

Alkaline Pulping of Portuguese *Eucalyptus globulus*: Effect on Hexenuronic Acid Content

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The effect of alkaline pulping parameters (temperature, effective alkali (EA) and sulphidity) on the hexenuronic acid (HexA) content of Portuguese Eucalyptus globulus pulps was studied. The HexA content was calculated on a pentosan basis. Unlike softwood pulping, the amount of this acid increased during kraft cooking and only started to decrease in the final stage of delignification (kappa number below 12). EA and temperature were shown to be the main factors affecting the amount of HexA in pulps. A good correlation of data with the consumption of EA, regardless of the cooking conditions, was observed. The HexA content was quite high (45–60 mmol/kg pulp) for the kraft pulps obtained in the common range of cooking conditions and it is not advisable to lower this content in the cooking stage to maintain pulp quality.

Nous avons étudié les effets des paramètres de la mise en pâte alcaline (température, alcali effectif, sulfidité) sur la teneur en acide hexénuronique (HexA) des pâtes d'Eucalyptus globulus portugais. La teneur en HexA a été calculée sur une base de pentosanne. À l'encontre de la mise en pâte de résineux, la teneur en HexA s'est accrue durant la cuisson kraft et n'a commencé à diminuer qu'à l'étape finale de la délignification (indice Kappa inférieur à 12). L'alcali effectif et la température ont été les principaux facteurs ayant eu une influence sur la teneur en HexA des pâtes. Nous avons observé une bonne corrélation entre les données et la consommation d'alcali effectif, peu importe les conditions de cuisson. La teneur en HexA était assez élevée (45 à 60 mmol par kg de pâte) pour les pâtes kraft obtenues dans l'échelle courante des conditions de cuisson, et il n'est pas recommandé de réduire cette teneur à l'étape de la cuisson afin de conserver la qualité de la pâte.

INTRODUCTION

During kraft pulping, polysaccharides undergo some undesirable reactions, such as the formation of hexenuronic acids (HexA), resulting from the elimination of methanol from the 4-*O*-methylglucuronic acids, which are side groups linked to the xylan backbone [1,2]. HexA is also degraded throughout the cooking but it has been shown that demethylation occurs faster than the loss of HexA groups [1,3]. Hence, HexA content increases with cooking time until a maximum is reached and then decreases.

This time-dependent profile is, however, influenced by the extent of HexA reactions, which are affected by the pulping conditions [2,3]. The xylan backbone also undergoes alkaline degradation, although this reaction is re-

tarded by the side groups of uronic acids. Additionally, alkaline hydrolysis of glycosidic bonds leads to the formation of xylan with a lower degree of polymerization, which can be removed from the pulp by dissolution in the liquor, carrying HexA groups with it. A topochemical effect was also referred to by Gustavsson and Al-Dajani [3] as another reason for HexA removal because xylan, with a higher degree of substitution (higher number of uronic acid or HexA groups), might be located on the surface of the pulp fibre and dissolved easily.

Consequently, HexA removal throughout cooking is due to not only its own degradation but also to its dissolution together with xylan degradation/dissolution, both reactions being dependent on cooking conditions. In softwood pulping, for instance, the rate of HexA degradation/dissolution increases with increasing [OH⁻], [HS⁻], temperature and ionic strength [3].

HexA provokes some harmful effects on the bleaching process because of its unsaturated structure [2]. HexA reacts with some

bleaching chemicals, increasing their consumption [2,4,5], and also binds transition metal ions, intensifying the use of expensive complexing agents in hydrogen peroxide bleaching [4]. In addition, the presence of HexA not only increases the difficulty of reaching a high degree of brightness, but also increases brightness reversion [4]. It was also found that HexA groups contribute to the pulp kappa number [4,6] because they consume part of the potassium permanganate used in this determination as well as lignin does. Therefore, this parameter does not reflect the true residual lignin content in the pulp, and this content is overestimated. In fact, it has been shown that for birch unbleached pulps, as much as 6 kappa number units were due to HexA contribution, depending on the cooking conditions [6].

