Steam explosion pulping: Effects of temperature and pressure on paper properties at constant pulp yield and ionic content

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ABSTRACT *Ultra-high-yield pulps—explosion pulps, chemithermomechanical pulps (CTMP), and chemimechanical pulps (CMP)—were prepared at the same yield (90%+) using identical chemical charges. Mechanical properties were highest for the explosion pulps. Explosion pulps also had a much lower specific refining energy than CMP and CTMP. Several of the explosion pulps were subjected to pressure of 25 atm following the regular cooking process. These pulps had the highest mechanical properties and the lowest specific refining energy. The results indicate that the high temperatures and pressures used in explosion pulping promote fiber softening, increase the portion of ordered Cellulose I, and facilitate fiber separation.*

KEYWORDS
Chemimechanical pulping
Chemithermomechanical pulping
High-yield pulps
Mechanical strength
Pulp properties
Refining
Steam explosion pulp

Ultra-high-yield pulps are produced from both softwoods and hardwoods. These high-yield mechanical pulps are made by refining wood chips after treatment with chemicals that react with the lignin but do not solubilize it (*1-5*). Chemimechanical pulping (CMP) and chemithermomechanical pulping (CTMP) are the two most widely used of these high-yield processes. Steam explosion pulping (SEP) is a recent innovation that is proposed as an alternative to the conventional CMP and CTMP processes (*6-8*).

The distinctive features of the SEP process include a short cooking time at high temperature and pressure in saturated steam. The cook is followed by explosive discharge of pulp when the pressure in the digester is instantaneously released. The exploded pulp is then beaten in an atmospheric refiner and, depending on the end use, bleached. Before the cook, the chips are impregnated with an antioxidant such as Na_2SO_3 to protect the yield, to retard chip oxidation, and to develop hydrophilic groups during the steam cook. A second chemical agent (NaOH, Na_2CO_3, $NaHCO_3$) can be added to promote fiber swelling and softening as well as improve the cook (higher ionic content, better regulation of pH) (*9*).

The short-duration cook at high temperature and pressure promotes permanent lignin softening, ultra-high yields, and excellent paper properties (*10-12*). The explosive pressure release enhances fiber separation, thus decreasing the required refining energy.

To date, the best SEP results have been obtained in semi-industrial trials, where chip impregnation in a press probably enhanced the results (*12, 13*). Previous research (*12*) showed that SEP (cooked at 180°C, 190°C, and 200°C) had lower specific refining energy than conventional CMP (170°C) cooked to the same yield (90%) and with the same chemical charge applied during both impregnation and cooking.

The work presented here was carried out to compare SEP, CMP, and CTMP at the same yield and chemical charge. Explosion pulps also were subjected to an additional high-pressure treatment following the initial cook in an effort to determine the effects of pressure on SEP.

I. Pulping conditions and yield for pulps depicted in Figs. 1-14

Pulp		Pulping conditions			Yield,	Ionic content, mmol/kg			Symbol used in
No.	Type	Time, min	Temp., °C	Pressure, atm	%	Sulfonic	Carboxylic	Total	Figs. 1-14
1	RMP	...	...	...	98.8	0	77	77	⊡
2	CTMP	10	128	1.8	95.3	35	143	178	◆
3	CTMP	60	128	1.8	93.1	39	141	180	■
4	CMP	55	150	4.0	89.6	37	128	165	◊
5	CMP	30	160	5.2	89.9	38	141	179	■
6	Water explosion	2.5	190	11.9	91.3	0	94	94	□
7	SEP	1.3	190	11.9	92.6	46	134	180	▲
8	SEP	2	190	11.9	90.4	43	137	180	△
9	SEP	1.5	195	13.5	90.1	46	134	180	⊠
10	SEP	1	200	15.5	89.9	45	121	166	+
11	SEP (nitrogen)	2	190	11.9-25	90.0	40	140	180	⊞
12	SEP (nitrogen)	1.5	195	13.6-25	89.7	51	132	183	×
13	SEP (nitrogen)	1	200	15.5-25	90.0	42	137	179	✳

Experimental

Raw material

Freshly cut aspen trees from Quebec were debarked and chipped at La Station Forestiere Duchesnay (Quebec). Chips were screened in our research center. The average chip size corresponded to industrial conditions, with mean chip dimensions of 31 mm × 15 mm × 5 mm.

