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A method to optimise birch CTMP pre-treatments by direct measurement of brightness on birch wood

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KEYWORDS: Birch, Pre-treatment, Pre-heating, Sodium sulphite, Sodium hydroxide, Brightness

SUMMARY: It is challenging and quite difficult to optimise the pre-treatment of birch CTMP with respect to brightness in large-scale trials. Because of the complexity of the system, it is necessary to go beyond a two-dimensional experimental approach (where not more than two variables are varied at the same time) to find optimal conditions. This paper presents a straightforward laboratory technique that may be used to study the effects on the brightness of wood by various pre-treatments. Combining this methodology with multivariate data analysis provides a powerful tool for optimising birch CTMP pre-treatment with respect to brightness. Recommendations within the experimental domain are given.

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We have previously demonstrated that a bulky pulp, with a low shive content, suitable for the middle ply of paperboard may be obtained by pre-heating chemically impregnated birch chips to a high temperature prior to refining using a low energy input (Vesterlind and Höglund 2005; Vesterlind et al. 2005). There were no signs of any negative effect on pulp brightness by a high pre-heating temperature. It is therefore reasonable to assume that pulp brightness is more dependent on a thorough impregnation and the quality of the raw material than of the pre-heating temperature or time. Therefore, in the study reported in this paper, the shavings technique described by Logenius et al. (2005) was used to find the optimal pre-treatment conditions regarding the brightness of birch chemi-thermomechanical pulp (CTMP). The wood shavings technique makes it possible to examine directly the brightness of the wood both prior to and after simulation of the conditions in a mill pre-treatment system.

Finding optimal conditions regarding brightness in the pre-treatment of CTMP has become increasingly important, even for pulp intended for the middle layer of multiply board. However, efforts to optimise the chemical impregnation of such pulp rarely concerns brightness, but instead focus on finding treatments that favour bulk and strength. Yet optimising the process with respect to brightness should not only help to reduce the cost of subsequent bleaching but should also help achieving the highest possible brightness of the final product.

It is generally acknowledged that high pre-heating temperature, prolonged pre-heating time and/or alkali impregnation are detrimental for the brightness of all types of mechanical pulps because of the formation of coloured groups in the pulps (Norrström 1969; Franzen et al. 1983; Jackson and Åkerlund 1983; Leask and Kocurek 1987; Granfeldt et al. 2001; Granfeldt and Suhonen 2003). Furthermore, the positive effect of sodium sulphite on brightness is well established (Gellerstedt 1983; Heitner and Tan 1987; Leask and Kocurek 1987) and it is often stated that the final brightness is the combined result of the darkening and the brightening reactions. A number of investigations have dealt with the effect of pre-treatments on the reflectance factor of wood (mainly spruce) by treating wood meal or chips prior to refining and then diluting the meal/pulp with dissolving pulp before measuring its optical properties (Engstrand and Hammar 1991; Johansson and Gellerstedt 2000). This method allows measurement of the reflectance factors of the wood/pulp at wavelengths shorter than the visible region of the spectrum. However, since the purpose of the current investigation was to optimise the reflectance factor at 457 nm, thus measuring the brightness that results from a process rather than determining what coloured substances are formed, the shavings method was judged to be a better approach.

The main advantage of this technique is that the evaluation is done directly on wood. Furthermore, there is no need for any extensive sample preparation, such as grinding of the wood to produce meal, refining it to produce pulp, or sample dilution. This eliminates any unwanted effects on brightness arising from these treatments, (e.g. from the high temperature that prevails during grinding and refining). Furthermore, as the experiments are simple to perform, an investigation can easily be expanded to cover a large experimental window.

Combining this technique with an experimental design and using multivariate data analysis, as is done in this paper, makes it fairly simple to identify sets of conditions (regions) that contain one or more areas with a good brightness response. Efforts were also made to estimate the yield as a result of various pre-treatments. Because environmental variations during storage and the like may affect the brightness of the green wood, efforts were also made to establish the sensitivity of the wood to various combinations of humidity and temperature.

