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Oxygen Delignification of High-kappa Number Pine Kraft Pulp

Abstract
The possibility of producing easy bleachable pulp (kappa number < 16) from high kappa number (68) pine kraft pulp through its oxygen delignification has been studied. The effect of the application of peroxy compounds in the oxygen delignification sequences of such pulps was also established. The yield and properties thus produced easy bleachable pulps were compared to the properties of pulp having similar bleaching susceptibility, produced in a conventional method (i.e. by wood pulping to kappa number approx. 30 and then oxygen delignification). The effect of factors such as: higher pH of medium, elevated temperature and presence of metal ions on stability of peracetic acid solution was also determined. The most important conclusion from carried out studies was finding that fibrous semi product produced in the process of oxygen delignification of pine kappa number 68 pulp with or without the use of peroxy compounds show higher yield (by approx. 2.5% on wood) compared with pulps produced by means of conventional method.

Key words: *high kappa number pine kraft pulp, oxygen delignification, degree of delignification, peracetic acid, hydrogen peroxide, fibre properties.*

Introduction

The production costs of high-grade bleached pulps with high and stable brightness and appropriate mechanical strength can be lowered above all by an increase in pulp yield [1] as well as an optimisation of the bleaching process technology [2]. Approx. 80% of the pulps used at present for the production of high-brightness, uncoated, non-wood printing papers are manufactured by means of the chemical method, the so-called sulphate method or kraft process, in which 50% of raw material is dissolved. The increase of pulp yield in this process by one per cent (e.g. from 42% to 43%) decreases wood consumption by approx. 2.4%, and thus lowers the raw material costs, which have been showing a steady upward trend [3]. At present, an oxygen delignification in alkaline medium (a method first used in 1970) is commonly used for the further delignification of kraft pulps before their bleaching by the ECF or TCF method. In order to optimise these processes in regard to pulp yield and quality, various modifications are investigated including an option which uses cooking at a relatively high content of residual lignin in pulp (corresponding to kappa number > 40), and then continuing the process of lignin removal by a more selective method of oxygen delignification [4]. The success of such a solution will depend on profit and cost balance. Preliminary estimates seem to support such modification [1].

The attractiveness of oxygen delignification of pulps also results from the possibility of applying different modifications to improve its effectiveness. These include the preliminary or inter-stage activation of lignin [5], the removal of heavy metal ions participating in the formation of hydroxyl radicals that take part in cellulose depolymerisation, and the reinforcement of the delignification effect by the addition of hydrogen peroxide [6] or the use of catalytic agents [7] and enzymes [8].

For lignin activation, peracetic acid (CH_3COOOH) [9], and inorganic persulphate compounds such as Caro acid (H_2SO_5), $2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$ (known as Oxone) [10] and sodium peroxysulphate (Na_2SO_5) can be used. Peracetic acid and Caro acid are produced on a commercial scale, although their price in comparison with the price of oxygen and hydrogen peroxide is much higher. Sodium peroxysulphate can be produced by a copper ion (Cu^{++})-catalysed oxidation of sodium sulphate with oxygen in a strongly alkaline medium [11].

According to literature, the amount of lignin removed from pulp in a single-oxygen stage usually does not exceed 40 to 50% [5]. In order to achieve higher delignification degrees of pulp without its deterioration (i.e. decreased yield and strength), it is suggested that preliminary lignin activation is applied by means of chemicals with higher oxidation potential than oxygen, or that delignification should be divided into two stages. Oxygen delignification of pulps with high lignin content (kappa number > 40) is characterised by special features. On the one hand, the amount of lignin to be removed is much higher than in the case of conventional pulps with a kappa number of 30; on the other hand, the residual lignin in pulp of a high kappa number may be less altered chemically, and thus easier to remove.

The replacement of part of the pulping process with oxygen delignification (which is considered to be more selective) should result in an improvement of pulp strength indices. Our earlier investigations proved that fibres of pulp with a kappa number of 68 are less damaged, longer, exhibit higher coarseness and larger width, and are less susceptible to cross-deformation. By means of using atomic force microscopy (AFM) differences have also been noted in the chemical composition of the fibre surface layers between this pulp and pulp of kappa number 28 [12].

