Process chemistry and control of rapid-displacement heating

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The upper-level digester-house management system is the crucial tool for maintaining and optimizing operation of the digester house.

The rebirth of kraft batch cooking has become a reality. The modified batch processes combine the versatility of batch-type operation and improved heat economy. High-quality kraft pulp can be produced by a batch-digester house with the following advantages:

- Sixty percent less primary heat consumption
- First washing stage in the digester
- Extended delignification
- Improved strength, with up to 15% more tear index.

These benefits are achieved by arranging the digester design and cooking cycle so that the heat of an individual cook can be recovered and used in subsequent cooks. The implementation involves:

- Digesters equipped with bottom and top strainers
- Tank "farm" for reusable liquor
- More cooking-cycle operations
- Effective process management.

Equipment suppliers have introduced very similar modified batch-digester-house designs to the market. The rapid displacement heating (RDH) concept by Beloit and Rauma-Repola and the Sunds-Celleco modified batch concept by Sunds Defibrator are currently the only designs with full-scale commercial installations.

The studies presented here were performed in the Joutseno pulp mill's RDH digester house, which represents the latest comprehensive modified batch-digester-house installation, with 11 digesters and up to 70 blows per day. The need for an upper-level optimizing control system was recognized during the early stages of design and engineering. After choosing the AFORA autocook control system, the mill initiated a thorough investigation into the process chemistry, which had never been comprehensively studied on a mill-scale basis.

Mill personnel were involved with startup operations and the optimization of a new cooking process, while AFORA was simultaneously working on the problem of process control. A cooperative research group was established to work in parallel with the installation-project group. This research group included the laboratory of pulping technology at the Helsinki University of Technology, Joutseno Pulp Oy, and AFORA Oy. The Laboratory of Pulping Technology provided a graduate student for experiment work. Joutseno's project and production engineers formed an advisory group and supported the initiative, and the mill's laboratory offered its research facilities. AFORA provided a second, supplementary cooking-liquor analyzer for off-line experiments and conducted research into data-handling and computing methods.

Cooking chemistry

Renewed interest in batch cooking has been attributed mainly to energy savings in the range of 2 to 3 GJ/a.d. metric ton of pulp, which is quite considerable and certainly offers a new perspective on the former competition of batch vs. continuous cooking. There have also been major technological developments in the batch-cooking process. The intensive research into the kraft batch-cooking process has been instrumental in developing new optimization strategies for the kraft process, which until now was straightforward. With its numerous phases, the kraft batch-cooking process offers many opportunities for optimization in the major cooking phases, during impregnation/pretreatment, and during bulk-phase and residual-phase delignification.

Separation of the cooking procedure into several stages where liquors are added and displaced represents a real departure from conventional batch cooking. The cooking chemistry is also different. We present here a short review of current knowledge on the chemistry of the modified kraft batch-cooking process. The different methods will be referred to by their abbreviated commercial names:

- RDH: rapid-displacement-heating method (*1*)
- SC: Sunds-Celleco method (*2*)
- WLI: Ekono's white-liquor-impregnation method (*3*).

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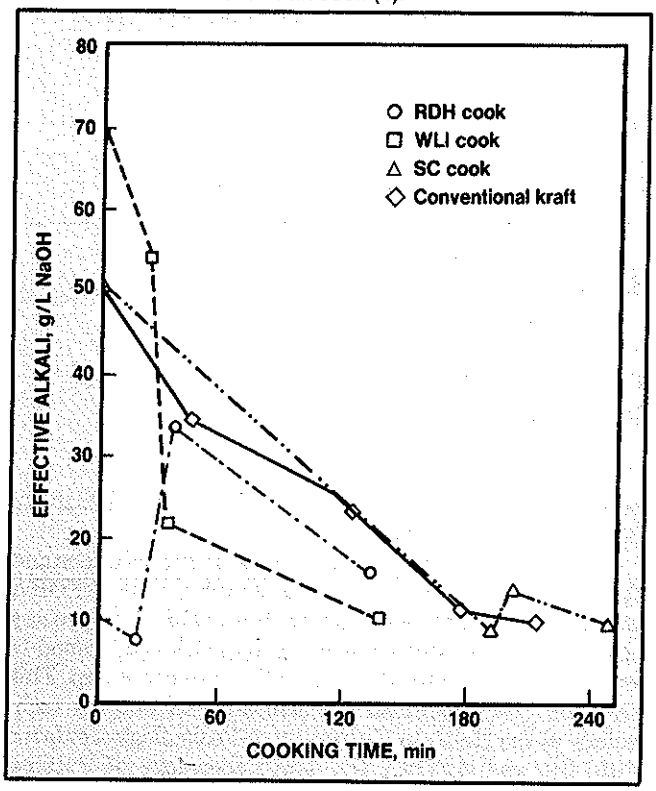
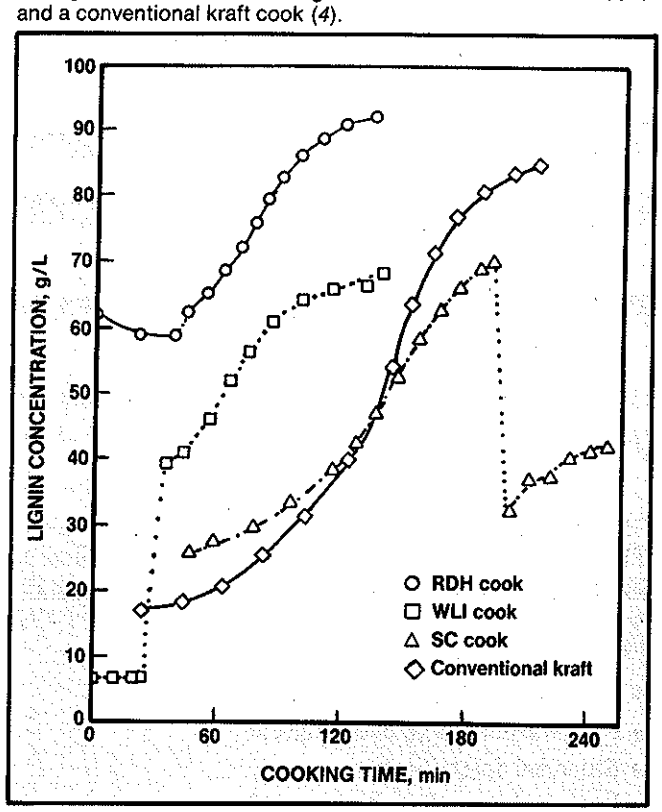
1. Effective-alkali concentration during three modified kraft batch cooks and a conventional kraft cook (*4*).



