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An Ester Chemirefinermechanical Pulping Feasibility Study

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AN ESTER CHEMIREFINERMECHANICAL PULPING FEASIBILITY STUDY

for

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INTRODUCTION

Conventional pulping processes (kraft and sulfite) utilize sulfur compounds which pose pollution problems for the environment. This environmental issue has served as a driving force in the development of a feasible sulfur-free pulping process which can deliver pulp of equal (or better) quality as compared to pulps from conventional pulping processes.

An answer to this problem has been searched for in the area of solvent pulping. A recent and promising extension of solvent pulping technology is the ester pulping process.

LITERATURE SURVEY

Solvent pulping, utilizing aqueous organic solvents and commonly referred to as organosolv pulping, is based on the simple principle of choosing the proper solvent to dissolve lignin in wood. Schuerch found that solvents with a Hildebrand solubility parameter of approximately 11.0 were most capable of dissolving lignin.(1,2)

The use of a combination of acetic acid and water as the solvent pulping medium has been studied and has proved capable of producing a kraft - like pulp from wood via dissolution of lignin. In order to maintain a solubility parameter of approximately 11.0 however, high concentrations of acetic acid are required in the process (greater than 85%).

The high acid requirement for this process (at high costs) requires a chemical recovery system of very high efficiency. Unfortunately, the methods available for recovery of organic acids are not economically feasible at the high concentrations of acid used.

To have an economical recovery system in organic acid pulping, a lower acetic acid concentration is needed. At the same time though, a concentration of 75% acetic acid is needed to maintain desirable pulp properties and cooking conditions.(1) This conflict of interests led Raymond Young of the University of Wisconsin to a modification of the acetic acid pulping system. It was proposed that the ester ethyl acetate (with a solubility

parameter of 9.1) be used as a substitute solvent for water in the aqueous acetic acid pulping process.(1,2) Young found that use of the ester pulping liquor provided many advantages over acid pulping including a reduction in the amount of water required in the system, enhanced solubility of lignin, reduced cooking time, higher strength pulps, higher yields, and simplified chemical recovery.(1,3). A liquor composed of equal volumes of ethyl acetate, acetic acid, and water was shown to readily delignify wood to low lignin content with high selectivity for lignin removal.(2) These advantages are obtained at a lower solvent requirement (67%) as compared to acetic acid pulping solvent requirements (>85%).

The use of esters in the chemical medium allows for chemical recovery based on phase separation. Liquor is reclaimed from the pulping process and ethyl acetate and water are added to produce two phases - one of which is the organic layer and the other the aqueous layer. The organic layer contains ethyl acetate, acetic acid, and dissolved lignin while the aqueous phase contains sugars. Lignin can be precipitated from the organic layer by addition of water to provide a sulfur - free form of lignin suitable for various commercial applications, unlike the lignin produced from kraft processes which lends itself only to burning.(4)

Thus, compared to conventional pulp liquor recovery systems (i.e. kraft and sulfite), ester pulping provides for reduced capital expenditures through simplified recovery systems.

Additionally, as economics would dictate the need for efficient chemical recovery, a reduction in pollution problems would result.(5)

Results thus far, as described by Young, indicate that this process is feasible for both hardwoods (aspen) and softwoods (spruce), albeit at stronger reaction conditions for the spruce species. The results of ester pulping of aspen show that the strength properties are generally superior to sulfite and kraft pulps however the tear strength of the ester pulp is lower than that of kraft pulp (yet higher than sulfite). Higher yields are also possible for ester pulping.(2)

Additionally, due to the ester solvent's selectivity for lignin removal, Young indicates that only minimal bleaching of ester pulps would be necessary, again resulting in reduction of costs and environmental problems.(2)

In a study by Aziz and McDonough (6), it was found that ester pulping of aspen does produce the results claimed by Young. It was also found, however, that other hardwood species did not respond as well to the ester pulping process while ester pulping of softwoods (spruce) was not likely to produce market-grade bleachable pulps.

Work done by Smith (7) also indicates that spruce does not avail itself to ester chemical pulping, however Smith did find (in his study of the reaction kinetics of ester pulping) a faster lignin removal rate in the early stages of ester chemical pulping of spruce as compared to the reaction rate (lignin removal rate)

exhibited in the early stages of kraft pulping of spruce. Based on his results, Smith intimated that although ester pulping may not be a feasible method by which to produce chemical pulp from spruce, there may be potential for the application of ester pulping in the arena of semichemical pulping. The short cooking time utilized in various semichemical procedures would expose the chips to a period of processing (cooking) at a rapid reaction rate, perhaps during which a small amount of dissolution of lignin would occur.