Aim of work

The aim of the work was to define the conditions for the oxygen delignification of high-kappa number pine kraft pulp that will enable easily bleachable semi-product to be obtained for the production of high-brightness, non-wood printing papers, and to determine the effects that can be achieved by the application of peroxy compounds in the oxygen delignification of such pulps.

Experimental

Pulps

For these experiments, the following pulps were used: high-yield pine kraft pulp (kappa number 68) produced in the laboratory at an average yield of 51.8%, and pine kraft pulp (kappa number 28) produced in the laboratory at an average yield of 45.4%. Pine wood chips were used as raw material for the production of these pulps. Pulping was carried out in a rotary Hatoo digester in 3l autoclaves. The chips were preliminarily vacuum-impregnated with cooking liquor of 30% sulphidity which in the case of the high-kappa number pulp contained 18.5% of active alkali (as NaOH) relative to o.d. wood, while this figure was 23% of active alkali relative to o.d. wood in the case of conventional pulp. The heating times and pulping temperatures were

the same in both cases, that is, 90 min and 164 °C respectively. The pulping time at maximum temperature was 90 min for high yield pulp, and 150 min for conventional pulp. After pulping, the pulp was preliminarily washed with distilled water on the wire, then by diffusion for 24 hours, and finally re-pulped and screened. After having been separated from pulp, any knots were refined in a laboratory impact mill and then added to the screened pulp. Next, the treated pulp was centrifuged, and after determining its dryness it was stored in a refrigerator in closed polyethylene bags.

Delignification and bleaching chemicals

Oxygen, distilled peracetic acid (CH_3--COOOH, Fluka Chemica), EDTA, hydrogen peroxide.

Oxygen delignification

The weighed portion of pulp (40 g o.d.) was placed in a polyethylene bag, and then the following chemicals were added: 0.5% magnesium sulphate relative to o.d. pulp (in the form of a 1% solution), and then an appropriate amount of sodium hydroxide. The bagged content was hand-mixed and then transferred in quantity to a Jayme digester. The autoclave was closed and filled with oxygen; the rotating mechanism was switched on, heated to a temperature of 100 °C within 30 minutes, and then heating was continued at this temperature for 60 or 120 minutes. The pulps were delignified in single- or two-stage processes, but in the second case the addition of sodium hydroxide was divided into equal parts between the two oxygen stages. At the end of the delignification time, the digester was degassed and emptied. After delignification, the pulp was washed with distilled water and filtered, and its dryness and yield were determined.

Pulp treatment with peracetic acid

A sample of pulp was placed in a polyethylene bag. Subsequently, heated distilled water in an amount necessary to ensure the assumed concentration of fibrous slurry, together with a proper amount of peracetic acid (calculated to active oxygen relative to o.d. pulp), and sodium hydroxide for determining the pH were added to a beaker. After a thorough mixing of the ingredients and determining the pH, the solution was poured into an ethylene bag containing heated pulp. The content was hand-mixed and heated in a water bath for 30 minutes at a temperature of 50 °C, and then for 90 minutes at 70 °C. At the end of the treatment time, the pulp was filtered in a Büchner funnel and then washed with distilled water.

Determining the pulp's properties

The pulping degree (kappa number) was determined by lignin oxidation with potassium permanganate (PN-70/P50095) [13]. The pulp brightness –was classified according to International Standard ISO No 36688 (1997) 'Pulp Measurement of diffuse blue reflectance factor (ISO brightness)' [14]. The fibre strength factor (FS-factor) was tested in a Zero-Span TroubleShooter Tester TS-100, Pulmac [15].

Discussion of results

Defining pulp treatment conditions with peracetic acid

As mentioned above, one promising method of lignin activation is the treatment of pulp with peracids, above all with peracetic acid. The amount, temperature and pH are important parameters of the pulp treatment process with this chemical. Considering this, prior to experiments with pulp, the effect of initial pH on the stability of a distilled peracetic acid solution at temperatures of 50 °C, 70 °C and 80 °C was studied. This effect was evaluated on the basis of determining the active oxygen concentration (A.O.) in a peracetic acid solution by means of the titration method [16]. The results of measurements are presented in Figures 1 and 2. According to the course of the curves shown in Figure 1, during the heating process of peracetic acid at 50°C over 60 min., the least losses of active oxygen were found in experiments in which the initial pH was 3.5, slightly higher in experiments with a pH of 4.5 and 5.5, and the highest in experiments with a pH of 7.0.

