# ON THE DEFORMATION PROPERTIES OF HIGH YIELD FIBRE MATERIALS BY UNDESTRUCTIVE METHODS

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Received September 16, 2008

High yield fibre material chemi-mechanical pulp (CMP), obtained after the treatment of poplar wood with sodium hydroxide solutions and green liquor, was studied at different degrees of grinding, as well as in composition with cellulose. The stress-deformation curves were derived and the effect of loading time on elastic deformation was shown. The time and rate of loading necessary to decrease the stress in the samples tested at constant deformation were studied. The slip modulus, characterizing the non-uniformity in cellulose and paper samples, was determined. The application of undestructive methods for the analysis of fibrous materials gives the possibility to predict their physico-mechanical properties and the behaviour of the paper under exploitation conditions.

Keywords: chemi-mechanical pulp (CMP), deformation properties, slip modulus, stress, deformation

## INTRODUCTION

The conventional methods applied for testing paper and cellulose fibrous materials estimate, by destructive tests, the paper properties, as determined by its structure at different levels (molecular, macrostructural, etc.). As the relative fraction of each structure is not established by these methods, they can be combined with the integral methods applied for macrostructural analysis, on considering the close correlation observed between the parameters found by both destructive and non-destructive methods. The former give an idea on the limit load value of each fibrous material tested, while the latter describe the behaviour of the fibrous material during loading.

The qualitative estimation of cellulosic fibrous materials, without destroying them, is a complex problem. The behaviour of the material and its ability to resist deformations and breaking under the action of external forces depend on its mechanical macroproperties, such as elasticity, flexibility, plasticity and strength. These properties are directly related to the structure of the material and to the intermolecular forces acting inside it. The ability of certain materials to resist depends not only on the magnitude of the forces, but also on the character of their application in time.

Numerous studies have been devoted to the behaviour of different types of cellulose ground to different degrees, as well as to its compositions with other materials.<sup>1,2,3</sup>

In the present study, the behaviour of chemi-mechanical pulp of poplar wood, obtained under laboratory conditions and treated by green liquor and sodium hydroxide solution, was studied by non-destructive analysis methods.<sup>4,5,6,7</sup>

Chemi-mechanical pulp (CMP), obtained after the treatment with green liquor, could be used for the production of different, either unbleached or bleached, types of paper or cardboards combined with cellulose. The application of undestructive methods may

Cellulose Chem. Technol., 43 (1-3), 37-42 (2009)

provide important information on the behaviour of fibrous materials.

#### EXPERIMENTAL

Chemi-mechanical pulp obtained by the below described method was used in the study. Poplar chops were treated with a sodium hydroxide solution enriched with 10% green liquor, at 140 °C, for 90 min. The following patterns were investigated:

1. low-ground (milled) CMP (14 °SR\*), SR – Shopper-Riegler;

2. high-ground (milled) CMP (62 °SR\*);

3. double-bleached CMP (first stage – bleaching with a 2% solution of  $H_2O_2$ , second stage – bleaching with a 1% solution of  $Na_2S_2O_2$ ), grinding degree – 64 °SR;

4. a fibrous composition of 50% doublebleached CMP and 50% bleached sulfite cellulose.

Tests on shear stress and deformation were performed with the universal test system "INSTRON" – model 1121, at 20 °C, relative air humidity of 50% ( $\phi = 50\%$ ) and velocity of 2 mm/min. Bands with a working length of 100 mm and width of 10 mm were prepared as samples.

The following methods were applied:

A. The method of active linear deformation (t = const), measuring the strain-deformation relationship during active linear stretching of the sample until its destruction (strain-deformation curves).

B. The method of strain relaxation ( $\varepsilon(t) = const$ ), followed by the elastic recovery of the sample, measuring strain  $\sigma$  for a certain time at constant deformation ( $\varepsilon(t) = const$ ), followed by a fast discharge at  $t = t_n$ . After completing the process of elastic recovery of the sample length at  $\sigma = 0$ , the value of the irreversible (residual) deformation component was measured. For samples 2 to 4, the applied deformation value was  $\varepsilon_p = 0.8\%$ , for sample 1,  $\varepsilon_p = 0.4\%$ .

The strain relaxation regime ( $\varepsilon(t) = \text{const}$ ) permitted to determine the following characteristics:

 the relaxation strain σ(t) – ratio between the internal stretching force in sample P(t) and its cross section area F:

$$\sigma(t) = \frac{P(t)}{F}$$

- the elastic component of deformation,  $\varepsilon_o$ , in time,  $t_n$ , calculated by the relation:

$$\varepsilon_o(t_n) = \frac{\sigma(t_n)}{E_o}$$

where E<sub>o</sub> is the initial elastic modulus

- the elastic component of deformation calculated by the relation:

$$\varepsilon_{el}(t_n) = \varepsilon_p - \varepsilon_o(t_n) - \varepsilon_{res}(t_n)$$

The slip modulus was found by the resonance method for unbleached CMP with different degrees of grinding, after its bleaching and mixing with bleached sulfite cellulose. The changes in the slip modulus may provide important information on the structural changes occurring in the studied samples.

