CHEMI-MECHANICAL PULP FROM RAPESEED STRAW

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This study deals with chemi-mechanical pulping of rapeseed straw (species *Brassica napus* L. convar. *napus*). Three cold chemi-mechanical processes, namely neutral sulphite, alkaline sulphite, and caustic soda, were applied under laboratory conditions. The chemi-mechanical pulping comprises four main operations, viz. chipping, grinding, leaching, and beating. The influence of varying charges of chemicals on the total yield, strength properties such as bending stiffness, bending modulus of elasticity, curvature in the region of elastic and plastic deformation, as well as the tensile index of pulp hand sheets was determined. The results obtained revealed that the cold caustic soda pulping has greater effect on the bending stiffness and bending modulus of chemi-mechanical pulps in comparison with neutral sulphite and alkaline sulphite pulping, although the total pulp yield for caustic soda pulping was lower than that for neutral sulphite and alkaline sulphite pulping. For all three cold processes, the bending stiffness, as well as the bending modulus of elasticity in the region of elastic deformation increased with increasing the chemical charge. However, the tensile index of chemi-mechanical pulps was found to be significantly lower in comparison with kraft softwood pulp and pulp from waste paper. The chemi-mechanical pulp was characterized by its degree of polymerization which was measured in the range of 75 to 110 with respect to relatively great amount of low molecular substances presented in pulps prepared by the cold pulping process.

Keywords: rapeseed straw, cold pulping process, pulp yield, bending stiffness, polymerization degree

INTRODUCTION

Nowadays, wood is the dominant resource for pulp and paper production which is increased continuously. However, since wood is not available in sufficient quantities in many countries, alternative new non-wood raw materials such as annual plants and agricultural waste are searched for exploitation as the potential substitution of wood.

Rapeseed (its spring cultivar knows as canola) is widely cultivated throughout the world and used as a major oilseed crop for vegetable oils and biodiesel production. With respect to growing demand for vegetable oils and biodiesel, the worldwide planted area for rapeseed increases ceaselessly. At present, the planted area in the Czech Republic achieved approximately 400 thousand hectares. After rapeseed seed collection, the total amount of straw produced per unit area varies from 2.8 to 4.5 t/ha (ref.[1]), depending for given genotype mainly on irrigation. Owing to the extreme coarseness of the rapeseed straw, it cannot be used as cattle feed, however, it could be used in various products, including pulp and paper manufacture.

The morphological analysis indicated that despite the thicker cell wall, the morphological properties of rapeseed straw fibres were comparable to those of non-woods and hardwoods fibres.[2] Rapeseed straw fibres having an average length of about 1 mm can be classified as short fibres, similarly as most of hardwood fibres. However, rapeseed stalks contain a pith portion, which is located in the centre of stem. The pith of non-wood plants is composed mainly of parenchyma cells and vessel elements and causes serious problems such as increasing chemical consumption in pulping and bleaching, washing problems, and drainage problems in papermaking.[2]

Various alternative pulping processes including conventional soda,[2,3] soda-AQ,[4,5] neutral sulphite semi-chemical[6,7] or chemi-mechanical[8,9] pulping were investigated. The soda and soda-AQ pulping results showed that, unlike to the other non-woods, canola stalks required higher chemical charge with higher cooking time. The relatively low screen yield of 35 to 39 % at kappa number between 24 and 54 for canola stalks,[2] as well as the total yield of 33 to 39 % at kappa number from 27 to 55 for conventional soda pulping of rapeseed straw,[3] and the screen yield of 35 to 41 % at substantially greater kappa number of 45 to 96 for rapeseed straw[4] may be attributed to high alkali solubility of rapeseed straw which lowers the selectivity of delignification and total pulping yield.

In order to achieve higher pulp yields, the neutral sulphite semi-chemical pulping[6,7] and chemi-mechanical pulping[8,9] of rapeseed residues were investigated. Ahmadi *et al*.[6] and Kasmani *et al*.[7] achieved the total yield of 58 to 72 % and of 66 to 77 %, respectively, using neutral sulphite semi-chemical process. Also, the chemi-mechanical pulping process has the advantages of a mild chemical treatment and of high pulping yield compared with the chemical pulping process. The canola straw was pretreated with various dosages of sodium hydroxide and sodium sulphite at a temperature of 125 °C for 15 min by Hosseinpour *et al*.[8] With increasing dosage of the chemicals, from 4 to 12 % of sodium hydroxide and sodium sulphite, the total pulp yield decreased from 69 to 60 %. Using cooking liquor containing sodium sulphite,[9] the total pulp yield from 75.2 to 69.7 % was reached with increasing pulping time from 30 to 50 min at a cooking temperature of 170 °C and sodium sulphite charge of 20 %.