The above-mentioned problems are more pronounced in the case of hardwood pulps because larger amounts of HexA are expected to be formed because of their higher xylan content. Furthermore, it has been reported recently [7] that the degradation/dissolution of HexA dominates over its formation only in the last

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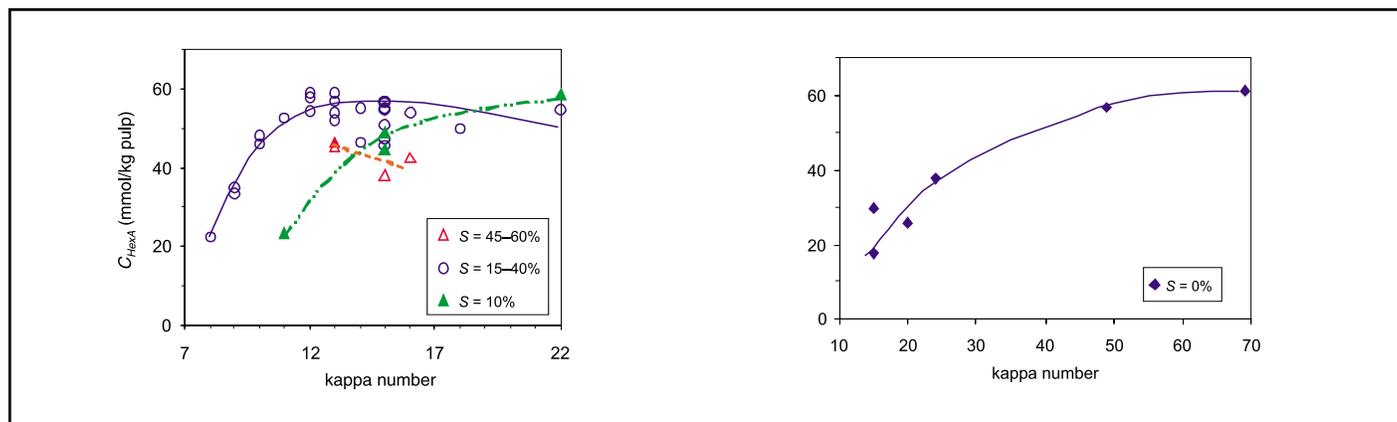


Fig. 1. HexA content as a function of the corresponding kappa number for all the pulps studied (S = sulphidity).

part of the hardwood cooking process and not in the beginning, as in the case of softwoods. As a result, the possibility of producing industrial hardwood pulps with a large amount of HexA is higher. It is therefore necessary to know how cooking conditions could minimize the HexA content of unbleached pulps, particularly in the case of hardwoods.

In the present work, the influence of kraft pulping parameters on both HexA and pentosan contents is evaluated for the main raw material of the Portuguese pulp industry – *Eucalyptus globulus* wood. In addition, a new approach to expressing results is addressed by calculating the HexA amount not only on a pulp or wood basis but also on a xylan (or pentosan) basis. This different way of quantification is meaningful because the dissolution of xylan fragments brings HexA into the cooking liquor, decreasing its amount in the pulp without being degraded.

EXPERIMENTAL

The unbleached pulps used in the present work were produced by the kraft process using *Eucalyptus globulus* wood chips, as described elsewhere [8]. The ranges of cooking conditions, yield and chemical characteristics for the studied pulps are summarized in Table I. These were selected to obtain one set of pulps with different kappa numbers and another set of pulps that were equally delignified (kappa number ~ 15). Pulps were characterized following standard procedures, as mentioned in previous studies [9,10], except for the HexA quantification. This was performed essentially according to the method established by Chai et al. [11] in pulps previously extracted with dichloromethane. This quantification is based on the HexA hydrolysis with a mercuric chloride (0.6%)–sodium acetate (0.7%) solution, combined with ultraviolet spectroscopy of the hydrolysate. Thus, a sample of pulp with known moisture content was weighed (~ 30 mg o.d.) and transferred to a 15 mL glass vial with 10 mL of $\text{HgCl}_2\text{-CH}_3\text{COONa}$ solution. Then the vial was closed, hand shaken (to disperse the pulp into the solution) and kept in a water bath at 70°C for 70 min. The reaction time and temperature were established after some preliminary experiments, as advised [11]. Thereaf-