2. Lignin concentration during three modified kraft batch cooks and a conventional kraft cook (*4*).



I. Comparison of lignin and alkali concentration with respect to conventional kraft batch process

	Initial phase		*Cooking phase*			
			Beginning		*End*	
Method	*Lignin*	*Alkali*	*Lignin*	*Alkali*	*Lignin*	*Alkali*
RDH	High	Low	High	Low	High	High
WLI	Low	High	High	Normal	Normal	Normal
SC	Normal	Normal	Normal	Normal	Low	High

These methods have been the object of numerous studies. However, the results of these various studies are not commensurable. This has been the main obstacle in conducting a logical comparison of these methods. Jokela (*4*) recently undertook a technical and economic comparison of these methods under continuous experimental conditions.

Alkali profiles

White liquor charge is introduced in different ways for each of these modified methods. Generally, the RDH cook has the smoothest alkali profile; the SC cook is the closest to a conventional batch cook; and the WLI cook has the largest variations. These trends are illustrated in **Fig. 1**.

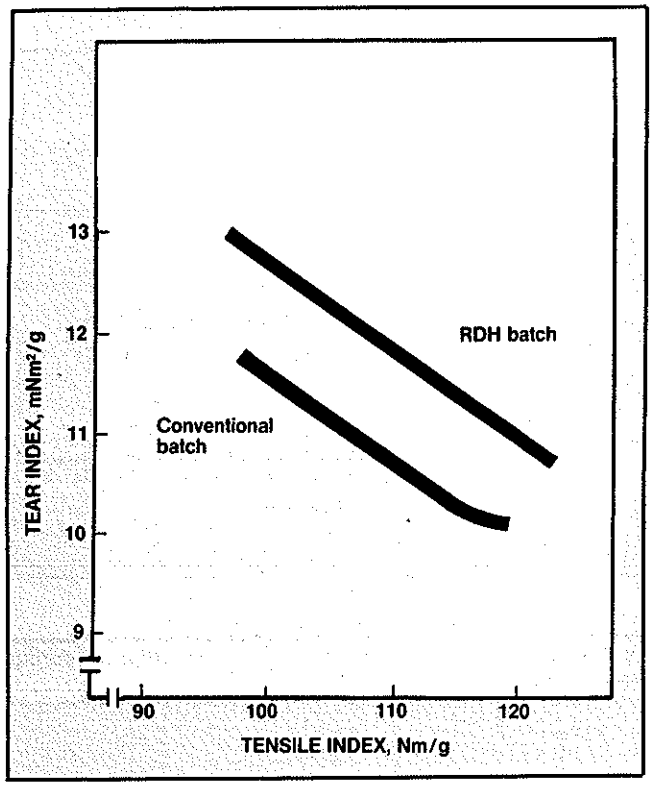
The methods offer a wide range of initial cooking conditions in the first stage: from the mildly alkaline black liquor in the RDH cook to the pure white liquor in a WLI cook. It had been assumed that the wide fluctuation in the concentration of cooking chemicals would lead to unselected delignification (*5*), but a more recent comparison (*4*) showed only minor differences in selectivity and pulp viscosity.

There is more similarity among the methods in the cooking phase, particularly in terms of effective-alkali concentration. The large difference between the effective-alkali concentrations at the start of the RDH and WLI cooks diminishes as the cook proceeds, and the alkalinity of the RDH cook begins to approach that of the conventional cook. At the end of the cook, the residual alkali concentrations for the WLI, SC, and conventional cooks flatten down to about 10–15 g/L. The alkalinity in the SC cook's second cooking phase remains slightly higher than in the conventional kraft cook, while alkalinity in the WLI cook is lower than in the conventional kraft cook. Residual alkali in the RDH cook remains on a slightly higher level, at approximately 17 g/L, which is quite reasonable, since the liquor is reused in forthcoming stages.

