1. Mill tests

11.1 Objectives of the work

The test runs were conducted for verifying the good results achieved by black and green liquor impregation in a laboratory scale and batch digester plants also in a mill scale using a continuous digester. By adding black and green liquor, the sulifidity and organic matter content of impregnation liquor were to be increased and their influence on the fiber product and chemical recovery was to be determined. The test runs were expected to increase the tear resistance and yield of the pulp. This would allow continuing the cooking till low kappa numbers without losing yield.

However, due to pulp quality it was decided that the tests will be carried out at a constant kappa level, and expectations were mainly directed at improving the technical properties of the papermaking pulp and possibly increasing the yield. All test runs were to be conducted at the normal production rate of the mill. When studying the influence of cooking variables, simultaneously non-disturbed production of uniform quality was to be ensured at the mill. The objective of the work was also to determine the influence of black and green liquor addition to the runnability and operational economy of the whole mill.

11.2 Test plan

The plan was made using a Taguchi 9-test matrix in soft wood and birch trial runs /46/. Aspen trial runs were conducted at two test points only. The Taguchi matrix is shown in Annex 2. The sequence of and changes in the tests are shown in Table 3.Table 3 also shows the amount of white liquor reduction, if the effective alkali of the semi-thickened black liquor is 30 g NaOH/l and that of green liquor is 43 g NaOH/l.

Taulukko 3.

Test No. / Kokeen numero	Semi-th. BL / Välimustalipeää l/t	Green Liq. / Viherlipeää l/t	White Liq. reduction / Valkolipeän väh. l/t
1	5	6	2,4
2	10	12	4,8
3	5	0	0,8
4	10	0	1,6
5	0	12	3,2
6	10	6	3,2
7	0	6	1,6
8	5	12	4,0
9	0	0	0,0

At the beginning, also cooking temperature was a variable, but it was left out upon the decision to run to a constant kappa level. The cooking temperature and the alkali dose change depending on the cooking hardness and residual alkali from extraction The tests were to be carried out only if oxygen bleaching and washing press were operating. The softwood and birch test cooks were to be carried out according to the test matrix, but those for aspen would be saved occasionally.

11.3 Chips and liquor used

11.3.1 Chips

The chips used in the tests for birch and aspen were pulpwood that was barked and chipped at the mill. Of the softwood chips about 30…40 % were purchased and further the spruce-pine –ratio varied freely. The average storing time of the chips was approximately one week. The chips were analyzed for each test cycle by conducting a sieve analysis and determining the solids content of all the screened chips going into the chip bin of the digester plant. The chips were usual winter chips. The piece size distribution was narrower for hard wood than for soft wood. The soft wood chips contained abundantly both over-sized and under-sized chips. The saw dust from screening was fractionated and the coarsest fraction was mixed into the chips going to digestion.

The dry solids content of the softwood chips was approximately 45% and that of birch 55%. The dry solids content of aspen chips was approximately 50%. The dry solids content of chips varies depending on the season and the size of the chip pile. The dry solids content of the chips has an influence on the alkali dose and the liquid-to-wood ratio in the digester. The chips analyses are shown in detail in Annex 3. The fluctuation range of the chips analyses is shown in Table 4.

Taulukko 4
Keittokokeissa käytetyn hakkeen laatu. Quality of chips

	Over-sized Ylisuurta >//8 %	Accepted Hyväksyttyä >D7 %	Under-sized Alimittaista <D7 %	Dry solids Kuiva-aine %
Havuhake Soft wood	7,2 - 21,4	70,5 - 78,9	5,5 - 14,3	43,2 - 48,7
Koivuhake Birch	3,1 - 7,8	78,4 - 89,4	5,3 17,0	45,5 55,4
Haapahake Aspen	5,9 - 8,5	85,4 - 86,2	6,6 - 7,9	49,7 - 51,1

11.3. 2 Liquors used

The white liquor was normal white liquor used at the mill, with an effective alkali of about 118 g/l NaOH and sulfidity varying in the range of 36...42%. The semi-thickened black liquor was obtained from the evaporation plant after the semi-thickened black liquor tank and its dry solids content was about 50%, effective alkali 30 g/l NaOH and sulfidity about 90...100%. Additionally, the semi-thickened black liquor had an organic matter content of about 300 g/l. The green liquor was obtained downstream of the green liquor clarifier, whereby its dry solids content was about 100 mg/l. The green liquor had an effective alkali of 45 g/l NaOH and sulfidity 72...77%. The analyses of the liquors used are shown in table 5.

