Solving soap and turpentine related process problems in softwood kraft mills

Making a case for soap audits

By P. Tikka, K. Kovasin and T. Laxén

Pulping processes have been developed and designed to deal with the removal of lignin from the wood chips and wood fibres at varying descending residual lignin concentrations, as if the wood raw material consists only of lignin and cellulosic fibre. The fate and co-processing of wood extractives is only sporadically addressed, usually after problems occur. This is especially true in softwood pulping, where soap (and turpentine) can cause severe losses of production, decrease pulp quality and reduce thermal and chemical efficiencies. Worries about lost production and pulp quality due to extractives often result in continuous injection of defoamers. This is generally accepted as normal: that is, taking an aspirin instead of curing the disease.

We recently evaluated the costs of extractives problems in the black liquor side of pulp mills. For a large modern softwood kraft mill the numbers are (US$/ton pulp):

- Additives, defoamers: 1.6
- Losses of chemicals, higher consumption of chemicals: 5
- Off-spec. pulp, lower income: 0.2
- Losses of turpentine and tall oil: 0.2
- Studies and process changes: 1

In addition to suffering this 8 US$/ton pulp in extra costs, some mills still continue to under-perform due to soap-related problems in the brownstock fibreline and the evaporation plant. If extractives were not an issue, these mills could increase their pulp production by 5-10%!

These estimates may be too high for the better-operated mills, but we believe they are typical of most older kraft mills. The message is clear: there is a lot of scope for better process solutions and better operation.

WHY SEPARATE SOAP?

Soap in the process strongly influences the whole mill operation. Besides degrading over-all performance, it can accumulate and cause sporadic upsets, which in turn lead to environmental consequences. Thus, it is of great importance for mill personnel to understand how extractives-related issues give rise to process disturbances, off-spec pulp and environmental excursions.

The list in Table I indicates the main areas affected by soap problems and disturbances. It is rather typical that upsets do not remain isolated in one area but often spread through the whole mill.

As discussed later, soap solubility is dependent on sodium and alkali levels in the liquor. In the first washing stage, with high concentrations of sodium, most of the soap remains in solid form and is not transferred to the filtrate. In the following stages, sodium is diluted and the soap is solubilized.

Washing — soap starts a chain of events: Dissolved soap starts foaming as soon as air or steam is present, either in the washing zone or in the filtrate tank. It is essential to collect foam as effectively as possible and to minimize the amount of soap in the counter-currently pumped filtrate. Foam increases filtration resistance and washing losses increase [1].

Insufficient washing results in soap accumulation in the filtrate of the following stage. Low efficiency factors in washing equipment are often exacerbated by great variations in the extractives content or in the composition of the wood furnish. Any washing losses carried over into an oxygen stage impair the kappa reduction and reduce pulp strength [2]. The damaged pulp suffers further during bleaching.

Soap & evaporation: Soap should be collected from weak liquor to avoid foaming in evaporators. Foaming increases chemical oxygen demand (COD) levels in condensates that are typically reused. If highly contaminated condensate is used for pulp washing, it has the same effect on pulp as a high washing loss — we are recycling the problem. Another difficulty is that soap greatly accelerates any Na_2SO_4-Na_2CO_3 scaling in the first effect of the evaporators. A decrease of soap content from 13 to 7 kg soap per ton of solids in the feed liquor to the first effect may decrease the scaling rate by 25% [3].

There is a further problem that may arise from soap in the evaporation area: in many softwood kraft mills where soap is not carefully skimmed, a thick, solid soap layer builds up in the black liquor storage tanks — effectively reducing the storage capacity and interfering with tank management.

Soap in recovery operations: There have been many warnings about blackouts in recovery boilers caused by the entry of slugs of soap containing pockets of weak liquor. To avoid this kind of hazard, it is recommended that if separated soap is to be burned in a recovery boiler, it should first be acidified to crude tall oil [4].

If the boiler is a bottleneck, feeding it with soap decreases the capacity of the mill, but this is frequently the only option. When soap is

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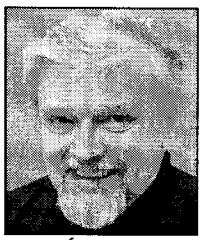

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TABLE I. Areas where soap influences the process by foaming or accumulation.

