# Evaluation of Hemp Root Bast as a New Material for Papermaking

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Industrial hemp is a non-wood fiber material that can be used for papermaking due to a high yield of hemp raw material and high-quality physical properties of its pulp. Hemp is already used as a raw material for papermaking and other industrial productions. The chemical composition of hemp root bast (HRB) was analyzed. HRBs were pulped using a soda-anthraquinone (Soda-AQ) process and bleached with oxygen (O), chlorine dioxide (D), and alkali (E) using the bleaching sequence $OD_0ED_1$. The results showed that HRB can be a suitable raw material for papermaking; the HRB pulp had a high viscosity (893 mL $g^{-1}$) and brightness (85.52% ISO-brightness).

*Keywords: Hemp root bast (HRB); Soda-AQ; Oxygen delignification; Viscosity; Bleaching*

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## INTRODUCTION

As production in the paper industry has increased, stricter environmental protection regulations have been implemented. The area of forest per capita is also decreasing, with the corresponding difficulties in obtaining sufficient amounts of the traditional raw material (*i.e.*, wood) to be used in papermaking in the future. As a consequence, research aiming to improve the yields of high-yield chemically pulped fiber products have focused on improving the selectivity of wood delignification and stability of polysaccharides and bleaching of high-kappa number pulps, which has led to the development of technology involving the use of polysulfides and anthraquinone, solutions that have been shown to facilitate more efficient wood processing (Cöpür 2007; Danielewicz and Surma-Ślusarska 2010). However, the presence of polysulfides and anthraquinone in the cooking stage could lead to a sharp decrease in the physical properties and bleachability (Wang *et al*. 2004). A relatively low increase in the pulp yield obtained from wood using the above methods has yet to solve the problem of a possible shortage of wood material for papermaking in the future.

Investigations searching for additional types of fibers for papermaking have focused on the development of quick-growing plantations, an increased degree of waste paper recycling, and new materials. As for the new materials, industrial hemp has a certain potential (Kane 2000), as the pulping yield of industrial hemp has been shown to be 5% higher than that of birch pulp and 14% higher than that of pine pulp (Danielewicz and Surma-Ślusarska 2006).

Industrial hemp is an annual plant generally cultivated for use in the textile industry (Sedelnik 2004; Cierpucha *et al*. 2004). Worldwide, industrial hemp is defined as plants of the *Cannabis sativa* species with less than 0.2% content of tetrahydrocannabinols (psychoactive substance), which makes such plants unsuitable for narcotic purposes. Some researchers have used different parts of industrial hemp (hemp core and hemp bast fibers) for papermaking (He and Liu 2010, 2011; Barbera *et al*. 2011; Danielewicz and Surma-Ślusarska 2010). Industrial hemp has the advantages of easy preparation, uniform material quality, fewer impurities, easy liquid penetration, easy cooking, and high-quality pulping performance (He and Liu 2011). The industrial hemp fiber is very long and closely linked; almost all relevant studies have used soda-AQ cooking methods (He and Liu 2010, 2011; Zhou 2012).

Many studies of industrial hemp pulp bleaching have been reported. He and Liu (2011) bleached a hemp core pulp obtained from soda-AQ cooking; a final ISO brightness of 86.1% was achieved using the $OD_1ED_2$ bleaching sequence. Danielewicz and Surma-Ślusarska (2011) used the bleaching sequence $OD_0EpD_1$ to bleach the kraft pulps of hemp wood and hemp core, with the brightness reaching 84% to 91% on the ISO scale; the final viscosity was 890 to 980 mL $g^{-1}$ (Danielewicz and Surma-Ślusarska 2011; Mehmet *et al*. 2010).

In this work, HRB was used as an alternative raw material for papermaking. Following the determination of the contents of hemicellulose, lignin, ethanol-benzene extractives, and ash, hemp root bast was pulped with classic reagents including NaOH and anthraquinone. The unbleached and bleached pulps were analyzed for yield, viscosity, Kappa number, and brightness. The objectives of the present work were as follows: to evaluate the potential of HRB as a raw material for papermaking and to find suitable conditions for cooking, delignification, and bleaching to obtain a suitable brightness (above 82% ISO-brightness), high viscosity (700 to 900 mL $g^{-1}$), and low Kappa number (below 5).