In contrast to the papers,[8,9] when chemical treatment of canola stalks was performed at a high temperature of 125 °C, and 170 °C, the objective of the present study was to manufacture chemi-mechanical pulp from rapeseed straw by cold pulping process. Three cold pulping processes, namely neutral sulphite, alkaline sulphite, and caustic soda ones, were conducted at a room temperature under laboratory conditions. Besides total pulp yield, the influence of chemical charge upon the bending stiffness of chemi-mechanical pulp sheets having a basis weight of 510 to 590 g m^{-2} was investigated using the three-point loading method.

MATERIALS AND METHODS

Rapeseed straw (*Brassica napus* L. convar. *napus*, line genotype Labrador) collected from the field in Polabian lowlands near city of Pardubice was used as a raw material to chemi-mechanical pulping experiments. The stalks and valves of silique were cut manually into small chips having a length of about 20 mm. After drying at 60 °C for 5 hours, the chips of stalks and of silique valves were ground for 20 – 25 s using a laboratory vibrating mill containing a rollar and collar in the milling space. Fine mass of accepts retained on +50 mesh size was used for leaching. The samples of fine material to be leached were blends of the stalks and silique valves in a mass ratio of 2:1. Three various cold pulping processes, viz. neutral sulphite, alkaline sulphite and caustic soda, were applied at three different levels of active alkali charges, namely 4, 9, and 16 mass % of Na_2O on oven-dried straw. For the liquor-to-straw ratio of 15:1, the leaching was performed for 18 hours at a temperature of 21 – 23 °C. For comparison, the leaching of a blend of stalks and silique valves into tap water was carried out as well.

After four-stage batch washing, the wet pulp was beaten to 40 – 46 °SR using a laboratory conical beater. The beating degree was measured by Schopper-Riegler method according to ISO 5267-1 Standard. The suspension obtained after beating process was used to prepare pulp hand sheets with basis weight of 510 to 590 g m^{-2} on a Rapid-Köthen sheet forming machine. To determine the stiffness properties, the stripes, 15 mm in width and 90 mm in length, were cut from the pulp hand sheets (Table 1). Using a TIRA test 26005 device, the bending stiffness was determined by the three-point loading method when the distance between supports was kept at 50 mm. Tensile properties such as tensile index, breaking length, and relative elongation were measured with strips, having a length of 150 mm and width of 15 mm, cut from hand sheets of basis weight ranging of 72 to 102 g m^{-2}. Strength characteristics were measured under a constant room temperature of 23±1 °C and relative humidity of 50±2 %. All the strength measurements were performed at least on 20 replicates per each tested sample.

To characterize chemi-mechanical pulps, the average degree of polymerization was determined by a viscosity test using FeTNa solution (iron (III) sodium tartrate complex) as a solvent for chemi-mechanical pulp according to ISO 5351/2-1981.

RESULTS AND DISCUSSION

Pulp yield

The total yield, Y, defined as a ratio between the mass of chemi-mechanical pulp reached after leaching and the mass of the raw material to be used for grinding is influenced mainly by the amount of fine fraction to be removed after grinding and the amount of solutes leached out of rapeseed straw into chemical solutions (Figure 1). Since the fine fraction rejected after grinding was approximately 24 %, the differences in the total yield are due to various amounts of solutes transferred from chips of rapeseed straw into aqueous solutions having different concentration of chemicals.

As it follows from Figure 1, where the yield of extraction, Y_E, defined as the mass of oven-dried solid raffinate after leaching divided by the mass of rapeseed straw accepted after grinding, the amount of solutes leached into aqueous solutions increased with increasing the concentration of chemicals. Cellulose is the principal component in cell walls and in fibres of annual plants and agricultural waste. The non-cellulose components of the cell walls include hemicelluloses, lignin, pectins, starch, proteins, as well as surface impurities like resins, fat, and wax substances, and in the epidermal cells also certain inorganic compounds which are essential for plant growth and development, but undesirable in pulping and papermaking. It is known that pectins, *i. e.* pectic polysaccharides, together tannins and inorganic salts are dissolved into cold water.[10] One may assume that xylans, mainly of lower degree of polymerization, were leached into sodium sulphite solutions, and furthermore some lignin into alkaline solutions.[11] Thus, the total yield, Y, as well as the yield of extraction, Y_E, decrease with increasing alkalinity of aqueous solutions, because sodium sulphite solution presented weak alkalinity as well (Table 1).

