A rapid method for measuring yield in semichemical pulping processes

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ABSTRACT *A rapid method for estimating pulp yield from pulp density is presented. The method can be used in conventional NSSC (neutral sulfite semichemical) mills as well as in mills that have switched to green-liquor pulping or to the no-sulfur method. Results can be obtained in 1-3 h and are accurate within ± 2.0 points once the method has been calibrated for a given system.*

KEYWORDS
Chemical analysis
Corrugating medium
Density
Green liquor pulping
NSSC pulping
Quantitative analysis
Semichemical pulping
Yield

The yield of semichemical pulp is a major factor determining product quality and production costs, but most mills producing corrugating medium do not control pulp yield as a routine operation (*1*). The main reason for this is the lack of a sufficiently simple, rapid, and accurate method of estimating digester yield for semichemical pulps.

Several NSSC (neutral sulfite semichemical) mills are reported to be measuring pulp yield (*2*) using the Robertson method (*3*), which is based on quantitative determination of organic solids dissolved during pulping. However, we believe that this procedure is not rapid enough for short-term correction of cooking conditions. In addition, the sampling procedure for this method can be impractical in some mills. In such cases, any rapid indirect method would be much more useful.

We have found that for conventional NSSC pulping of birch wood, yield can be estimated based on the pulp's residual lignin content, determined indirectly as kappa number (*4, 5*). Such estimates are accurate enough if the species and quality of the wood raw material are stable.

However, with so many NSSC mills, mainly in North America, switching to green-liquor or nonsulfur processes (*6*), estimating yield has become much more difficult. The semichemical pulps produced using these unconventional processes have a very dark brown color, indicating a high degree of lignin condensation. For such pulps, yield cannot be established reliably using the indirect estimates of lignin content obtained from rapid tests such as kappa number or chlorine number. This is seen in **Fig. 1**, which shows pulp yield vs. kappa number for pulps from three semichemical pulping methods. Only the NSSC pulps show a linear correlation.

This article presents an alternative rapid method of estimating semichemical pulp yield. The method is based on linear correlation between pulp yield and the density of dry pulp. The method was proposed by Duranti (*7-9*), improved by Kovacs and Pavlik (*10*), and successfully applied in a Slovakian NSSC pulp mill in Sturovo, Czechoslovakia.

Fundamentals

From a crystallographic perspective, pulp consists of two fractions:

1. Crystalline components, consisting of high-molecular-weight cellulose and a portion of the hemicellulose
2. Amorphous components, consisting of lignin and the balance of the hemicellulose.

Each fraction has a distinctly different density, 1.588 g/cm^3 for the crystalline components and 1.330 g/cm^3 for the amorphous components (*8, 11*).

It is mainly the amorphous fraction

of wood that is solubilized in the course of pulping, especially semichemical pulping. Based on this fact, Duranti proposed that pulp yield could be determined by measuring pulp density (*7-9*). Duranti adopted the Hermans flotation method of density determination (*11*), which uses chloroform or carbon tetrachloride as a suspending medium.

Hermans (*11*) measured the density of cotton, which has a value close to the density of carbon tetrachloride, by adjusting the temperature of this suspending liquid until the dry fibers tend neither to sink nor float. This simple and rapid method requires only a prior determination of the temperature-density relationship for the suspending liquid. It neglects any slight effect of temperature on the density of pulp substances.

Kovacs and Pavlik (*10*) recognized that the density of carbon tetrachloride at ambient temperature was higher than that of semichemical pulp. The density of chloroform, on the other hand, is lower, so they measured pulp density using a mixture of both solvents as the suspending liquid.

Procedure

The procedure used by Kovacs and Pavlik (*10*) to determine yield for semichemical pulps is as follows: Approximately 50 small (3-5 mg) pellets are formed by fingers from well-washed sample of pulp. Pellets should be dried as swiftly as possible to satisfy the requirements of routine mill control. It is recommended that the pellets be dried in a stream of hot air.

Pulp density is determined by immersing 8-10 dry pellets in a mixture of carbon tetrachloride and chloroform (1:2 by volume) placed in a test-tube. The pellets are deaerated by heating them in a small volume of the suspending mixture (ca. 5 cm^3) to its boiling point. After approximately 20% of the liquid has evaporated, another 5 cm^3 of mixture is added. The glass tube with pulp pellets and liquid is then plugged, cooled, and placed in a thermostatically controlled water bath. The bath is heated gradually, and the temperature at which the pulp starts to sink is recorded to the exact 0.1°C. Pulp density is determined using the established temperature-density relationship depicted in Fig. 2. A fresh portion of suspending liquid should be used in each successive test.

Time of the yield determination, according to Kovacs and Pavlik (*10*), does not exceed 3 h. According to our experience, it can be as short as 1 h if the pulp pellets are dried rapidly enough.