EFFECTS OF DISTURBANCES	
AREA:	**IMPACTS:**
Brownstock washing	Soda loss, bleach demand
Oxygen delignification	Pulp quality
Evaporation condensates	Chemical oxygen demand (COD)
Evaporation	Scaling, black liquor tank management
Recovery boiler	Performance
Effluents	Biological and chemical oxygen demand (BOD, COD)

TABLE II. Parameters to be optimized for effective soap separation.

SOAP SEPARATION: KEY PARAMETERS
Ratio fatty acids: resin acids
Residual effective alkali (EA)
Sodium content
Dry solids content (EA)
Retention time in skimmer (superficial liquor velocity)
Temperature

being burnt with the black liquor, it is important to ensure a uniform flow: random fluctuations of soap content in black liquor can cause an unstable recovery boiler operation.

So far, defoaming agents have not been mentioned. The agents are a good remedy for foaming both during washing and during weak liquor evaporation, but they are not miracle medicines. Extensive use of them is both expensive and, according to our observations, can cause silicon accumulation in the lime cycle. Primarily, measures should be taken to establish the soap balance and to define and eliminate the weak spots in the balance. Fine-tuning may then be done by using defoamers.

WHY SEPARATE TURPENTINE?

The motivation for good separation of turpentine is two-fold: turpentine is classified as a flammable liquid and may cause hazardous situations in the process if not effectively removed and, secondly, it is a saleable product. The combustion properties of turpentine/air mixtures are as follows [5]:

- Explosive limits: 0.8 vol% (lower), and 6.0 vol% (upper);
- Flame speed:150 m/s

Turpentine is vaporized from wood during steaming and digester degassing in the early stages of a kraft cook. In processes that do not apply steaming, such as displacement batch cooking, turpentine is contained in the off-gases from warm and hot liquor accumulators. These gases also contain other volatile compounds, non-condensable gases (NCG), methanol and water vapour.

Turpentine is separated from the gas mixture by condensation in a surface cooler, but much of the turpentine can be found in the NCG collection system. Although the flame speed is low, several accidents have occurred in these systems. In addition to that, we have observed that turpentine also readily attaches to soap micelles in black liquor from which it can volatilize in unexpected places. Soap solubility may also be affected in unexpected ways by turpentine attached to soap micelles, [6]. By establishing the turpentine balance for a mill, one can design improved turpentine-collection systems.

SOAP SEPARATION PARAMETERS

Effective separation of soap is performed at three positions in the process. Foam (containing soap) is separated from the filtrate tank in washing stage No. 2, while solid soap is separated from weak liquor tank feeding into evaporation and also from the intermediate liquor tank in the evaporators.

The reason for separating foam is primarily to enhance pulp washer performance. Foam separation results in the recovery of only a small fraction of the total soap mass. In order to establish effective soap skimming arrangements from black liquor, some preconditions must be fulfilled. The critical parameters are shown in Table II.

Uloth et al [7-9] and Gullichsen and Lindeberg [10] have thoroughly reported the optimal conditions for soap separation. The most important factors affecting soap solubility are the ratio of fatty acids to resin acids and residual effective alkali (EA) [9]. The lower the fatty/resin acid ratio is, the greater the solubility. Optimal concentration of residual effective alkali in the digester extraction liquor (17-20% solids concentration) is in the range of 10-13 g NaOH/L. At a given effective alkali content, a high sodium content is also beneficial for good soap separation. A good dry solids range to minimize solubility is 26-30%. Below that range, the soap is rather soluble; above it, increasing liquor viscosity hinders the soap separation. Above 35% solids, the retention time required for separation increases very rapidly [9]. The optimum temperature is quite low, preferably 80-85°C.

Optimization of soap separation is a unique problem in each mill. It depends on the wood raw material, which sets both the over-all extractives content and the ratio of fatty acids to resin acids. It also depends on cooking conditions, on weak black liquor solids content, as well as on evaporation conditions. For many mills, but far from all, the best solution is to increase the dry solids content of weak liquor to 23% by mixing in strong black liquor and then to skim off, at the weak liquor tank, 70-80% of the total recoverable soap. A second skimming takes place in the intermediate liquor tank in the evaporators before feeding the liquor to the next effect. For other mills, depending on process and equipment design, most of the soap is recovered from the intermediate liquor. To prevent foaming in the initial evaporation stages, it is always better to skim as much as possible from the weak liquor. The best separation conditions can be established by performing a soap audit at the mill.