Experimental

A sledge microtome (Leica SM2000R) was used to cut shavings approximately 100 μm in thickness from a frozen block of birch wood (*Betula verrucosa*). The block (6×6 cm) was taken from a log of birch that was stored in a freezer at –19°C to avoid discolouration.

In order to investigate the effects of temperature and humidity on green wood brightness at atmospheric conditions, the shavings were not allowed to dry prior to the treatment. The samples were immediately put into a climate test cabinet (VC 4081 from Vötsch Industrietechnik), where they were exposed to different temperatures and humidities for one hour (see *Table I*). After the treatment, the shavings were dried in airflow at room temperature and measured for brightness as described below.

Based on the results of the experiments with green wood, the shavings to be used in the pre-treatment experiments at pressurised conditions were dried rapidly in an airflow, weighed and measured for brightness at 457 nm. The light absorption (k) and light scattering (s) coefficients were calculated from two measured values of the reflectance factor at 457 nm for a single shaving over an opaque pile of shavings and over a black background, using a standard instrument for measuring brightness (Elrepho SE 071/070R from Lorentzen and Wettre, ISO 2469). Four shavings were used in each run, and the response values were taken as the mean values of the four samples in each test point. However, as four samples were not sufficient to produce an opaque pile, eight freshly cut, untreated and dried shavings were placed underneath the four samples as an additional background. Any effect of this background on the measured brightness does not affect the final result of this investigation because the error will be the same in all measurements and the study aimed at detecting discrepancies. The grammage of the shavings was within the range of 50–125 g/m^2.

Industrial pre-treatments were simulated by placing the shavings in Petri dishes with impregnation liquor. Two samples were placed in each dish. In the case of TMP simulation, only milli-Q water was used. The wood-to-liquor ratio was 1:4 by weight. The dishes were placed in a steam vessel (Logenius et al. 2005) where they were quickly heated in water-saturated steam to the chosen temperature, at which they were kept for a given time (the time to reach the right temperature was 15–20 seconds and was not included). After the heat-treatment, the samples were immersed in milli-Q water at a pH of ~7 in less than 30 seconds to cool them to room temperature in order to stop any chemical reactions.

The samples were boiled for three minutes before they were dried in an airflow, weighed and again measured for brightness (wood-to-water ratio was approximately 0.2%). The ratio of the weight before and after the washing was used as a rough estimation of the yield.

In the model, all responses except yield were given as delta values, $\Delta y = y_{after} - y_{before}$ (i.e. as the change in response due to the treatment). This approach was necessary since birch wood is not homogenous and the initial values of the responses (brightness, k and s) vary from shaving to shaving.

Milli-Q water was used throughout this investigation. The active chemicals in the pre-treatment were Na_2SO_3 p.a. and NaOH p.a., supplied by Sigma-Aldrich.

A full-factorial experimental design was used for the pre-treatments (see *Table II*). The levels of chemicals were chosen to be of industrial relevance, but without making the experimental window too small. When CTMP is produced from any type of hardwood, the alkali charge should be kept low to maintain the yield and the bulk of the pulp at sufficiently high levels. A NaOH addition of less than 3% was judged to be relevant for this investigation. A high sulphite charge reduces the shives content, but contributes to an increased environmental load if the charge is too high. Thus 5% Na_2SO_3 was the maximum sodium sulphite charge in this investigation. The total number of experimental runs amounted to 147, three of which were replicates at the lowest level. The design was formulated using Modde 5.0 software for multivariate data analysis supplied by Umetrics AB, Sweden. The model was fitted using partial least squares regression. Interaction terms and quadratic terms were included in the model in the event of non-linear behaviour. The resulting model was used to identify the factors significant for brightness, and also to predict the final brightness as the factors during pre-treatment were altered. Predictions can be made providing the quality of the fit and the predictive ability are considered good enough. The goodness of the fit (i.e. how much of the variation can be explained by the model) is expressed by the R2 value. The predictive ability (i.e. how well the model will predict the responses under new experimental conditions) is expressed by Q2 value. R2 > 0.9 and Q2 > 0.5 are generally judged to be sufficient (Eriksson et al. 2001).

