspen wood chips were impregnated with aqueous ethanol in the presence of a second chemical as NaOH, Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub> or MgCl<sub>2</sub> at various concentrations. Cooking was carried out with saturated vapor at high temperature (190 °C) for short residence time, mostly 2 and 4 min. The ratio of ethanol to aqueous solution used in this study was 50 : 50, 30 : 70, 15 : 85 and 0 : 100. The influence of the nature of the chemicals, their concentration and cooking time on pulpyield and properties was also examined. The presence of NaOH in impregnation solutions consisting of aqueous alcohol, or water, remarkably improved the mechanical properties of the resulting pulp. The presence of alcohol appears to protect pulp brightness. For a similar yield range, pulp obtained with ethanol + NaOH provided superior pulp properties in comparison to the pulp obtained with ethanol + Na<sub>2</sub>CO<sub>3</sub> or NaHCO<sub>3</sub> or MgCl<sub>2</sub>. The pH of the impregnation liquor influenced the pulp yield as well as pulp properties.

**KEY WORDS:** - Alcohol pulping. Ultra-high-yield pulping. Non-sulfuric Pulping. Pulp yield. Paper properties. Pulp brightness. Chemical impregnation. Aspen wood. High-temperature cooking. Explosion pulping.

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## INTRODUCTION

Today, a great deal of research effort is aimed at developing new pulping processes that are cost effective and less damaging to the environment. Among the many approaches to environment-friendly pulping processes, alcohol pulping processes are slowly advancing toward commercialization through numerous lab and pilot-scale studies. Among these

Centre de recherche en pates et papiers  
Universite du Quebec a Trois-Rivieres  
C.P. 500, Trois-Rivieres (Quebec)  
Canada G9A 5H7

\* Departament d'Enginyeria Quimica  
Universitat de Girona  
17003 Girona  
Spain

processes, Alcell, organocell, MTU. And ASAM processes are very close to commercialization. The alcell process (1, 2) includes the pretreatment of chips with low pressure steam followed by extraction of pretreated chips with a liquor consisting of 60% alcohol at a temperature of 190 to 200 °C. In the modified single-stage organocell process (3), wood chips are cooked with alkali, methanol and catalytic amounts of anthraquinone at 160-170 °C. The MTU process (4) includes pretreatment of chips in aqueous alcohol with NaOH followed by pulping in a liquor of ethanol and water, catalyzed by SO<sub>2</sub> at 140-150 °C. In the ASAM process (5), wood chips are cooked with alkalisulfite (mixture of sodium hydroxide, sodium sulfite and sodium carbonate) and methanol in anthraquinone which serves as a catalyst at temperatures of 160-170 °C. All of these processes produce low-yield chemical pulps aimed at replacing the well-known kraft pulps for environmental and economic purposes. Until now, research efforts concerning alcohol and non-sulfuric pulping were entirely devoted to low-yield chemical pulping.

Our research group at the University of Quebec at Trois-Rivieres has been working since 1990 to develop a non-sulfuric high-yield/ ultra-high-yield pulping process based on alcohol for environmental and economic reasons (6-9). The new process will be an alternative to conventional CTMP and CMP processes. Recently, we reported (8, 9) and ultra-high-yield pulping process where chips were impregnated with methanol-aqueous NaOH, and then cooked at vapor phase at 190 °C for short periods of time. The resulting pulp quality was equivalent to that of other ultra-high-yield pulps prepared with sulfur-based chemicals in conventional processes.

This paper deals with the preparation of ultra-high-yield pulp from aspen chips by using ethanol in the presence of various non-sulfuric chemicals. The effect of ethanol concentrations on pulp properties was also studied.

## EXPERIMENTAL

### Materials

Aspen (*populus tremuloides*) used in this study

was fresh cut and supplied in log form. The aspen log was debarked and chipped at "La Station Forestiere Duchesnay, Quebec". The chips were shredded at the "Centre de Recherche et Developement, Consolidated - Bathurst Co., Grand-mere, Quebec" and screened in our laboratory. The average chip size after screening was as follows : length = 2.5 - 3.7 cm., width = 1 - 2 cm. and thickness 1 - 9 mm. with maximum distribution at 5 mm. The chips were shredded to enable better penetration of the impregnation liquor. The chemicals used in this study were of a technical grade supplied by Aldrich Chemical Co.