EVALUATING & SOLVING EXTRACTIVES-RELATED PROBLEMS

Motivation for a soap audit: A well-planned and conducted soap/extractives audit is the necessary prerequisite for evaluating and solving extractives-related problems. As already mentioned, each mill is an individual case, both from the raw material and process point of view. For this reason, there is basically no shortcut in soap/extractives matters. Depending on whether the focus is on soap, pitch or turpentine, the nature and extent of the mill audit will vary. This definition of the specific problem further sharpens the target area from the process point of view. The following discussion will illustrate the general features of a mill audit.

Preparations and sampling arrangements: Before starting the mill work and sampling,

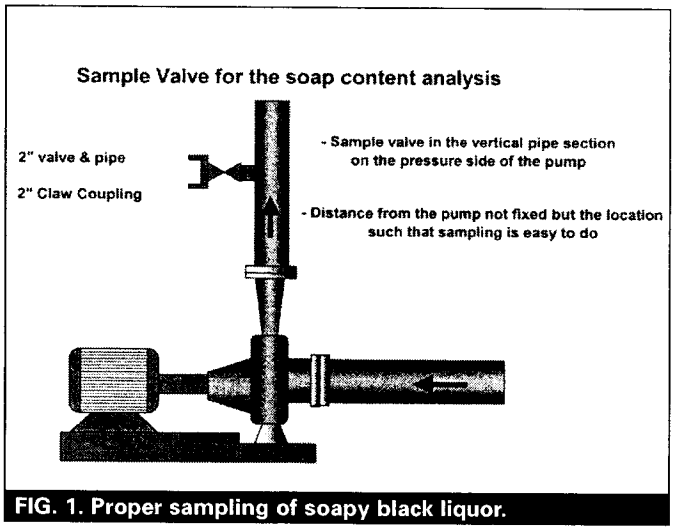

FIG. 1. Proper sampling of soapy black liquor.

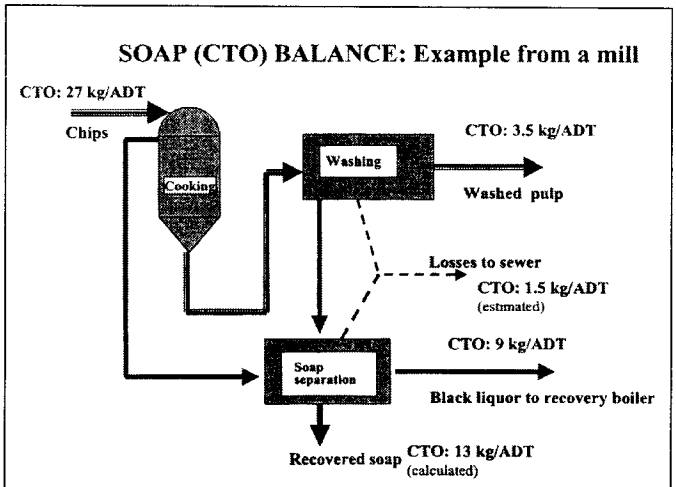

FIG. 2. Example of a soap balance.

a thorough investigation of the flow sheet and layouts of the mill departments (e.g. cooking, washing and evaporation) needs to be done. This may sound self-evident, but with experience, soap "hot spots" can be identified at this point. The sampling program (location of the sampling points and frequency of sampling) can then be tentatively planned. Very often, the available sampling arrangements do not match the requirements for obtaining representative samples. This holds especially true for black liquor samples that contain insoluble soap or turpentine.

When the locations of the sampling points have been defined, each position has to be discussed by the testers and mill staff. If the existing valves are not appropriate or if there are no adequate sampling points available at the desired position, the mill may be able to prepare them prior to the actual mill audit.

Figure 1 shows how a sample valve has to be positioned for obtaining a representative soap-black liquor sample. The valve has to be on the pressure side of the pump (to ensure thorough mixing), and not too far from the pump (preferably only a couple of meters). Placing the valve in a vertical pipe section is the next requirement in order to prevent separation of insoluble soap from the liquor. If turpentine-containing process streams have to be sampled, a separate sample cooler has to be used in order to prevent gaseous turpentine from escaping during the sampling. A cooler is always needed when sampling any stream with a temperature above 100°C.

It is worth mentioning that the worst possible sampling arrangement — often the easiest available existing one — is on the suction side of a pump and off the bottom of the horizontal pipe section. Due to the risk of soap separation, no representative sampling can be guaranteed with such a valve arrangement.