The model was not further improved by removal of insignificant factors.

Table I. Levels chosen to study the impact of temperature and humidity at atmospheric conditions on green wood brightness of birch.

Temperature, °C	10	25	50
RH, %	30	60	90

Table II. Levels chosen to study the impact of pre-treatment under pressurised conditions on green wood brightness of birch.

Factors (Abbreviation)	Levels
Na_2SO_3, % on dry pulp (S)	0/ 3 /5
NaOH, % on dry pulp (OH)	0/1/2/3
Time, min	1/3/5
Temperature, ∞C (Temp)	125/140/155/165

Results and Discussion

Influence of temperature and humidity on the brightness of birch green wood

The brightness of green birch wood in this investigation was around 57% ISO (see Table III). The light absorption coefficient, k, of the birch wood was 2.7 m^2/kg, which is low compared to the 6.9 m^2/kg that has previously been reported for birch wood meal (Engstrand and Hammar 1991). The very large differences in light absorption may partly be explained by the major differences between the methodologies used in the two studies. Engstrand and Hammar used birch wood meal that was diluted with a bleached sulphite pulp. The grinding required to produce the meal causes local temperature increases that have a negative impact on wood brightness. The shavings technique used in the present study is gentler to the samples. The *k* value of 6.9 m^2/kg, found in the Engstrand and Hammar study would yield a brightness of around 41% ISO, which is at least 15 units lower than that found in the present study (with both studies applying the same light scattering coefficient of around 17 m^2/kg).

There is an increase in colour, the *k* value, during refining. However, if the *k* value of native wood found in this investigation could be preserved and the scattering coefficient increased from 17 to 35 m^2/kg in the pulping process, the brightness would be increased from 57% ISO to 67% ISO, giving a gain of as much as 10 units. This gain would have an enormous impact on the subsequent bleaching, resulting in a reduced cost and/or a higher final brightness.

Table III. Optical properties of untreated birch green wood.

Responses	Mean ± Standard dev.
Brightness, % ISO	57.3 ± 2.1
s_{457}, m^2/kg	16.7 ± 0.9
k_{457}, m^2/kg	2.7 ± 0.3

Initial experiments were carried out to determine the effects of environmental variations in temperature and humidity on brightness. *Fig 1* shows how the brightness of green wood from birch, aspen and spruce was affected by changes in relative humidity and temperatures. The maximum standard deviation in *Fig 1* was ± 2.1% ISO for birch at 50°C and RH 90%.

As can be seen in the figure, the brightness of birch green wood was much more sensitive to increases in temperature and humidity than was spruce (*Picea abies*) or aspen (*Populus tremula*). This finding has implications for the storage of birch green wood. For example, heat is released when chips are stored in piles (Hajny 1966; Springer and Hajny 1970). The observed sensitivity of birch green wood to heat means that the brightness loss during storage will be more pronounced in birch wood than in aspen or spruce.

The finding also has implications for the pre-steaming of the chips. The pre-steaming of birch chips in a CTMP process to ensure good uptake of the impregnation liquid proved to be far from optimal from a brightness perspective, which was an unexpected observation. But these negative effects can be reduced by performing the pre-steaming under slightly pressurised conditions, which enables much shorter steaming times. In *Fig 2* it is shown that the brightness of birch green wood was lowered by steaming under atmospheric conditions, whereas pressurised steaming had no effect on the brightness. The standard deviations for spruce and aspen were < 0.5% ISO at all test points. A major difference between the atmospheric and the pressurised steaming was that the amount of air was significantly lower in the pressurised environment as there was a constant flow of steam through the steam vessel. It is therefore reasonable to assume that the discolouration that took place when no pressure was applied can at least partly be explained by the presence of air, or most likely by the oxygen in the air. This assumption is supported by the fact that the loss in brightness was more pronounced when the birch shavings were conditioned at 50°C and 90% RH than when steaming was performed (compare *Figs 1* and *2*). *Fig 2* also shows that for atmospheric steaming|, it is especially important to limit the steaming time as much as possible.