Impregnation

Aspen chips (150 g, moisture content = 50%) were mixed in plastic bags and impregnated with a solution containing 8% Na_2SO_3 for 24 h at 60°C using a liquor-to-wood ratio of 6:1. Impregnated chips were drained prior to the cooking process. Because of same chemical charge during the impregnation, the chemical uptake was the same for all pulps. Two pulps—refiner mechanical pulp (RMP) and "water explosion" pulp—were processed without any added chemicals.

Cooking

The explosion pulps were flushed with steam for 1 min at atmospheric pressure and then cooked in a 300-mL laboratory batch reactor (Stake Tech Co.) using saturated steam. All SEPs were discharged explosively into the blow vessel, washed thoroughly with tap water, and stored in a refrigerator at 3°C prior to further treatment.

Cooking times, temperatures, and pressures for all pulps are presented in **Table I**. Pulp 2 is a CTMP prepared under conventional conditions (10 min at 128°C) to a yield of 95%. Pulp 3 is a CTMP that was cooked for a longer duration (60 min at 128°C) to reduce yield to about 93%. This was done to bring the yield within the range of one of the SEPs (Pulp 7), which was cooked for a shorter time than the other SEPs (1.3 min at 190°C and 11.9 atm) to increase yield from 90% to about 93%. The CMPs, Pulps 4 and 5, were cooked somewhat longer than industrial conditions to obtain the 90% yield needed for direct comparison with the SEPs. Pulps 8-13 are all SEPs produced under three sets of conditions to obtain a 90% yield. The cooking conditions for Pulps 8-10 were replicated for Pulps 11-13, which were subjected to a pressure of 25 atm (under nitrogen) immediately after the cook and then discharged explosively from the digester. Nitrogen was chosen as the pressurizing agent because of its inert character.

Yield calculation

Exploded chips were washed with 1 L of tap water and subsequently defibered for 90 s in a laboratory blender at 2% consistency. The resulting pulp was washed again and dried at 105°C to constant weight, which was then compared with the initial weight of o.d. wood.

Refining

Laboratory refining was carried out in a domestic blender (Osterizer B-8614) at a consistency level of 2%. Refining times were chosen to obtain at least three levels of freeness (approximately 100, 300, and 500 mL CSF). Refining energy was measured using an EW-604 wattmeter. Relative specific refining energy was calculated by subtracting the blending energy of fully beaten pulp from the total consumed energy.

The decision to use a blender was based on Shaw's work (*14*), which showed that blenders are the most suitable device for low-scale laboratory evaluation of high-yield pulps. Blenders are preferred in this situation because they do not cut the fibers to the extent that other laboratory refiners do (PFI mill, Valley Beater, etc.). Shaw's work and our previous research confirm that there is very good agreement between results obtained using a laboratory blender and those obtained in semi-industrial trials.

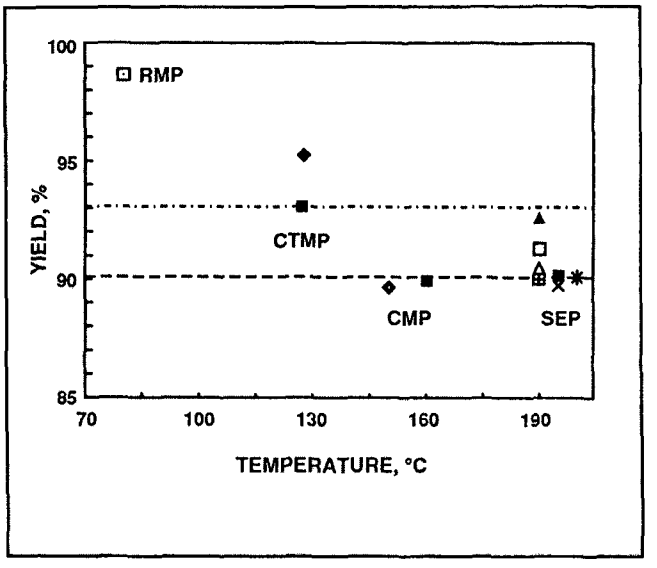
Bleaching

Bleaching was carried out on all pulps using 4% hydrogen peroxide. Bleaching conditions were 3 h at 80°C (*12*).