Estimation of soap and extractives potential: It is good to start a soap/extractive audit by estimating the total extractives input from the wood raw material. Such a study might have its own use, for example, knowing the total extractives input allows a mill to discover what fraction it is removing as skimmed soap (or tall oil) — a useful figure for comparison between mills. A mill with no soap skimming equipment will understand what the soap recovery potential is — this would help with the design of a skimmer, if that is the plan.

A representative composite sample of the chips is first taken. The total content of extractives is then determined from the chips. Considering the soap content, it is recommended that the chips be cooked with an excess white liquor charge in order to release all available soap into the cooking liquor. After cooking, the pulp is thoroughly washed with hot water in order to release the more sticky extractives. The soap content of the combined mother liquor and wash filtrate is then measured. The pitch-like part of the extractives, which cannot be washed away, is determined by extracting the washed pulp with acetone.

In order to measure the turpentine potential of the chips, it is necessary to arrange for chips that are as fresh as possible so as not to lose turpentine during chip storage. The analysis itself comprises steam distillation in alkaline conditions and measurement of the turpentine content of the condensate obtained.

Establishing the soap/extractives balances: Whether an over-all soap balance, or a more detailed study of a process department is planned, the key process streams still have to be selected and sampled. The samples to be analysed may be single samples or composites of a number of individual samples. Often, we prefer to analyse single samples instead of making composites. The reason is that a well-mixed composite of soap-containing liquor or pulp samples is difficult when insoluble soap is present. Furthermore, there may be time-dependent trends in the soap behaviour, and catching them implies analysing individual samples collected during a longer period of time.

In preparing an over-all soap balance — for example, to estimate the soap production potential — the necessary sampling points include the intermediate black liquor in the evaporation plant and the washed pulp from the last brownstock washer.

The intermediate liquor at dissolved solids content of 25-35% usually corresponds to the minimum soap solubility and the optimum point for soap separation. The washed pulp represents, on the other hand, the outlet and loss of extractives to bleaching. This soap output also significantly contributes to pitch problems in bleaching and in the final pulp, and therefore may require even more thorough extractive component analysis than the total soap content. An over-all soap balance obtained by the method described above is illustrated in Fig. 2 and represents a real mill case. With mills like this one, having soap skimmers, the "balance" obtained by measuring the input in the wood raw material and the two specified streams (skimmed intermediate liquor and washed pulp) will show a deficit between the input and outputs. This deficit represents the sum of recovered and lost soap (e.g. overflows and spills). Considering the fact that the direct measurement of recovered soap may be quite difficult and cannot be performed very accurately, the above approach most often produces a more accurate figure than any attempt at measuring directly the yield of recovered soap.

Measurement of soap solubility and separation conditions: Once the extractive "hot spots" have been identified, more thorough sampling and specially designed tests are employed. Soap separation characteristics in weak liquor or intermediate liquor are usually fruitful areas for investigation at this point, as is the beginning of the brownstock washing plant, is not

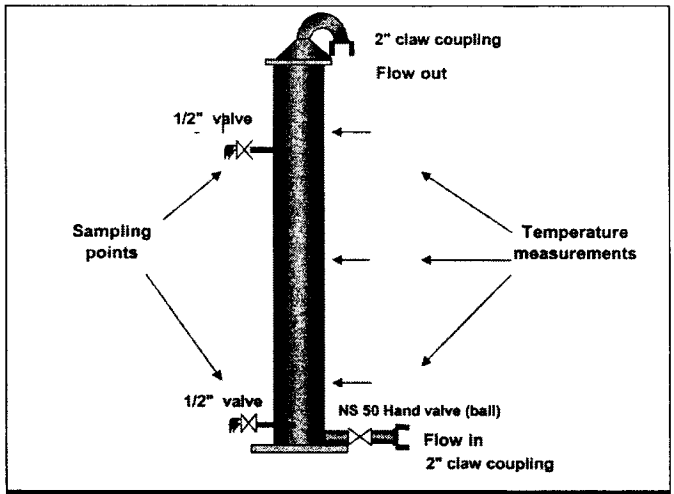

FIG. 3. Device for determining the soap rising velocity.