### Impregnation

150 g. of chips with a 50% moisture level were mixed in a plastic bag with 150 g. of a solution made up of ethanol and a NaOH aqueous solution. The chips were also impregnated with a Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub> or MgCl<sub>2</sub> · 6H<sub>2</sub>O aqueous solution and ethanol. The impregnation of chips was conducted at 25 °C for 48 h.

### Cooking

Cooking was carried out in a 340 ml. batch reactor by using saturated steam. The impregnated chips were cooked with saturated steam at 190 °C for 2 to 4 min. Cooking was preceded by one minute steam flushing at atmospheric pressure to remove the air from the reactor in order to reduce the oxidation reaction. At the end of the cooking period, the pressure of the reactor was released suddenly, and the cooked chips were washed with tap water. Washing was performed to ensure that the chips contained no initial chemical or products liberated during pulping. The washed chips were then stored in a cold room and refined later on.

### Yield Measurement

Cooked chips obtained from 75 g. (o.d. basis) of the initial chips were washed with one liter of tap water and subsequently defibrated for 90 s in a laboratory blender at a 2% consistency level. The pulp was then washed again with water until the releasing water was visually found clear, then dried in an oven at 105 °C to constant weight. The resulting

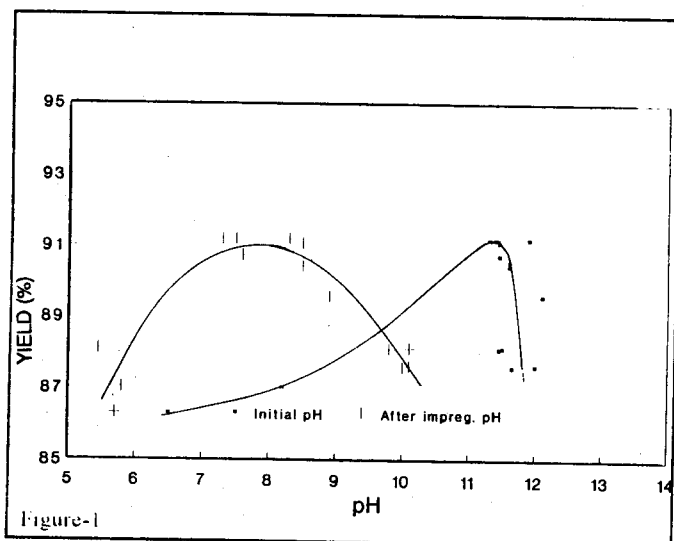
weights were then compared to the initial oven-dried weights of chips (10).

### Refining and Property Evaluation

Laboratory refining was done with a domestic blender osterizer B-8614 at a 2% consistency level. Paper sheets of a basic weight of 60 g/m<sup>2</sup> were prepared and tested according to standard CPPA testing methods. Brightness was evaluated on sheets prepared with tap water.

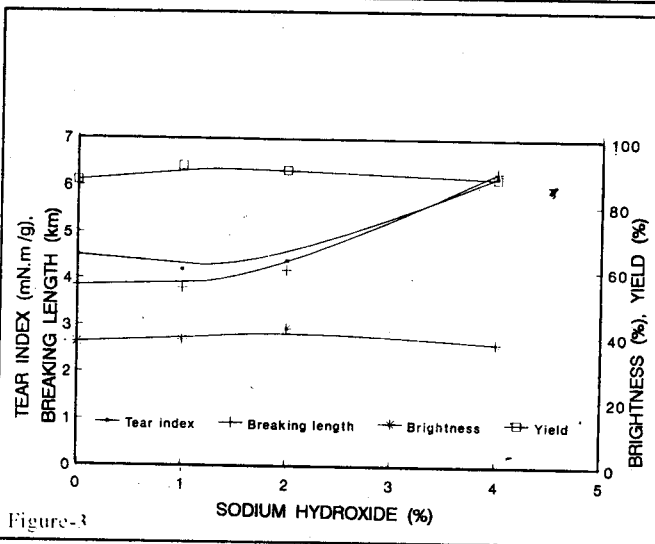
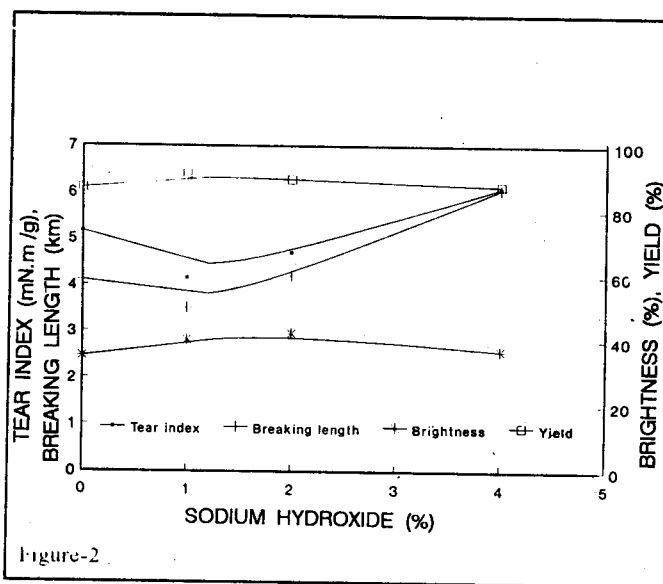
### RESULTS AND DISCUSSION