Results

Kovacs and Pavlik (*10*) state that this method is capable of determining yield for NSSC pulps within ±1.5 points, even when blends of many different hardwoods of variable and uncontrolled composition are cooked, which is the case at the Sturovo mill. We attempted to apply the method to such unconventional semichemical pulping processes as green-liquor pulping and the no-sulfur modified soda ash method.

For each pulping method (NSSC, green liquor, no sulfur), 18 laboratory cooks were performed. Chemical charge and time of digestion were varied to obtain different pulp yields. Figure 1 shows yield as a function of kappa number for the three pulping methods. **Figure 3** shows yield as a function of pulp density.

As seen in Fig. 1, yield for the two unconventional semichemical pulping processes cannot be estimated from the pulp's kappa number. Only the NSSC process showed a linear correlation between kappa number and pulp yield. The best-fit regression equation for the NSSC process was

$$y = 7.57 + 0.673x \qquad r^2 = 0.986$$

The standard error of the estimate for the regression equation is 1.16.

Pulp density, however, proved to be an indirect measure of the yield of semichemical pulps in all the investigated processes, as seen in Fig. 3. This is confirmed by **Table I**, which shows the results of regression analysis of data obtained in our study. Determination of NSSC pulp yield from pulp density is less accurate than the kappa-number procedure. Nevertheless, the method is reliable and can be applied to NSSC pulp as well as pulps produced from the green-liquor and no-sulfur processes.

Since each of the evaluated processes had different regression equations, the flotation method of determining pulp yield cannot be treated as independent of the pulping method, as has been suggested in previous studies (*9, 10*). The regression lines for the unconventional green-liquor and no-sulfur processes are distinct from that of the NSSC process, as seen in Fig. 3.

Experimental

Birch (*Betula verrucosa*) mill chips were used as the basic wood raw material of Scandinavian and Polish semichemical pulp mills. The chemical composition of the wood is given in **Table II**.

Green liquor of sulfidity 38.5% was taken from a kraft mill. NSSC and no-sulfur carbonate cooking liquors were prepared in the laboratory from technical-grade chemicals. Liquor compositions were typical for the respective processes. NSSC cooking liquor consisted of 85% Na_2SO_3, and liquor for the modified soda ash process contained 10% NaOH.

Pulping was carried out in six 1-L autoclaves heated in a rotating air bath. Cooking conditions were as follows: impregnation at 80°C and 3.25 L/kg for 2 h, liquor-to-wood ratio 2.5 L/kg, time to temperature (80°C to 175°C) 1.5 h. Total alkali charge and time at temperature were changed in the ranges of 6-11% Na_2O and 10-110 min, respectively.

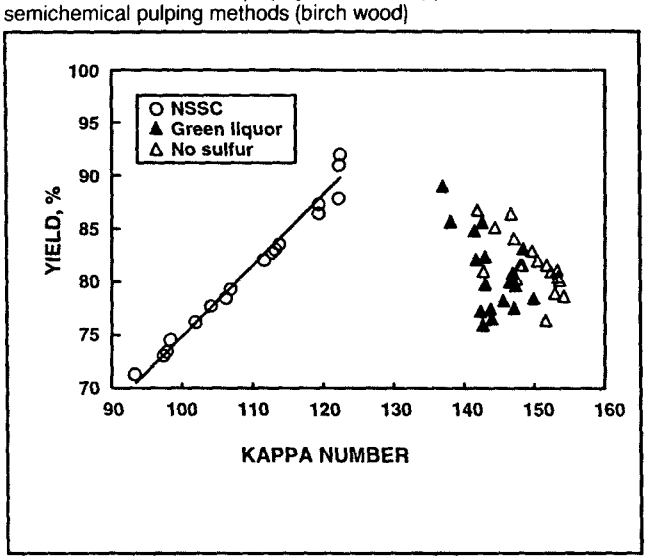
After digestion, chips were washed thoroughly by overnight diffusion in hot water and then dried for yield determination. Cooked chips were soaked in water and then refined in double-pass operation in an 8-in. laboratory Bauer disk refiner at plate clearances of 0.50 mm and 0.25 mm.

Kappa number was determined using TAPPI Test Method T 236 cm-85.