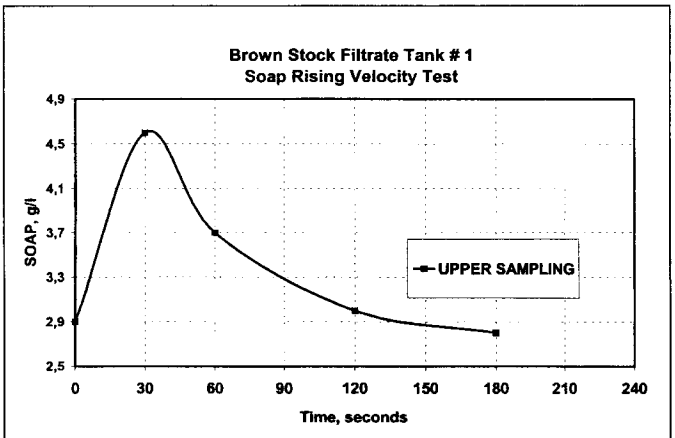

FIG. 4. Example of the soap concentration measurement from a soap rising test.

uncommon for insoluble soap to be present in the feed pulp suspension and the filtrate from the first stage of brownstock washing. Arranging proper conditions to skim soap and foam in such a position may be a key improvement for the operation of the entire washing line.

Determining the amount of insoluble soap in relation to the total soap concentration is important when evaluating the potential for skimming soap at a certain position. Moreover, the need for pumping and other handling systems for the skimmed soap will be defined by the actual quantity of the insoluble soap. Measuring insoluble soap in black liquor is not difficult:

A liquor sample is retained in a thermos bottle for 10 to 15 minutes at the process temperature, allowing the insoluble soap to rise to the surface. The bottom of the thermos bottle now contains saturated black liquor without insoluble soap. Using a special pipette, a sample of liquor is carefully withdrawn from the bottom of the bottle and analysed for soap content, yielding the soluble soap content at the process temperature. Comparing this with the analysis of the total soap content from the same sample position, the percentage insoluble soap can be determined.

Once the potential for soap skimming has thus been determined at a given process position, the next step is to measure the "soap rising velocity" corresponding to that position. Note that the soap rising velocity is dependent on the size distribution of the insoluble soap "particles," the density and viscosity of black liquor, as well as the air content and the size of the air bubbles in the liquor (according to Stokes's law, e.g. [9]). For this reason, each sampling position may have very different soap rising velocities.

We have developed a device to determine the soap rising velocity in actual process conditions. This device, nicknamed "The Rocket," is illustrated in Fig. 3. The idea is to take a soapy black liquor stream from the pipeline (preferably after the pump) and feed it to the bottom of The Rocket.

The liquor is allowed to flow upwards through the device until the temperature has stabilized to match the process temperature and then the flow is stopped. Soap immediately starts to rise upwards in the stagnant black liquor inside The Rocket. Black liquor samples are then collected at a scheduled rate from the sampling port in the upper part of the Rocket, and the total soap concentration is analysed in these samples. Finally, after all the insoluble soap has certainly risen to the top, a sample is taken from the bottom — this represents the saturated black liquor.

Figure 4 shows the data from such a measurement made at the first filtrate tank of a brownstock washing line. In this case, the soap separated much faster than, for example, in an intermediate liquor tank, due to low black liquor viscosity and the presence of a significant amount of air. The plot of the total concentration of soap in the samples as a function time can be used as the basis of mathematical operations that finally produce the rising velocity of soap. These involve combining Stokes's law with a selected soap particle size distribution. A differential equation for the soap concentration is produced and is solved at the sampling level by using the measured soap concentration profile [11]. The resulting soap rising velocity is defined as the time for 95% of the soap to separate and the velocity figure so obtained is applicable to the design of liquor tanks suitable for effective soap skimming. In the case illustrated by Fig. 4, the calculated design velocity for soap rising was 33 m/h. In case of intermediate liquor tanks, the rising velocity is typically 3-5 m/h.

Analysis of soap content in black liquor: Before discussing the actual analytical methods, it is worth re-emphasizing that a representative sample is absolutely the most important precondition for accurate results. The sampling arrangements have been already mentioned. The second key matter lies in the analysis of the sample: the black liquor sample must be used in its entirety, because insoluble soap easily collects at the vessel and liquor surfaces. For this same reason, when filling a sample bottle, the contents must not be allowed to overflow.