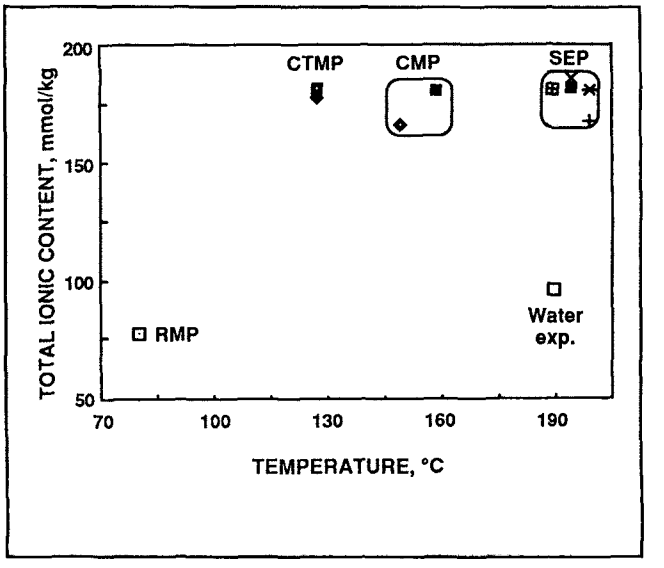
Property evaluation

Handsheets were prepared and tested according to CPPA test methods

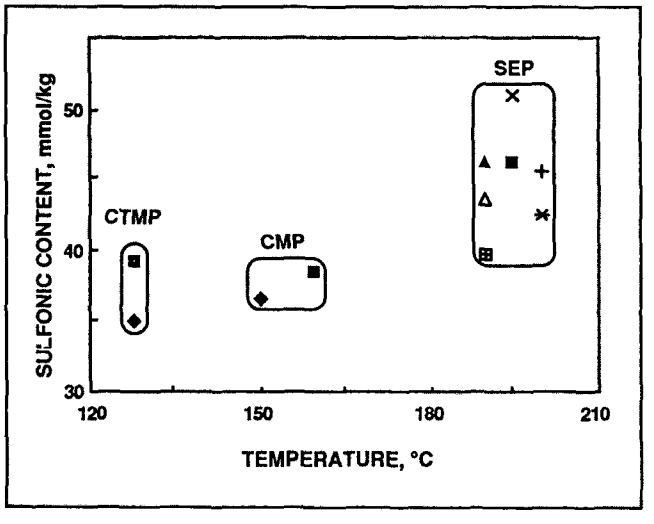
1. Pulp yield as a function of pulping temperature for the 13 pulps in Table I. CMP and SEP were prepared at 90% yield. CTMP was prepared under usual conditions (95% yield) and at extended cooking time (93% yield). Cooking time for one SEP was reduced (93% yield) for comparison with CTMP.



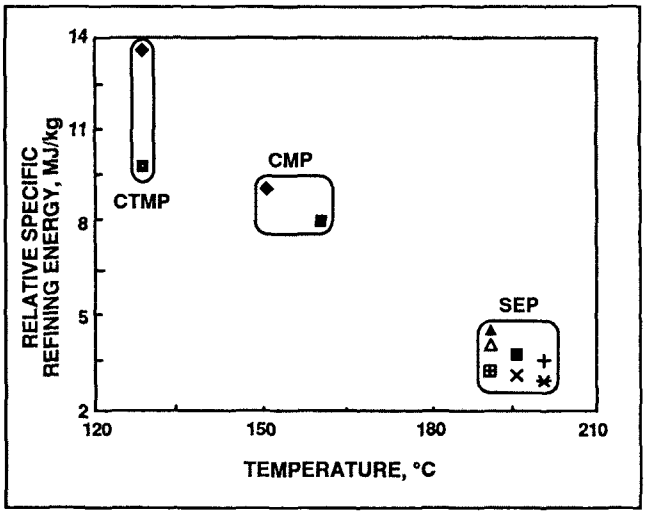
2. Total ionic content as a function of pulping temperature for all pulps



3. Sulfonic content as a function of pulping temperature for chemically treated pulps



4. Relative specific refining energy as a function of pulping temperature for chemically treated pulps (100 mL CSF)



[1.2-g sheets (60 g/m²) prepared with tap water]. Brightness was also measured on 3.0-g sheets (150 g/m²) prepared with demineralized water. Sulfonate and carboxylate content were determined by conductometric titration (*15*).