Taulukko 5

Käytettyjen lipeiden analyysit

	EA Tehollinen alkali NaOH g/l	Sulfidity Sulfiditeetti %	Sodium carb. Natriumkarbon. g/l	Organic matter Org.aine g/l
WL Valkolipeä	113,9 - 125,0	37,2 - 40,98	28	0
GL Viherlipeä	39,4 - 46,5	72,6 - 76,2	143,2 - 152	0
S-T BL Välimustalipeä	26,4 - 32,6	90 - 100	0	300
Trans. circ. Siirlok.lipeä Liquor	38,9 - 42,6	52,5 - 57,1	10 - 20	48,0 - 65,8
Paisuntamustal. Extraction BL	10,6-11,6	90 - 100	0	90 - 110

From the transfer circulation, liquor is fed to the upper part of the impregnation tower and this is called compression circulation. The purpose thereof is to compress the chip column downwards. The properties of the compression circulation liquors depend on the distribution of the liquors and the properties of liquors fed into the impregnation tower. Weak black liquor is liquor obtained from digester extraction the properties of which are dependent on the liquor doses into the impregnation tower and into the transfer circulation, as well as reactions in the digester. Additionally, the conditions of oxygen bleaching have an influence on the effective alkali of extraction liquor and the composition of dissolved solid matter. The properties of extraction liquor are equalized only after the pulps have passed oxygen bleaching.

11.4 Analyses conducted

The test runs were in principle carried out based on laboratory analyses relating to normal operation control. Additional analyses were some analyses on liquor and chips and determination of flow viscosities. All refinings carried out on the test pulps were additional analyses. The pulps were tested in the Central Laboratory by means of PFI-refinings at two test points. The refinings were interpolated to a constant tensile strength of 65 Nm/g instead of 70 Nm/g, because not for all pulps did the strength develop in the manner that was desired for comparison.

11.5 Data collection and result processing

The measuring and laboratory data on the process were gathered by means of a process computer as hourly average values. The results were processed with an excel spreadsheet computation program, which was also used for processing the data. Confidence determination was conducted by means of a Taguchi test matrix (confidence interval 95%).

11.6 Performing of the tests

11.6.1 Description of the digester plant

The mill tests were performed at Metsä-Sellu Oy Äänekoski Mill and the main purpose was to carry out the tests without disturbing the pulping.

In the test runs, black and green liquor were dosed through a Kamyr-digester's high-pressure feed into the impregnation tower. The Kamyr-digester of Metsä-Sellu has originally been designed for "continued cooking". The digester is provided with a possibility to dose white liquor into several feed points:
feed
impregnation
transfer
countercurrent and
washing circulation.

The planned production of the digester has been 1100 ADt/d, but it has increased remarkably, the present production being about 1400 ADt/d soft wood pulp and 1700 ADt/d birch pulp. Originally the digester has been run in "continued cooking" mode by means of a countercurrent zone and distribution of white liquors for two years, after which the use of countercurrent circulation was abandoned. Countercurrent cooking succeeded with very good quality chips only and a production rate of 1100 t/d.

The digester was thereafter modified to continued co-current mode, which enables also distribution of alkali to several points and cooking liquid can be heated indirectly in transfer circulation, leveling circulation, in continued co-current circulation and washing circulation.

About 60...70% of the white liquor is dosed into the impregnation tower and the rest to the transfer circulation. White liquor addition to continued co-current circulation and to washing circulation has also been tried, but the results were not encouraging, and those additions have been given up.