## EXPERIMENTAL

### Materials

The HRB was purchased from Yun Nan Province, China. The HRB was the bast from the hemp root above the soil. The average length of HRB fiber was 10.8 mm, the average diameter of HRB fiber was 27.6 μm. The chemical compositions of HRB are listed in Table 1.

**Table 1.** Main Chemical Compositions of the HRB unit: %

| α-cellulose | Pentosans | Klason Lignin | Acid-soluble Lignin | Ash | Alcohol-benzene extractives | 1% NaOH solubility |
|---|---|---|---|---|---|---|
| 46.60 | 7.52 | 16.01 | 1.65 | 6.52 | 5.43 | 40.25 |

**Fig. 1.** Raw material of HRB

## Methods

### Pulping

Pulping of the HRB was carried out in a 15-L Santasalo-Sohlberg rotary digester. The active alkali charge, respectively, was 10%, 12%, 14%, and 16% (on the basis of oven-dried HRB). For cooking, the liquor-to-HRB ratio was 5:1 for the HRB. The temperature was raised to 170 °C within 90 min and held at 170 °C for 150 min in all of the cooking tests. After cooking, the pulp was washed first on a wire mesh and then by diffusion with deionized water for 24 h and finally disintegrated. The pulp was centrifuged, after its dryness had been determined, and then the pulp was stored in closed polyethylene bags under wet conditions in a refrigerator.

### Oxygen pulp delignification

HRB pulp delignified with oxygen in alkaline media was considered in this work. A Jayme reactor was used to delignify pulps using this method. All the experiments were carried out at the pulp consistency of 15% and oxygen pressure of 0.6 MPa at 110 °C. At the beginning of each experiment, a 0.5% charge (on the basis of oven-dried HRB pulp) of $MgSO_4$ was first dissolved in 50 mL of water and mixed with the pulp; then, a 3% charge (on the basis of oven-dried pulp) of NaOH was added with the rest of the water. The experiment of the oxygen delignification on alkali charge also used 2%, 4%, and 5%. The pulp was mixed with chemicals and placed in the reactor, and oxygen pressure was applied. The HRB pulp delignified with oxygen in alkaline media was in 45 min, 60 min, 75 min, and 90 min. Finally, the experiment used 60 min for the bleaching. After bleaching, the oxygen pressure was released, the reactor was opened, and the pulp was discharged, filtered, washed with deionized water, and placed into a plastic bag for further treatment.

### Chlorine dioxide bleaching

A water bath was used for the chlorine dioxide bleaching and alkali extraction process. First, a total of 100 g (oven-dried pulp) of disintegrated pulp was placed in a double polyethylene bag with deionized water. After mixing the pulp thoroughly, charges of 1.5%, 2.0%, 2.5%, and 3.0% $ClO_2$ were used for the bleaching. Finally, the experiment used a charge of 2.5% $ClO_2$ for the bleaching. The chlorine dioxide bleaching ($D_0$) stage was carried out at a pulp consistency of 6%, and the pH of the pulp suspension was adjusted to 3.5. After 90 s of further mixing, the pulp was placed into a water bath with a constant temperature of 70 °C. The bag was mixed every 15 min. After 150 min of bleaching in the $D_0$ stage, the pulp was then washed to a pH of 7 and subsequently placed in a new plastic bag for the next treatment.

After the $D_0$ stage, the pulp was subjected to an alkali extraction process (E stage). Deionized water and a 3% NaOH charge were added to the pulp suspensions at a 10% consistency. After kneading, the bag was placed into a water bath (60 °C) and mixed every 10 min. After 60 min of treatment, the pulp was washed using the same method as described for the $D_0$ stage.

The second chlorine dioxide bleaching stage ($D_1$ stage) was conducted using bleach liquor consisting of 0.4%, 0.6% , 0.8%, 1.0%, and 1.2% chlorine dioxide (on the basis of oven-dried pulp) at a pH of 3. In the $D_1$ stage, the experiment used a charge of 0.6% $ClO_{2.}$ Each pulp sample (20 g of oven-dried pulp) and accompanying bleaching chemicals were mixed with deionized water in a polyethylene bag to obtain a final pulp consistency of 10% before being immersed in a water bath of 70 °C, in which they were held for 150 min and mixed every 15 min by hand. At the end of the treatment, the pulps were washed as described previously.