STATEMENT OF OBJECTIVE

The starting point for this project was found in Smith's closing recommendations - application of ester pulping to a semichemical pulping process. As ester pulping is a relatively new procedure, the main goal of this project was development of information in a complimentary avenue of ester pulping - application of the ester pulping chemical mixture to a chemirefinermechanical pulping process (CRMP).

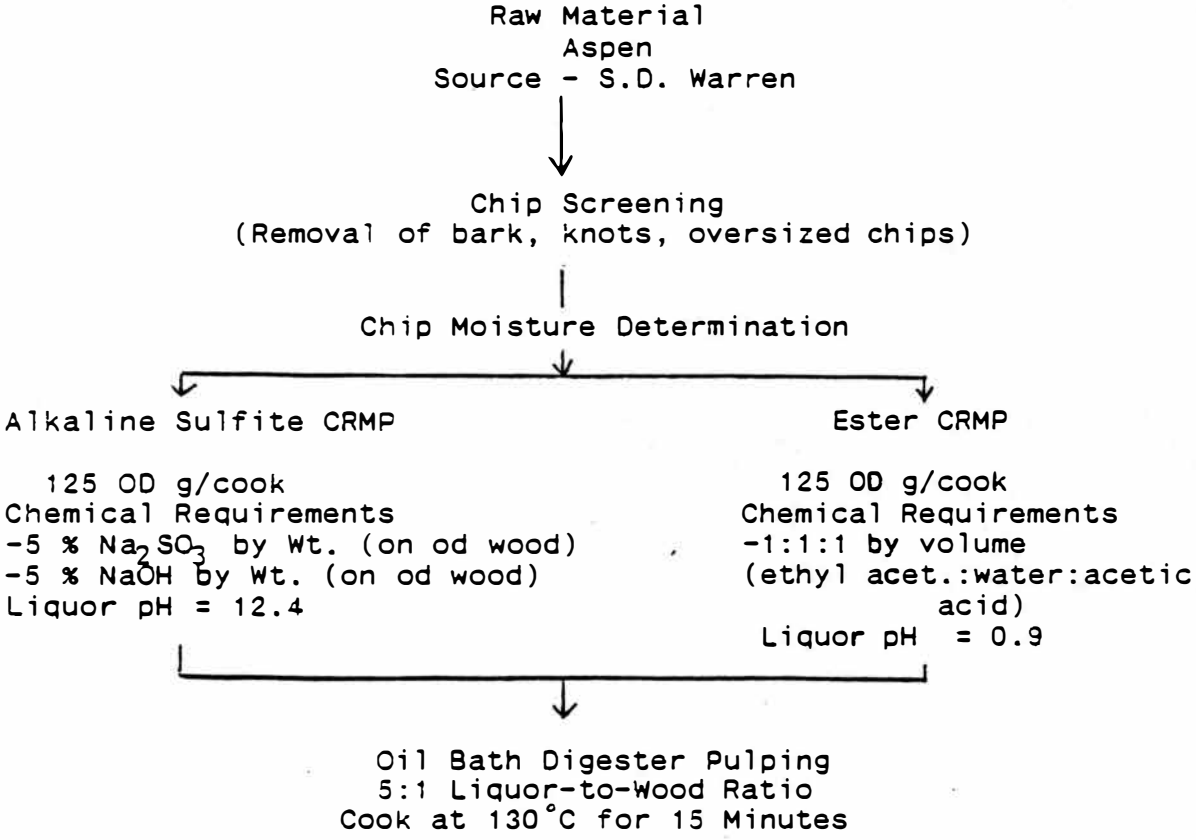
The focus of this project was on development of a profile of an aspen ester CRMP. The project also included a feasibility study of the ester CRMP. The feasibility study involved a comparison of properties of the resultant pulp and paper from an ester CRMP process to those of a conventional (alkaline sulfite) CRMP process.