Cooking conditions		Results	
EA (% Na_2O)	10.9–22.8	kappa number	7.5–69.3
S (%)	0–60	VISC (dm^3/kg)	596–1472
ACT (%)	90	PENT (%)	13.2–16.7
L:W	4:1	C_{HexA} (mmol/kg)	17.9–61.2
T ($^\circ\text{C}$)	152–181	TY (%)	48.1–64.4
t (min)	40–195**	REJ (%)	0–60
H-factor	260–3600	PY (%)	4.6–53.5

*See Nomenclature **In the majority of the cases, the cooking time was 90 min.

ter, the suspension was cooled to room temperature, centrifuged for 5 min at 2700 rpm, and filtered. The HexA content of the filtered solution was quantified by measuring the absorbance at wavelengths of 260 and 290 nm in a 1 cm path length silica cell. The $\text{HgCl}_2\text{-CH}_3\text{COONa}$ solution was used as the blank. HexA content was calculated by:

$$C_{\text{HexA}} \text{ (mmol/kg pulp)} = 0.287 \frac{(A_{260} - 1.2A_{290})V}{G} \quad (1)$$

where A_{260} and A_{290} are the absorbances at 260 and 290 nm, respectively; V is the volume of the filtered solution (mL), G is the oven-dry weight of the pulp (g) and 0.287 is the calibration factor [11]. For each pulp, the experiment was performed at least four times and a repeatability of 4.8 mmol/kg pulp was obtained.

RESULTS AND DISCUSSION

The statistical analysis of the data (~ 40 experiments under different conditions) revealed that only 59% of the HexA content variations are explained by pentosan content variations. This fact certainly results from different reaction kinetics concerning HexA formation/degradation as well as xylan degradation/dissolution. Therefore, to minimize the direct effect of xylan dissolution on the evaluation of HexA formation/degradation, it is advisable to determine the HexA content reported to the xylan amount in the pulp (here as mmol HexA/kg pentosan). Whenever

needed, the HexA content on pulp or wood basis is shown as well.

The pulp kappa number is a control parameter commonly used in industrial pulping to describe the degree of delignification. As previously mentioned, this parameter is not only related to the lignin content in the pulp but also includes the contributions of other oxidizable groups, mainly HexA. To assess the contribution of each chemical structure, a statistical treatment of twelve pulps with known kappa number (K), total lignin (TL , % in pulp) and HexA content (mmol/kg pulp) was performed using the STATISTICA software, Statsoft Inc., Tulsa, OK, USA, 1995. The following equation was obtained with a standard error of estimation of 0.7:

$$K = 5.3 TL + 0.09 C_{\text{HexA}} \quad (2)$$

($r^2 = 0.9994$; $F = 10\,000$)

in the range of $7.5 \leq K \leq 69$, $1.2 \leq TL \leq 12$ and $18 \leq C_{\text{HexA}} \leq 61$. Thus, 11 mmol of HexA contribute to one pulp kappa number unit, which is similar to the result obtained by Gellerstedt and Li [6].

In Fig. 1, all data are plotted as a function of the uncorrected pulp kappa number for soda ($S = 0\%$) and kraft pulps produced with different sulphidity ranges (low, medium and high). With respect to the normal range of industrial sulphidities (15–40%), Fig. 1 shows that the HexA content slightly increases as the kappa number decreases, from 22 to ~ 12 , below