Results and discussion

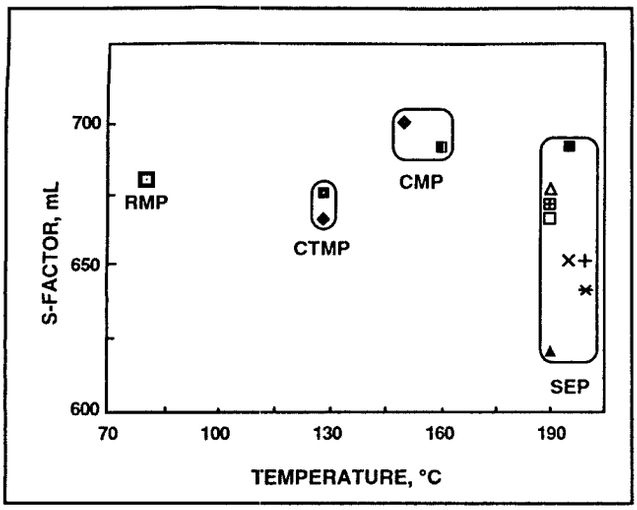
Experimental design

High-yield pulps and even ultra-high-yield pulps (90%+) can be prepared over a wide range of process conditions. Based on results of our previous research, as well as on theoretical considerations, we believe that high temperature and pressure are the most influential factors contributing to the superior performance of explosion pulps. We focused on the influence of temperature and pressure by manipulating the pulping conditions to eliminate some of the process variables.

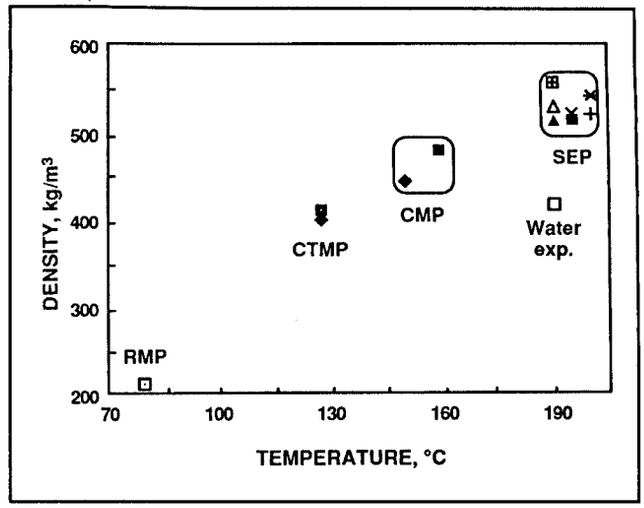
First, we tried to cook the pulps to the same yield. **Figure 1** shows that pulp yield for the two CMPs and most of the SEPs was 90%. Explosion pulps were cooked under typical conditions, while the CMP cooking times were extended somewhat to obtain a yield of 90%. In the case of CTMP, conventional cooking gave a yield of 95% (Pulp 2). However, we managed to prepare a CTMP and an explosion pulp at comparable yield (about 93%) by extending the CTMP cook and shortening the SEP cook.

Second, all pulps received the same degree of chemical treatment, except for the RMP and water explosion pulp, where no chemicals were added. The chemical charge and the resultant chemical uptake were the same for all chemically treated pulps. **Figure 2** shows that the total ionic content was similar for most of the

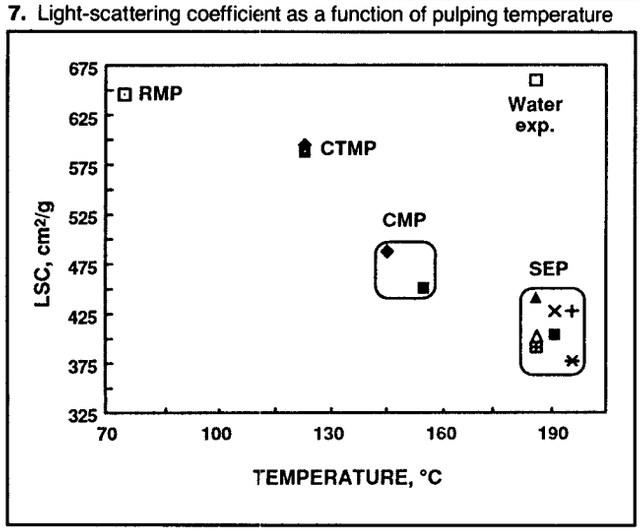
5. S-factor as a function of pulping temperature for all pulps