*Analytical methods*

Standard methods were used to measure both the kappa number of pulps, with ISO 302-2004, and the intrinsic viscosity of pulps, with ISO 5351-2010. The pulp brightness (R457 nm) was determined according to the ISO 2470 standard using an ELREPHO device.

## RESULTS AND DISCUSSION

### Raw Material Analysis

The main chemical composition of HRB is shown in Table 1. The high α-cellulose (46.60%) and low lignin (17.66%) contents of HRB suggest that a high yield and viscosity should be achievable after pulping and bleaching. The chemical compositions and residual lignin in lignocellulosic materials greatly influence both pulping yield and lignin removal rate, which are important issues for papermaking. Lower Klason lignin (16.01%) content would suggest a decrease in the requirement for alkali and the feasibility of using gentle cooking conditions.

The alcohol-benzene extractives (5.43%) and other extractives show the presence of many pectin, fatty oil, and pigment. The abundance of plant pigment would affect the results of subsequent bleaching operations.

The HRB was close to the soil and could accumulate various metal ions. The ash content of HRB was higher than those of agricultural residues. The iron ion content (255.8 ppm) and the manganese ion content (208.5 ppm) of HRB, would affect subsequent bleaching treatments.

### Pulping of HRB

The main purpose of pulping HRB fibers as a raw material was to obtain a high-yield chemical pulp product characterized by low reject content and low residual lignin, as required for further processes, namely oxygen delignification and bleaching. Such properties of pulps are usually obtained at a residual lignin content corresponding to a Kappa number in the range of 7 to 20.

The susceptibility of HRB to delignification with soda-AQ cooking was investigated. The cooking condition is an important key to delignification. When the alkali charge increased from 10% to 16%, the Kappa number decreased from 17.58 to 7.23. The results of studies on pulping of HRB conducted during this investigation are given in Table 2. NaOH charges of 12% active alkali and below gave pulps that had high Kappa numbers and contained too much uncooked material that was difficult to disintegrate into fibers; thus it was not possible to make suitable HRB pulp. The optimal amount of NaOH charge was judged to be 14%. The Kappa number and the yield decreased with increasing active alkali charge. After the application of 14% alkali charge, the Kappa number decreased to 7.76, with a yield of 57.93%.

**Table 2.** Effect of NaOH Charges on the HRB Pulp

| NaOH Charge /% | Yield /% | Kappa number | Brightness/%ISO | Residual Alkali/g $L^{-1}$ |
|---|---|---|---|---|
| 10 | 63.08 | 17.58 | 24.06 | 0.35 |
| 12 | 59.66 | 11.44 | 24.49 | 1.57 |
| 14 | 57.93 | 7.76 | 24.85 | 2.76 |
| 16 | 48.02 | 7.23 | 24.88 | 5.81 |

The viscosity of HRB after soda-AQ cooking was 1234 mL $g^{-1}$ at the alkali charge of 14%. The high viscosity data indicated that the HRB could yield a pulp with suitable physical properties. The HRB fiber's SEM image after cooking is shown in Fig. 2, and the HRB fiber's SEM image after bleaching stages is shown in Fig. 3. These images show that the HRB fibers are linked closely and are of high strength.