As shown in Figure 2, peracetic acid also maintains relatively good stability when heated at temperatures of 70 and 80 °C at pH 3.5. In such conditions, after raising the pH to 5.5 and especially 7.0, a significant increase in CH_3COOOH decomposition can be observed. Initially, pH 7 was difficult to accept in practice also because of the considerable amount of sodium hydroxide necessary to adjust the pH at this level. Apart from that, Figure 3 shows that the pH of peracetic acid solution initially alkalised to pH 7.0 quickly decreases at 70 °C anyway. Taking into consideration the significant effect of pH on the stability of peracetic acid, it is evident that the pulp should be treated with this acid at the lowest possible pH.

In order to follow the process of the peracetic acid consumption in the presence of pulp, some experiments were carried out using pulp of kappa number 68, which was delignified in single-stage oxygen delignification with and without a chelation stage (Q). The individual series of experiments, whose results are represented by the curves in Figure 4, differed from each other in the initial pH in the peracetic acid treatment, although a constant temperature of 70 °C was maintained. The amount of residual active oxygen was determined after different reaction times within the range of 5 to 120 minutes. The course of the curves in Figure 4 shows above all that the consumption of peracetic acid during the treatment of pine kraft pulp with a kappa number of 42.2 is faster in those experiments in which the pulp was not treated with a chelating agent, or/and when the treatment of pulp was performed at a higher pH (5.5 or 7). In all probability, the faster consumption of peracetic acid in these experiments is the result of transition metal catalysed decomposition of peracetic acid, or its spontaneous decomposition taking place at higher pH values. Thus, the obtained results confirm the earlier stated tendency of peracetic acid decomposition under the influence of heavy metal ions [17], and the higher stability of this agent at lower pH [18].

Taking into consideration the results of the experiments obtained at this stage of work, the treatment of pulps with peracetic acid was applied following the chelation stage, at pH 5.5 and 3.5, first

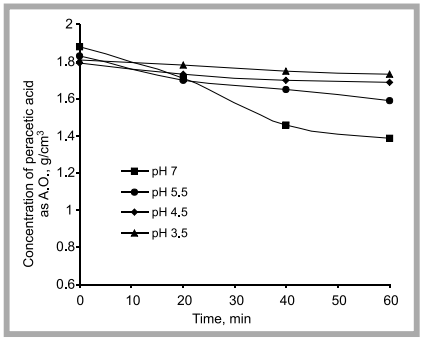

Figure 1. *Effect of pH on decomposition kinetics of distilled peracetic acid at temperature 50* °C.

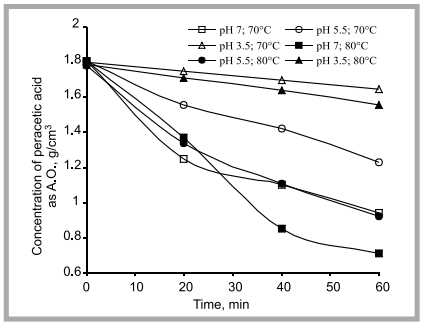

Figure 2. *Effect of pH on decomposition kinetics of distilled peracetic acid at temperatures of 70 °C and 80 °C.*

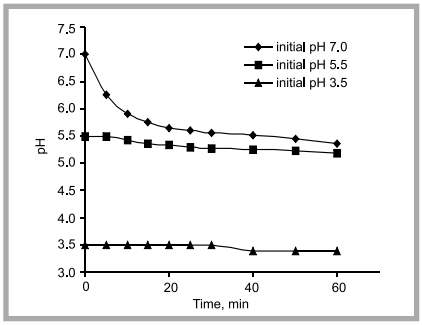

Figure 3. *Variations of pH of peracetic acid solution initially alkalised to pH 3.5; 5.5 and 7.0 during heating at temp. 70 °C.*