Figure 1 shows the relation between pulp yield and the pH of the initial impregnation solution and liquor after impregnation. Maximum pulp yield is obtained when the pH of the initial impregnation



solution is around 11.5 and the pH of the liquor after impregnation stays between 7 and 8.5. The pH of the initial solution drops due to the release of acid groups during impregnation of chips. pH further drops during the cooking period. This means pulp yield can be controlled by adjusting the liquor pH at different stages for the same cooking time and temperature.

Figure 2 shows the influence of the NaOH concentration in an impregnation liquor consisting of 50% ethanol and a 50% aqueous NaOH solution on pulp yield and properties, such as tear index, breaking length and brightness. The pulp's mechanical properties increase following an initial drop accompanying increased concentrations of NaOH in the impregnation liquor, whereas both pulp yield and



brightness drop following an initial increase. Similar relations between the NaOH concentration and resulting pulp yield and properties are also observed for impregnation solutions consisting of 30% ethanol and 70% NaOH aqueous solutions (Figure 3). We observed similar phenomena when methanol was used in impregnation solutions (8, 9).

The change in breaking length with the variation of pulp yield obtained with impregnation solutions containing various proportions of ethanol is shown in Figure 4. As found in previous studies (11), breaking length of ultra-high-yield pulp increases with the drop of pulp yield. It is difficult to conclude the actual influence of ethanol on pulp yield and breaking length. However, it is interesting to note that when the cooking time is reduced from

4 min. to 2 min., both pulp yield and breaking length of the pulp increase dramatically for the same impregnation solution (for example, an impregnation liquor of 50% ethanol and a 50% NaOH aqueous solution). It appears that more drastic conditions not only reduce pulp yield but are also detrimental to pulp properties, especially breaking length.