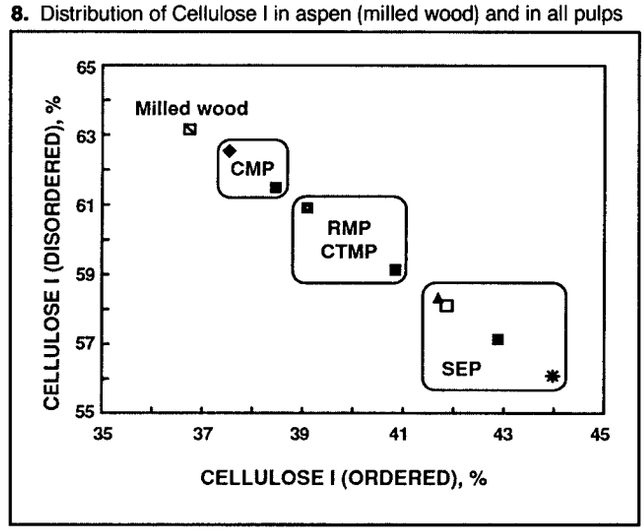
6. Pulp density as a function of pulping temperature for all pulps (100 mL CSF)



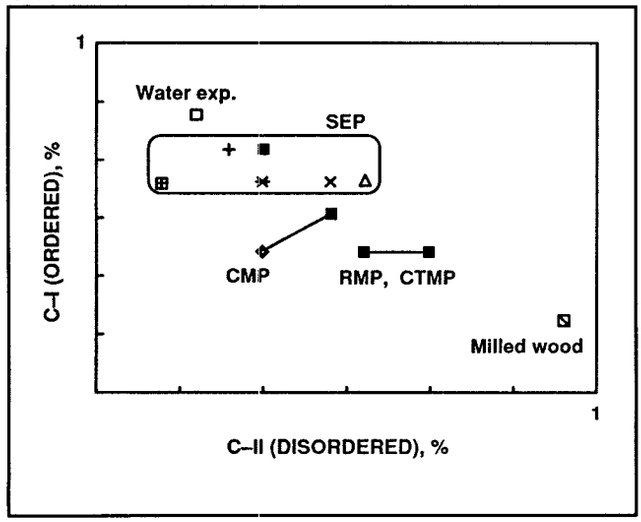
7. Light-scattering coefficient as a function of pulping temperature



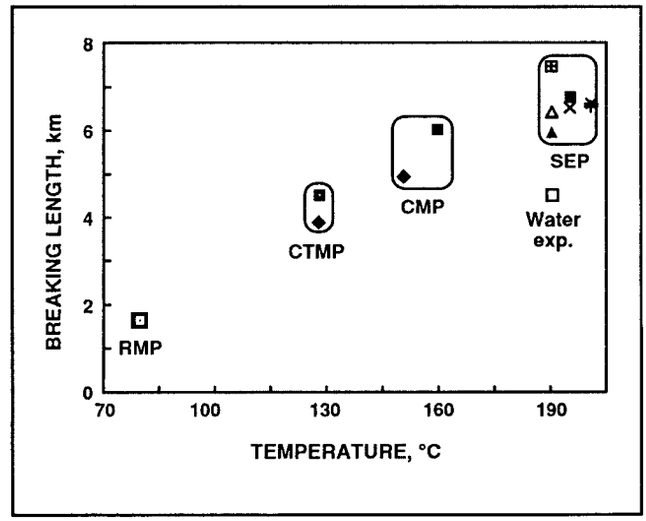
8. Distribution of Cellulose I in aspen (milled wood) and in all pulps



9. Relative distribution of cellulosic material in aspen (milled wood) and in all pulps. SEPs had a higher portion of high-quality ordered C-I.



10. Breaking length as a function of pulping temperature for all pulps (100 mL CSF)



chemically treated pulps. However, **Fig. 3** shows that the degree of sulfonation was higher in the explosion pulps.

Ionic content is one of the most important factors contributing to the pulp's mechanical properties. As shown previously (*12*), mechanical properties of explosion pulps are superior to those of other high-yield pulps at the same ionic (sulfonic) content. Similarly, explosion pulps provide comparable mechanical properties at a lower ionic content. This was confirmed in our study and is discussed later in this report.