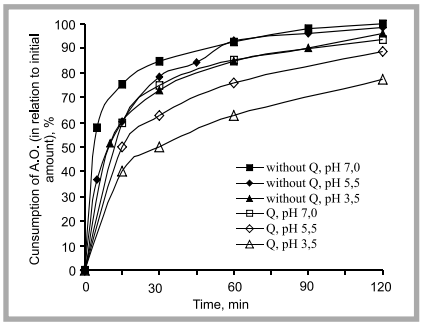

Figure 4. *Effect of reaction time and initial pulp treatment by a chelating agent and pH on the consumption of active oxygen (A.O.) during pine kraft pulp treatment with peracetic acid at temp. 70 °C.*

for 30 minutes at 50 °C and next for 90 minutes at 70 °C. Because when peracetic acid was added in an amount of 1% some part of it was not consumed, its amount was decreased to 0.66% of A.O. in relation to o.d. pulp.

Oxygen delignification

Oxygen delignification becomes especially significant in the case of pine kraft pulps with a kappa number higher than 20, because such pulps are not qualified directly for bleaching, especially in sequences where the elemental chlorine is completely substituted by chlorine dioxide. In this work, the kraft pulp of high-kappa number (68) was delignified in the single- and two-stage medium consistency oxygen delignification to decrease the lignin content to 14-16 kappa number units, which would enable the pulp to be bleached fully with a reasonable amount of bleaching agent. The pulps were pre-treated with a chelating agent (Q stage).

A variable total amount of NaOH (4, 6, 8 and 9% relative to o.d. pulp) was used, which in the case of two-stage processes was divided into two equal parts between the two oxygen stages. In some experiments, peracetic acid and hydrogen peroxide were used in both single- and two-stage oxygen delignification processes. In oxygen delignification experiments with hydrogen peroxide, this chemical was added in equal amounts at each oxygen stage. Peracetic acid was applied at a separate stage marked Pa. Table 1 shows the experiment number, the oxygen bleaching sequence, the total amount of added NaOH, treatment conditions at the Pa stage or the total amount of added hydrogen peroxide in experiments in which this chemical was used.

As shown in Figures 5 and 6, the two-stage oxygen delignification of pulp with a kappa number of 68 pulp with the use of NaOH in the amounts of 4, 6, 8 and 9% (exp. 1-4, table 1) makes it possible to lower the kappa numbers of this pulp to 33, 24, 18 and 16 respectively, that is, to achieve delignification of 52, 65, 73 and 77%. The degree of delignification in a single-stage process, with a specific amount of added NaOH, proved to be higher by approx. 3-6% than the degree of delignification achieved in a two-stage process. In this case, the decrease of the kappa number of the initial pulp to a level below 16 was achieved at a 1% lower amount of added NaOH in comparison with the two-stage process. Taking into consideration the fact that the time and temperature conditions in single- and two-stage oxygen delignification processes were identical (100 °C, 120 min.), the result obtained may be due to the application of a smaller amount of magnesium sulphate in the single-stage process (0.5%, in comparison with 1% in the two-stage process), and to the fact that in the two-stage process pulp delignification is interrupted by inter-stage washing, which probably decreases the amount of alkali by a small but significant part for the second stage of oxygen delignification.

Figures 5a and 5b show that similar results were obtained from the oxygen delignification of kappa number 68 pulp with peracetic acid treatment added in an amount of 0.66% A.O. to o.d. pulp in three applied variants (inter-stage treatment at pH 5.5 and 3.5 and pre-treatment at pH 3.5). In comparison with the reference experiments (Table 1, exp. 1, 2 and 5), a higher decrease in kappa number was achieved at a specific amount of used sodium hydroxide. For example, at 6% of added sodium hydroxide to o.d. pulp in the two-stage oxygen delignification, the kappa number reduction was higher by 10-11, while in the single-stage oxygen delignification by only approx. 6 (Figure 5a). As a result, the degree of delignification of peracetic acid-treated pulp was higher by approx. 23% for the two-stage process, and approx. 14% for the single-stage process (Figure 5b). It should be noted that although the amount of used NaOH was lower by 2-3%, the kappa number of the oxygen delignified pulps treated with peracetic acid was lower by approx. 2-3 units than the kappa number of pulps delignified only with oxygen in an alkaline medium.