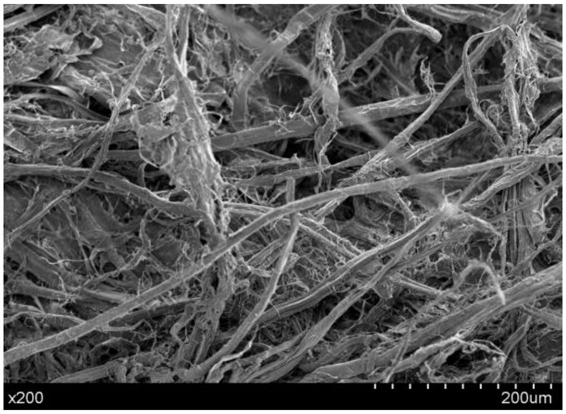

**Fig. 2.** SEM image of the HRB pulp's fiber (*200)

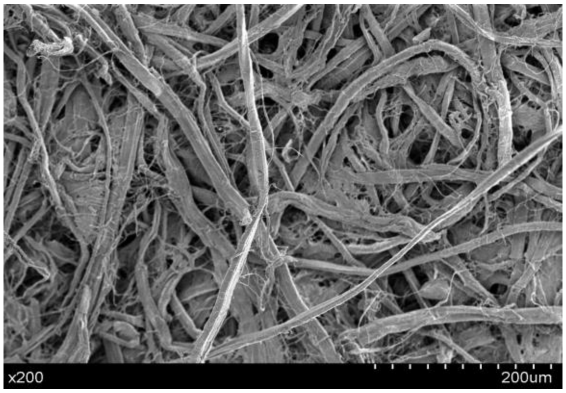

**Fig. 3.** SEM image of the HRB bleached pulp's fiber (*200)

## Oxygen Delignification of HRB Pulp

In this part, the aim was to study the oxygen delignification and bleaching performance of HRB pulps obtained via Soda-AQ pulp with a 14% NaOH charge. The pulps obtained were subsequently subjected to oxygen delignification at different alkali charges and contact times. The results are presented in Table 3. The bleachability of the pulp produced from HRB is very important. The pulp brightness was increased from 33.08 to 43.15% ISO-brightness with an increase in alkali charge from 2.0% to 5.0%, while the yield decreased from 91.08% to 86.47%. However, the viscosity of bleached HRB pulp changed significantly. When the alkali charge was increased from 2% to 5%, the viscosity decreased from 1191 to 996 mL $g^{-1}$. The Kappa number of HRB was very low, as shown in Table 3 and Fig. 4. The residual lignin content was also very low, which shows that most of the lignin was removed in the process of cooking and oxygen delignification. The bleaching yield was slightly reduced with increasing active alkali charge.

**Table 3.** Oxygen Delignification Stage

| Conditions | | | Pulp Properties | | | | Black Liquor |
|---|---|---|---|---|---|---|---|
| T/°C | t/min | Alkali charge/% | Yield/% | Kappa number | Viscosity /mL·$g^{-1}$ | Brightness/%ISO | pH |
| 110 | 60 | 2 | 91.08 | 5.04 | 1191 | 33.08 | 12.43 |
| | | 3 | 88.76 | 4.48 | 1147 | 38.30 | 12.50 |
| | | 4 | 87.00 | 1.90 | 1069 | 42.08 | 12.65 |
| | | 5 | 86.47 | 1.65 | 996 | 43.15 | 12.59 |
| 110 | 45 | 3 | 90.12 | 5.66 | 1164 | 37.50 | 12.17 |
| | 60 | | 88.76 | 4.48 | 1147 | 38.30 | 12.50 |
| | 75 | | 87.77 | 2.27 | 1069 | 38.68 | 12.47 |
| | 90 | | 87.03 | 1.39 | 984 | 42.77 | 12.64 |

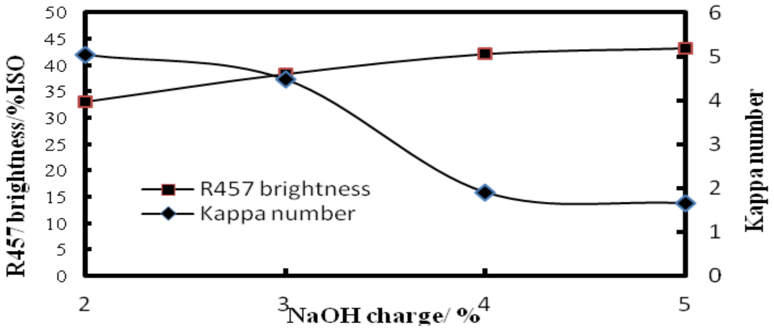

**Fig. 4.** Relationship between the Kappa number, brightness, and the amount of active alkali applied in the delignification process of HRB pulp