11.6.2 Black and green liquor feed into impregnation

The variables were black and green liquor doses, due to which the composition of the impregnation liquid changed as to sulfidity, organic matter content and sodium carbonate. The attempt was to keep the alkali dose of the cooking constant by taking into account the amount of effective alkali of black and green liquor when dosing the alkali. The dosing was carried out in accordance with Figure 25.

The temperature at the top of the digester and in the co-current circulation was changed based on cooking kappa. The sulfidity of white liquor was determined to a certain level by the combined effect of the temperature of pressurized heating, the alkali dose of oxygen bleaching and the feed of sodium sulfate. During the test runs the sulfidity of the white liquor varied between 36–42%. The white liquor sulfidity level before the implementation of oxygen bleaching was about 42...45%, to which level it increased as oxidized white liquor replaced sodium hydroxide in the feed of oxygen bleaching in autumn 1995.

The cooking variables are limiting in relation to each other when the goal is uniform production. Increasing the temperature increases alkali consumption. Often it is necessary to change the cooking temperature and alkali charge simultaneously. The desired residual alkali in extraction liquor was 8–10 g NaOH (EA) / l.

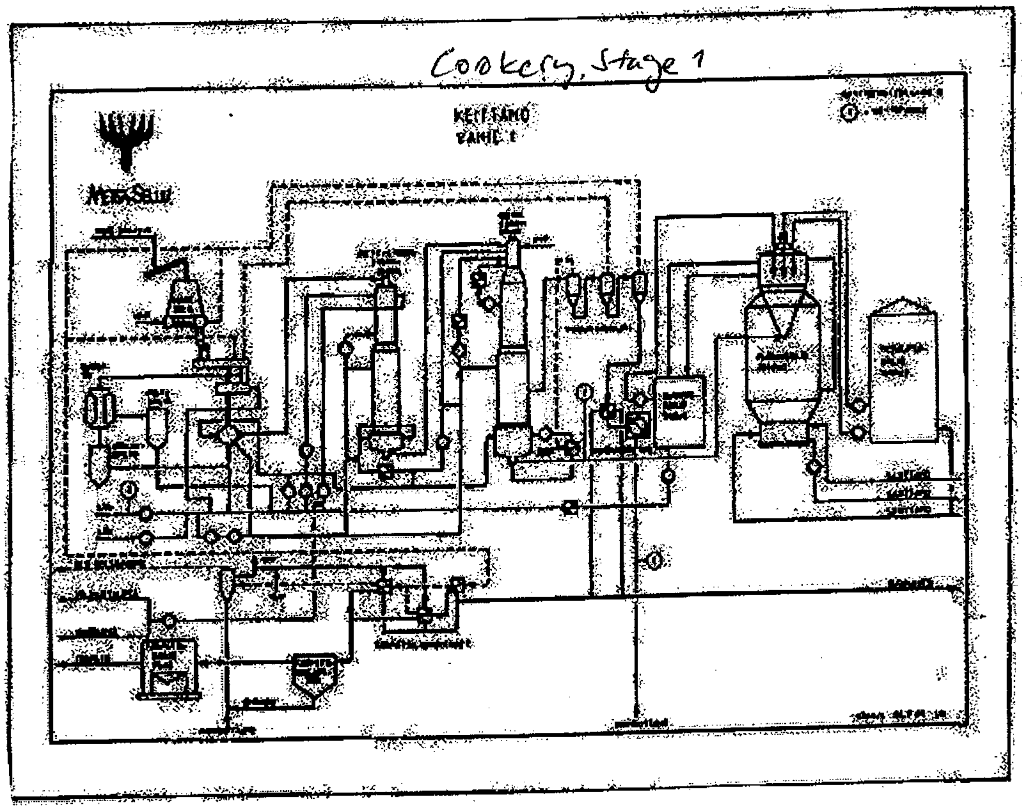
Further, the attempt was to keep the conductivity of the transfer circulation in the level of about 30 g NaOH (EA) /l. The liquid-to-wood ratio of the digester was made constant by changing the amount of weak black liquor introduced into the impregnation tower in accordance with test doses. The amount of weak black liquor was 5...18 l/s. The concentration of the impregnation solution is also dependent on the amount of flashing steam, the dry solids content of the wood and liquid originating from melted snow.

