CHLORINE DIOXIDE BLEACHING OF PULP FROM CROP RESIDUES: BAGASSE, KASH AND CORN STALKS

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This investigation describes the effect of hot chlorine dioxide delignification (D_{HT}) of bagasse, kash and corn stalk pulps on pulp properties and effluent quality. The pulps were subjected to D_{HT} at 85 °C for 45 min and the results were compared with those of the D_{0} process carried out at 70 °C for 45 min. The kappa numbers after the alkaline extraction (Ep) stage in D_{HT} bleaching were always lower and brightness was higher, compared to the corresponding parameters in D_{0} bleaching, without impacting pulp viscosity. The final brightness of the corn stalk pulp was 84.8% at a kappa factor of 0.25 in the D_{0} process, while in the D_{HT} process, the same type of pulp reached the brightness of 87.2% at a kappa factor of 0.15, saving 40% ClO_2 in the first stage. Similarly, kash pulp exhibited 90% brightness at a kappa factor of 0.15, which also saved 40% ClO_2, compared to the conventional D_{0} process. The brightness of bagasse pulp in D_{HT} and D_{0} processes was almost similar. Oxygen delignified pulp had a lower effluent discharge than unbleached pulps in subsequent ECF bleaching. The COD value in D_{HT} was lower than that in D_{0}.

Keywords: bagasse, kash, corn stalk, high temperature ClO_2 bleaching, Kappa number, brightness, COD

INTRODUCTION

Nowadays, elemental chlorine free (ECF) bleaching is the dominant bleaching technology used for chemical pulp.1 Chlorine dioxide (ClO_2) is a specific oxidizing agent for lignin removal and provides good quality pulp of high brightness, without carbohydrates degradation.2 ClO_2 has a higher oxidizing potential when compared to Cl_2. Molecular chlorine generates substantially more chlorinated organics, particularly aromatics, via electrophilic aromatic substitution reactions, whereas ClO_2 reacts via radicals and mainly oxidation, not electrophilic aromatic substitution. This accounts for the reduction in chlorinated aromatics and lower environmental impact of the effluent.3,5 However, ClO_2 is still enough to pollute the environment. Therefore, many attempts have been made to reduce ClO_2 consumption.6-8 Studies showed that xylanase, oxygen, peracid or acid treatments improved subsequent ClO_2 bleaching. Splitting the ClO_2 charge of DED sequences into DEDED sequences increased brightness and reduced the effluent load.6-9 Hot acid treatment and D_{HT} bleaching have been thoroughly investigated by Ragnar and Lindström, who showed that D_{HT} achieved superior results for many of the investigated parameters.10 The delignification efficiency of ClO_2 in the D_0 stage depends on the reaction time and reaction temperature, while reducing the effluent load of bleaching chemicals and also improving the bleached pulp properties.8,11 Introducing hot chlorine dioxide (D_{HT}) in ECF bleaching reduced the ClO_2 consumption and had a positive impact on bleached pulp properties. Ventorim et al. revealed that a partial acid hydrolysis of hexeneuronic acid (HexA) occurred in D_{HT} treatment, resulting in decreased generation of chlorinated organic halides, which is not a possible reaction in D_{0} with an increase in time and temperature.12 Ventorim et al. also found that at a similar ClO_2 dose (kappa factor 0.20 at pH 3.0) in bleaching produced 46.3% and 9% less AOX and OX in the filtrate and pulp, respectively, in relation to D_{0}.12

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Brogdon in his review showed that hot A-stages, when applied ahead of ECF bleaching, can reduce the amount of HexA entering into the bleach plant by 30 to 90% and some lignin was removed as well. Most applications of hot A-stages are combined with D₂ into a single operation, (A/D₂) or “hot D₂-Stage.” These processes can be very effective in decreasing the amount of ClO₂ required for ECF bleaching of hardwood pulps (20 to 50%).

Buchert et al. showed that, when bleaching with chlorine dioxide, the HexA side groups of pulp xylan were degraded, whereas the MeGlcA and arabinose side groups were relatively stable toward the bleaching chemicals. The hydrolysis of HexA occurs rapidly at high temperature and low pH, and it can be removed easily from the pulp. This lowers the consumption of chemicals during bleaching, leading to lower generation of chlorolignin compounds. The studies revealed that the D₂₇₅ stage is able to dissociate the phenolic lignin and HexA, and to limit the generation of organochlorides. Much research has been carried out on D₂₇₅ in the initial stage of bleaching of hardwood pulp, but the literature available on non-wood/agricultural residues pulps is scarce.