which the HexA content decreases rapidly. Similar profiles have been reported by Chai et al. for aspen [7], while for softwoods, the HexA content begins to decrease at a much higher kappa number [3,12]. The results obtained therefore suggest that the HexA degradation/dissolution is higher than the HexA formation only in the final stage of delignification, which does not usually occur in industrial kraft pulping. As shown in Fig. 1, as much as 60 mmol/kg pulp of HexA can be found in an *E. globulus* pulp for the usual range of delignification degrees. However, for pulps produced under extremely unfavourable cooking conditions (0–10% sulphidity), the HexA content always decreases in the range of kappa numbers studied (70–15), even though the maximum value of ~60 mmol/kg pulp is reached as well. Taking into account that the presence of HS⁻ ions increases the delignification rate, lower temperatures and/or alkali charges are needed for higher sulphidity cooks to reach the same kappa number. Under these softer conditions, the HexA is formed probably at a lower rate and its content increases continuously as delignification proceeds, until the HexA degradation/dissolution overcomes its formation at the end of the cook. The hypothesis of a covalent bond between HexA and lignin, suggested by Jiang et al. [13],

could also explain partially why the HexA content decreases mainly at high degrees of delignification. Figure 1 also highlights that pulps with the same kappa number have different HexA contents, depending on the cooking parameters. Thus, their effect on the remaining amount of HexA will be analyzed in a set of pulps with different kappa numbers and in a set of pulps that are delignified equally.

Although the effect of cooking time was not studied fully, the few results obtained (Fig. 2) show that the HexA content of kraft pulps either increases or decreases as cooking time is extended, depending on the cooking conditions. These conditions determine the position of the maximum HexA content in the C_{HexA} -time profile.

The results shown in Tables II–IV were selected to study the effect of temperature, effective alkali (EA) or sulphide charge variations on HexA behaviour, while keeping constant the other cooking parameters. The data of Table II indicate that the effect of cooking temperature on the HexA content after 90 min of cooking time depends on the EA level. For the lower one (EA = 12.8%), an increment in cooking temperature raises the HexA content (because this acid is formed at a higher rate). The opposite is observed for the upper EA values. For these cases, the maximum HexA con-

tent of the C_{HexA} -time profile was reached probably before 90 min of cooking because of, firstly, the higher rate of HexA formation and, secondly, the increase in the HexA degradation/dissolution.

Identical behaviour was found for the influence of EA charge at different temperature levels (Table III). The increment of EA leads only to higher amounts of HexA (due to its faster formation) when the levels of temperature and EA are low (first 5 cooks of Table III). In the other cases, HexA reactions probably occurred at higher rates, causing lower HexA contents for the same cooking time. With regard to the effect of sodium sulphide charge (0–10%, which corresponds to sulphidities between 0 and 60%), small variations in the HexA content that are within the experimental error can be observed in each set of cooks presented in Table IV, except for cook E81. Thus, the effect of HS⁻ ions on the HexA reactions seems to be insignificant, as opposed to previous results in softwood pulping [3].

In agreement with the above results, the statistical analysis of data corresponding to 40 experiments identified temperature and EA as the main factors that influence the HexA content of the pulps, even though the interaction between these variables has the highest effect. It should be mentioned that, for the same

TABLE II
EFFECT OF COOKING TEMPERATURE ON THE HexA CONTENT CALCULATED ON A PENTOSAN BASIS, FOR SETS OF COOKS WITH DIFFERENT LIQUOR COMPOSITION ($t = 90$ min). SEE NOMENCLATURE

Cook Reference		T (°C)	K	C_{HexA} (mmol/kg pentosan)
E96	EA = 12.8%	156	18.2	302
E22	S = 30%	161	14.8	334
E43		166	12.8	333
E86		170	11.6	367
E65	EA = 14.5%	150	22.3	335
E53	S = 30%	156	15.2	355
E46		161	12.9	365
E73		166	11.5	329
E77		170	9.8	286
E54	EA = 17%	152	14.8	364
E48	S = 30%	161	11.4	344
E95		170	8.9	216