The comparison of the results of the two-stage oxygen delignification experiments performed with peracetic acid shows that the variant of treatment at pH 3.5 proved to be slightly better than the variant of treatment at pH 5.5. Apart from that, the peracetic acid treatment at pH 3.5 is supported by the argument that a considerably lower amount of NaOH is required to settle pH at 3.5 than at 5.5.

Figure 5a shows that the reinforcement of the oxygen delignification process with hydrogen peroxide in an amount of 3% relative to o.d. pulp gives similar results, regardless of the process sequence. In

Table 1. *Conditions in experiments performed on kraft pine pulp with kappa number 68.*

Exp. number	Oxygen delignification sequence	Total amount of added NaOH	Pa and O_P stages
1	QOO	4	-
2	QOO	6	-
3	QOO	8	-
4	QOO	9	-
5	QO	6	-
6	QO	8	-
7	QOPaO	4	0.66 % A.O., pH 5,5
8	QOPaO	6	0.66 % A.O., pH 5,5
9	QOPaO	4	0.66 % A.O., pH 3,5
10	QOPaO	6	0,66 % A.O., pH 3,5
11	QPaO	4	0,66 % A.O., pH 3,5
12	QPaO	6	0,66 % A.O., pH 3,5
13	QO_PO_P	6	3% H_2O_2
14	QO_PO_P	8	3% H_2O_2
15	QO_P	6	3% H_2O_2
16	QO_P	8	3% H_2O_2

comparison with the reference experiments (Table 1, exp. 2, 3 and 5, 6), at a specific amount of NaOH added, the kappa number was decreased in the following degrees: for pulps delignified in two-stage oxygen delignification by approx. 5, and for pulps delignified in single-stage by only 2-3. So, the degree of delignification has increased, as shown in Figure 5b, by approx. 8% for the two-stage process, and by approx. 2-3% for the single-stage process; that is, considerably less than in the case of peracetic acid-treated pulps, and at the same time at a consumption of sodium hydroxide higher by 2%. Thus the oxygen delignification variant with the addition of hydrogen peroxide is less favourable than the variant with the peracetic acid treatment of pulp.

Figure 5c shows the relationship between the yield and kappa number for different variants of oxygen delignification of pine kraft pulp, kappa number 68. As for the yield, it appeared that the variant in which the peracetic acid treatment was applied at pH 5.5 proved most favourable. At a kappa number of approx. 14 units, the pulp yield was approx. 89.5%. The two-stage oxygen delignification of pulp with a kappa number of 68 with inter-stage treatment with peracetic acid in pH 3.5, or single-stage oxygen delignification according to the QO, QPaO and QO_P sequences, led to a pulp yield which was lower by approx. 0.4%. The lowest yield of pulp was achieved in experiments in which the initial pulp was delignified only by oxygen in aqueous alkali, and in the two-stage oxygen delignification with the addition of hydrogen peroxide.

Comparing the corresponding curves in Figure 6a, it can be stated that at a comparable kappa number, the modified method including pulping to a kappa number of approx. 70, followed by a single- or two-stage medium consistency oxygen delignification, with or without the application of peroxy compounds, leads to the obtaining of a semi-product with a higher yield by approx. 2.5% in comparison to kraft cooking to the kappa number of 30, followed by a single-stage oxygen delignification.

An estimate of the effect of oxygen delignification of pine kraft pulp kappa number 68 on fibre strength was made by determining the zero-span fibre strength factor (FS-factor) with a Troubleshooter TS-100 tester. Strips of paper were rewetted with water before the tests. According to the literature, the value of this index can be significantly influenced by fibre curl [19, 20]. Hence, this property was determined for beaten pulps in laboratory conditions. It is known that pulp fibres are straightened during beating, and in test conditions they can simultaneously