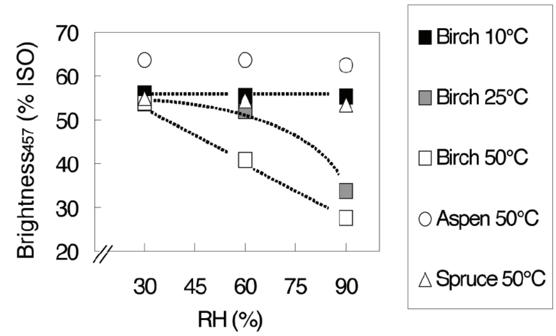

Fig 1. Brightness of wood shavings from birch, spruce and aspen, measured after conditioning for 1 h.

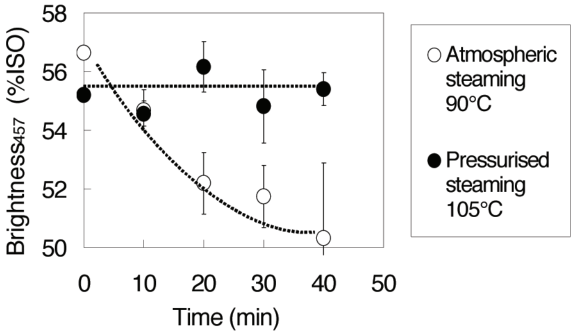

Fig 2. Brightness of wood shavings from birch, measured after atmospheric and pressurised steaming.

Influence of process conditions on the brightness of birch green wood

The subsequent investigation aimed at establishing the effects of different processing conditions on the brightness and yield of birch wood. It was performed on shavings that had been dried as explained. *Fig 3* shows the quality of the model for the four responses (ΔBrightness, Δ*k*, Δ*s* and *yield*). The y-axis shows two values, R2 and Q2, indicating how well the model is fitted to the data and the predictive ability, respectively. It is important to realise that the outcome of the study is a reflection of the specific levels chosen for each factor. As can be seen in the figure, the changes in *brightness* and *k* were fairly well fitted and could also be relatively well

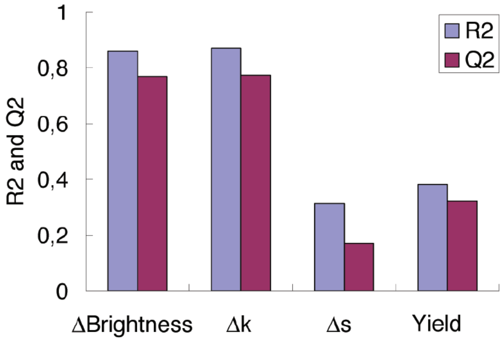

Fig 3. The quality of the model as described by R2, which shows how well the model is fitted to the data, and Q2, which shows the predictive ability of the model.

predicted, whereas *s* and *yield* were less well fitted and impossible to predict using this method.

The poorly fitted yield may probably be attributed to the very low grammage of the samples, which unfortunately made weighing a critical measurement causing large experimental variations. The changes in the light scattering coefficient, *s*, were small (0.9 ± 0.8 m^2/kg). This result could be expected, as the light scattering coefficient is a structural property that should not be affected by this process. Consequently, light scattering will also be poorly fitted.

Due to the small variations in *s*, it was only the changes in the light absorption coefficient, *k* that had any significant impact on wood brightness. *Fig 4* shows the overall result for the changes in ISO brightness caused by the changes in pre-heating temperature, pre-heating time, charge of sulphite and alkali, and the combined effects of these changes. The changes in *brightness* (and *k*) depended on the temperature and also marginally on time and on the combined effect of time and temperature at the specific levels chosen in this investigation. As expected, the brightness was also affected by the amounts of Na_2SO_3 and NaOH charged in the impregnation; on the interaction of sulphite and temperature, and on the interaction of sulphite and pre-heating time. The significance of the negative quadratic terms S × S implies that the charge of sulphite has a non-linear effect on brightness, which may give a brightness maximum, although this maximum does not necessarily fall within the experimental domain. The same reasoning applies to the alkali charge, but since the quadratic term OH × OH is positive, a brightness minimum is implied.