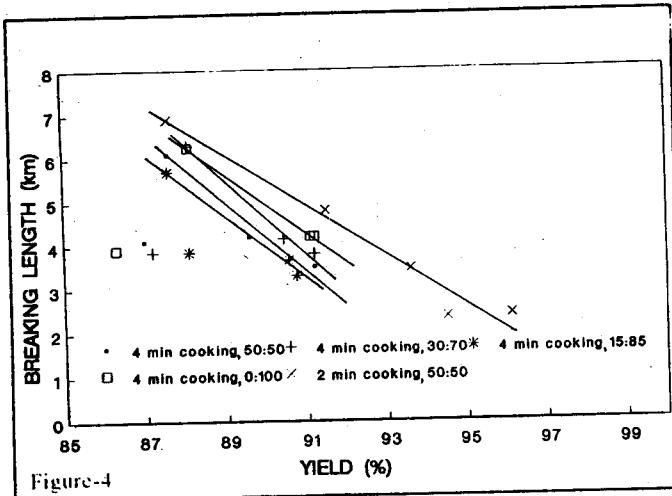


Figure-4

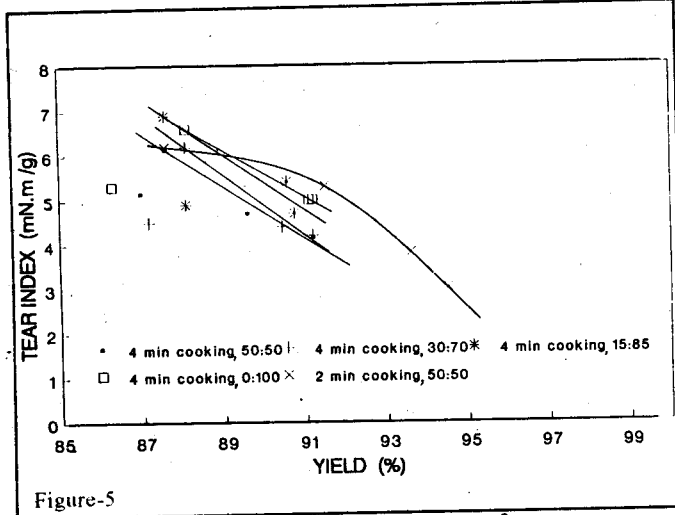


Figure-5

The influence of various proportions of ethanol on pulp yield and tear index is shown in Figure 5. Breaking length and tear index of the pulp increase with the drop in pulp yield, except for pulp yield obtained with an impregnation solution containing 0% NaOH. For similar pulp yield, tear values increase with the decrease of ethanol proportions in the impregnation solution. It is also interesting to mention that for the same impregnation solution (for example, 50% ethanol and 50% NaOH), a decrease in cooking time from 4 min. to 2 min. improves both pulp yield and tear index.

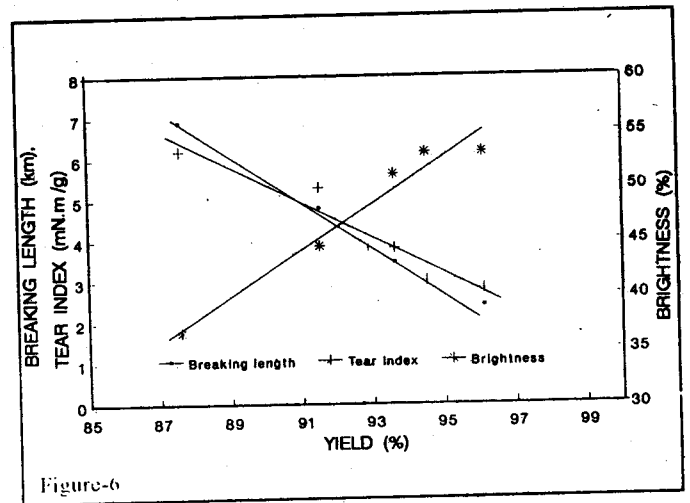


Figure-6

As shown in Figure 6, breaking length and tear index values as well as brightness depend on pulp yield. Both tear index and breaking length increase with the drop in pulp yield. Brightness decreases sharply with the drop in pulp yield. At 91.5% yield, tear index, breaking length and brightness are 4.75 mN.m<sup>2</sup>/g, 5.3 km and 46% respectively.

The influence of the NaOH in impregnation solutions on pulp yield and breaking length, pulp yield and tear index, and pulp yield and brightness is shown in Figures 7, 8 and 9 respectively. All these figures show the relative advantage of using 2 min cooking time instead of 4 min. The more drastic the conditions, the more they affect seriously pulp yield and brightness, but favour tear index and breaking length. Therefore, it is important to study the influence of both cooking temperature and time on pulp properties and yield for various impregnation chemicals to find optimum operating conditions. Our

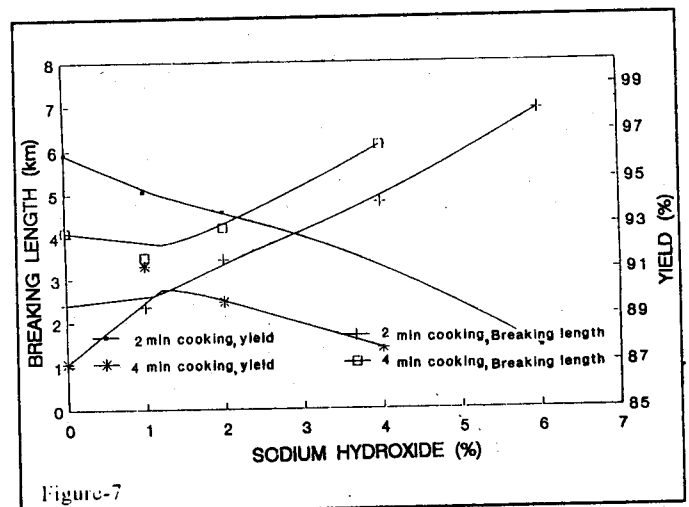


Figure-7

objective is to produce high pulp yield and brightness, as well as acceptable mechanical properties.