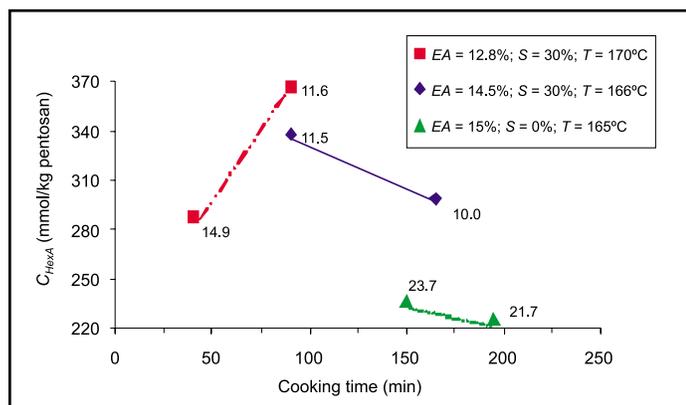


Fig. 2. Effect of cooking time (after the heating period) on HexA content reported to the pentosan content (the figures near the data points are the kappa numbers). See Nomenclature.

TABLE III
EFFECT OF EA ON HexA CONTENT CALCULATED ON A PENTOSAN BASIS ($t = 90$ min) FOR THREE SETS OF COOKS. SEE NOMENCLATURE

Cook Reference		EA (% Na ₂ O)	K	C_{HexA} (mmol/kg pentosan)
E96	S = 30%	12.8	18.2	302
E53	T = 156°C	14.5	15.2	355
E81		12.0	15.8	321
E22	S = 30%	12.8	14.8	334
E46	T = 161°C	14.5	12.9	365
E48		17.0	11.4	344
E98		20.4	9.3	244
E86	S = 30%	12.8	11.6	367
E77	T = 170°C	14.5	9.8	286
E95		17.0	8.9	216
E99		20.0	7.5	155

TABLE IV
EFFECT OF Na₂S CHARGE ON THE HexA CONTENT CALCULATED ON A PENTOSAN BASIS ($t = 90$ min) FOR THREE SETS OF COOKS. SEE NOMENCLATURE

Cook Reference		Na ₂ S Charge (%)	K	C_{HexA} (mmol/kg pentosan)
E81	EA = 12%	4.0	15.8	321
E38	T = 161°C	6.1	14.0	283
E67		10.1	12.7	277
E92	EA = 14.5%	0.0	49.4	349
E93	T = 161°C	1.5	21.9	359
E46		5.1	12.9	365
E58		7.5	12.2	355
E2	EA = 20%	0.0	15.1	136
E99	T = 170°C	7.1	7.5	155

pulps, EA and the interaction between temperature and EA were also the main factors that explained the intrinsic viscosity variations [8, 9]. According to the data of Chai et al. [12] for Loblolly pine, the formation/degradation of HexA does not depend on sulphidity being related to the amount of alkali consumed.

The results obtained in the present study show this interesting dependence for three temperatures in Fig. 3. As in the work of Chai et al. [12], these data fall on a single curve despite the fact that the pulps were produced under very different conditions. In fact, 87% of the C_{HexA} variations are explained by the variations of the EA consumed. As known, this consumption is due to mainly the neutralization of the degradation products of polysaccharides (cellulose and hemicelluloses) and extractives. Moreover, a minimum amount of EA has to be consumed to produce pulps suitable for industrial applications (~27 g Na₂O/L for the Portuguese *E. globulus*). On the right side of the curve (Fig. 3), the increase of EA consumption is a result of higher polysaccharides degradation/dissolution (including HexA reactions) due to high temperatures and/or high EA charges. Furthermore, lower yields and pulps with poorer properties are obtained [8]. The data on the left side of the curve correspond to very high sulphidity cooks, which are analyzed next.