Most of the studies used a temperature of 95 °C for longer time, and consequently obtained reduced viscosity and a slight drop in final pulp brightness. The lower brightness obtained after D₂₇₅/E is explained by the brightness reversion reactions caused by maintaining the pulp at high temperature/time in the complete absence of chlorine dioxide (Eiras et al.). The hot acid treatment also formed new lignin phenolic hydroxyl groups, which may form new chromophores. Therefore, the purpose of the present study was to use ClO₂ treatment at higher than conventional temperature (70 °C and lower than 95 °C, in order to reduce ClO₂ consumption and effluent load, while achieving improved brightness.

In this study, D₂₇₅ bleaching of crop residue pulps obtained from bagasse, kash and corn stalks was carried out at 85 °C for a short time, and the results were compared with those of D₁ bleaching. The final pulp properties and the effluent quality were also studied.

**EXPERIMENTAL**

**Materials**

Pulps from bagasse, kash and corn stalks were prepared in the laboratory by the soda-AQ process. The cooking experiment was conducted in an electrically heated, thermostatically controlled digester of 20 litre capacity. 1 kg of crop residues was used for all the experiments. All three types of crop residues were cooked with 18% alkali charge and 0.1% AQ for 2 h at 150 °C. The material to liquor ratio was 1:6.

**Oxygen delignification**

Oxygen delignification (OD) was carried out in a thermostatically controlled digester, rotating at 1 rpm. The OD conditions were as follows: 110 °C, retention time 60 min, pulp consistency 10%, NaOH 2%, MgSO₄ 0.3% and O₂-pressure 3.5 kg.cm⁻².

**Evaluation of pulps**

The kappa number, viscosity, brightness and HexA of the resulting pulps in unbleached and oxygen delignified state were determined in accordance with Tappi Test Methods, namely, kappa number (T 236 om-99), pulp viscosity (T 230 om-99), pulp ISO brightness (T 452 om-92) and hexenuronic acid content (T282 pm-07). Three replicates of all the experiments were performed, and the average reading was taken.

**D₀(Eₚ)D₁ and D₂₇₅(Eₚ)D₁ bleaching**

Unbleached and oxygen delignified pulps were bleached by D₀(Eₚ)D₁ and D₂₇₅(Eₚ)D₁ bleaching sequences (where D represents chlorine dioxide and (Ep) represents peroxide reinforced alkaline extraction). The chlorine dioxide charge in the D₁ and D₂₇₅ stages was varied by kappa factor of 0.15, 0.2 and 0.25. The temperature was 70 °C in the D₁ stage for 45 min. Pulp consistency was 10%. The pH was adjusted to 2.5 by adding dilute H₂SO₄. In the D₂₇₅ stage, the bleaching temperature was 85 °C and all the other parameters remained the same. The alkaline extraction stage was performed under the following conditions: temperature of 70 °C for 60 min, a water solution of 2% NaOH and 0.5% H₂O₂ (on o.d. pulp). After the (Ep) stage, kappa number, viscosity and brightness were determined in accordance with Tappi Test Methods, as mentioned above.

In the D₁ stage, the pH was adjusted to obtain a final pH of 4.5. The ClO₂ charge in the D₁ stage was fixed to 1%. The brightness and viscosity of the bleached pulp, as well as COD of the mixed effluent collected from the D₀, Eₚ and D₁ stages, were determined in accordance with PAPTAC Methods H.3.

**RESULTS AND DISCUSSION**

Table 1 shows the basic properties of the starting pulps obtained from bagasse, kash and corn stalks. Oxygen delignification reduced the kappa number by 40.7%, 51.3% and 31.6% and increased the brightness by 24.59%, 30.33% and 7.2% ISO points for bagasse, kash and corn stalk
pulps, respectively. However, the pulp viscosities were not changed significantly.