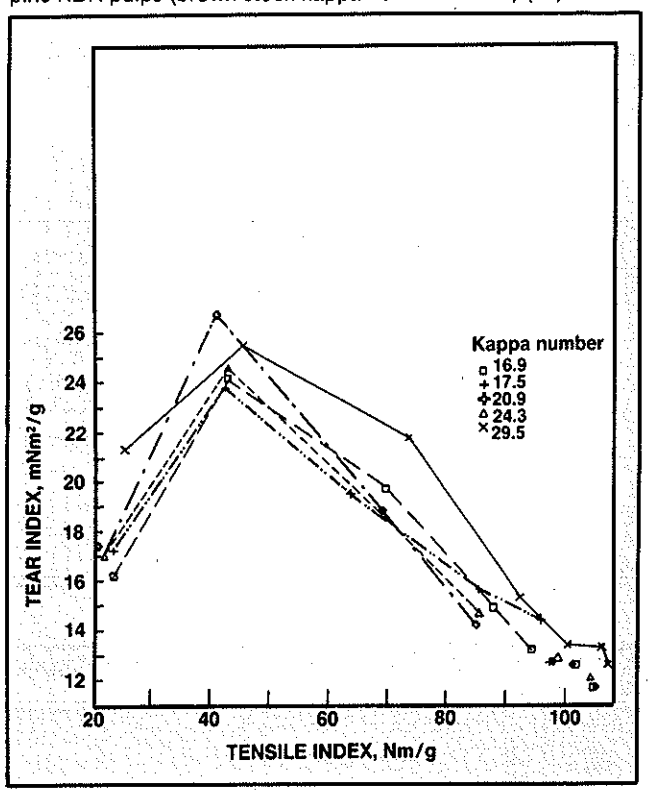
The most important feature in RDH and WLI cooks is the separate chip-impregnation stage. This assures a more uniform delignification over the chip particle's cross section, resulting in less rejects. The thorough impregnation of the chips also neutralizes the wood acidic groups, which helps decrease fluctuation in alkali consumption during the cooking phase and promotes cook uniformity.

The special feature of the RDH cook is what happens inside the chip particle during black liquor impregnation. Following consumption of the black liquor's residual alkali, the conditions prevailing in the chip particle's interior can resemble those of the so-called hydrogen sulfide pretreatment. The hydrogen sulfide pretreatment is well known for

3. Tear index vs. tensile index for RDH-cooked and conventionally cooked bleached pulps (brown stock kappa no. × 30) (*9*).



4. Tear index vs. tensile index of (C+D)EDED-bleached Finnish pine RDH pulps (brown stock kappa no. × 16.9–29.5) (*10*).



its carbohydrate-stabilizing effect, which occurs through the reduction of polysaccharide end groups at near-neutral pH. The increased carbohydrate yield results in higher yield and good strength properties (*6, 7*). A complementary hypothesis states that the achieved sulfide concentration in the chip's interior during the early stage of the cooking phase is beneficial to delignification selectivity. Whatever the case, the apparent sulfidity in the cook is high, and the net impact of this chemical phenomenon is not yet empirically known.

A further stabilizing feature of the RDH cook is the high liquor-to-wood ratio resulting from operation of the hydraulic digester. This keeps alkali concentration low while retaining a large buffer capacity.

One common feature for all of the modified batch methods is that the shorter cooking time—the result of rather high initial temperatures following liquor charge—lessens the exposure of carbohydrate material to alkaline degradation.

Lignin profiles

Delignification during the various cooking phases has been studied with a cooking-liquor analyzer, which monitors lignin concentrations during pilot cooks (*4*).

Figure 2 shows the development of lignin concentration during the modified kraft batch-cooking processes. The different cooking conditions for each of the various methods are evident in the dissolved-lignin curves, which reflect the corresponding alkali curves in Fig. 1. Once again, the difference between the beginning phases of the RDH and WLI cooks is clearly illustrated. The lignin concentration in the beginning phase is high in the RDH cook and low in the WLI cook. The SC cook's first cooking stage resembles a conventional cook.

The development of lignin concentration at the end phase of the cooks is characteristic as well. In RDH and WLI cooks, lignin concentration increases swiftly following the hot liquor charges and steaming up. In the final stage, the SC cooking curve drops dramatically when some of the cooking liquid is displaced by hot new liquor (white liquor and wash liquor), which lowers the lignin concentration considerably. In this case, the similarity between the SC batch cook and the modified Kamyr continuous cook is obvious (*8*).

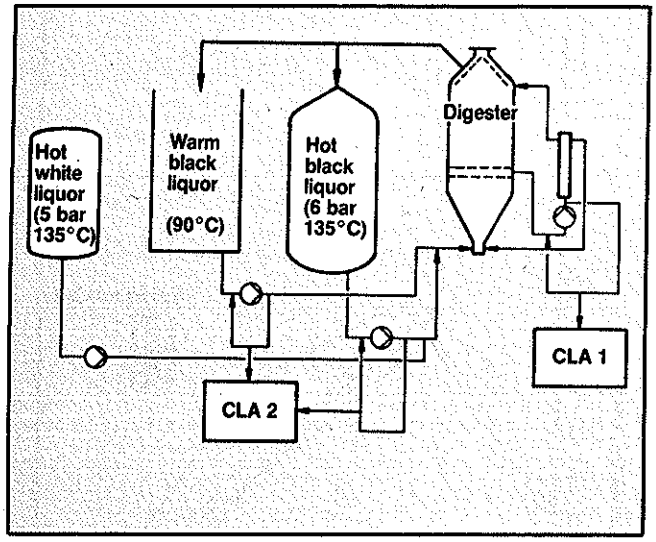
As a group, the modified pulping processes cover a broad range of possible lignin concentrations. **Table I**, based on the data in Figs. 1 and 2, provides a relative comparison of alkali and lignin concentrations during the modified cooks.