The total yield of chemi-mechanical pulp was much greater than that of soda rapeseed pulp. In our previous paper, [5] the total soda pulp yield ranging from 28 % to 42 % was reached, depending on the degree of pulp delignification. Thus, our results are similar to those obtained by Ahmadi *et al.*[6] and by Kasmani *et al.*[7] for neutral sulphite semi-chemical process, and by Housseinpour *et al.*[8] who used cooking liquor contained sodium hydroxide along with sodium sulphite for chemi-mechanical pulping.

Strength characteristics

The bending stiffness is a property of paper and board which expresses its rigidity or resistance to bending. A typical dependence between specimen deflection, y, and acting force, F, for chemi-mechanical pulp made from rapeseed straw using cold caustic soda process is illustrated in Figure 2. The bending stiffness, S, is defined as

$$S = \frac{F l^3}{48 y} \tag{1}$$

where F/y corresponds to the slope in the region of elastic deformation, at low acting forces, when the dependence of the acting force on the deflection is straight, and l is the distance between supports.

Although the bending stiffness increases strongly with increasing the thickness of test specimen, theoretically with the third power of paper sheet thickness, *e. g.*, Potůček *et al.*[12] found that $S \approx h^{2.74}$ for groundwood, the bending stiffness measured for neutral sulphite, alkaline sulphite, and caustic coda pulps are greater in comparison with that measured for mechanical pulp after leaching into tap water (Figure 3). For comparison, the results obtained earlier[13] for cautic soda leaching of rapeseed stalks only are shown in Figure 3 and in further figures illustrating strength properties of chemi-mechanical pulps. Moreover, one may assume that the bending stiffness has an increasing trend how the charge of chemicals increases, mainly for neutral sulphite and caustic soda pulps. It is worth nothing that pulp hand sheets made mainly by neutral sulphite process from rapeseed straw containing silique valves had a much higher bulk in comparison with hand sheets made form rapeseed stalks by the cold caustic soda process earlier[13] when specimen thickness of 1.19 and 1.08 mm for basis weight of 521 and 508 g m^{-2}, respectively, were determined.

The bending modulus of elasticity in the region of reversible deformation, E, defined as

$$E = \frac{F l^3}{4 y b h^3} \tag{2}$$

where b is the specimen width, h is the specimen thickness, and a meaning of other symbols is the same as in equation (1), is illustrated as a function of chemical charge in Figure 4. Our previous results[12] showed that the bending modulus of elasticity is not appreciably different for groundwood specimens with various thicknesses. For all cold leaching processes, the bending modulus of elasticity increased unambiguously with increasing the charge of chemical. The greatest values of the bending modulus are achieved for cold caustic soda process. Comparing the bending modulus measured for pulps made from rapeseed straw and from stalks only, it is evident that the presence of silique valves in straw has a negative effect upon both bending stiffness and bending modulus of elasticity. It is worth mentioning that the bending stiffness and bending modulus measured for chemi-mechanical pulps including pulp made from stalks only lie within the limits of 0.4 to 1.8 kN mm^2, and of 0.06 to 1.1 kN mm^{-2}, respectively, and are lower than those of 2.6 to 12.3 kN mm^2, and of 1.3 to 1.6 kN mm^{-2}, respectively, reported by Potůček *et al.*[12] for unbleached spruce groundwood sheets having a thickness of 1.09 to 1.97 mm.