**D_{HT}(Ep) and D_{0}(Ep) pulp properties**

The effect of kappa factor on residual ClO₂, (Ep) kappa number, brightness and viscosity are shown in Table 2. As shown in Table 2, the residual ClO₂ in conventional D₀ was found to be of 66-158 ppm for bagasse, 60-100 ppm for kash and 54-82 ppm for corn stalk unbleached pulps, which decreased significantly in the D_{HT} treatment. The residual ClO₂ was found to be lower than for the unbleached pulp and nil for the oxygen delignified kash pulp after D_{HT} bleaching, where a lower dose of ClO₂ was used. At higher temperature in D_{HT}, most of the ClO₂ was consumed during bleaching and its effect was also reflected in the kappa number measurement after the (Ep) stage.

As expected, an increasing kappa factor resulted in a decreased kappa number and viscosity, as well as increased brightness, in both D₀ and D_{HT} processes. D_{HT} bleaching is more efficient than D₀ and supported a reduction of the chlorine dioxide dose during bleaching (Table 2). The kappa number after the (Ep) stage decreased with increasing kappa factor and oxygen delignification. The kappa numbers after the (Ep) stage in D_{HT} bleaching were always lower than in D₀ bleaching. This is related to the acid leaching of lignin, as described and reported in earlier studies. For pulp bleaching at a kappa factor of 0.2, the (Ep) kappa numbers were 2.4 and 1.4 for bagasse, 1.2 and 0.9 for kash and 2.5 and 2.1 for corn stalks, for unbleached and oxygen delignified pulps, respectively, which further decreased upon D_{HT} to 1.4 and 0.7 for bagasse, 0.9 and 0.8 for kash and 1.8 and 1.6 for corn stalks. The higher kappa number decrease in the D_{HT}(Ep) stage, as compared to the D₀(Ep) one, can be explained by the higher removal of HexA. This can also be explained by the degradation of hemicelluloses during the D_{HT} treatment, which breaks down the bonds of the lignin-carbohydrate complex (LCC) and increases the extraction of the lignin from the surface of the fiber, as described by Zhang and coworkers. On the other hand, according to Ikeda et al., the temperatures are not high enough to cleave the C-O or C-C bonds that link lignin to carbohydrates in LCC. McDonough and co-workers speculated that the proportion of HexA contributing to the pulp’s kappa number entering the D₀ stage affected the bleachability of a red oak pulp that underwent hot acid hydrolysis.

Pulp brightness after the D₀(Ep)/D_{HT}(Ep) stage is also shown in Table 2 and Figure 1 (a, b and c). Oxygen delignified pulp showed better pulp brightness than the unbleached pulp, regardless of D₀ or D_{HT} bleaching. The brightness of D₀ treated pulps was better than that of the pulps treated by the D_{HT} process using an identical amount of chlorine dioxide (Fig. 1). As shown in Figure 1, the higher brightness advantage in the D_{HT} stage than in the D₀ stage after the (Ep) stage was of 3% for corn stalks and kash unbleached pulps. Ventorim et al. found the first chlorine dioxide stage at high temperature (D_{HT}) decreased the brightness by 2.5% ISO and kappa number by 46% (1.9 units) in the extraction stage, as compared to the conventional D stage. Their results were also in agreement with other findings. The target of this study was to improve brightness and kappa number reduction at 85 °C for shorter bleaching time, which was achieved.

As shown in Table 2, oxygen delignification reduced pulp viscosity significantly. There was no significant change in post-extraction pulp viscosity after D_{HT} bleaching. However, other studies showed that the D_{HT}E treatment at 95 °C resulted in a significant drop in post-extraction pulp viscosity, as compared to the DE one. A slight carbohydrate hydrolysis may occur when the pulp is exposed to high time/temperature reaction and acidic pH. The significant viscosity loss is related to the hot acid hydrolysis of the carbohydrates at 2.5 pH.
### Table 1

Pulp properties of unbleached and oxygen delignified pulps from bagasse, kash and corn stalks

| Pulp          | Kappa number | Viscosity (mPa.s) | HexA (µmole/g) | Brightness (%) |
|--------------|--------------|------------------|----------------|---------------|
|              | Unbleached   | O₂-delignified   | Unbleached     | O₂-delignified | Unbleached   | O₂-delignified |
| Bagasse      | 14.3         | 8.5              | 15.1           | 15.5          | 25.9         | 26.1            | 27.9            | 52.5          |
| Kash         | 14.8         | 7.2              | 13.9           | 12.2          | 21.2         | 22.7            | 21.1            | 51.4          |
| Corn stalks  | 10.0         | 8.2              | 33.6           | 32.3          | 31.2         | 30.5            | 21.4            | 28.6          |