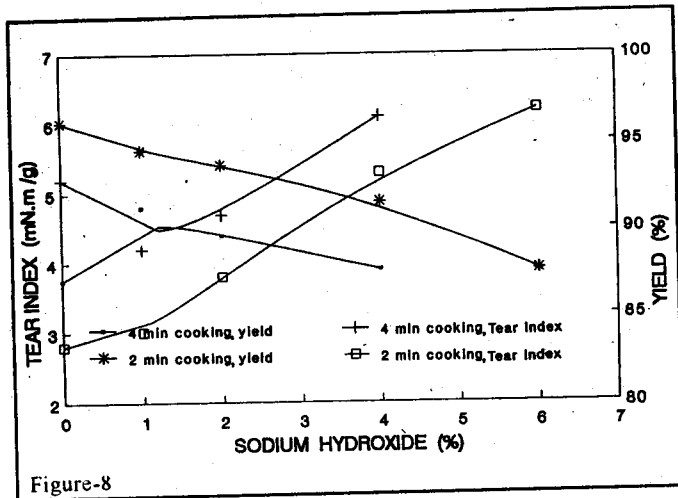


Figure-8

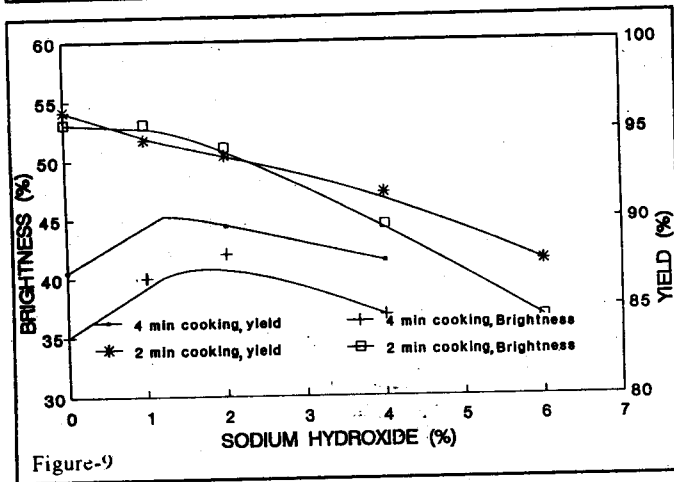


Figure-9

The influence of different chemicals in impregnation solutions on breaking length and pulp yield appear in Figure 10. At high yield range, the effect of different chemicals on breaking length is similar.

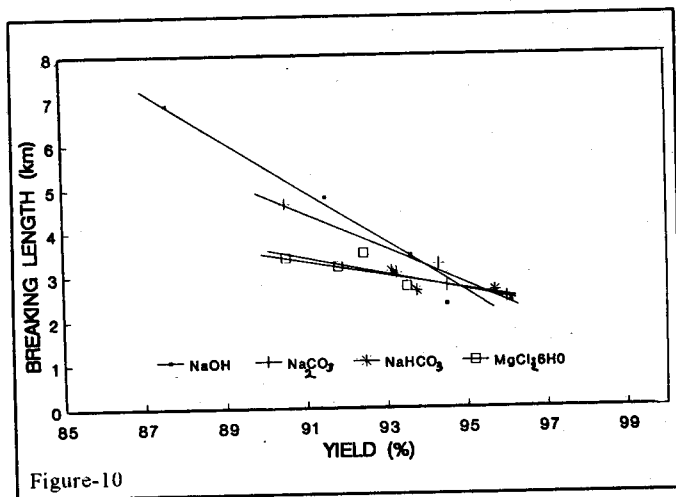


Figure-10

However, in the yield range of less than 93%, NaOH in impregnation liquor favors breaking length, followed by  $\text{Na}_2\text{CO}_3$  and  $\text{MgCl}_2$ . It is important to mention that the effectiveness of different impregnation chemicals depends on cooking temperature and time (12). As shown in Figure 4, for an impregnation liquor containing NaOH, pulp obtained at  $190^\circ\text{C}$  for a cooking period of 2 min. show higher breaking length values than that obtained at  $190^\circ\text{C}$  for 4 min at similar yield levels.