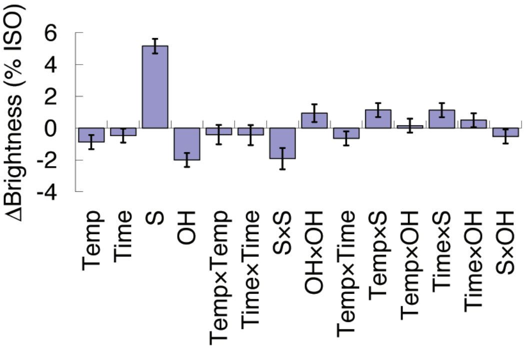

Fig 4. Changes in ISO brightness for thermally treated wood shavings from birch in response to changes in temperature, time, charge of sulphite and alkali, as well as the combined effects of these changes.

The interaction effect of NaOH and temperature on brightness was not significant, and the interaction effect of NaOH and time was only marginally significant. The reason for this could be that most of the alkali darkening reactions occur very fast and are almost completed within the first minute of the pre-heating, after which the brightness curve tends to level out. This can be seen in *Fig 5*, where the experimental result of using 2% NaOH in the impregnation is shown. The maximum standard deviation in *Fig 5* was ± 2.1% ISO at 165°C and 5 min. At all other test points, it was less than ± 1.5% ISO. The reaction with alkali did not require a high temperature; even at room temperature, there was a considerable loss in brightness. Unfortunately, the model did not allow for any conclusions to be drawn about yield. Yet, on the basis of common knowledge, it is reasonable to assume that the yield is reduced by the combined effects of increases in alkali, pre-heating temperature and pre-heating times.

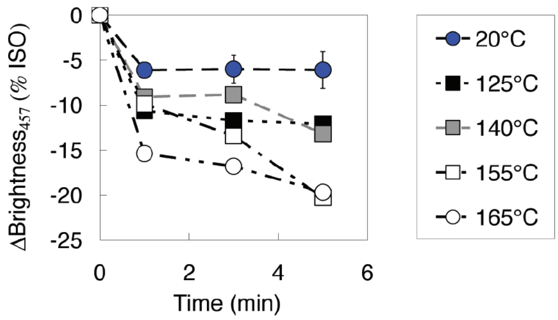

Fig 5. Changes in brightness of thermally treated wood shavings from birch impregnated with 2% NaOH.

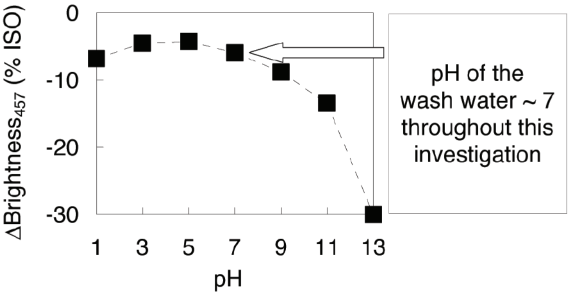

Fig 6. Change in brightness of thermally treated wood shavings from birch impregnated with 2% NaOH and washed with water with different pH.

However, the final brightness is also affected by the pH of the washing water or, in these experiments, the boiling water, which was 7 throughout this investigation. A more acidic wash would be more favourable from a brightness perspective, as indicated in *Fig 6* where the impact of the pH of the washing water on the brightness is shown. The pH interval where the brightness peaks seem to be somewhere in the range of 3–5, which is consistent with what is reported in the literature for unbleached spruce TMP (Lai et al. 1993). The maximum standard deviation in *Fig 6* was ± 1.4% ISO at pH 13.