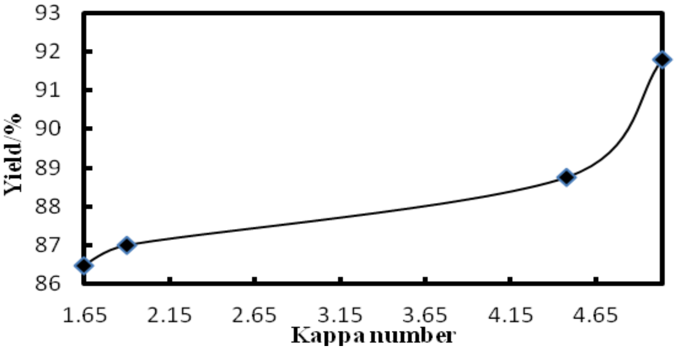

**Fig. 5.** Yield of HRB pulp in the process of oxygen delignification *vs*. Kappa number

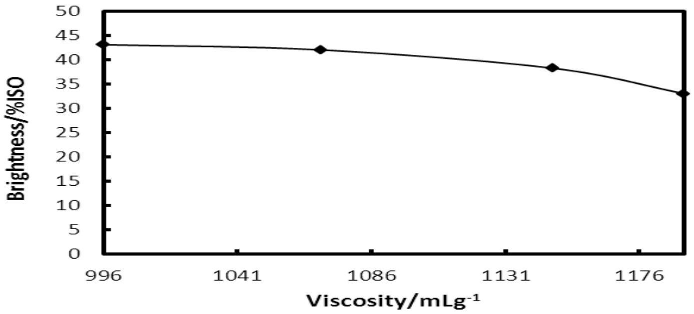

**Fig. 6.** Brightness of HRB pulp in the process of oxygen delignification *vs*. viscosity

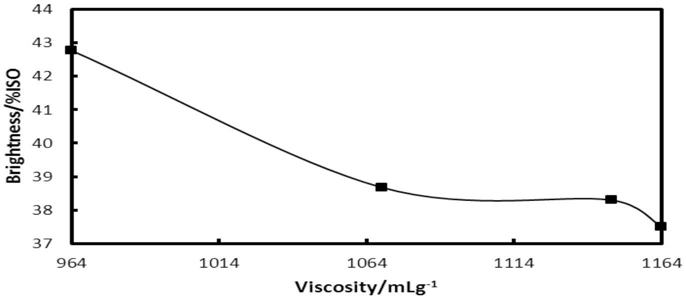

**Fig. 7.** Brightness of HRB pulp in the process of oxygen delignification *vs*. viscosity

The bleaching time of soda-AQ pulp affected the yield of the bleached pulp, Kappa number, and viscosity. With an extension of the bleaching time, the yield of the bleached pulp, Kappa number, and viscosity were decreased significantly. The brightness improved slightly. Table 3 shows that the alkali charge (3%) and bleach time (60 min) were much better than other alkali charges for the oxygen delignification process. These conditions were employed to obtain a higher yield and brightness when using the lower alkali charge. When the alkali charge was 4%, the viscosity and yield of HRB pulp decreased sharply, and the brightness was improved slightly comparing to the 3% alkali charge. Considering the above reasons, the 3% alkali charge and 60 min bleaching were conducted in this stage.

## Chlorine Dioxide Bleaching

The brightness of the pulp was determined by the effect of $ClO_2$ charge in the $D_0$ stage on the bleaching performance. An increase in the $ClO_2$ charge can be expected to increase the delignification degree and allow for easier final pulp bleaching. When the $ClO_2$ charge was increased from 1.5% to 3.0%, the Kappa number and yield of the HRB bleached pulp didn’t improve very significantly. The minimization of chlorate formation by controlling pH can improve the efficiency of the chlorine dioxide bleaching process (Strumila and Rapson 1979). In this sequence, the pH was about 3.5. When the $ClO_2$ charge ($D_0$ stage) was increased from 1.5% to 3.0%, the brightness of the HRB pulp was increased from 68.15% to 71.07% ISO-brightness. The brightness improved slightly, and the viscosity of the pulp decreased sharply. When the $ClO_2$ charge was increased from 1.5% to 3.0%, the viscosity decreased from 1152 mL $g^{-1}$ to 998 mL $g^{-1}$. Thus, the effect of $ClO_2$ charge in $D_0$ stage of bleaching sequence on cellulose degradation was found to be very significant.