Pulp quality

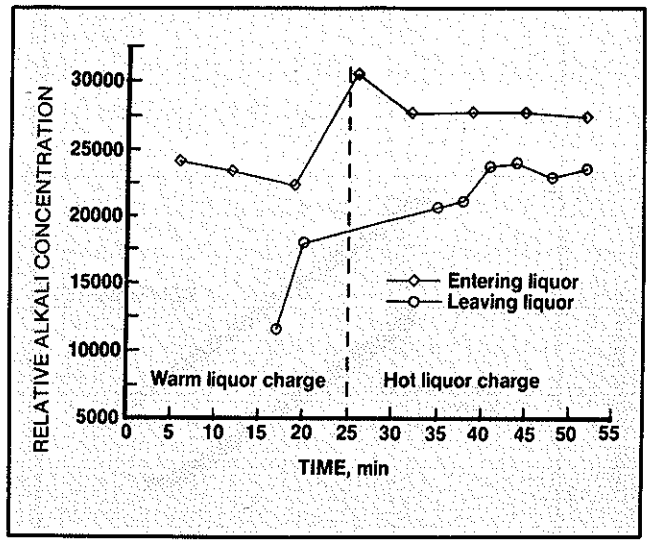
The fundamentals for each of the modified processes have been derived from a wood-chemistry point of view. It is claimed that through this assumption various benefits are gained with respect to pulp quality via different mechanisms. Without going into the details of these arguments which have been discussed elsewhere (*1, 2, 5, 8*), some general statements have emerged. The first problem deals with the definition of pulp quality.

In trying to assess the effectiveness of different pulping processes, pulp viscosity is not a reasonable criterion in judging pulp quality. It is quite evident that the relationship between viscosity and strength is valid, but only in the broadest sense. It is possible to define a viscosity range within which the pulp quality is good, but the absolute readings are not an indication of strength properties of RDH pulps, nor are they valid in comparison with pulps obtained from other modified processes. The only remaining criterion for judging pulp quality is a tear-vs.-tensile

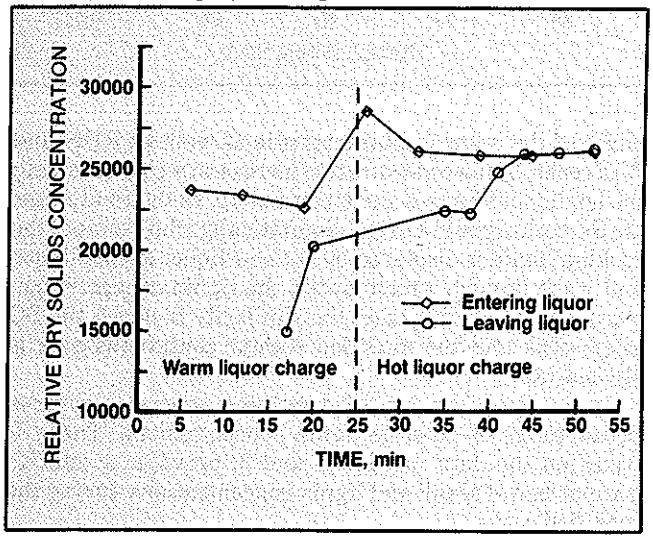
5. Schematic view of the Joutseno digester house and the cooking-liquor analyzer connections.



6. Relative residual alkali concentration for incoming and outgoing liquors during liquor charge.



7. Relative dry-solids concentration for incoming and outgoing black liquors during liquor charge.



8. Development of pH for black liquor leaving the digester during liquor charge.

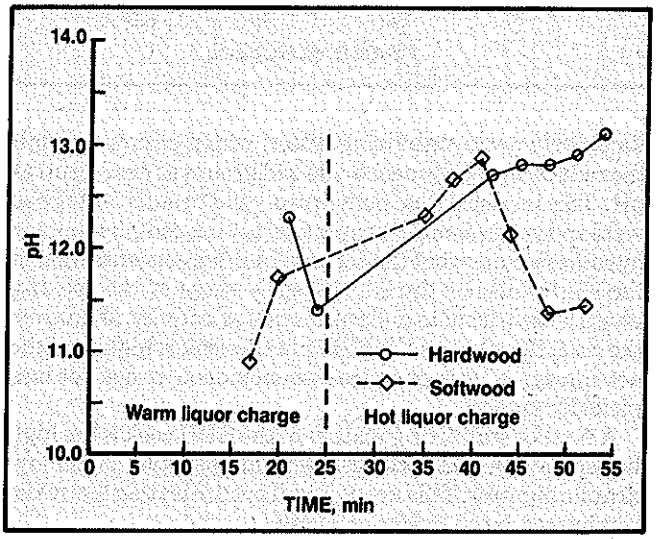


diagram for pulps at a specific kappa number.