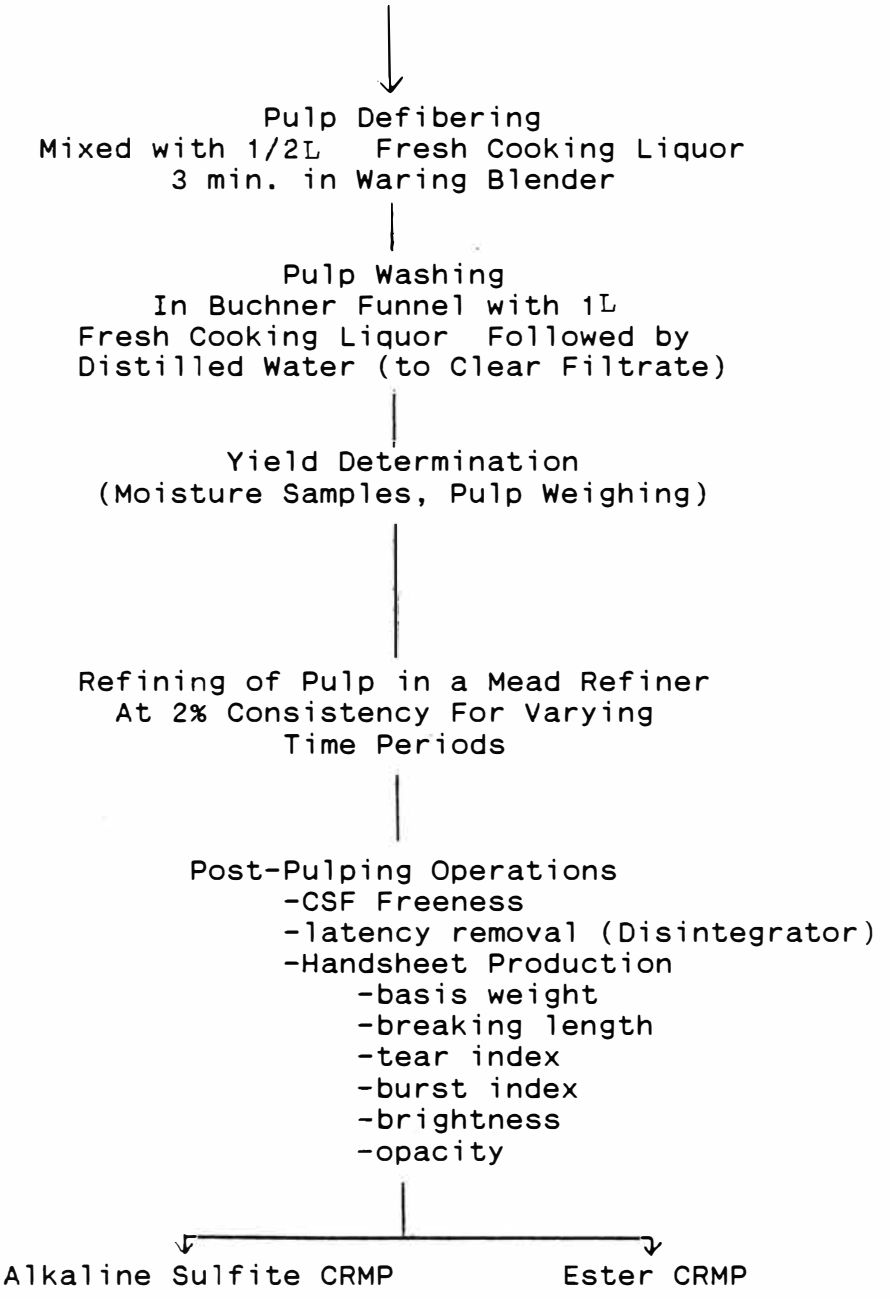
EXPERIMENTAL PROCEDURE

The experimental schematic is given in Figure 1 below. Pulping was conducted using the WMU Pilot Plant Oil Bath Digester Unit.

While two separate CRMP processes were carried out (one as ester and one as alkaline sulfite), the pulping process variables were kept constant.

Figure 1. Experimental Schematic





Two separate ester pulp samples were refined in the Mead Refiner (2.3 kg load) - 1 sample for 3 minutes, and 1 sample for 7 minutes. Three separate alkaline sulfite samples were refined for 1, 2, and 3 minute time periods. The CSF freeness of each pulp sample was then measured.