**Table 4.** The $ClO_2$ Bleaching ($D_0$) Stage

| Conditions | | | | Pulp properties | | | | Black liquor | |
|---|---|---|---|---|---|---|---|---|---|
| $ClO_2$ /% | T/°C | t/min | Bleaching pH | Yield/ % | Kappa number | Brightness %ISO | Viscosity /(mL·g$^{-1}$) | Bleached pH | Residual $ClO_2$ /g·L$^{-1}$ |
| 1.5 | 70 | 150 | 3.53 | 93.46 | 3.78 | 68.15 | 1152 | 5.88 | 0.18 |
| 2.0 | 70 | 150 | 3.49 | 93.42 | 3.02 | 68.22 | 1140 | 5.73 | 0.42 |
| 2.5 | 70 | 150 | 3.48 | 92.86 | 2.98 | 70.45 | 1080 | 5.64 | 0.80 |
| 3.0 | 70 | 150 | 3.47 | 91.48 | 2.41 | 71.07 | 998 | 5.73 | 1.12 |

The results presented in Table 4 show that when the $ClO_2$ charge was 2.5%, the pulp yield was 92.86%, and the viscosity was 1080 mL g$^{-1}$. The higher the $ClO_2$ charge in the $ClO_2$ bleaching sequence, the lower the yield of the bleached pulp was in this test. The residual $ClO_2$ was very high. At higher residual $ClO_2$, the chlorine dioxide was not fully involved in the bleaching reaction. Residual chlorine dioxide caused great adverse effects in the subsequent alkali treatment. In the $D_0$ stage, the viscosity was the most affected parameter, since the yield and brightness showed little improvement. The addition of $ClO_2$ affected the yield, viscosity, and residual $ClO_2$. The results suggest that a 2.5% $ClO_2$ charge was the optimal dose of $ClO_2$ and could improve brightness effectively and achieve suitable viscosity under these conditions. The 2.5% $ClO_2$ charge was chosen in this stage.

The purpose of alkali treatment (E stage) is to cause the dissolution of the remaining chlorinated lignin and to remove organic acids. After the alkali treatment, the brightness of the bleached pulp did not improve substantially. The bleached pulp in the E stage had a slight decrease in Kappa number compared to that of the $D_0$ stage, with only a 0.36-unit decline. The brightness of the HRB pulp in E stage showed a slight improvement of 0.24 points in comparison to $D_0$ stage. The viscosity in the E stage declined by 168 mL g$^{-1}$ compared to that of the $D_0$ stage. This shows that the alkali treatment obviously degraded the cellulose.

The results listed in Table 5 show when the $ClO_2$ charge increased from 0.4% to 1.2%, there was little effect on the viscosity of bleached pulp. The Kappa number of the bleached pulp was low. The residual lignin was so small that it was possible to ignore removing the lignin by the $ClO_2$. The main work of the $D_1$ stage was improving the brightness of the HRB pulp. In the $D_1$ stage, when the $ClO_2$ charge was increased from 0.4% to 1.2%, the viscosity was decreased from 912 mL g$^{-1}$ to 857 mL g$^{-1}$ and the ISO brightness increased from 79.03% to 91.05%. The addition of $ClO_2$ influenced the yield and viscosity of HRB pulp. $ClO_2$ could remove the chromogenic groups; thus the pulp could achieve a better bleaching result after $D_1$ stage. In $D_1$ stage, the yield of HRB bleached pulp at the 0.6% $ClO_2$ charge was the highest. When the $ClO_2$ charge increased from 0.6% to 1.2%, the viscosity decreased slightly. The 0.6% $ClO_2$ charge was chosen as the optimum for this bleaching sequence.