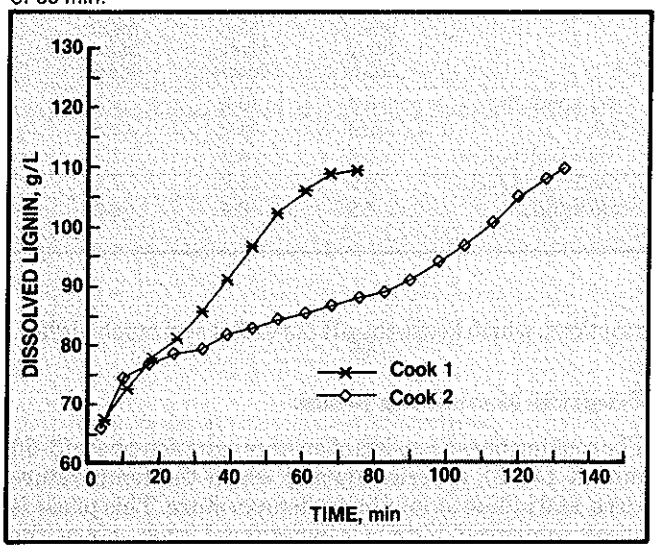
All of the modified methods seem to provide pulp of good quality. This highlights the need for new explanations for some strange results. For example, theories about the effect of low lignin concentration on residual delignification selectivity and on extended delignification can be justified in terms of viscosity and strength for SC grades (*5*). However, pulp from an RDH-type extended delignification, which has extremely high terminal lignin concentrations, shows exceptionally good strength properties in spite of slightly lower viscosities (*9, 10*). **Figure 3** illustrates the strength properties for RDH-cooked and conventional batch pulps.

Figure 4 shows the results of one test trial for mill-scale pine cooks with kappa numbers ranging from 16.9 to 29.5. It is quite evident that strength development for the softwood bleached pulp in the kappa number range 17–30 differs drastically from that of conventional kraft pulps and is a sign of new mechanisms in the development of fiber composition and nature during the split-up cook of the RDH process.

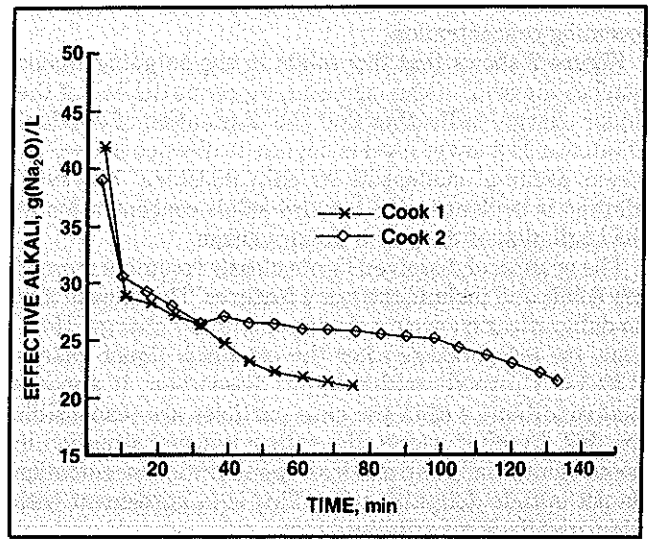
Apart from the cooking chemical point of view, the role of cold, nonflashing blow must be kept in mind. The importance of controlled, nonaggressive cold blow—or dump—has been well established in the development of the continuous cook, and the cold blow certainly contributes to the unique features of the modified batch processes. The effect is qualitatively known, but many questions remain unanswered.

- Why is the quality of conventional batch blow and Kamyr cold blow almost equal?
- Why does the quality of RDH pulp exceed that of Kamyr-cooked pulp (Scotch pine)?
- Where is the dividing line between the beneficial effects of modified cooking phases (chemistry) and gentle, nonflashing blow (fiber physics)?

9. Development of dissolved lignin concentration during two RDH cooks with different impregnation times. Cook 1 at minimum impregnation time of 3 min. Cook 2 at maximum impregnation time of 60 min.



10. Development of residual alkali concentration during RDH cooking phases for cook 1 (minimum impregnation time) and cook 2 (maximum impregnation time).



- What would happen if a conventional kraft batch was displaced and dumped cold?

RDH digester house

The RDH digester house at the Joutseno mill consists of 11 digesters. Four of these are new 200-m³ digesters, and seven are old, modified 140-m³ digesters. The tank farm includes tanks for warm and hot black liquor, hot white liquor, and displacement liquor. Heat exchangers are used to produce hot white liquor and hot water. Produced grades are pine and birch with kappa number targets of 32 and 20, respectively. Production can be as high as 70 blows/day, with total pulp production at 950 a.d. metric tons/day (*9*).

The digester house has an Alcont digital instrumentation system and a higher-level AFORA Autocook process control. Autocook operates as the optimizer of production, economy, and pulp quality (*11*). Quality control is based on dynamic cook models, with data provided by an on-line AFORA cooking-liquor analyzer (CLA). **Figure 5** provides a simplified flow scheme of the digester house and the analyzer connections.

CLA 1 is an integral part of the Autocook process-management system. The second analyzer (CLA 2) was an extra installation for research purposes. CLA 2 is connected to the liquor lines between warm and hot black liquor tanks and digesters. CLA 1 normally analyzes two samples per cook. During the monitoring trials, it was used to analyze up to 20 samples per cook, with a sampling frequency of 5-min. CLA 2 was used to monitor the liquor entering and leaving the digesters.

The CLA provides rapid multimeasurement analyses of cooking liquids. In kraft cooking mode, the CLA takes a single sample and within 5 min provides measured concentrations of

- Dissolved lignin
- Dry solids
- Residual alkali.

The CLA measurement technique has been described in another paper (*12*).