In order to produce handsheets that could be couched from the British sheet mold screen, each pulp was mixed with a chemical pulp composed of approximately 85% cedar (unbleached Kraft). The chemical softwood was refined in the Mead Refiner for 3 minutes (to a CSF of 685 mL). Ten handsheets (60 g/m^2 each) were made from a 60/40 mixture (60% CRMP/40% chemical) of each pulp type. The handsheets were conditioned for one day and then weighed to determine the basis weight. Tensile was measured on the Instron tensile tester on 1 sample of each of 5 sheets, in order to calculate breaking length. Burst was measured on 2 samples per 5 handsheets, from which burst index was calculated. Tear was measured on each pulp sample using a pad of five handsheets. Brightness of the various pulp samples was measured on a pad of 10 handsheets (1 test per sheet), while opacity was also measured - again 1 test per sheet for a total of ten measurements per pulp type.

Beater curves were then constructed in order to study pulp strength properties at comparable freeness values.

The various tests were conducted as per the standard test methods listed in Table 1.

Table 1. Testing Procedures

Test	Test Standard	Equipment
Consistency	T 240 om-88	Buchner funnel, drier
Brightness	T 452 om-87	Brightness Tester (457 nm filter)
Opacity	T425 om-81	Opacity meter
Handsheets (Phys.Props)	T 205 om-88	Disintegrator, British sheet mould
Physical Testing of H.S.	T 220 om-88	
Tensile	T 404 om-87	Instron Tensile Tester
Tear	T 414 om-82	Elmendorf Tear Tester
Burst	T 403 om-85	Burst Tester
Pulp Moisture	T 210 m-58	Oven
Latency Removal	T 262 pm-81	Disintegrator

Following are the results of the above described pulping processes.

TABLE 2. PULP PROPERTIES

PULP TYPE *	OPACITY %	BRIGHT. %	FREEN. CSF mL	BREAKING LENGTH km	TEAR INDEX mN m^2/g	BURST INDEX kPa m^2/g	BASIS WEIGHT g/m^2
EP 3	92.4	37.7	397	1.01	6.74	0.82	65.8
EP 7	93.9	37.5	205	1.25	8.27	0.92	59.8
AS 1	92.6	33.8	248	1.75	9.54	1.33	65
AS 2	93.9	33.2	107	1.94	10.31	1.62	67.5
AS 3	92.9	32.6	95	2.43	10.16	2.03	66

TABLE 3. % YIELD

ESTER PULP	92.9
ALKALINE SULFITE PULP	91.4

TABLE 4. BEATER CURVE RESULTS

Strength Properties at 240 ml CSF

PULP TYPE	BREAKING LENGTH km	TEAR INDEX mN m^2/g	BURST INDEX kPa m^2/g
ESTER	1.22	8.03	0.905
ALKALINE SULFITE	1.77	9.73	1.35

* EP = ESTER PULP
AS = ALKALINE SULFITE PULP
NUMBER INDICATES TIME PROCESSED IN MEAD REFINER

Figure 2.