Fig 7 shows how the interaction of pre-heating time and temperature affected brightness when 5% sulphite was charged in the impregnation. The maximum standard deviation in *Fig 7* was ± 1.6% ISO at 165°C and 3 min. At all other test points, it was less than ± 1.1% ISO. The experimental results reported in *Fig 7* show that at the

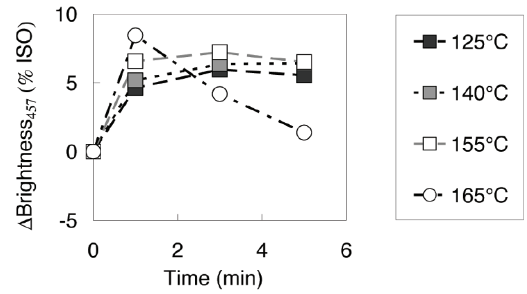

Fig 7. The change in brightness of thermally treated wood shavings from birch impregnated with 5% Na_2SO_3 at pH 9.5.

highest temperature (165°C) it is important to keep the pre-heating time short to maintain maximum brightness.

Modelling of the change in brightness with the changes in process conditions

When alkali is used, optimisation of the pre-treatment becomes even more complicated because sulphonation is affected not only by the pre-heating time and temperature but also by the pH. Given that there is an existent and applicable methodology to simulate the brightness response in the CTMP process, the use of multivariate data analysis can facilitate optimisation immensely. By using multivariate tools to analyse the experimental data regarding how native wood responds to different conditions in the pre-treatment (chemicals, pre-heating time and pre-heating temperature), it becomes possible to predict the outcome for a given set of conditions. The modelled or predicted response can then easily be illustrated by a contour plot, which serves as a topographical map. *Figs 8* to *11* and *13* show examples of contour plots with the change in brightness as the predicted response. To facilitate comparison between the plots, all share the same colour code, which ranges from red (gain in brightness) to blue (loss of brightness). The extent to which the brightness was changed is shown in the legend. The chemical charges referred to in *Figs 8* to *10* were chosen because of their perceived industrial relevance.

Fig 8 displays a case where 1% NaOH was used and *Fig 9* displays a similar case with 2% NaOH (compare with *Fig 5* where the actual experimental result is shown). No sulphite was used in either case. The loss in brightness became slightly more pronounced as temperature and time increased simultaneously, and the effect was enhanced with the higher alkali dosage. Vesterlind and Höglund (2005) showed that if the alkali charge is kept below 1.5%, yield is not adversely affected by a raised pre-heating temperature (160°C in comparison to 130°C). Combining their findings with the data reported here, it is recommended that the dosage of NaOH be kept below 2% in the impregnation to maintain both sufficiently high brightness and yield.

Fig 10 shows a case where 3% Na_2SO_3 was charged and *Fig 11* a case when 5% Na_2SO_3 was charged (compare with *Fig 6* where the actual experimental result is shown). The pH of the sulphite-liquor was 9.5 in both cases as no extra alkali was charged. Due to the bleaching effect of sulphite, both figures display a large interval where the brightness response could reach a maximum, that is, where the sensitivity to variations in temperature and time in the pre-heater was low.

Fig 12 shows that with no sulphite charged at all in the impregnation, the sensitivity to pre-heating temperature and time was more pronounced than when the highest sulphite charge (5%) was used. Factors not included in the figure were kept at their mean level. With the high sulphite charge, the brightness proved to be more or less unaffected by the increases in pre-heating temperature and pre-heating time, and the overall brightness response was notably increased.

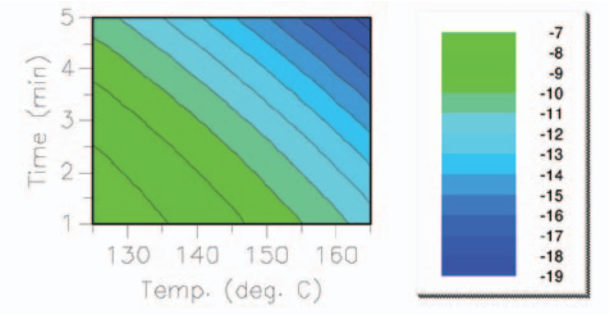

Fig 8. Contour plot of the predicted change in brightness of thermally treated wood shavings from birch impregnated with 1% NaOH.