Quality of recirculated liquors

After the cook, black liquor is displaced by wash liquor to a black liquor tank. During liquor displacement, the outgoing liquor flows either into a warm black liquor tank ($T < 105°C$) or into a hot black liquor tank ($T > 105°C$). Hot black liquor is conducted to the warm black liquor tank through heat exchangers. This is why recirculated black liquor contains high concentrations of dissolved lignin and other dissolved organic substances together with high-sulfidity residual alkali. The composition of these liquors defines the chemical conditions for cooking. Dry-solids content, sulfidity, and dissolved lignin are maintained at a high level throughout the RDH cook in a hydraulic environment with a liquid-to-wood ratio of about 5:1. **Table II** presents some typical laboratory values for recirculated liquors.

The quality of warm black liquor is subjected to continuous oscillation as a result of successive tank fills from the hot black liquor tank and from displacement liquors under 105°C. The magnitude of fluctuation in tanks could not be measured directly, but liquor quality can be estimated from profiles of the incoming and outgoing liquors. These profiles were monitored, and the results are presented in the following section.

Progress at liquor charge

During the liquor charge, the black liquor entering and leaving the digester was analyzed by the CLA, which provided measurements that were used to determine the reactions occurring in this stage. Curves indicating the concentrations of residual alkali and dry solids and the pH during the first part of a typical RDH cook are shown in **Figs. 6–8**, respectively. The dissolved-lignin profile closely resembles that of dry solids.

Figure 6 shows that residual alkali in liquor leaving the digester is lower than in the incoming liquor. The dif-

ference between incoming and outgoing warm black liquor is quite large initially. Finally, during the hot liquor charge, the outgoing residual alkali concentration increases and flattens out at a level about 25% less than the incoming concentration.

Figure 7 shows that dry solids in the outgoing liquor reaches the level of the incoming liquor at the end of the liquor charge, indicating that the diminution of residual alkali to a 25% lower level is not due to dilution caused by steam packing condensate or chip moisture. However, dilution is evident in the low dry-solids concentration at the beginning of the warm liquor charge.

The profiles of dissolved lignin closely resemble those of dry solids and include the same features. This means that no delignification occurs during the liquor charge. However, the temperature after the hot black liquor charge (130–140°C) would enable lignin dissolution. It appears that conditions within the chip particles are responsible for this. In other words, the rather unusual situation in terms of internal alkali and hydrogen sulfide concentration do not initiate delignification. This is in agreement with discussed literature findings (*13*).

Figure 8 shows that the development of pH is different for hardwood (birch) and softwood (pine). Neutralization reactions with carboxylic groups in the wood material are responsible for the characteristically low pH readings.

pH during liquor charge for softwood

Figure 8 shows the outgoing black liquor at pH 11 during warm liquor charge, indicating a rather high consumption of alkali. From this point, the pH rises as larger quantities of percolated warm black liquor leave the digester, indicating that a large portion of the liquor's alkali does not react.

A rapid drop in pH can be observed when the hot black liquor charge reaches the outlet. This indicates another wave of rapid alkali-consuming reactions following the exposure of wood material to hot black liquor. After the first reaction front, pH increases slightly before the outlet is closed, and the cooking phase starts, followed by mixing of the hot white liquor.

pH during liquor charge for hardwood

Figure 8 shows a rapid, 1-unit drop for pH at the beginning of liquor outlet for hardwood pulp. After that, pH rises steadily until the liquor charge is over. A hypothetical explanation for the initial drop in pH might include the fact that birch cooks are not filled with the Svensson-steaming, and the first front of percolating liquor removes air, penetrates into chips, and warms up the chip charge. Shortly after this, rapid neutralization reactions begin to lower the pH.

The interesting difference between the softwood and hardwood curves at the hot black liquor front (softwood pH drops, while hardwood pH continues to rise) may be due to the difference in hemicellulose composition. The softwood galactoglucomannan material starts to react at temperatures exceeding 100°C, and the dissolution is caused mainly by rapid peeling reaction. The arabinoglucuronoxylan of hardwood reacts only at temperatures exceeding 140°C, and the dissolutive peeling reaction is hampered by arabino grafts (*13,14*). The net result is that the larger-scale consumption of hemicellulosic alkali starts in softwood liquor charge with the introduction of hot liquor, while hardwood hemicelluloses are more resistant to rapid reactions, which keeps the pH curve flat and much higher.

II. Typical composition of recirculated black liquors

	Warm black liquor	*Hot black liquor*	*Displacement black liquor*
Dry solids, %	15.3	16.7	7.3
pH	12.9	13.0	11.5
NaOH, g(Na_2O)/L	4.0	4.0	1.6
Na_2S, g(Na_2O)/L	6.9	8.8	7.3
g(Na_2O)/L	7.5	8.4	5.3

Progress at cooking phase

The cooking phase consists of an impregnation phase (3–60 min at 140°C), a period during which the temperature rises, and a time at maximum temperature. This phase is short compared with a conventional cook, since the charging liquors are already close to the cooking temperature. At the beginning of the cooking phase, the temperature is between 135°C and 145°C, pressure is 5 bar, dry-solids concentration of liquor is 17–18%, and effective-alkali concentration is close to 40 g(Na_2O)/L. Sulfidity is high because of the high concentration of recycled black liquor.