When the objective is to achieve a target kappa number using different cooking conditions, at least two variables have to be changed. In the case of Portuguese *E. globulus*, an increase in temperature counterbalanced by a decrease in cooking time (and keeping the H-factor constant), leads to a slight decrease in both yield and pentosan content but to a high reduction in pulp viscosity [9,10]. The remaining HexA amount is also lower, as shown in Table V (either on a wood, pulp or pentosan basis). However, while the pentosan content diminishes ~3%, HexA content decreases 17%. As the HexA formation increases with temperature, as discussed earlier (Table II, cooks E22 and E86), the reduced cooking time (from 90 min in cook E86 to 40 min in cook E78) leads to a lower HexA amount (although this acid is

formed at a higher rate than in cook E22), i.e. the cooking was stopped at the increasing side of the C_{HexA} -time profile.

If, in turn, a higher temperature is compensated for with a lower EA, the yield and pentosan content increase without a significant difference in pulp viscosity [9,10]. In addition, the HexA content is not significantly affected, except for soda cooks (Table VI). In the latter cases, the effect of a temperature increase was not counterbalanced by the effect of an EA decrease, and the lowest HexA content was obtained for the pulp produced with the highest EA level. When the sulphidity is modified, both the EA and Na₂S charge could be changed with or without a simultaneous variation in temperature to reach a target kappa number. In all these cases, a sulphidity increase allows a decrease in the other parameters, resulting in higher yield, pentosan content and pulping selectivity (higher viscosity at the same kappa number) [8–10]. The corresponding HexA content is

shown in Table VII. The highest values were obtained for the sulphidity range close to industrial practice (15–30%). The lowest ones were achieved for pulps produced using either very low or very high sulphidity liquors. In the first case, the high temperatures and/or high EA charges that are needed increase the HexA formation rate as well as its degradation, resulting in lower HexA amounts for the same cooking time. On the other hand, very high sulphidities also lead to lower HexA contents because the wood is cooked under soft conditions (low EA), thereby resulting in slow HexA reactions. However, the latter conditions intensify the smell problems, increase the amount of chemicals circulated in the system and reduce the optical properties of the pulps. Tables V–VII, as well as Fig. 1, also depict that in the industrial range of sulphidities used for kraft pulping, the HexA content is high and its variation with pulping conditions is quite small. Thus, if the objective is to remove the HexA before the

TABLE V
EFFECT OF TEMPERATURE ON HexA CONTENT FOR PULPS EQUALLY DELIGNIFIED ($K \sim 15$, $S = 30\%$, $EA = 12.8\%$). SEE NOMENCLATURE

Cook Reference	T (°C)	t (min)	TY (%)	PENT (%)	C_{HexA} (mmol/kg pulp)	C_{HexA} (mmol/kg pentosan)	C_{HexA} (mmol/kg wood)
E22	161	90	53.5	16.4	54.8	334	29.3
E78	170	40	53.2	15.9	45.7	287	24.3

TABLE VI
HexA CONTENT OF PULPS EQUALLY DELIGNIFIED ($K \sim 15$): EA AND T WERE MANIPULATED IN OPPOSITE DIRECTIONS ($t = 90$ min). SEE NOMENCLATURE

Cook Reference	EA (% Na ₂ SO)	S (%)	T (°C)	C_{HexA} (mmol/kg pentosan)
E22	12.8	30	161	334
E53	14.5		156	351
E54	17.0		152	365
E64	15.1	0	181	198
E2	20.0		170	135

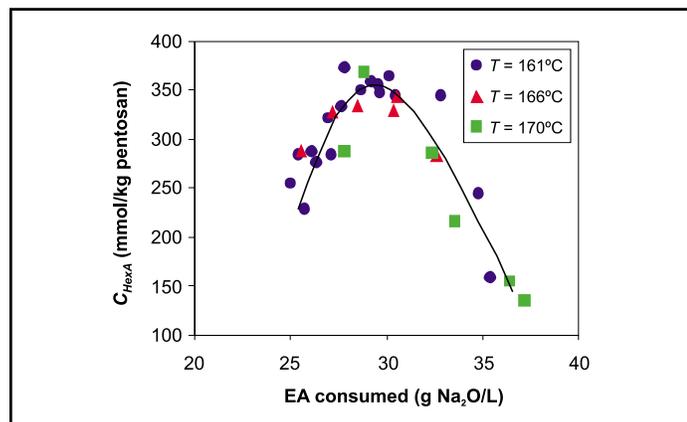


Fig. 3. Effect of effective alkali consumed on HexA content of three sets of pulps obtained at different cooking temperatures (161, 166 and 170°C). Pulps of each set were produced using liquors with very different sulphidities and EA charges ($S = 0$ –60%, $EA = 11$ –23%, $t = 90$ min).