#### **RESULTS AND DISCUSSION**

The CMP obtained after the treatment with green liquor, under optimal working conditions, with different grinding degrees, either bleached or mixed with cellulose, was studied through undestructive analysis methods.

Figure 1 plots the strain-deformation curves illustrating the relationship between the deformations obtained at a given strain, for the following samples: 1 - low-ground (14 °SR) unbleached CMP; 2 - high-ground (62 °SR) unbleached CMP; 3 - double-bleached CMP and 4 - bleached CMP mixed with 50% bleached sulfite coniferous cellulose.



Figure 1: Strain-deformation curves: 1 - low-ground (14 °SR) unbleached CMP; 2 - high-ground (62 °SR) unbleached CMP; 3 - high-ground (62 °SR) double-bleached CMP; 4 - 50% bleached CMP + 50% bleached sulfite coniferous cellulose

The graph evidences an initial linear part, which becomes curvilinear after the increase in the strain applied. A possible explanation of the linear part of the curves is the occurrence of elastic deformation in the samples, determined by the rearrangement of the segments inside the material structure during the load.

Here, according to Hooke's law, the value of the deformation is proportional to the strain applied. From the slope of this first linear part of curves  $\sigma/\epsilon$ , the initial elastic modulus  $E_o$ , characterizing the value of the strain after taking away the load at which the samples return to their initial state, can be determined.

The observation was made that the samples containing 50% bleached CMP and 50% sulfite bleached cellulose presented the highest value of the elastic modulus.

The total deformation of the paper samples under the applied strain is composed of:

- elastic Hookean deformation, characterizing the change of intermolecular spaces in the structure of both paper and separate fibres;

- highly-elastic deformation, changing the configuration of the macromolecules in the fibres and the distance between the molecules connected on the fibre surface, as well as fibre configuration;

- plastic deformation, during which the fibres shift irreversibly and the intermolecular bonds between the surfaces are disturbed.

Figure 2 plots the effect of the loading time on the elastic deformation component for different CMP and CMP–cellulose samples, at a constant deformation  $\varepsilon_p = 0.8\%$ .

elastic Hookean deformation The instantly arises and disappears on charge application or removal. Over the domain of the elastic deformations of the paper samples, changes occur in the form and magnitude of the fibres and their contact zones. There is no break of the interfibrous bonds (i.e., bonds between fibres). During continuous charge, elastic deformation occurs, gradually increasing and totally disappearing after the discharge. The elastic deformation changes are presented in Figure 3. The largest elastic deformation occurs in the composition of cellulose with bleached CMP, followed by unbleached CMP, which contains more elastic fibres than the bleached CMPs.

The further increase in charge leads to the appearance of irreversible plastic deformations. Figure 4 illustrates the change in residual deformation. CMP addition to sulfite cellulose leads to serious increases in total deformation, as well as in its component –

residual deformation. From the same figure it is evident that the critical time for all three CMP samples is the same ( $t_{cr} = 0.1$  min).

Despite CMP bleaching and addition of 50% sulfite bleached cellulose, the working limit does not change. Although the collected residual deformation is the highest for the samples containing 50% CMP and 50% sulfite bleached cellulose, it cannot be asserted that plasticity increases with cellulose addition. The residual deformation component,  $\varepsilon_{res}$ , at moment  $t_n$ , includes a plastic component, due to mechanical destruction and forced (constrained) elastic deformation, with a relaxation nature. Figure 5 plots the deformation change in unbleached CMP with a 14 °SR grinding degree.

Stretch deformation includes the sum of deformations obtained after straightening the twisted fibres and the deformation of the bonds between fibres.

Table 1 lists all characteristics of the samples. The breaking strain studied characterizes the mechanical strength of the samples and their limit strength value, which is consistent with the mechanical strength determined through destructive analysis tests. To assure a normal paper production, the material should not be exposed to charges exceeding the breaking strain. The deformation of the samples, attained at breaking strain, characterizes their maximal ability to change their length under its influence. No charges, causing deformations even near to the limit ones, should be permitted during paper production.

The strain relaxation method permitted to study the time (t) necessary to decrease the strain ( $\sigma$ ) in the samples studied at constant deformation. It was found out that, for constant deformation values of  $\varepsilon = 0.8\%$  for samples 2, 3 and 4, the strain sharply decreases and, after a certain time, it becomes linear (Fig. 6). Therefore, the investigated material possesses elasticity and strength and can overcome the sharp loads imposed on it. An important feature of the paper materials is their relaxation velocity, *i.e.* the velocity with which the material will overcome the strain exerted on it and would return to its equilibrium state. These velocity values are calculated from the slope of the straight lines of the relationship between strain and time, in log coordinates  $\sigma/\log t$ , as presented in Figure 7.