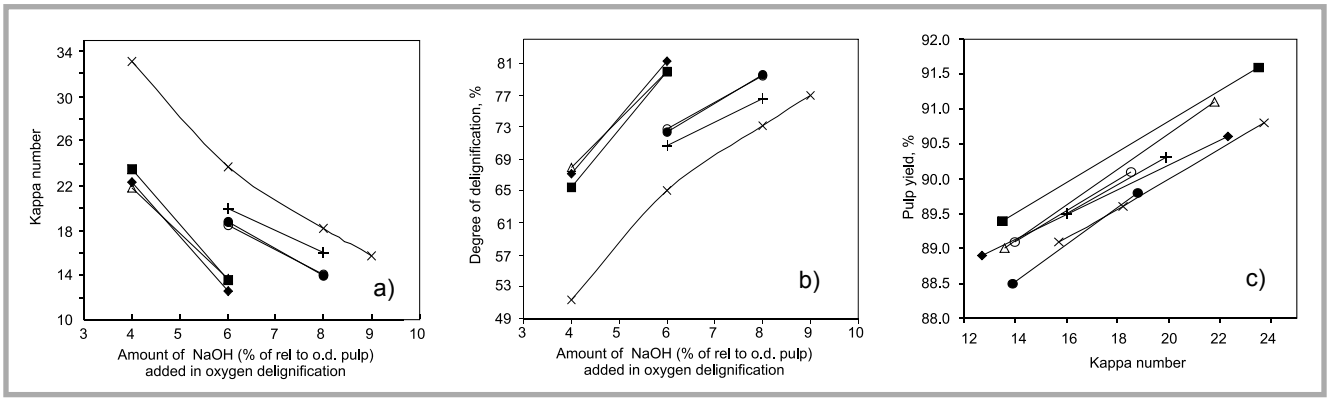

Figure 5. *a) Relationship between the kappa number and the amount of NaOH added in different variants of oxygen delignification of pine kraft pulp, kappa number 68 (the standard deviation of the kappa number determination was 0.1); b) Relationship between the delignification degree and the amount of NaOH added in different variants of oxygen delignification of pine kraft pulp, kappa number 68; c) Relationship between yield and kappa number in different variants of oxygen delignification of pine kraft pulp, kappa number 68 (standard deviation of yield amounted to 0.14%).Exp. numbers according to Table 1: × - 1, 2, 3 & 4, + - 5 & 6; ■ - 7 & 8; ♦ - 9 & 10; Δ - 11 & 12; ● - 13 & 14; o - 15 & 16.*

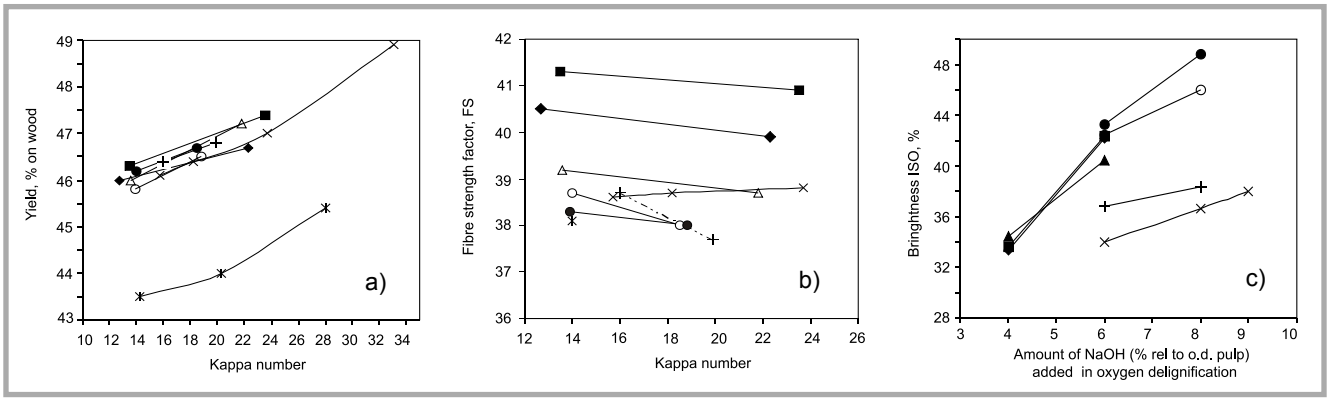

Figure 6. *a) Relationship between pulp yield (% on wood) and kappa number in different variants of oxygen delignification of pine kraft pulp, kappa number 68, against yield of conventional pulp, kappa number 28 oxygen delignified in a single-stage process, b) Relationship between FS-factor and kappa number in different variants of oxygen delignification of pine kraft pulp, kappa number 68, after beating in Jokro mill (beating time 12 minutes, freeness 21°SR) (standard deviation of FS-factor was 1.1), Relationship between pulp brightness and the amount of added NaOH in different variants of oxygen delignification of pine kraft pulp, kappa number 68; Exp. numbers according to Table 1: × - 1, 2, 3 & 4, + - 5 & 6; ■ - 7 & 8; ♦ - 9 & 10; Δ - 11 & 12; ● - 13 & 14; o - 15 & 16, ⁎ - conventional pulp, oxygen delignified .*

transfer the load exerted by tester jaws. For this reason, the FS-factor values obtained are higher and closer to the actual fibre strength.