Liquor from the transfer circulation was introduced into the impregnation tower for improving its operation. This so-called compression circulation increased significantly both the liquid-to-wood ratio of the impregnation circulation and the alkali dose introduced thereto. The alkali concentration in the liquor in the impregnation circulation was in the same range as that of the rest of the impregnation liquid. The amount in the compression circulation was about 30 l/s. Due to the compression circulation, the liquid-to-wood ratio in the impregnation tower was

3.9...4.8 for pine and 3.1...3.9 for birch. The attempt was to keep, when possible, the alkali dose constant and in the limits of target values 19.5...21.0% NaOH effective alkali on wood. The need for alkali in the cooking varied depending on the quality changes of the chips. The alkali dose of the feed circulation was changed during the series of tests from 14% to 13%. An even lower dose was experimented in the feed circulation, but it was given up due to discharge disturbances in the impregnation tower. The extraction screens would have started to limit the production, if the amount of black liquor into the feed circulation would have increased.

The long retention time (4.4–5.5 h) of the digester complicates both making changes and maintaining a constant run. Changes are made mainly based on blow kappa analyses and alkali concentrations of the circulations. The effect of the changes on the cook result is not seen until after several hours.

Figure 25. The dosing of semi-thickened black liquor and green liquor into impregnation in a Kamyr-digester



When studying the effects of the cooking variables, the attempt was to simultaneously ensure maximum undisturbed production with uniform quality at the mill. This set limitations to the cooking variables and the duration of the test cycles: too rough changes were to be avoided. The test runs were disturbed by frequent wood grade changes, which took place after two days of birch cycle and three days of soft wood cycle.

A precondition for the test runs was regarded to be a relatively even run at a constant speed and the presence of oxygen bleaching. Data collection on the test runs was performed after about 8 hours' leveling time after change of wood grade. The cooking temperature was changed for maintaining a constant cooking hardness and by means of alkali dosing the residual alkali of extraction was regulated to conform to the target value.

The amount of effective alkali in black and green liquor was underestimated, by using lower concentrations than in reality (for green liquor 30 g NaOH EA/l and for semi-thickened black liquor 15 g NaOH EA/l) and as a result of this, the alkali contents of the circulations increased in the beginning of every test. The liquid-to-wood ratio was kept constant by changing the amount of weak black liquor going to impregnation.

The most important cooking parameters are shown in Table 6.

1) Chip feed t/h
2) White liquor to impregnation l/s
3) White liquor to transfer circulation l/s
4) Concentration of white liquor g/l
5) Sulfidity of white liquor %
6) Compression circulation l/s
7) Conductivity of transfer circulation mS/cm
8) Liquid-to-wood ratio
9) Temperature at the top of the digester °C
10) Cooking kappa

Taulukko 6

Tärkeimmät keittoparametrit

	Mänty	Koivu	Haapa
1) Hakesyöttö t/h	211 - 234,7	223 - 251	202 - 214
2) Valkolip. imeytykseen l/s	26,3 - 38,1	32,7 - 38,7	27,4 - 29,9
3) Valkolip. siirtokiertoon l/s	13,9 - 18,6	15,4 - 20,3	13,0 - 14,1
4) Valkolip. väk. g/l	108,0 - 125,0	117,2 - 122,5	113,8 - 120,6
5) Valkolip. sulfiditeetti %	37,2 - 41,0	36,7 - 41,4	37,8 - 40,0
6) Painatuskierto l/s	27,5 - 36,8	19,7 - 30,7	28,7 - 33,2
7) Siirtokierron johtok. mS/cm	24,3 - 33,2	27,9 - 32,2	25,0 - 28,9
8) Neste-puusuhde	3,8 - 4,2	2,9 - 4,0	3,0 - 3,3
9) Keittimen huipun lämpötila °C	163,0 - 167,0	151,2 - 153,7	145,1 - 149,5
10) Keittokappa	23,9 - 26,1	14,9 - 17,3	18,9 - 21,1

The tests were started on Dec. 5 and they were continued at a relatively fast tempo until Christmas, after which the collection of reference material took place until Jan. 3, when the tests were restarted. The tests were interrupted on Jan. 11, because the increase of inorganic dry solid matter caused by green liquor resulted in a decrease in the caloric value of strong black liquor and steam was needed in the mill integrate. This was due to the elevation of the boiling point and increased liquid flow. During the following week it was decided that the tests will be continued in short 16-hour cycles, whereby reference tests would be possible immediately. The tests were then continued on Jan. 20 and 25. By the end of March the tests had been completed.