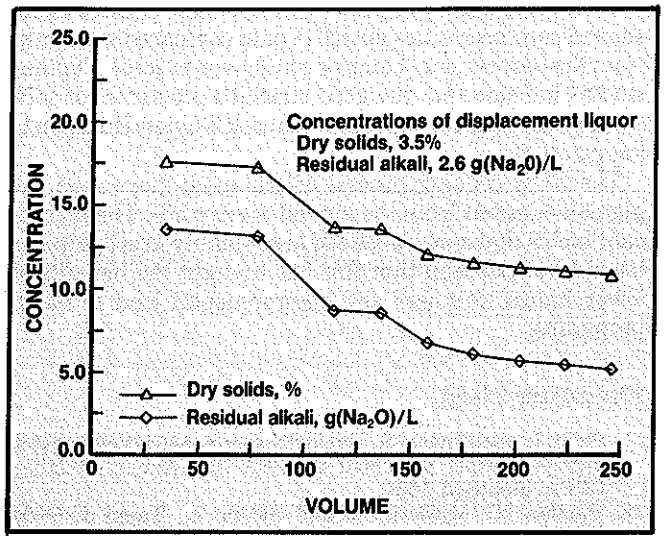
Delignification starts immediately after white liquor charge, when the alkali concentration increases in the hot-black-liquor-soaked chips. **Figure 9** shows dissolved lignin curves for two cooks. Cook 1 was conducted at the minimum impregnation time of 3 min. Cook 2 was conducted at the maximum impregnation time of 60 min. In cook 1, bulk delignification starts rapidly as soon as the cooking phase reaches 155°C. Normally, this takes 10–15 min. In cook 2, bulk delignification is only faintly visible and starts at a somewhat higher temperature. Slow delignification and alkali consumption proceed at a constant rate during the impregnation. The long cooking-phase impregnation is not recommended for the following reasons:

- A very low reject percentage of RDH pulp is achieved with the minimum of cooking-phase impregnation.
- The slow delignification during impregnation is unselective.
- The production rate decreases.

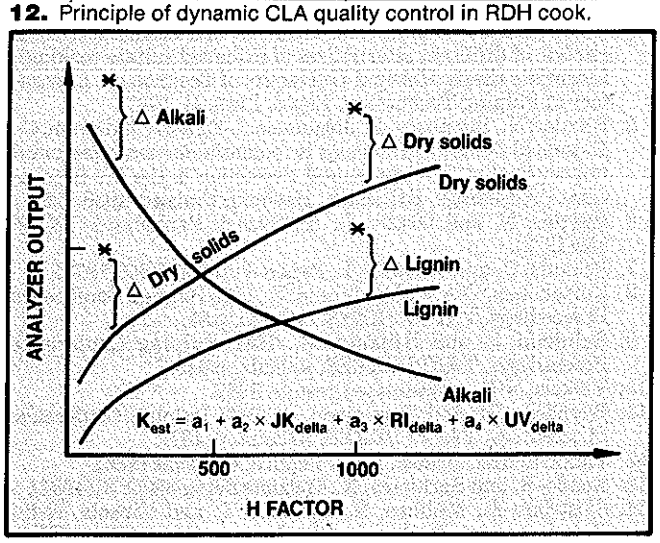
The target kappa number of cooks 1 and 2 was 30. There is almost no residual delignification to be seen after the rapid bulk delignification. In some cooks, there was no change in the slope of the lignin curve. The high liquid-to-wood ratio probably tends to flatten the curves downward somewhat, but the delignification still seems to continue at a high reaction rate to kappa no. 30. This is in agreement with the finding that softwood RDH pulps can be cooked easily down to kappa no. 20 without severe losses in selectivity and pulp strength.

Figure 10 shows that the alkali concentration during cooks 1 and 2 dropped by 30% during the first 15 min. This is less than in conventional cooking and can be explained by the higher liquid-to-wood ratio and the already-reacted alkali in the black liquor charges. Following the rapid drop, alkali consumption slows down and continues on a slightly descending slope to the end of the cook. The

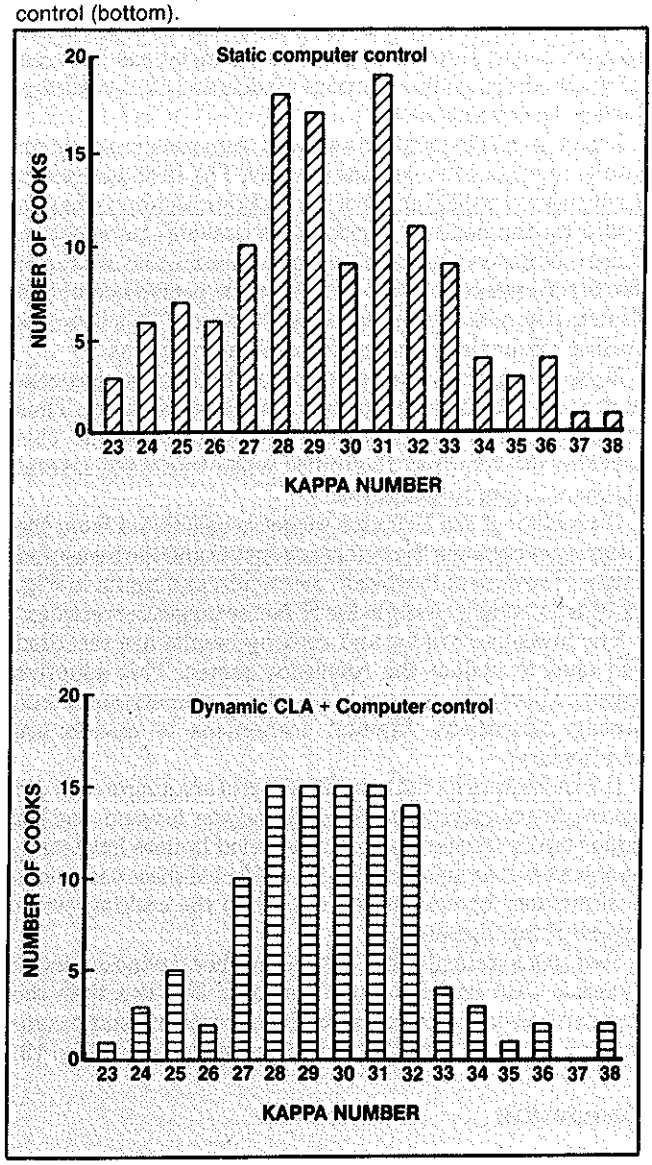
11. Dry solids and residual alkali concentrations in outgoing black liquor during displacement.