The maximum curvature in the region of reversible deformation, C_E, defined by the following relationship

$$C_E = \frac{12 y_{E_{\max}}}{l^2} \tag{3}$$

is plotted against the chemical charge in Figure 5. For alkaline sulphite and caustic soda pulps, the maximum curvature increases with increasing the chemical charge, however, these pulps are less elastic in comparison with mechanical pulp when the tap water with zero chemical charge was used in leaching process. Surprisingly, the greatest values of the maximum curvature were achieved for neutral sulphite pulp when the thickness of specimens was higher than that of alkaline sulphite and caustic soda pulps. Nevertheless, except for 16% charge of Na_2O, the maximum curvature of specimens from rapeseed straw comprising silique valves was lower than that measured for caustic soda pulp made from rapeseed stalks only in our previous paper.[13]

It should be noted that, for neutral sulphite, alkaline sulphite, and caustic soda pulps, the maximum curvature in the region of reversible deformation reveals an increasing trend with increasing the chemical charge even if the thickness of the specimens decreases (Table 1). The results obtained in the previous paper[12] showed that the maximum curvature decreased with increasing the thickness of specimens made from unbleached spruce groundwood when the maximum curvature decreased from 7.7 to 4.6 m^{-1} how the thickness rose from 1.09 to 1.97 mm.

The influence of chemical charge on the critical curvature for tensile crack is illustrated in Figure 6. The critical curvature, C_F, is given by the following equation

$$C_F = \frac{12y_F}{l^2} \tag{4}$$

Tensile crack occurs in the region of irreversible or plastic deformation and is evident in the convex side of specimen below the neutral plane. The quantity of the critical curvature was found to increase with the increment of chemical charge in all pulps made by cold chemi-mechanical process. For chemical charge of 4 %, the critical curvature is comparable to that for mechanical pulp when rapeseed straw was leached into tap water. However, further increment of chemical charge brought an increase in the critical curvature for all pulp made from rapeseed straw comprising silique valves. Similarly as the bending stiffness and bending modulus of elasticity, the critical curvature for tensile crack was found to be the greatest for cold caustic soda pulps in which the presence of silique valves in rapeseed straw had a negative effect on the critical curvature comparing with pulp made from stalks only. Likewise as the maximum curvature in the region of reversible deformation, the critical curvature revealed a decreasing trend with increasing the thickness of hand sheets made from unbleached spruce groungwood[12] when the critical curvature dropped from 63.1 to 36.8 m^{-1} how the thickness rose from 1.09 to 1.97 mm. Thus, the critical curvature measured for chemi-mechanical pulps was found to be lower in comparison with that for groundwood in the previous paper.[12]

The strength characteristics determined for chemi-mechanical pulps made by three cold processes included the tensile index, *TI*, defined as

$$TI = \frac{F}{BW\ b} \tag{5}$$

where *BW* is the basis weight, and a meaning of other symbols is the same as in equation (2).

For pulp hand sheets having basis weight within the interval from 72 to 102 g m^{-2} and the thickness ranging of 0.25 to 0.42 mm, the tensile index increased with increasing the charge of chemicals (Figure 7), but the values obtained were much lower than those reported previously by Potůček *et al.*[14] who measured 78.4 and 37.4 N m g^{-1} for virgin kraft softwood pulp beaten to 19 °SR and waste paper from postconsumer corrugated board, respectively. Also, the relative elongation of 0.3 to 0.8 % measured for chemi-mechanical pulp from rapeseed straw is much lower than that of 3.2 % and 2.1 % for virgin kraft pulp and waste paper, respectively, determined earlier.[14] These results confirmed that chemi-mechanical pulp from rapeseed straw is not a raw material convenient for production of wrapping papers.

Furthermore, the stiffness results obtained for rapeseed straw were compared with those measured for unbleached spruce groundwood and for moulded fibre products, which were published earlier.[12,15] At almost constant thickness of the pulp hand sheets made from rapeseed stalks by the caustic soda chemi-mechanical process (CMP), the bending stiffness and bending modulus of elasticity in the region of reversible deformation measured for the highest charge of caustic soda are comparable to those measured for unbleached spruce groundwood and are much greater in comparison with those obtained for moulded fibre products made from waste paper (Table 2). However, the bending stiffness and bending modulus of elasticity determined for mechanical pulp (MP) made from rapeseed straw comprising silique valves, when leaching into only tap water was carried out, are somewhat lower than those published for moulded fibre products previously.[15] It is worth mentioning that the presence of silique valves in rapeseed straw led to much higher bulk of hand sheets having lower density when leaching into tap water was performed.