The influence of chemicals such as NaOH,  $\text{Na}_2\text{CO}_3$ , or  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  in impregnations solution on pulp yield and tear index is shown in Figure 11. Pulps obtained with NaOH in an impregnation liquor shows highest tear values followed by those of pulp obtained with  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ,  $\text{Na}_2\text{CO}_3$  or  $\text{NaHCO}_3$  in an impregnation liquor.

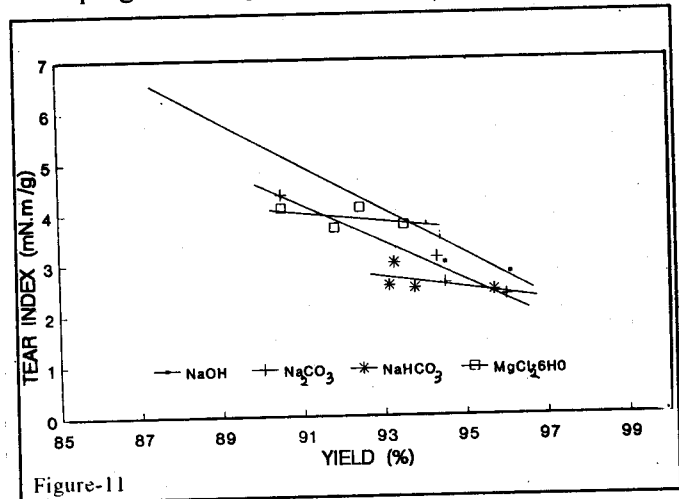


Figure-11

Figure 12 shows the influence of different chemicals in an impregnation solution on resulting pulp yield and brightness. Pulp yield and brightness

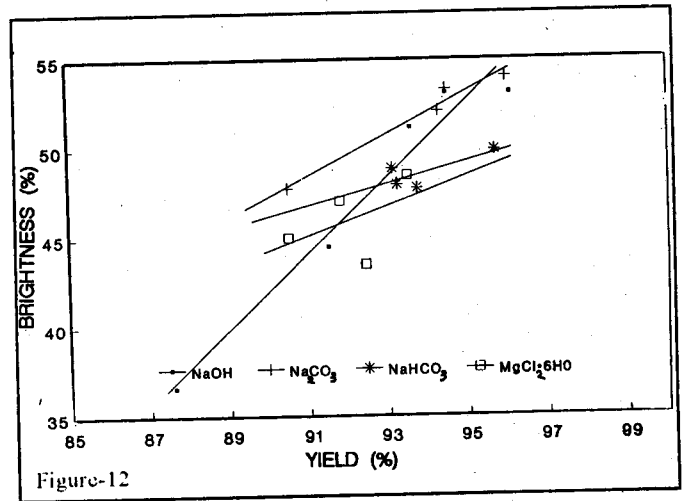


Figure-12

are very sensitive to NaOH in an impregnation liquor compared to other chemicals, such as Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub> or MgCl<sub>2</sub> · 6H<sub>2</sub>O.

## CONCLUSIONS

Ultra-high-yield pulp of good mechanical and optical properties can be prepared from aspen by using ethanol and NaOH as impregnation solutions. Pulp yield can be controlled by adjusting the pH of the impregnation liquor. Pulp yield and brightness drop and mechanical properties increase with the concentration of NaOH in the impregnation liquor. For the same impregnation solution, pulp yield, breaking length and tear index increase dramatically when cooking time is reduced from 4 to 2 min.

Pulp breaking length, tear index and brightness depend on pulp yield. At a 91.5% yield level, tear index, breaking length and brightness are 4.75 mN.m<sup>2</sup>/g, 5.3 km and 46% respectively. In the yield range of 93% and lower, NaOH in an impregnation liquor favors breaking length followed by Na<sub>2</sub>CO<sub>3</sub> and MgCl<sub>2</sub>. More work is necessary to study the influence of both cooking temperature and time on pulp properties and yield for various impregnation chemicals in order to determine optimum operating condition.

## ACKNOWLEDGEMENTS

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