### Table 2

Effect of D₇₅₀ and D₀ bleaching on pulp properties after (Ep) stage

| Pulp          | Temp. | Kappa factor | Residual ClO₂ (ppm) | Kappa No | Brightness (%) | Viscosity (mPa.s) |
|--------------|-------|--------------|---------------------|----------|----------------|-------------------|
|              |       |              | UB                  | OB       | UB             | OB                |
| Bagasse      | 70    | 0.15         | 66                  | 55       | 69.43          | 70.31             | 18.4             | 16.57         |
|              |       | 0.2          | 105                 | 68       | 70.76          | 72.51             | 18.5             | 16.49         |
|              |       | 0.25         | 158                 | 73       | 71.00          | 72.96             | 18.9             | 16.12         |
|              | 85    | 0.15         | 67                  | 50       | 70.10          | 72.41             | 18.7             | 14.50         |
|              |       | 0.2          | 67                  | 61       | 73.92          | 73.28             | 17.8             | 15.24         |
|              |       | 0.25         | 68                  | 62       | 76.85          | 75.51             | 16.5             | 10.79         |
| Kash         | 70    | 0.15         | 100                 | 54       | 66.0           | 84.20             | 14.4             | 11.07         |
|              |       | 0.2          | 110                 | 60       | 82.0           | 85.35             | 15.4             | 12.74         |
|              |       | 0.25         | 112                 | 64       | 84.38          | 85.5              | 13.5             | 13.05         |
|              | 85    | 0.15         | 65                  | nil      | 81.09          | 83.10             | 14.4             | 10.8          |
|              |       | 0.2          | 67                  | nil      | 84.48          | 85.15             | 14.6             | 10.2          |
|              |       | 0.25         | 82                  | nil      | 85.02          | 86.2              | 13.9             | 10.7          |
| Corn stalks  | 70    | 0.15         | 54                  | 52       | 66.9           | 83.4              | 22.8             | 18.7          |
|              |       | 0.2          | 74                  | 62       | 82.29          | 85.2              | 22.6             | 18.2          |
|              |       | 0.25         | 82                  | 74       | 83.6           | 85.52             | 23.0             | 18.4          |
|              | 85    | 0.15         | 45                  | 34       | 76.03          | 80.58             | 22.5             | 18.6          |
|              |       | 0.2          | 64                  | 57       | 83.1           | 83.4              | 22.1             | 18.2          |
|              |       | 0.25         | 74                  | 64       | 84.2           | 86.75             | 21.4             | 18.0          |
D$_{HT}$(Ep)$D_1$ and $D_0$(Ep)$D_1$ pulp properties

The effect of the $D_{HT}$ on the residual HexA was also studied and shown Figure 2. The residual HexA content in $D_{HT}$ bleached pulps was much lower than in the $D_0$ pulps. At a kappa factor of 0.2, the residual HexA contents were 4.25, 2.93 and 6.21 µmol/g for the $D_0$ pulps, and 2.89, 1.21 and 3.23 µmol/g for the $D_{HT}$ pulps from bagasse, kash and corn stalks, respectively. This is explained by the degradation of HexA in the $D_{HT}$ process. HexA could not react with chlorine dioxide, but reacted with its intermediates, such as hypochlorous acid and molecular chlorine, therefore increased chlorine dioxide consumption and affected pulp bleachability.

As shown in Figure 3, the COD value of the combined effluent from the $D_0$, Ep and $D_1$ stages in $D_{HT}$(Ep)$D_1$ bleaching was lower than that of the effluent from $D_0$(Ep)$D_1$. Oxygen delignified pulp always exhibited a lower COD load in the effluent. The $D_{HT}$ process at a kappa factor of 0.2 decreased the COD value from 1120 mg/L to 1060 mg/L and from 1040 mg/L to 760 mg/L for bagasse pulp, from 1044 mg/L to 860 mg/L and from 828 mg/L to 700 mg/L for kash pulp, and from 1270 mg/L to 1011 mg/L and from 717 mg/L to 558 mg/L for corn stalk pulp, for unbleached and oxygen delignified pulps, respectively. Similar COD value reductions were reported in other studies.