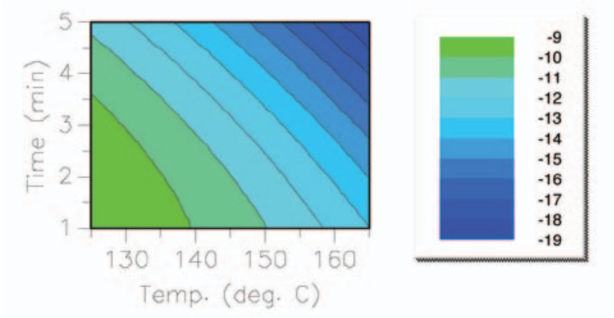

Fig 9. Contour plot of the predicted change in brightness of thermally treated wood shavings from birch impregnated with 2% NaOH.

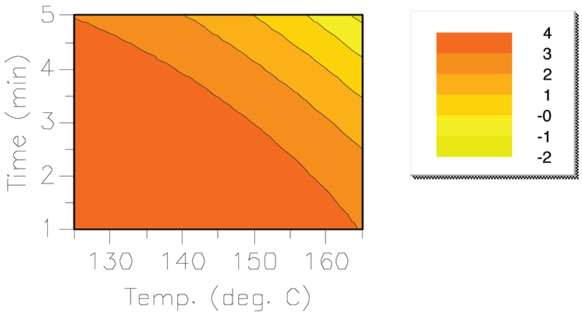

Fig 10. Contour plot of the predicted change in brightness of thermally treated wood shavings from birch impregnated with 3% Na_2SO_3 (pH ~ 9.6).

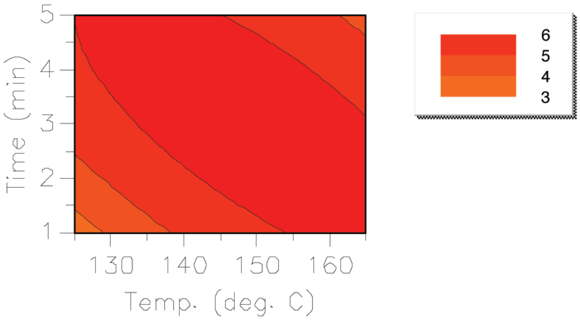

Fig 11. Contour plot of the predicted change in brightness of thermally treated wood shavings from birch impregnated with 5% Na_2SO_3 (pH ~ 9.6).

The case was more complicated when the chemical charge was made with a combination of NaOH and Na_2SO_3. This is exemplified in *Fig 13*, where the chemical charge in the model was 2% NaOH and 3% Na_2SO_3.

In *Fig 13* it is also clear that the sensitivity within this region was quite small – the loss was smallest in the upper left-hand corner of the figure but, apart from that, the brightness loss was fairly constant. The results are consistent with what was found in previous study of birch

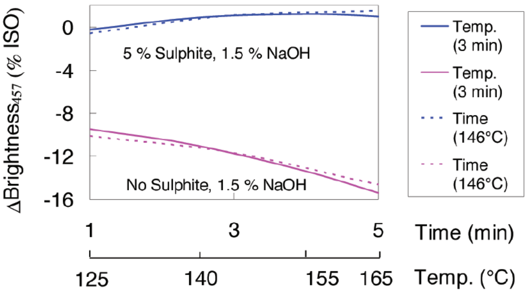

Fig 12. The modelled change in brightness of wood shavings from birch impregnated with 1.5% NaOH without sulphite or with 5% sulphite in the impregnation.

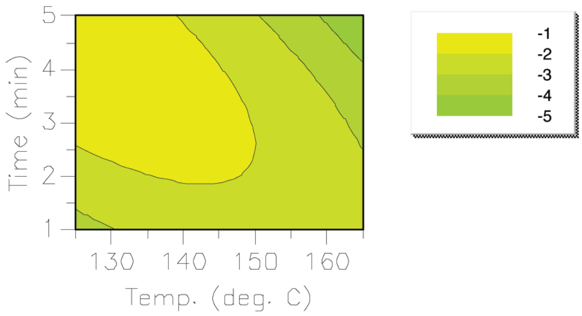

Fig 13. Contour plot of the predicted change in brightness of thermally treated wood shavings from birch impregnated with 2% NaOH and 3% Na_2SO_3.