EFFECT OF FREENESS ON

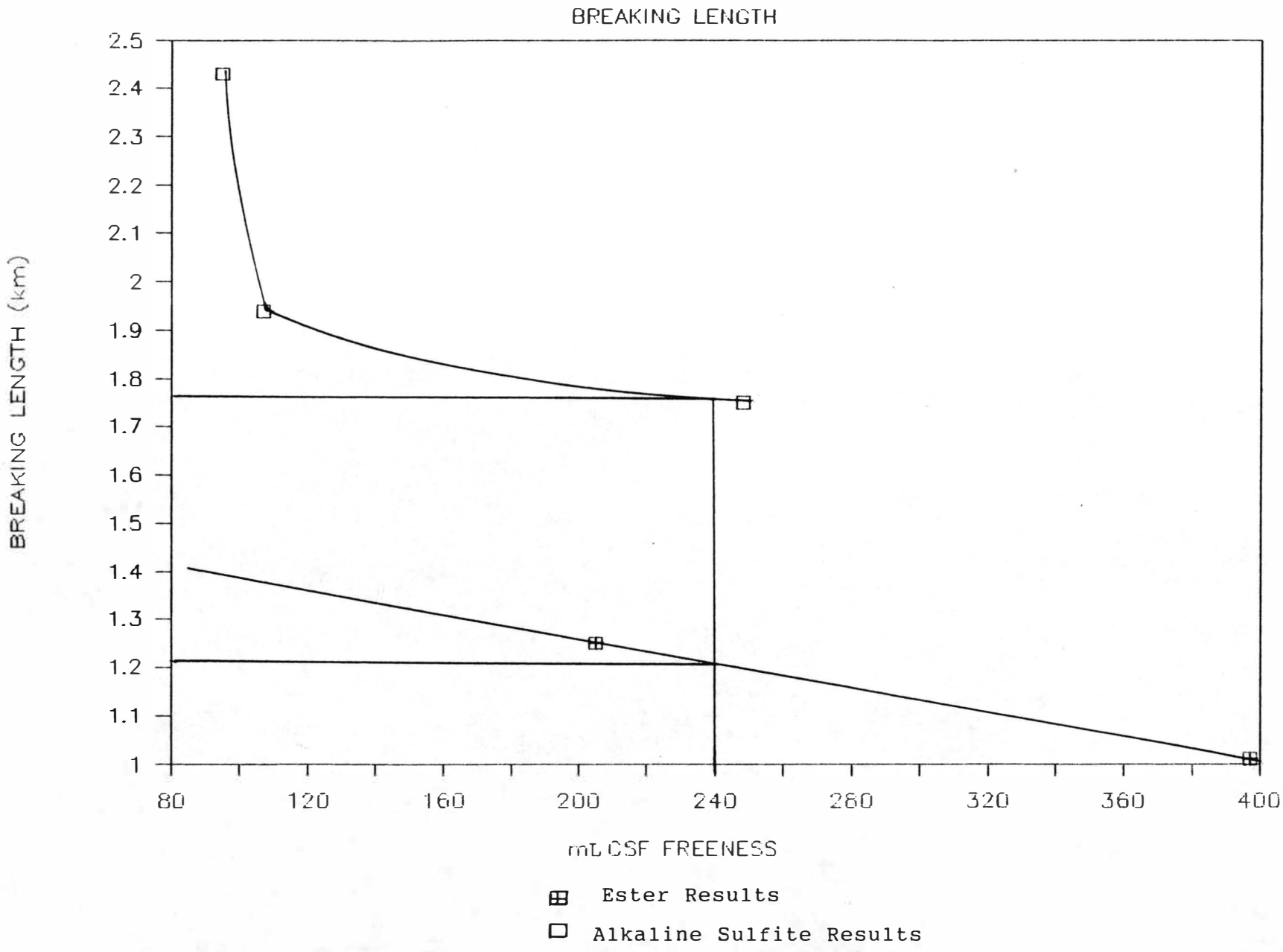


Figure 3.