Traditionally, the total concentration of soap in black liquor has been determined by using the Saltsman-Kuiken method [12].— The related TAPPI and SCAN standards are both based on this method. In the original Saltsman-Kuiken method, the determination of soap (as crude tall oil, CTO) is based on the titration of fatty and resin acids, but the neutral component was not considered at all. This drawback has been overcome by using the gravimetric determination of the tall oil extracted from the sample [13]. It can also be overcome by using the acid number of the tall oil produced from the skimmed soap as recommended in the new Pulp Chemicals Association test procedure [14].

The Saltsman-Kuiken method and its modifications are accurate but very time-consuming to do. For this reason, improved methods have been suggested. Probably the best and most useful of these alternative methods is based on gas chromatographic analysis [15, 16]. The entire analysis is much faster than the previously mentioned methods and also more accurate [16]. In addition, another clear benefit is that besides the total soap concentration, the distribution of the main soap components (fatty and resin acids and the neutral component) can also be obtained. This aspect is very useful in following the behaviour of soap and extractives through the fibreline. At the beginning, in the brownstock washing area, the main extractives-related problems are associated with soap, but later on, when the sodium salts of the fatty and resin acids are largely washed away, the pitch-type prob-

lems can emerge. When detecting the transition from a "soap-problem" to a "pitch-problem," the relative distribution of the three main soap components is indeed useful.

When analysing pitch or turpentine, gas chromatographic analysis can be recommended for both quantitative and qualitative analysis.

CONCEPTUAL ENGINEERING PHASE

Any mill audit or study, regardless of the topic, should lead to practical recommendations for solving the problem in question. This may be called the conceptual engineering phase, which results in suggestions for process modifications, possible new equipment and/or modified operational principles. Properly performed, conceptual engineering will also give the basic design data, e.g. main dimensions of tanks and process equipment, to be used in the detailed engineering phase.

In the case of the conceptual engineering for extractives-related process modifications, the soap/extractives audit is able to pinpoint the key areas for modification and also produce the basic design data needed.

Figure 5 shows how an optimal soap collection system for a pulp mill should be configured. Using the results of the soap audit, a decision is made on whether or not existing filtrate and weak/intermediate liquor tanks are sufficient for effective soap skimming. If new tanks are needed, the previously described soap solubility and soap rising tests are able to give the required design parameters. It is not uncommon to find that existing skimming arrangements do not perform properly. Due to layout reasons, the tanks may have a totally wrong diameter-to-height ratio. In many cases, the current production level is far above the original design capacity of the mill and the residence time in the tanks is simply too short. Additional arrangements for facilitating effective soap separation (such as baffles, soap troughs and floating skimmers) may be needed as well. An excellent overview of these matters has been given by Foran [17].

When considering a continuous cooking fibreline, the performance of the digester hi-heat washing normally allows efficient soap washing, and practically all the soap is transported with the extracted weak liquor directly to the evaporation plant. In such a case, the soap and foam problems in the brownstock washing area are not a major concern. However, whenever the hi-heat washing becomes overloaded, for example, due to increased pulp production, the situation can dramatically change. In conventional batch cooking, and sometimes also in modern displacement batch cooking systems, a

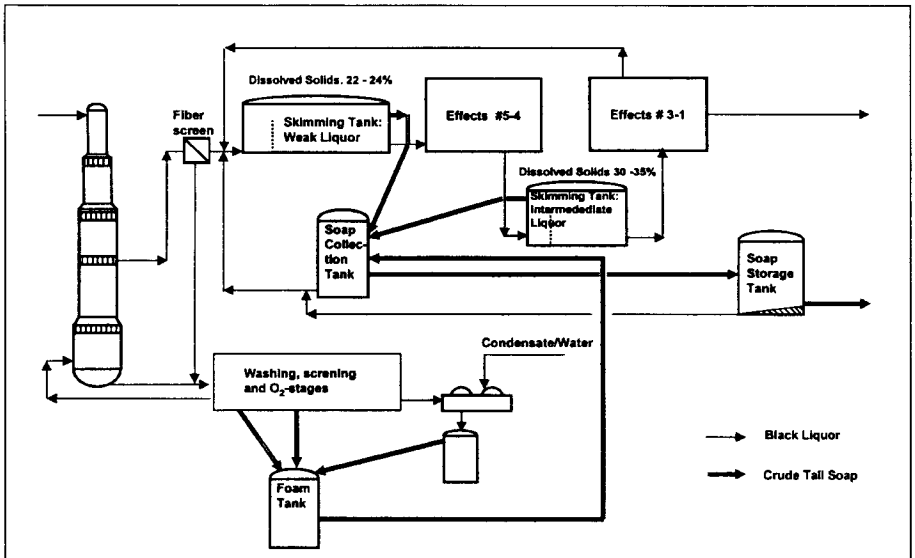

FIG. 5. An example of an optimal soap collection scheme at a pulp mill.

significant soap input to the brownstock washing area makes it necessary to carefully design the soap and foam separation systems in connection with the filtrate tanks.