At the digester plant the tests were carried out smoothly without major disturbances. The liquid-to-wood ratio was made constant by decreasing the amount of black liquor to dilution when possible. The chips and liquor levels remained within the desired values and the extraction screens stayed free of clogging.

The target kappa for pine was 26–27 and for birch 16–17. Normal disturbance-free cooking cycles between the test cycles were selected as reference points.

11.6.3 Pulp washing efficiency

Increased inorganic and organic matter resulted in decrease of washing quality during the test cycles and this was not reacted upon by increasing washing liquid. The washing coefficient of the digester changed depending on the conditions in the weak black liquor tank. The washing coefficient was too low due to high production speed and the pulp entered oxygen bleaching and bleaching in excessively dirty condition in view of chemical consumption. The production was run depending on the level in the weak black liquor tanks.

The washing efficiency was observed based on the washing loss of the wash press, the conductivity of the twin thickener and also the COD-amount in the bleaching effluent. Also, the chemical consumption of the D_0 –stage reacted directly upon the washing loss when running to constant intermediate kappa, if the kappa number after oxygen bleaching also remained constant.

11.6.4. Bleaching costs of the pulps

In connection with the test runs, also the effect of black and green liquor addition on the bleaching costs, i.e. the bleaching efficiency of pulp was determined. The bleaching cost differences between test and reference pulps were calculated based on delay, from hourly averages of chemical consumption collected by a process computer and laboratory analyses. The production rate was calculated from the amount of chips using a grade-specific yield and taking into account the level changes in pulp towers. As the washing loss changes during a running cycle, the calculation of bleaching chemical consumption does not give full certainty on the effect of the test runs. It should have been possible to wash the pulps to the target washing loss. The oxygen bleaching dosing changed also during the tests, but the attempt was to eliminate its effect by means of adjacent reference runs.

The bleaching has been carried out using a normal sequence $D_oE_{op}E_pD_2$. Hydrogen peroxide was used in the E_p –stage during some running cycles due to high washing loss. Bleaching the pulp to full brightness succeeded in every test run cycle.

11.6.5 Survey of the results in the recovery line

The addition of black and green liquor has on effect, either positive or negative, on the properties of black liquor, too, such as viscosity, boiling point and reduction heat, and thus the operation of the whole recovery line.

The use of green liquor in impregnation increases the water amount to be evaporated in proportion to the extra liquid amount added into the digester. The amount of liquor to the evaporation plant can be decreased by weakening the wash. Green liquor dosing causes boiling point elevation due to the effect of dissolved inorganic and organic matter and this way decreases the capacity of the evaporation plant. Green liquor decreases the calorimetric value of black liquor as the inorganic loading increases, and simultaneously the total loading of the recovery boiler increases.

The changes taking place in the recovery line were observed by means of reports from the department. Black liquor flow from the digester plant to the evaporation plant was included in the computer reports, as well as the extraction flow of the evaporation plant, and they disclose the changes in the loading of the evaporation plant during the test runs. In the recovery line, the changes in the test points were mixed up, because uniform production of the recovery boiler allows to take care of the energy supply of the whole mill integrate. Thus, changes in the burning and the energy production do not correspond to the situation at test run points. The birch and pine liquors were mixed in the liquor tanks, as well as liquors from different test cycles. The causticizing plant and the lime kiln operated largely depending of the green liquor state, so that the oil consumption of the lime kiln can not be addressed to any specific running mode. Savings to the causticizing plant are calculated directly for each test run in proportion to the amount of saved white liquor.