Degree of polymerization

The chemi-mechanical pulp made by three cold pulping processes was also characterized by its average degree of polymerization which is directly proportional to the chain length of macromolecular substances and has impact upon mechanical properties of pulp fibres. The degree of polymerization was determined viscosimetrically for unbeaten pulp when FeTNa solution was used as a solvent. Figure 8 illustrates the effect of Na_2O charge upon the average degree of polymerization. The low values of degree of polymerization can be attributed to the presence of low molecular substances, mainly hemicelluloses, in chemi-mechanical pulps. However, the degree of polymerization slightly increases with increasing the chemical charge because of the reduction of hemicelluloses and other low molecular weight components contained inside chemi-mechanical pulp fibres. Using conventional soda pulping, [5] the average degree of polymerization was found to be 917 and 943 for soda pulp from stalks only and from rapeseed straw comprising silique valves (line genotype Labrador), respectively, when Cadoxen was used as a solvent for cellulose. However, it should be taken into account that the solvent properties can influence the average polymerization degree.[16] Similarly, Enayati *et al*.[2] (2009) measured the degree of polymerization ranging of 1,408 to 1,579 for soda pulp cooked from canola stalks. For comparison the degree of polymerization of 1,234, 1,098, and 481 was also determined for beech unbleached kraft pulp (kappa number of 14.1), unbleached kraft pulp from softwood (blend of spruce and pine, kappa number of 24.9), and bleached kraft pulp from softwood (blend of spruce and pine), respectively. The lower value of the degree of polymerization in case of unbleached softwood pulp may be ascribed to the presence of much amount of hemicelluloses in this pulp in comparison with the unbleached beech pulp cooked to low degree of delignification.

CONCLUSIONS

The results obtained in the scope of our study proved that, owing its papermaking properties exhibiting slight tensile strength, the chemi-mechanical pulp from rapeseed straw is not sufficient for the production of wrapping papers with a basis weight below 100 g m^{-2}. In spite of this fact, the bending modulus of elasticity of chemi-mechanical pulp hand sheet with a basis weight above 500 g

m^{-2} prepared by cold caustic soda process was found to be greater in comparison with that for moulded fibre products manufactured from secondary fibres.

Nevertheless, the preliminary results obtained offer a possibility to utilize chemi-mechanical pulp from rapeseed straw, at least partially, in the pulp and paper industry, *e. g.*, in a blend with secondary fibres to manufacture moulded fibre products. However, with respect to current knowledge on chemi-mechanical pulping of rapeseed straw, further studies should be developed to confirm the suitability of rapeseed as a future non-wood fibre source. Thus, besides chemical pulping of rapeseed straw, cold chemi-mechanical pulping offer another possibility of rapeseed straw treatment.

SYMBOLS

b	specimen width, mm
BW	basis weight, g m^{-2}
C_E	maximum curvature in the region of elastic deformation, m^{-1}
C_F	critical curvature in the region of plastic deformation, m^{-1}
E	bending modulus in the region of elastic deformation, N mm^{-2}
F	force, N
h	thickness of specimen, mm
l	distance between two support points, mm
S	bending stiffness, N mm^2
TI	tensile index, N m g^{-1}
y	deflection, mm
y_{Emax}	maximum deflection in the region of reversible deformation, mm
y_F	deflection attained in the region of non-reversible deformation where tensile crack occurs, mm
Y	total yield
Y_{E}	yield of extraction

Abbreviations

AQ	anthraquinone
CMP	chemi-mechanical pulp
MP	mechanical pulp
o. d.	oven-dried

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Table headings

Table 1

Pulp hand sheet characteristics

Table 2

Comparison of bending stiffness, S, and bending modulus of elasticity, E, measured for various pulp hand sheets with different thickness, h

Table 1

Pulp hand sheet characteristics

Charge of chemicals g Na_2O / 100 g o.d. straw	pH of extract after leaching	Beating degree °SR	Basis weight g m^{-2}	Thickness mm
0	6.3	45	537	1.81
Cold neutral sulphite				
4	8.0	42	540	1.90
9	8.2	40	537	1.72
16	8.3	43	547	1.76
Cold alkaline sulphite				
4	9.2	40	557	1.77
9	11.2	40	515	1.36
16	12.0	46	514	1.17
Cold caustic soda				
4	10.0	40	520	1.47
9	12.2	40	514	1.26
16	12.4	46	584	1.12

Table 2

Comparison of bending stiffness, S, and bending modulus of elasticity, E, measured for various pulp hand sheets with different thickness, h