EFFECT OF FREENESS ON

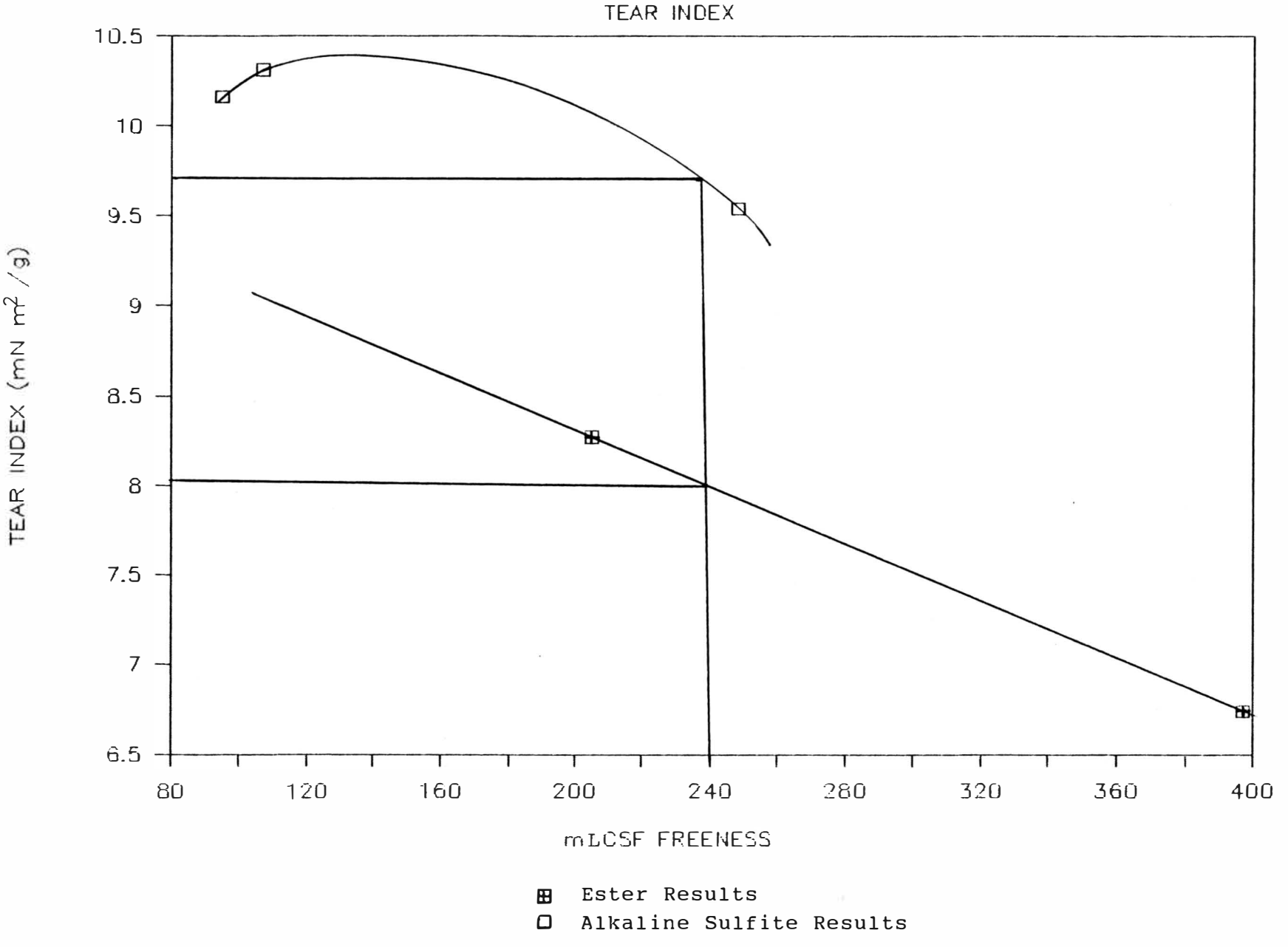
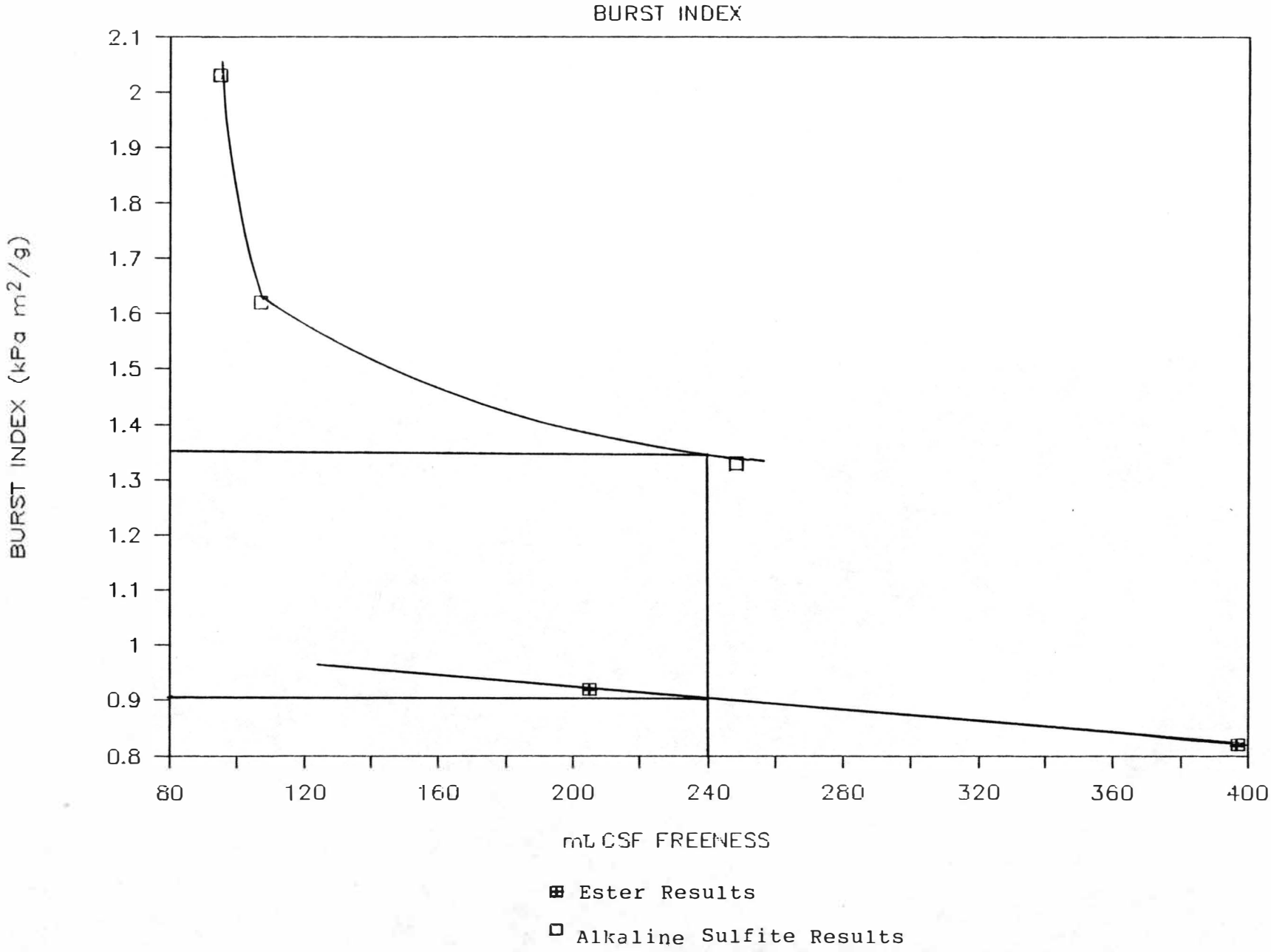