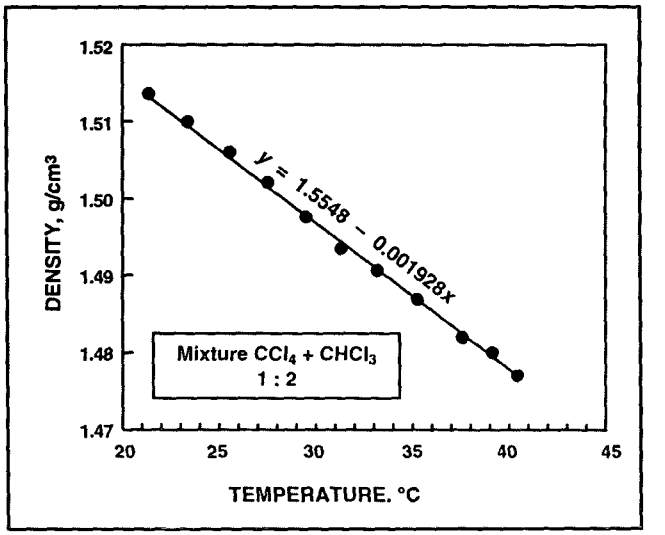
Conclusion

The rapid method for estimating pulp yield from pulp density can be used in any mill producing corrugating medium. This includes mills using the NSSC process as well as those using the green-liquor or no-sulfur semichemical pulping processes. Before the method is used at a given mill, results must be calibrated against pulp yields (mill or laboratory pulps) obtained using some other method of greater accuracy.□

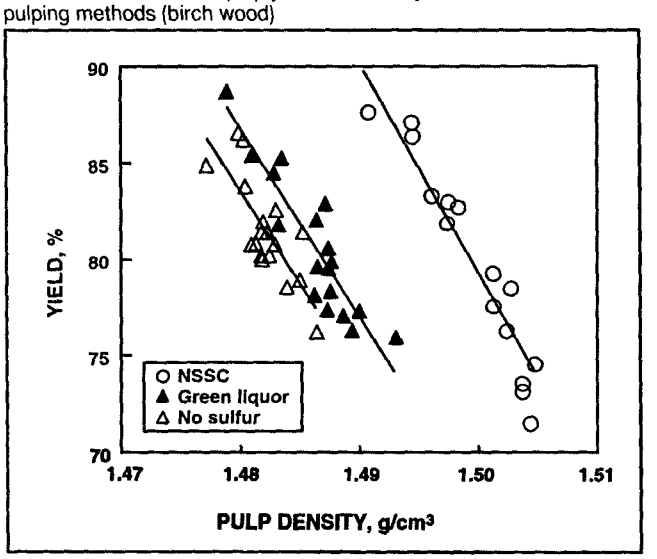
1. Correlation between pulp yield and kappa number for three semichemical pulping methods (birch wood)



2. Density-temperature relationship for 1:2 mixture of carbon tetrachloride/chloroform



3. Correlation between pulp yield and density for three semichemical pulping methods (birch wood)



I. Equations for predicting pulp yield from pulp density for three semichemical pulping methods (birch wood)

Pulping method	*Regression equation*	R^2	*Standard error of the estimate*
NSSC	*y*: 1730.46-1100.98*x*	0.939	1.70
Green liquor	*y*: 1553.07-990.72*x*	0.814	1.64
No sulfur	*Y*: 1507.70-962.18*x*	0.612	1.71

II. Chemical composition of birch wood chips

Component	*Fraction, %*
Total lignin[a]	24.6
Seifert cellulose[b]	40.3
Pentosan	20.0
Solubility in alcohol/ benzene (1:1)	2.2
Ash	0.4

[a]Source: (*12*)
[b]Source: (*13*)

Literature cited

1. Ingruber, O. V., Kocurek, M. J., and Wong, A., Pulp and Paper Manufacture, Vol. 4, Sulfite Science & Technology (3rd edn.), Joint Textbook Committee of the Paper Industry, Atlanta/Montreal, 1985, p.155.
2. Temler, J., and Bryce, J., Pulp Paper Can. 76(2): 92(1975).
3. Robertson, J. D., Tappi 43(3): 192A(1960).
4. Surewicz, W., and Modrzejewski, K., Przeglad Papier. 34(9): 347(1978).
5. Surewicz, W., Modrzejewski, K., Mroz, W., and Gizowski, M., Przeglad Papier. 35(1): 38(1979).
6. Hanson, J. P., Pulp Paper Intern. 20(5): 50(1978).
7. Duranti, D., and Colapietro, M., Cellulosa Carta 18(8): 3(1967).
8. Duranti, D., Cellulosa Carta 18(12): 25(1967).
9. Duranti, D., Cellulosa Carta 19(2): 13(1968).
10. Kovacs, V., and Pavlik, S., Papir Celuloza 37(3): 39(1982).
11. Hermans, P. H., Contribution to the Physics of Cellulose Fibers, Elsevier, New York, 1946.
12. Surewicz, W., Plonka, A. M., and Wandelt, P., Zellstoff Papier 23(11): 327(1974).
13. Seifert, K., Papier 10(13/14): 301(1965).

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