The design of the filtrate tanks is crucial in this respect and the pumping of foamy soap also requires special consideration. Modern gas-removing centrifugal pumps can be recommended for soap pumping in the brownstock area because the separated soap always contains foam and black liquor. However, pumping dense soap that is skimmed from the evaporation line tanks makes it necessary to use gear or clove rotor-type positive displacement pumps.

Concerning the conceptual design for turpentine collection, even more mill specific features can be found here than in the case of soap separation. The means by which the available turpentine is collected in continuous and batch cooking processes are profoundly different.

Inefficient turpentine recovery in the digester area may lead to unexpected problems in the brownstock washing area (for example, odd behaviour of soap, inefficient washing) [18]. A smell of turpentine at the brownstock washing plant is always a hint that the turpentine recovery system may need a closer look!

CONCLUSIONS

The softwood kraft process is very vulnerable to soap and extractives disturbances. Pulp quality, the energy balance and smoothness of the process operations are all upset if soap and foam separation are not correctly managed. For similar reasons, turpentine must also be effectively separated. Most of the problems are highly mill-specific.

It is the experience of the authors that in many mills, the necessary attention has not been given to the design of soap and turpentine separation systems, partly due to the fact that general solutions are not always applicable to a specific mill. This is why soap and turpentine audits are of great help for the mills. A successful audit combined with practical recommendations on how to improve the situation solves many problems both by recommending improvements in the process and by transferring understanding of soap behaviour to the mill personnel.

To correctly perform an audit requires experienced people to carry out the work at the mill. The sampling points must be chosen correctly, as are the sampling intervals. There is only one correct way to take samples of black liquor containing soap — often partially in solid form — from process pipes. Determining soap rise velocity under process conditions also requires experience. The analytical procedures must be reliable.

It is best if two rounds of measurements are taken. The first set pinpoints the "hot spots" and identifies the most necessary steps to be taken. The second round seeks the optimum level of soap or turpentine separation considering separation performance in relation to investment costs. The consequent recommendations give a picture of what can be achieved at the specific mill.

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Résumé: La séparation effective du savon et de la térébenthine est un facteur important, mais particulier à chaque usine, de la performance dans l'ensemble du procédé de pâte kraft de résineux. La présente communication décrit une façon systématique d'évaluer et de résoudre divers problèmes de procédé reliés aux matières extractives. Lorsqu'une "vérification" est effectuée dans l'usine, on évalue d'abord le potentiel en savon et en matières extractives des matières brutes du bois de l'usine, on procède à des essais des principaux circuits de fabrication, et les bilans savon et matières extractives obtenus sont utilisés pour évaluer le chemin suivi par les matières extractives dans l'usine. On procède à un échantillonnage plus approfondi et à des essais spécialement conçus pour les "secteurs chauds" identifiés lors de la vérification du savon.

Abstract: Effective separation of soap and turpentine is an important, but mill-specific aspect, regarding the entire softwood kraft process performance. This paper describes a systematic approach to evaluate and solve various extractives-related process problems. In a mill "audit", the soap and extractives potential of the mill's wood raw material is first estimated, the key process streams are tested and the resulting soap and extractives balances are used to clarify the routes taken by the extractives in the mill. More thorough sampling and specially designed tests are employed at the "hot spots" identified in the soap audit.

Reference: TIKKA, P., KOVASIN, K., LAXÉN, T. Solving soap and turpentine related process problems in softwood kraft mills. *Pulp Paper Can.* 103(6): T149-154 (June 2002). Paper presented at the PacWest 2001 Conference in Whistler, BC, on May 16 to 19, 2001. Not to be reproduced without permission of PAPTAC. Manuscript received July 13, 2001. Revised manuscript approved for publication by the Review Panel November 16, 2001.

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