12. Principle of dynamic CLA quality control in RDH cook.



13. Distribution of kappa number for RDH cooks controlled with conventional computer control (top) and CLA plus computer control (bottom).



residual alkali concentration obtained can be as high as 20 g/L as Na_2O.

The dry-solids content starts at a rather high level of 17–18% and rises only about two percentage points during the cook. This is again due to the high liquor-to-wood ratio and the fact that a portion of the early reaction products has been removed from the digester along with the overflow of the black liquor charge.

Progress at displacement phase

After the cooking phase, the wash liquor displaces the cooking liquor in the digester. The purpose of this displacement is to save the energy of the black liquor. At the same time, displacement is the first washing stage. Dry-solids and residual-alkali concentration curves in outgoing black liquor from the digester are presented in **Fig. 11.** Here we can see that ideal displacement continues until approximately half of the free volume of the digester has been pumped out. The proportion of wash liquor in the outgoing liquor increases steadily, and displacement proceeds without any difficulties.

Control aspects

There are numerous demands placed on the Autocook process-management system:

- Maximum production
- Optimized operation of tank farm
- Economical operation during both high and low rates of pulp production
- Smooth steam-consumption profiles
- Low deviation of pulp quality despite uneven chip material.

These demands partly contradict each other. Maximum production and low deviation in pulp quality cannot take place at the same time when there is fluctuation in chip quality. It was obvious from the beginning of the modification project that the demands for quality control could not be fulfilled through conventional computer control.

The dynamic features in static control were introduced by integrating the CLA analyzer with mathematical

reference model control. The implemented quality control eliminates long-term variations via static kappa number feedback control, which corrects the H-factor target of the digester house. Individual short-term variations between cooks are corrected by reference models and CLA readings during the ongoing cook.

The CLA carries out two analyses from each cook, which results in up to 140 analyses per day. The first analysis is performed at the beginning of the cook during impregnation or during the heat-up period. This provides information about cooking conditions after impregnation and mixing of the liquor charges. A second analysis is performed at 300 H-units before the estimated end of the cook. This provides feedback information about the cooking reactions.

Measurement results are compared with updated concentration reference curves as a function of H-factor. Thus sampling points are not tied to precise time points. This provides the required flexibility when there are several digesters requesting analysis.

Correction of the H-factor target is calculated from the differences between the reference curves and the measured values. The curves for alkali, dry solids, and lignin in **Fig. 12** illustrate the principle for H-factor target corrections.

The measured values and cooking results are recorded and used to update the reference curves. This adaptive feature allows the system to be adjusted to accommodate process alterations. Manual corrections to models are unnecessary.

It was discovered that quality control of a single cook did not require connection of a second analyzer to warm and hot liquor tanks. Concentration of recycled liquors have long- and short-term variations, but the effect of these variations is eliminated by connecting CLA 1 to the cooking circulations of the digesters.

Test trials indicate that kappa number deviation under dynamic CLA-based quality control is 30% less than the standard deviation obtained with conventional static computer control. Results of one trial are presented in **Fig. 13.**

Conclusion

The joint study conducted on the Joutseno mill's rapid-displacement-heating system revealed the versatile chemical conditions in the various stages of the modified kraft batch-cooking process. In general, separation of the kraft batch-cooking process into multiple stages seems to enhance the kraft process. The partial consumption of black liquor residual alkali in the first black liquor stage neutralizes most of the wood's acidic groups and seems to generate unique reaction conditions in terms of pH and the role of sulfur. The net effect is stabilization of the subsequent cook, which starts rapidly at approximately 140°C, resulting in a shorter cooking time than in a conventional cook. Delignification does not seem to have a distinctive residual phase, but lignin concentration increases at a more or less constant slope until the end of the cook. Subsequent displacement of hot black liquor and simultaneous washing with wash filtrate proceed without any problems. The conventional flashing blow is replaced by a gentle, cold dump using pressurized air.

The upper-level digester-house management system is the crucial tool needed to maintain and optimize the complex operation, which involves high production rates and numerous intermediate events. The quality control aspect of the system is based on a new dynamic cooking control system that uses feedback from an on-line cooking-liquor analyzer.

The RDH pulp is of superior quality. Tear-tensile results show record properties for RDH pulp compared with pulp from the conventional batch or continuous process. Further studies indicate that the good strength properties of softwood pulps do not drop, even if the cook is extended down to kappa no. 20.

The RDH cooking process offers much more than the gigajoules saved from the annual energy bill. The significant fact is that a new grade of high-quality kraft pulp has been developed and that this has brought the benefits of lower kappa numbers and environmental load to batch pulp mills.

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