As shown in Figure 6b, the FS-factor of pulp with an initial kappa number of 68, delignified in a two-stage oxygen delignification with peracetic acid inter-stage treatment at pHs of 3.5 and 5.5, reached a kappa number level higher by 3-7% than the FS-factor value obtained in all the other experiments, and also by approximately the same in comparison with conventional pulp fibres (pulped in a digester to kappa number 28, and then oxygen-delignified in a single-stage process).

Brightness is a significant parameter which characterises the course of the pulp delignification process. With the increase of added NaOH, which is the main factor deciding the level of delignification in an oxygen-alkaline process, and the decrease of the kappa number, brightness should increase. However, the brightness increase obtained in oxygen delignification process is not great, which indicates that a large amount of lignin chromophores are resistant to oxygen. As shown in Figure 6c, the brightness of pine kraft pulp oxygen delignified with peracetic acid treatment at a definite amount of added sodium hydroxide is higher by approx. 5% in comparison with the brightness of the pulp delignified only with oxygen in an aqueous alkali (Tab. 1, exp. 2 and 6) and only somewhat lower than the pulp brightness after hydrogen peroxide-reinforced oxygen delignification.

Conclusions

1. In order to produce, by oxygen delignification from pine kraft pulp with a kappa number of 68, a bleachable semi-product free of residual lignin to a sufficient degree (in view of its subsequent complete bleaching according to the ECF method, i.e. at kappa number < 16) 8-9% of NaOH should be used relative to o.d. pulp. Single-stage delignification ensures a slightly higher decrease of the pulp's kappa number in comparison with the two-stage delignification process.
2. The application of initial or inter-stage pulp treatment with peracetic acid enables the required level of delignification to be reached at a 25-33% lower amount of added sodium hydroxide. The pulps produced in such conditions, in comparison to pulp delignified only with oxygen, are characterised by even lower kappa numbers (2-3 units), comparable or slightly higher yield (depending on the applied variant) as well as FS-factors higher by 4-7% and brightness higher by approx. 5%.
3. Peracetic acid exhibits good stability at 50 °C. However, at temperatures higher than 70 °C, its stability is much lower. To avoid losses due to its decomposition, it is necessary to maintain a low environmental pH (e.g. approx. 3.5).
4. The removal of heavy metal ions from pulps prior to peracetic acid treatment by means of a chelating agent treatment decreases the amount of decomposed peracetic acid. For this reason, some additional part of the peracetic acid can participate in lignin activation.
5. Good results are achieved in the case of peracetic acid treatment at pH 3.5 because the degree of pulp delignification obtained and the pulp strength are almost the same as in the case of peracetic treatment at pH 5.5, and the amount of NaOH consumed for pH control is relatively low.
6. The following results were produced by oxygen delignification of kappa number 68 pulp, with the addition of hydrogen peroxide to a kappa number level of approx. 14-16, at the same amount of added NaOH as in experiments in which the initial pulp with a high kappa number was delignified only with oxygen in an alkaline medium: a kappa number lower by approx. 11% [rel.] and ISO brightness higher by approx. 8-11% at comparable or slightly higher yields and similar fibre strengths.
7. Oxygen delignification of pulps characterised by a high kappa number to approx. 14-16 units (both with and without peroxide treatment), enables the production of fibrous semi-product characterised by higher yield (by approx. 2.5%) on wood, in comparison with the yield achieved in case of conventional pulps. The FS-factor of this pulp is comparable or only somewhat higher (4-7% rel.) than the FS-factor of conventional pulp.

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Received 30.06.2005 Reviewed 08.02.2006