Although pulp brightness after the (Ep) stage was higher in oxygen delignified pulp, the final pulp brightness of unbleached and oxygen delignified pulps was almost similar. Also, the final brightness of the pulp that underwent the $D_{HT}$ process was better or similar to that of the pulp subjected to conventional $D_0$. 
Table 3
Effect of D_{HT} and D_{0} bleaching on final pulp properties

| Pulp      | Temp. | Kappa factor | Brightness (%) | Viscosity (mPa.s) |
|-----------|-------|--------------|----------------|-------------------|
|           |       |              | UB  | OD  | UB  | OD  |       |
| Bagasse   | 70    | 0.15         | 87.2| 86.0| 14.85| 13.0|       |
|           |       | 0.2          | 89.1| 87.1| 14.2 | 12.5|       |
|           |       | 0.25         | 89.3| 89.1| 13.12| 11.4|       |
|           | 85    | 0.15         | 85.6| 85.0| 12.7 | 12.6|       |
|           |       | 0.2          | 89.3| 88.6| 12.5 | 11.1|       |
|           |       | 0.25         | 89.2| 88.3| 12.1 | 11.5|       |
| Kash      | 70    | 0.15         | 83.0| 87.0| 14.79| 10.45|       |
|           |       | 0.2          | 88.6| 90.4| 14.2 | 10.49|       |
|           |       | 0.25         | 89.0| 90.2| 14.24| 13.23|       |
|           | 85    | 0.15         | 90.0| 89.6| 11.03| 10.5 |       |
|           |       | 0.2          | 90.2| 90.0| 10.8 | 10.2 |       |
|           |       | 0.25         | 90.5| 90.9| 10.5 | 10.0 |       |
| Corn stalks | 70    | 0.15         | 83.5| 83.8| 15.3 | 14.5 |       |
|           |       | 0.2          | 83.7| 83.7| 15.5 | 14.8 |       |
|           |       | 0.25         | 84.8| 84.7| 15.2 | 14.2 |       |
|           | 85    | 0.15         | 87.2| 87.8| 13.7 | 12.5 |       |
|           |       | 0.2          | 87.1| 87.9| 13.5 | 12.7 |       |
|           |       | 0.25         | 87.4| 87.7| 13.5 | 12.4 |       |

Figure 3: Effect of D_{HT} and D_{0} bleaching on COD value after final stage

Unbleached corn stalk pulp produced a final brightness of 84.8% at a kappa factor of 0.25 in the D_{0} process, while in the D_{HT} process, it achieved a brightness of 87.2% at a kappa factor of 0.15, and saved 40% ClO_{2} in the first stage. Similarly, kash pulp exhibited 90% brightness at a kappa factor of 0.15, and also saved 40% ClO_{2}, compared to the conventional D_{0} process. The brightness of bagasse pulp in D_{HT} and D_{0} processes was almost similar, which has been one of our targets in this study. Kumar et al.\textsuperscript{11} showed that the use of D_{HT} reduced by 15% the ClO_{2} requirement and improved pulp brightness and brightness stability, in comparison with the reference sequences D_{0}. In another study, it was observed that the final pulp brightness was directly correlated with the percentage of HexA removal during D_{HT}.\textsuperscript{33}

CONCLUSION

Bagasse, kash and corn stalk pulps were bleached by hot chlorine dioxide bleaching. In the first stage, bleaching at 85 °C for 45 min (D_{HT}) produced pulp with a lower kappa number, higher brightness and similar viscosity after the (Ep) stage, compared to the same parameters achieved after the conventional D_{0} stage. The final pulp brightness reached 87.2% for corn stalk pulp and 90.0% for kash pulp, i.e. higher than the values obtained in D_{0} bleaching, while using 40% less ClO_{2}. The COD values and residual HexA content in the D_{HT} bleached pulps were much lower than those of the D_{0} pulps. Therefore, D_{HT} bleaching at 85 °C for 45 min improved the brightness of bagasse, kash and corn stalk pulps, while diminishing the effluent load.
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