**Table 5.** $ClO_2$ Bleaching ($D_1$) Stage

| Conditions | | | | Pulp properties | | | | Black liquor |
|---|---|---|---|---|---|---|---|---|
| $ClO_2$ /% | T/°C | t/min | Bleaching pH | Yield/ % | Kappa number | Brightness /% ISO | Viscosity /(mL·g$^{-1}$) | Bleached pH |
| 0.4 | 70 | 150 | 3.52 | 97.73 | 0.97 | 79.03 | 912 | 3.71 |
| 0.6 | 70 | 150 | 3.47 | 98.50 | 0.86 | 85.52 | 893 | 3.68 |
| 0.8 | 70 | 150 | 3.43 | 94.08 | 0.40 | 87.48 | 885 | 3.72 |
| 1.0 | 70 | 150 | 3.44 | 95.12 | 0.40 | 89.48 | 880 | 3.65 |
| 1.2 | 70 | 150 | 3.46 | 95.47 | 0.40 | 91.05 | 857 | 3.69 |

In this bleaching $D_1$ stage, the yield and brightness should be the principal affected quantities. When the $ClO_2$ charge exceeded 0.8%, the brightness of the HRB bleached pulp did not improve significantly, whereas the yield decreased sharply. The results listed in Table 5 reveal that the $ClO_2$ bleaching in the $D_1$ stage resulted in a high yield, viscosity, and brightness at a 0.6% $ClO_2$ charge.

The conditions of different stages for the HRB pulp were as follows:

**Table 6.** Conditions of HRB Pulp at Different Stages

| Stage | Alkali charge/ % | $MgSO_4$/ % | T/°C | t/hr | C/% | P/MPa | pH |
|---|---|---|---|---|---|---|---|
| Cooking | 14 | 0.15(AQ) | 170 | 4.0 | 5:1(liquor -to-HRB) | — | — |
| O | 3.0 | 0.5 | 110 | 1.0 | 15.0 | 0.6 | — |
| $D_0$ | — | — | 70 | 2.5 | 6.0 | — | 3.5 |
| E | 3.0 | — | 60 | 1.0 | 10.0 | — | — |
| $D_1$ | — | — | 70 | 2.5 | 6.0 | — | 3.0 |

**Table 7.** Properties of HRB Pulp at Different Stages

| Stage | Yield/% | Viscosity/mL·g$^{-1}$ | Brightness/%ISO | Kappa number |
|---|---|---|---|---|
| Pulp | 57.93 | 1234 | 24.85 | 7.76 |
| O | 88.76 | 1147 | 38.30 | 4.48 |
| $D_0$ | 92.86 | 1080 | 70.45 | 2.98 |
| E | 95.36 | 912 | 70.69 | 2.62 |
| $D_1$ | 98.50 | 893 | 85.52 | 0.86 |

The results in Table 7 show that HRB can be used as a raw material for pulping. The final viscosity of HRB pulp can reach 893 mL g$^{-1}$. The HRB bleached pulp's iron ion content (204.9 ppm) and manganese ion content (15.0 ppm) were rather high. In this work, the HRB bleached pulp was found to create difficulties in the bleaching stages, and this could be attributed to the metal ions and high content of ash. But the ISO brightness of the HRB pulp was also above 85%. The Kappa number of HRB bleached pulp was only 0.86. After cooking, the yield of the pulp was 57.93% with the soda-AQ cooking of 14% alkali charge.

## CONCLUSIONS

1. Hemp root bast (HRB) can be processed into pulps, and the pulp can be bleached by the conventional methods used for non-wood fiber materials.

2. HRB has a high cellulose content (46.60%) and a low lignin content (17.66%), but also a high ash content (6.52%). Although the high content of ash, as well as high contents of iron and manganese ions will lead to problems in bleaching, the results showed that the HRB was still a high-quality material for papermaking.

3. For the soda-AQ pulping of HRB, an increase in alkali charge resulted in a lower total yield, a lower Kappa number, and a slightly lower viscosity and brightness. The suitable cooking conditions were 14% alkali charge, a cooking temperature of 170 °C, a heating up time of 1.5 h, a holding time of 2.5 h, and a 0.15% AQ charge.

4. After the soda-AQ cooking of HRB, a difficult-to-bleach pulp could be produced having a Kappa number of 7.76, a 57.93% yield, and a 1234-mL $g^{-1}$ viscosity. This result shows that HRB pulp has a low Kappa number, suitable yield, and suitable viscosity.

5. For the $OD_1ED_2$ bleaching sequences of HRB pulp, a high viscosity (893 mL $g^{-1}$) and brightness (85.52% ISO-brightness) of the bleached pulp can be reached.

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