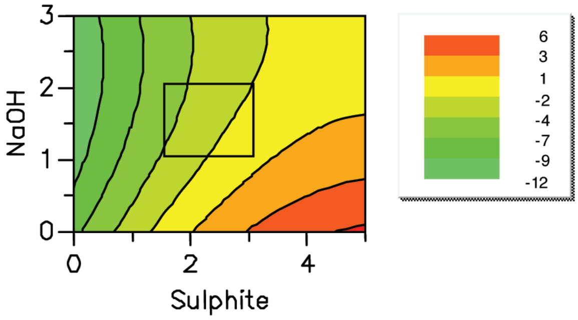

Fig 14. Contour plot of the predicted change in brightness of chemically treated wood shavings from birch pre-heated at 146°C for 3 minutes. The area judged to be of main industrial relevance is marked by a square.

CTMP (Vesterlind and Höglund 2005; Vesterlind et al. 2005) which demonstrated that the darkening (light absorption) of birch pulps produced with a short pre-heating time (1 min) and with the same amount of chemical charge (2% NaOH and 3% Na_2SO_3) varies more or less randomly on a small scale with the temperatures used in the investigation. The same study demonstrates the positive effect of a high pre-heating temperature (>140°C) on the energy consumption and the shive content in first stage refining.

Fig 14 shows the change in brightness as a function of the chemical charge of NaOH and sulphite when the temperature was kept constant at 146°C with a pre-heating time of 3 minutes. The brightness loss in the area of main industrial relevance (marked in the figure by a square) ranges from –6 units (with 1.5% sulphite and 2% NaOH) to no loss at all (with 3% sulphite and 1% NaOH). This means that, within the area of industrial relevance, it is possible to find conditions at, or close to, this pre-heating temperature and time where the brightness loss is about zero.

It can therefore be recommended that temperatures around 140–155°C should be used for the production of birch CTMP. The process can then benefit from low energy consumption and low shive content, as described by Vesterlind and Höglund (2005) and Vesterlind et al. (2005). Brightness can be maintained if the chemical treatment is made with proper charges of sodium sulphite and sodium hydroxide.

In summary, combining the knowledge obtained from large-scale trials, such as pilot trials or mill trials (Vesterlind et al. 2005), and the use of the methodology presented in this paper provides a powerful tool for optimising the CTMP process with respect to runnability, mechanical pulp properties and brightness.

Conclusions

Optimising the pre-treatment of birch CTMP with respect to brightness is obviously quite a delicate matter as all factors in the pre-treatment (i.e. temperature, time, alkali charge and sulphite charge), may affect the final brightness. The shavings methodology, which examines changes in the brightness of green wood, can greatly facilitate this optimisation. Multivariate data analysis makes it possible to identify regions of larger or smaller stability, or in other words, to establish combinations of process conditions that yield high brightness, and where the brightness is not very sensitive to variations in the conditions. The experimental window used in this study can easily be widened, making it comparatively easy to perform investigations of many variables at many levels. Furthermore, because the evaluation is done directly on the wood, it gives valuable information that might be lost when the pulp is produced in authentic trials.

The investigation reported here shows that the brightness of birch green wood is sensitive to increases in relative humidity and temperature. This finding has implications for the steaming of the chips and suggests that this operation is best performed under pressurised conditions in order to maintain the initial wood brightness.

An evaluation of the effect of varying the temperature, time and chemical charge in the pre-treatment showed that, within the experimental domain of this study, the change in brightness was mainly related to the amount of chemicals and the pre-heating temperature. The pre-heating time had less influence. The negative effect of alkali on brightness was evident and so was the positive effect of sulphite. Furthermore, the combined effects of alkali with pre-heating temperature and pre-heating time were not as significant for brightness as the combined effects of sulphite with pre-heating temperature and pre-heating time.

It can be concluded that using a relatively high pre-heating temperature (~140–155°C) in the manufacturing of birch CTMP is not detrimental to pulp brightness. However, at very high pre-heating temperatures (>160°C) it is important to keep the pre-heating time short to avoid discolouration.

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