Figure 4.

EFFECT OF FREENESS ON



DISCUSSION

It can be seen from the results in Table 2 that the ester CRMP was not as responsive to refining as the alkaline sulfite CRMP. This characteristic can be seen in the CSF freeness results. At 3 minutes of refining, the ester pulp developed a freeness of 397 mL as compared to the alkaline sulfite pulp which exhibited a 95 mL CSF. Even at 7 minutes of refining, the ester pulp was not able to develop a freeness comparable to the alkaline sulfite pulp refined for 2 and 3 minutes (205, 107, and 95 mL CSF, respectively). These results imply that the alkaline sulfite chemical medium is more effective at softening the chips than the ester chemical medium. A softer chip would be more susceptible to refining and thus more responsive to such action.

This discrepancy in response to refining is further illustrated in the strength property results obtained for the various pulps. As the results show (Table 2 and Figures 2 through 4) the strength properties (i.e. breaking length, tear index, and burst index) of the ester CRMP are consistently lower than those of the alkaline sulfite CRMP.

The beater curve results compare the ester CRMP to the alkaline sulfite CRMP at a CSF of 240 mL. These results, too, show consistently lower strength properties for the ester CRMP. In the comparison of the ester pulp curves to the alkaline sulfite pulp curves, it is important to note that the limited data available for the ester pulp hamper one's ability to construct an accurate curve.

In terms of optical properties, opacity appeared relatively consistent from pulp to pulp. The brightness of the ester pulp however, does appear to be higher than the alkaline sulfite pulp brightness. The lower brightness for the alkaline sulfite pulp may be due to the high alkalinity of the cooking liquor applied in the alkaline sulfite CRMP process (12.4 pH), as compared to the pH applied in the ester pulping process (0.9).

In examining the yield properties of the two pulps (prior to refining), it was seen that the ester CRMP process is comparable to the alkaline sulfite CRMP process in this respect.

CONCLUSIONS

In conclusion then, these results imply that the ester CRMP process produces a weaker pulp than the alkaline sulfite CRMP process, even at longer refining times (3 and 7 minutes as opposed to 1, 2, and 3 minutes). This effect is due to the ester pulp's limited response to the refining action, perhaps due to the limited chip-softening ability of the ester chemical medium.

In contrast to the strength property comparison, it was seen that the ester CRMP process produced a brighter pulp than the alkaline sulfite CRMP process (likely due to the highly alkaline conditions of the chemical medium applied in the alkaline sulfite CRMP process).

Finally, while the application of ester pulping to a CRMP process does not appear feasible (based on the strength properties exhibited by the ester CRMP), ester pulping of aspen has proved feasible in full chemical applications. These results imply that perhaps there may be potential for the application of ester pulping in other semichemical pulping processes which utilize more intense heat and pressure applications i.e. those semichemical processes more closely approximating a chemical process, namely a CTMP process.

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