Refining energy

The blender may not be the ideal method for refining pulp, but it has been reported as the best method for refining on a small scale (*14*). Pulp properties and refining energies obtained with the blender compared well with the semi-industrial results (*12, 13*).

Figure 4 shows the relative specific refining energies for chemically treated pulps at a freeness of 100 mL CSF. The decrease in refining energy in the case of SEP can be attributed to the influence of temperature and explosive discharge from the digester. The cooking temperature in the SEP process is above the glass-transition temperature for lignin, which leads to additional permanent softening of the chips. The explosive discharge helps to separate the softened chips into individual fibers as well as fibrillating the individual fibers themselves (*16*). Exploded chips are more flexible than CMP or CTMP chips and can be easily separated into fibers and refined. The shorter refining time also helps preserve fiber length.

Fiber properties

Because of the permanent lignin softening and the explosive discharge, explosion pulps need substantially less energy for fiber separation. Most of the energy is thus applied to fiber development. The higher average fiber length of explosion pulps can be explained by the shorter refining time and by the ease of fiber separation. (The blade does not cut into the shives or chip particles, treating separated fibers instead.)

The surface development of fibers can be evaluated using the S-factor. (S-factor is a measure of the freeness of the fraction that has passed through a 48-mesh wire but has been retained on a 100-mesh wire on the Bauer-McNett fiber classifier.) **Figure 5** shows that the SEPs had a lower S-factor than the CMPs, indicating good fiber development. Most of the SEPs also had a lower S-factor than CTMP and RMP. S-factor for CTMP and RMP is skewed downward by the presence of fines and debris, both of which significantly retard pulp drainage.

The longer fibers in the SEPs are also more flexible because of the lignin softening that occurs at high temperature. **Figure 6** depicts pulp density as a function of cooking temperature for pulps at 100 mL CSF. High density is evidence of fiber flexibility, and it is clear that pulp density increased with the severity of the cooking process. Even the water explosion pulp (no chemicals) had a density comparable with that of CTMP.

The higher flexibility of the longer SEP fibers dramatically improves the bonding capacity, which explains the superior mechanical properties of explosion pulps. Light-scattering coefficient (LSC) is another way of measuring bonding capacity. **Figure 7** shows LSC as a function of pulping temperature. The low LSC for the SEPs confirms their superior bonding capacity. As in Fig. 6, pulp quality increases with increasing severity of the cooking process, favoring the explosion pulps over the conventional CMP and CTMP.

The chemical changes that take place during the cook are another important consideration. Tanahashi (*16*) recently reported an increase in the crystallinity of cellulose following steam treatment at high temperature and pressure. Using Sukhov's qualitative methods for evaluating cellulosic materials via FTIR (Fourier-transform infrared reflectance) and Raman spectroscopy (*17*), we found that explosion pulps have the highest content of ordered Cellulose I (C-I). The results in **Fig. 8** show that crystallinity of C-1 increased with pulping temperature. Ordered C-I is the highest quality cellulose (highest bonding capacity) and contributes significantly to pulp mechanical properties.

Disordered Cellulose II (C-II), on the other hand, is a lesser quality material composed of a mercerized type of cellulose and hemicelluloses. **Figure 9** shows the distribution of C-I ordered and C-II disordered cellulose in the samples of SEP, CMP, CTMP, RMP, and in milled aspen wood. It is apparent that cellulose structure improved with increasing temperature and pressure of cooking. From this point of view, the best C-I/C-II ratio was found in the water explosion pulp.

The high C-I/C-II ratio in the water explosion pulp (no chemicals) can probably be explained by the fact that these chips were not chemically protected by an antioxidant. In this case, the predominant reaction is hydrolysis of the unprotected hemicelluloses, which produces a higher proportion of C-I compared with C-II in the resultant pulp.

According to another analysis (*18*), the concentration of cellulose and sulfur on the fiber surface is higher for explosion pulps than for conventional pulps. The fibers in explosion pulps also were reported to have less lignin on their surfaces than the fibers in conventional pulps, even though the concentration in bulk was the same for both types of pulp.