The highest values of the relaxation rate were recorded for the bleached CMP + cellulose



Figure 2: Effect of loading time on the elastic Hookean deformation component: 2 - high-ground (62 °SR) unbleached CMP; 3 - high-ground (62 °SR) bleached CMP; 4 - 50% bleached CMP + 50% bleached sulfite coniferous cellulose



composition, followed by unbleached CMP ground to 62 °SR. The lowest relaxation rate was recorded for low-ground CMP (14 °SR).



Figure 3: Effect of loading time on the high elastic deformation component: 2 - high-ground (62 °SR) unbleached CMP; 3 - high-ground (62 °SR) bleached CMP; 4 - 50% bleached CMP + 50% bleached sulfite coniferous cellulose



Figure 4: Accumulation kinetics of the residual deformation component: 2 - high-ground (62 °SR) unbleached CMP; 3 - high-ground (62 °SR) bleached CMP; 4 - 50% bleached CMP + 50% bleached sulfite coniferous cellulose

Figure 5: Time dependence of the Hookean deformation component: 1 - high elastic deformation component; 2 - residual deformation component; 3 - for unbleached CMP with a 14 °SR grinding degree

№ of sample	Cross section area F, mm	Breaking strength (effort), N	Breaking deformation, %	Breaking strain, MPa	Work for breaking, A, J	Elastic modulus, E, MPa·10 <sup>-3</sup>
1	1.70	11.92	0.70	7.00	0.0065	1.03
2	1.10	45.15	1.25	41.13	0.0354	4.18
3	1.00	47.88	1.45	47.88	0.0488	4.05
4	1.06	47.28	1.56	44.71	0.0498	4.70

Table 1 Physical and mechanical characteristics of the CMP samples

1 – unbleached chemi-mechanical pulp with a grinding degree of 14  $^{\circ}SR$ 

2 - unbleached chemi-mechanical pulp with a grinding degree of 62 °SR

3 - double-bleached chemi-mechanical pulp with a grinding degree of 64 °SR

4 - 50% bleached CMP + 50% sulfite bleached cellulose



Figure 6: Strain relaxation for different samples:  $1 - \text{low-ground} (14 \text{ }^{\circ}\text{SR})$  unbleached CMP;  $2 - \text{high-ground} (62 \text{ }^{\circ}\text{SR})$  unbleached CMP;  $3 - \text{high-ground} (62 \text{ }^{\circ}\text{SR})$  double-bleached CMP; 4 - 50% bleached CMP + 50% sulfite bleached cellulose

The analysis of the results obtained by the relaxation method shows that they are valid when the deformation applied occurs inside the linear part of the strain-deformation curves. Obviously, applying deformations exceeding this linear part on the paper samples would not result in stress relaxation, because the deformation values would be beyond the elastic deformations domain and the samples will not return to their initial state. The practical application of the method refers to the fact that, once known the elastic modulus of the paper materials, the domain of the possible deformations they can bear, without approaching their breaking limit, will be more obvious.

The slip modulus is a quality characteristic of the non-uniform structure of both cellulose and paper materials. The



Figure 7: Strain relaxation for different samples: 1 -low-ground (14 °SR) unbleached CMP; 2 -high-ground (62 °SR) unbleached CMP; 3 -high-ground (64 °SR) double-bleached CMP; 4 - 50% bleached CMP + 50% sulfite bleached cellulose

physical sense refers to the creation and concentration of the stress on the crosssection of the sample, during its structure formation. The higher the values of the slip modulus, the higher the paper quality will be.

Table 2, presenting the results on the slip modulus of different samples, shows that the lowest value of the slip modulus is recorded for low-ground (14 °SR) unbleached CMP. The existence of rough, untreated and weakly fibrillated fibres explains the structural non-uniformity of the samples. By increasing the grinding degree, the structure is improved and therefore, a sharp increase occurs in the slip modulus. CMP bleaching has a certain effect on the improvement of samples structure, by contributing to a better bonding between the treated and fibrillated fibres. The highest value of the slip modulus is recorded for the 50% bleached CMP + 50% bleached sulfite cellulose composition,

which testifies the good cellulose – CMP compatibility.

Table 2						
Slip modulus values for different samples						

Sample	Slip modulus, MPa	
100% low-ground (14 °SR) unbleached CMP	224	
100% high-ground (62 °SR) unbleached CMP	528	
100% high-ground (62 °SR) bleached CMP	562	
50% bleached CMP + 50% sulfite bleached cellulose	581	

## CONCLUSIONS

1. The high value of the elasticity modulus in samples consisting of 50% high-ground (64 °SR) bleached CMP and 50% sulfite bleached cellulose expresses the improvement of its material properties.

2. The CMP studied shows good strength and elastic properties and can overcome the loads applied.

3. Information on the elasticity modulus of the fibrous material will elucidate the possible resistance of the material to certain loads, without exceeding the breaking limit.

4. The slip modulus is a quality characteristic of the structural nonuniformity of both cellulose and paper samples. The higher the slip modulus, the higher the quality of the paper sample will be.

The application of undestructive methods in the analysis of fibrous materials permits to predict their physico-mechanical properties and the behaviour of the paper under exploitation conditions.

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