Investigation of the thermal ageing of chemical mechanical pulps obtained from different types of hardwood

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The basic raw materials used in papermaking processes are obtained from fibrous materials produced by chemical treatment of various types of wood or annual plants.

In this research were obtained chemical mechanical pulps (CMP) of wood chips from Paulownia tomentosa and Populus deltoides clon-235-15 with improved density (474 kg/m³).

The obtained chemical mechanical pulp was bleached in two stages by using H₂O₂ and Rongalyt C (NaHSO₃·CH₃O·2H₂O). The investigation of changes of brightness and yellowness of samples in the process of artificially thermal ageing at 105°C have been performed.

The main goals of the present work are studying of artificial thermal ageing of bleached and unbleached CMP obtained in the laboratory from Populus and Paulownia woods.

Key words: wood, thermal ageing, fiber materials.

INTRODUCTION

During the last years we observe that the production of fibrous materials lags behind from the demand of the market. That trend requires to fabricate higher quality papers, cardboards and packaging obtained by high-grade initial fibrous materials. Therefore, in this research we aim to investigate not only the well knows species Populus deltoides clon-235-15, but also the relatively new fast-growing species Paulownia, which are suitable for the production of high-yield fibrous materials (HYFM) [1, 2, 3].

Genus Paulownia (fam. Scrophulariaceae) includes nine species wood [4, 5]. All species of Paulownia are fast growing, with excellent timber, but the most promising ones are P. elongata (emerald tree) and P. Fortune which for a period of 8-10 years reach 20 m in height and 30-40 sm diameter of the stem [6, 7].

EXPERIMENTAL

Production of CMP

The utilized CMP is obtained from the fast-growing poplar timber Populus characterized by increased density of and hardwood timber of the specie P. Tomentosa [6, 7]. The two types of wood were cleaned from the root parts and later shredded into slices with 20mm thickness. After roots removal the wood was chopped into chips of standard dimensions of 15x20x3 mm.

The fibrous materials utilized in this study are:
- CMP – derived by poplar wood of the specie Populus deltoides clon - 235-15, obtained in laboratory conditions;
- CMP – from timber of P. tomentosa, obtained in laboratory conditions.

During the process of production of CMP are used: Na₂SO₃, analytical grade, NaOH. During the bleaching are utilized: H₂O₂, analytical grade, Rongalyt C (NaHSO₃·CH₃O·2H₂O), produced by the company BASF.

Into the composition of the bleaching solution we included the substances Na₂SiO₃ and MgSO₄ which serve as stabilizers of the H₂O₂. Further more NaOH was added to reach the required level of approximately pH 10.5. In order to sequester the ions of the heavy metals - solution of ethylenediaminetetraacetic acid (EDTA) was utilized. The following parameters of the produced CMP have been determined:
- Yield [%], determined by weight method in comparison to the mass of the absolutely dry timber;
- Milling degree, as determined by the device Schopper – Riegler (“SR) as per EN ISO 5267 – 1/AC:2004;

For all used fibrous materials the degree of

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brightness $R_{457}$ (ISO 2470:2002) was determined before and after ageing thanks to the appliance by Spectrophotometer Gretag Magbeth Spectroeye.

Each sample consists of 100g absolutely dry timber of *Populus* and wood of *Paulownia*.

The preliminary weighted chips have been placed in a thermostatic container with the aim to temper it and to maintain the required permanent temperature. Later on they were soaked into required quantity of solution of NaOH and Na$_2$SO$_3$. After retention for a specified period, the used solution was removed and the chips have been washed to reach pH 7. The treatment continued by refined in a Sprout-Valdron laboratory mechanical refiner till the moment of production of fibrous materials. Further on it was washed away and sorted out manually between two sieves.

For production of a CMP is performed according to preliminary determined optimization regime: NaOH 7%, Na$_2$SO$_3$ 5%, temperature 80°C, treatment duration – 120 min and liquor-to-wood ratio 1:5 [4].

The yield of CMP is calculated by the mass method. After soaking for 24 hours in distilled water, the treated chips were washed to reach neutral pH and dried into a drying apparatus at 105°C to achieve absolute dry state.

**Bleaching of the different types of fibrous materials**

The samples of CMP are destined to two-stage bleaching:

I stage – bleaching with H$_2$O$_2$, II stage – bleaching with Rongalyt C.

The conditions of bleaching are given in Table 1.