TABLE VII
HexA CONTENT OF PULPS EQUALLY DELIGNIFIED ($K \sim 15$): EA AND S OR T AND S (BY KEEPING ACTIVE ALKALI CONSTANT) WERE MANIPULATED IN OPPOSITE DIRECTIONS; ($t = 90$ min). SEE NOMENCLATURE

Cook Reference	EA (% Na ₂ SO)	S (%)	T (°C)	C_{HexA} (mmol/kg pentosan)
E68	15.5	18		345
E22	12.8	30		334
E88	11.2	40	161	284
E85	10.9	45		256
E44	11.3	50		229
Active Alkali = 15%				
E64	15.1	0	181	198
E8	14.4	10	165	303
E18	13.5	20	163.5	349
E22	12.8	30	161	334

bleaching process, it would be advisable to take measures on the unbleached pulp instead of changing the cooking conditions.

CONCLUSIONS

In this study, the HexA content of *E. globulus* kraft pulp was calculated not only on a pulp or wood basis but also relative to the pentosan content to minimize the effect of xylan dissolution on the HexA removal. The amount of this acid has shown a close dependency on the extension of the delignification process and, unlike softwood pulping, only decreased at kappa number below 12, which is not achieved usually in industrial practice. The study provided data on the contribution of both lignin and HexA to pulp kappa number. The HexA content of kraft pulps was found to depend on the cooking conditions that determine the position of the maximum in the C_{HexA} -time profile. EA and temperature were shown to be the main factors affecting the amount of HexA in pulps, while the effect of sodium sulphide charge seems to be insignificant, as opposed to findings in other works, and should be studied further. In addition, a good correlation of the data with the consumption of EA, regardless of the cooking conditions, was observed. For the ranges of variables and target kappa numbers where the pulping selectivity is high, the HexA amount in the pulps is also quite high (45–60 mmol/kg pulp) and its variation with pulping conditions is quite small. Lower values were obtained by using soda and very low or very high sulphidity liquors that reduce the pulp quality or increase process problems. These findings indicate that the HexA content in pulp is a complex result of the cooking parameters, which, in turn, not only affect the delignification rate but also other pulp characteristics that are relevant for the final product behaviour.

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NOMENCLATURE

ACT activity ($=100 \times (\text{NaOH} + \text{Na}_2\text{S})/(\text{NaOH} + \text{Na}_2\text{S} + \text{Na}_2\text{CO}_3)$) (%)

EA effective alkali charge ($= 100 \times (\text{NaOH} + \frac{1}{2}\text{Na}_2\text{S})/W$) (%; g $\text{Na}_2\text{O}/\text{o.d.}$ wood weight in grams)
 C_{HexA} hexenuronic acid content (mmol/kg pulp) or (mmol/kg pentosan) or (mmol/kg wood)
 K pulp kappa number
 Na_2S charge $100 \times \text{Na}_2\text{S}/W$ (%; g $\text{Na}_2\text{O}/\text{o.d.}$ wood weight in grams)
 PENT pentosan content (% pulp basis)
 PY pulp yield (%; kg of screened pulp/kg wood)
 REJ rejects yield (%; kg/kg wood)
 S sulphidity ($= 100 \times (\text{Na}_2\text{S})/(\text{NaOH} + \text{Na}_2\text{S})$) (%)
 T temperature ($^{\circ}\text{C}$)
 t time (min)
 TL total lignin (insoluble plus soluble, % in pulp)
 TY total yield (%; kg of unscreened pulp/kg wood)
 VISC intrinsic viscosity (dm^3/kg)
 W Weight of wood (g)

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