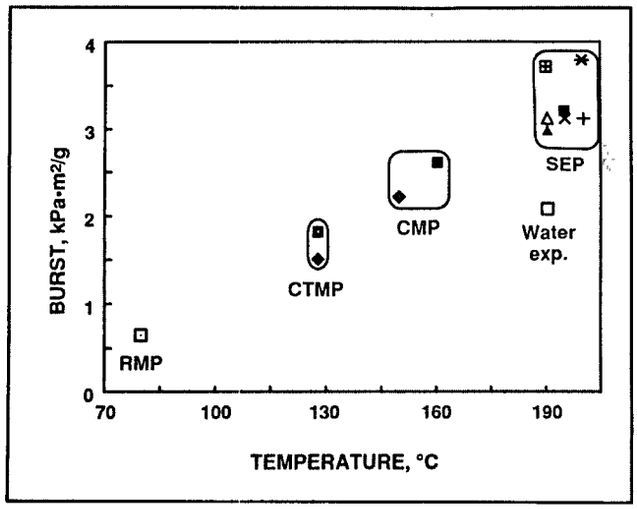
Mechanical properties

The results presented so far show that the physical and chemical changes that take place during the high-temperature-and-pressure cook and subsequent explosive discharge are beneficial and should provide a pulp with superior mechanical properties. This was experimentally confirmed, and results for breaking length, burst index, and tear index are presented in **Figs. 10–12**, respectively.

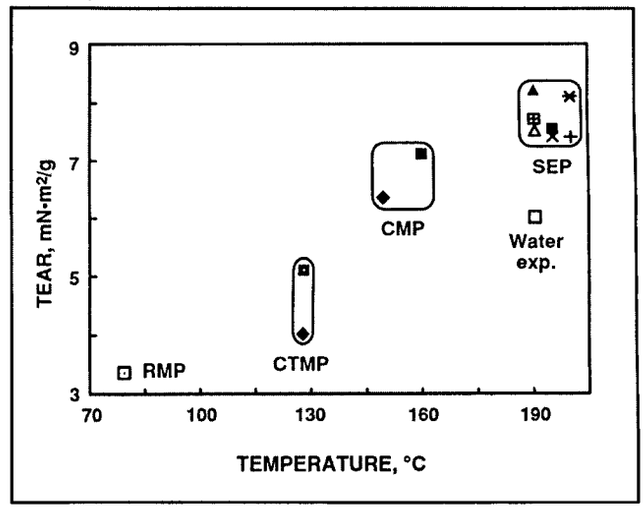
If we compare CTMP and SEP, the superiority of explosion pulps is obvious in any regard: higher mechanical properties (breaking length, burst, and tear), higher density and C-I crystallinity at lower LSC, and lower refining energy. This was accomplished either at conventional CTMP conditions (Pulp 3) or at a similar yield (Pulp 4) and at the same chemical treatment and similar ionic content. The only difference between the CTMP and SEP pulps was the cooking conditions (temperature, pressure, and time), which emphasizes the influence of these parameters on pulp quality.

While the differences between CMP

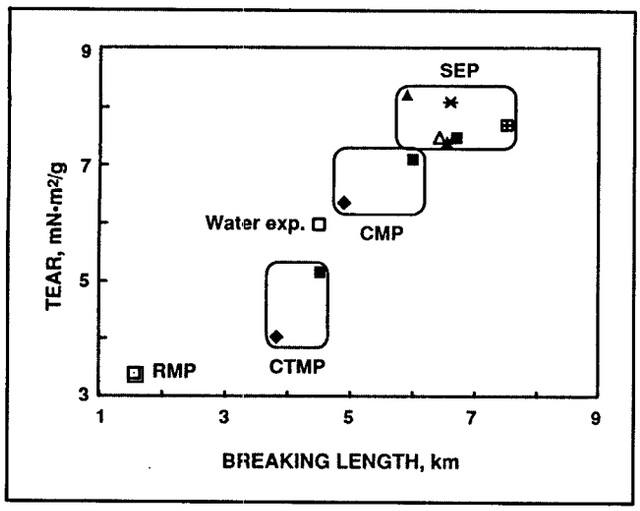
11. Burst index as a function of pulping temperature for all pulps (100 mL CSF)