**Table 1. Conditions of bleaching CMP**

<table>
<thead>
<tr>
<th>Type of Bleaching</th>
<th>Quantity of reagent, (%)</th>
<th>$T$, °C</th>
<th>Duration of process, (min)</th>
<th>Concentration of fibrous materials, (%)</th>
<th>pH of the solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>I stage</td>
<td>2% H$_2$O$_2$</td>
<td>80</td>
<td>120</td>
<td>10</td>
<td>10.5</td>
</tr>
<tr>
<td>II stage</td>
<td>1.5% Rongalyt C</td>
<td>80</td>
<td>60</td>
<td>6</td>
<td>5</td>
</tr>
</tbody>
</table>

The milling degrees are SR$_{12}$.

During the first stage of bleaching, the degree of whiteness is increased. After the completion of the first stage, the fiber material is washed properly to obtain pH 7 and in this manner it is able to undergo the next phase.

The fibrous mass is placed in a polyethylene bag where the bleaching solution is poured. The additional reagents are added and the solution is mixed till reaching complete homogenization. Later the bag is placed in thermostatic container which ensures constant temperature during the whole process of bleaching. In order to achieve better degree of degree of whiteness, it is required to ensure mixing of the fiber material, thus facilitating the uniformly distribution of whitening mixture.

**Ageing of fibrous materials**

The samples of both bleached and unbleached fiber materials, are dried and then they undergo artificial ageing in thermal cupboard at 105°C. In order to characterize the ageing process for bleached and unbleached samples, the degrees of brightness and yellowness for periods of 0, 2, 4, 6, 12, 24, 36, 48 and 72 hours since the commencement of the artificial thermal ageing are determined.

**RESULTS & DISCUSSION**

The yield of CMP from the specie *Paulownia tomentosa* is 86%, while for the specie *Populus*: the yield is 88%. The milling degrees are: CMP from *P. tomentosa* - 13°SR, and CMP from *Populus* - 12°SR.

The utilization of HYFM in various brands of paper and cardboard is limited because of the low level of whiteness. This is the reason to perform two-staged process of whitening. Furthermore artificial thermal ageing at 105°C is conducted. In order to investigate the impact of this ageing over the parameters of the different fiber materials, bleached and unbleached ones, the levels of whitening and yellowness are measured after various periods of time 0, 2, 4, 6, 12, 24, 36, 48 and 72h. Figures 1 and 2 represent the kinetic correlations between the changes in the degree of of brightness and yellowness.

During the process of ageing (Fig. 1) together with the increase of time, the degree of brightness...
decreases for both CMP - bleached and unbleached ones. The change in the degree of brightness is most drastically expressed in the beginning of the ageing. The degree of whiteness (Fig. 1) diminishes in various levels for all samples independently of the way of obtaining of fiber materials. The higher degree of brightness of the bleached samples is kept even during the process of artificial thermal ageing.

Even at equal conditions of production and bleaching of fibrous masses, the higher degree of brightness [%] is observed for the timber from *Paulownia*.

During the continuation of the ageing (fig. 2) the degrees of yellowness increase for all samples. Independently of the origin of the sample in bleached and non-bleached, their yellowness increase with time. The yellowness is higher for the sample of unbleached CMP from *P. tomentosa* timber. Almost identical are the recorded levels of yields for the unbleached CMP from *Paulownia* and the two-staged bleached CMP from *Populus*.

Figures 1 and 2 give clearly shows that the CMP from *Paulownia* in their bleached and unbleached form, provides higher degree of brightness, while the level of yellowness is lower in comparison with the bleached and unbleached CMP from *Populus*.

Finally it became clear that at equal conditions of production, bleaching and ageing, the CMP derived from *Paulownia* explicitly proves better results.

**CONCLUSIONS**

During the study we obtained CMP from the species *Populus* and *P. tomentosa*, under preliminary determined regime the yield obtained was 88% and 86% accordingly, while the degree of milling was 12°SR and 13°SR:

Because of the low level of brightness, the CMP was treated by two-stage bleaching. During stage I the agent utilized was an H₂O₂, while during stage II reduction agent was applied - Rongalyt C. The final degree of brightness achieved for *Paulownia* was 66.4% and for *Populus* - 59.0 %.

- During the processes of bleaching of the two types of fibers materials, it became clear that when treated under the same conditions for preparation and bleaching, the degree of brightness for the specie *Paulownia* is higher.
- The obtained high-yield fibrous materials originating from different timbers are suitable and successfully could be included into compositions of various quality papers and cardboards.

**Fig. 1.** Change of the degree of Brightness of different bleached and unbleached samples during artificial thermal ageing at 105°C.

**Fig. 2.** Change of the degree of Yellowness of different bleached and unbleached samples during artificial thermal ageing at 105°C.
Abbreviations:
CMP - Chemical Mechanical Pulp
EDTA - Ethylenediamine tetraacetic acid
HYFM - High-yield fiber materials

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REFERENCES
1. V.B. Woods, AFBI Agri-Food and Biosciences Institute, 7, 2, 2008.