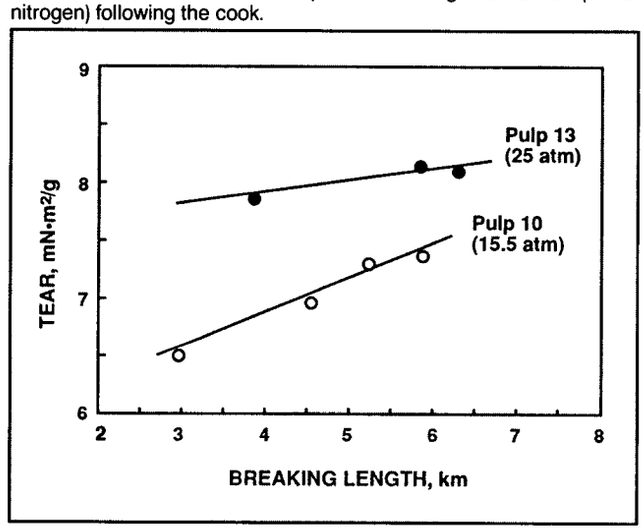
12. Tear index as a function of pulping temperature for all pulps (100 mL CSF)



13. Tear index vs. breaking length for all pulps (100 mL CSF)



14. Tear index vs. breaking length for explosion pulps prepared at 200°C, 1 min, and 15.5 atm. Pulp 13 was brought to 25 atm (under nitrogen) following the cook.



and SEP were not as dramatic as between CTMP and SEP, Figs. 10–12 clearly show that the mechanical properties of SEP were superior. This is also apparent in **Fig. 13**, which relates tear index and breaking length. This relationship is most useful in evaluating the mechanical strength of pulp, and we can see that strength increases almost linearly with the severity of the cooking process.

The CMP and SEP pulps were prepared at the same yield using the same chemical treatment during impregnation and cooking. The total ionic content was almost identical for CMP Pulp 4 (150°C, 55 min) and SEP Pulp 10 (200°C, 1 min). This is also true of CMP Pulp 5 (160°C, 30 min) and SEP Pulp 8 (190°C, 2 min) and SEP Pulp 9 (195°C, 1.5 min). The results in Fig. 13 thus confirm previous research (*12*) showing that explosion pulping provides comparable strength at lower ionic (sulfonic) content and higher strength at the same ionic content compared with conventional high-yield pulping processes.

Both ionic content and pulp yield are predominant parameters in the pulp evaluation. However, it has been shown that attempts to explain pulp properties as a function of a single parameter would be statistically incorrect (*19*). The system is so complicated that we need at least two or three parameters to explain satisfactorily the effect on individual pulp properties. We chose pulping temperature as the independent variable in Figs. 1–7 and Figs. 10–12 as a simple manner for better visualization of our results. In reality, pulp response is a complex function of multiple influences that could not be presented in detail in this limited space. Evaluation of the relationship between pulp properties and fiber characteristics using multiple linear regression is the subject of another paper (*19*).

Optical properties

Brightness exceeding 60% can be obtained if chip quality is satisfactory. Water explosion (no chemicals) was

the only process where brightness dropped significantly (to about 42%) because of the condensation reactions. Otherwise, brightness values ranged from 60% to 66% MgO for the unbleached and from 78% to 84% MgO for bleached pulps. These results are in agreement with those reported previously (*12*). Explosion pulps showed slightly higher brightness and brightness stability. For both unbleached and bleached pulps, the order in brightness was as follows: water explosion < CMP < CTMP < RMP < SEP.

Influence of pressure

The cooking conditions used to produce SEP Pulps 8-10 were replicated for Pulps 11-13. These latter pulps were subjected to a pressure of 25 atm under nitrogen immediately after the cook and then discharged explosively. The high-pressure explosion increased the mechanical properties, as seen in **Fig. 14**, which shows the results for SEP Pulps 10 and 13, both cooked at 200°C for 1 min at 15.5 atm. These pulps showed the greatest difference in mechanical properties, with the pulp discharged at high pressure showing an increase in tear index at constant breaking length. Increasing the pressure prior to the explosion also leads to better fiber separation and hence lowers refining energy consumption. The effect of pressure on explosion pulps is now being researched over a wider pressure range.

Conclusion

Explosion pulping shows promise as a viable ultra-high-yield pulping process. Compared with the conventional methods of chemimechanical and chemithermomechanical pulping, explosion pulping is very fast (usually 2 min or less) and requires less chemicals.

Comparison of high-yield pulps at the same chemical charge, yield, and ionic content showed that the explosion pulps had superior mechanical and optical properties and lower refining energy. The superiority of the explosion pulps can be attributed to the chemical changes (higher crystallinity, more cellulose on the fiber surface, better lignin softening) that occur following the high-temperature-high-pressure cook as well as the physical changes (better and easier fiber separation) that occur following explosive discharge from the digester.□

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