Material	*h* mm	*S* kN mm^2	*E* kN mm^{-2}
CMP from stalks only (15.9 % Na_2O)[13]	1.08	1.76	1.11
CMP from rapeseed straw (16% Na_2O)	1.12	1.29	0.74
MP from stalks only (0 % Na_2O)[13]	0.99	0.22	0.18
MP from rapeseed straw (0 % Na_2O)	1.81	0.56	0.07
Spruce groundwood[12]	1.01	1.62	1.27
Waste paper (moulded fibre products)[15]	1.01	0.34	0.26

Figure captions

Figure 1: Influence of Na_2O-charge on the total yield, Y, and yield of extraction, Y_E

Figure 2: Typical dependence between specimen deflection, y, and acting force, F, measured for chemi-mechanical pulp hand sheet

Figure 3: Influence of Na_2O-charge on the bending stiffness, S

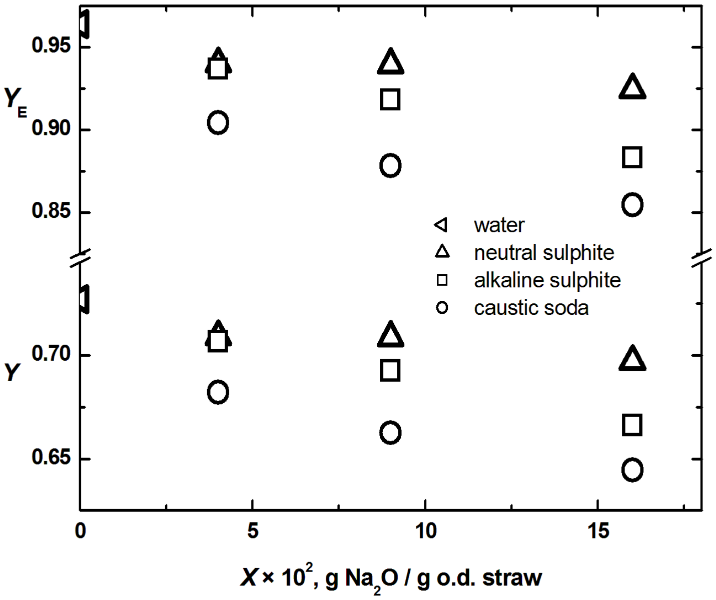
Figure 4: Influence of Na_2O-charge on the bending modulus of elasticity, E, in the region of reversible deformation

Figure 5: Maximum curvature, C_E, in the region of reversible deformation as a function of Na_2O-charge

Figure 6: Curvature for tensile crack, C_F, as a function of Na_2O-charge

Figure 7: Tensile index, TI, as a function of Na_2O-charge

Figure 8: Degree of polymerization, DF, as a function of Na_2O-charge



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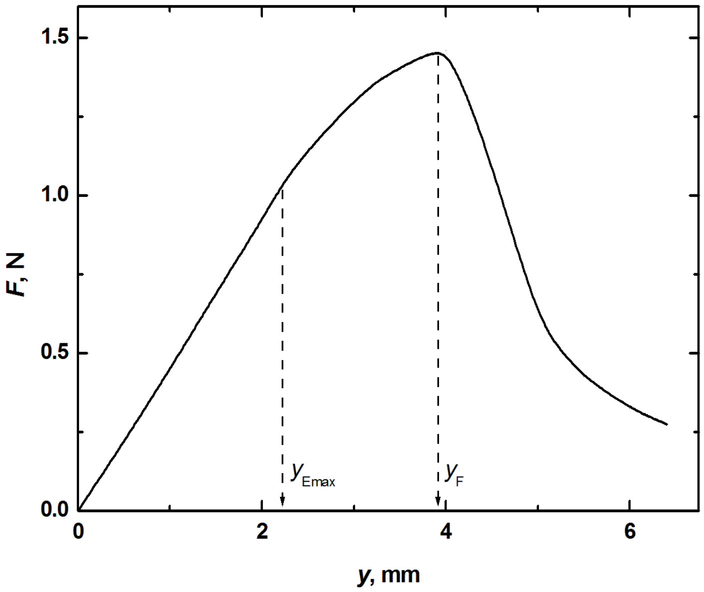

Figure 2: Typical dependence between specimen deflection, *y*, and acting force, *F*, measured for chemi-mechanical pulp hand sheet

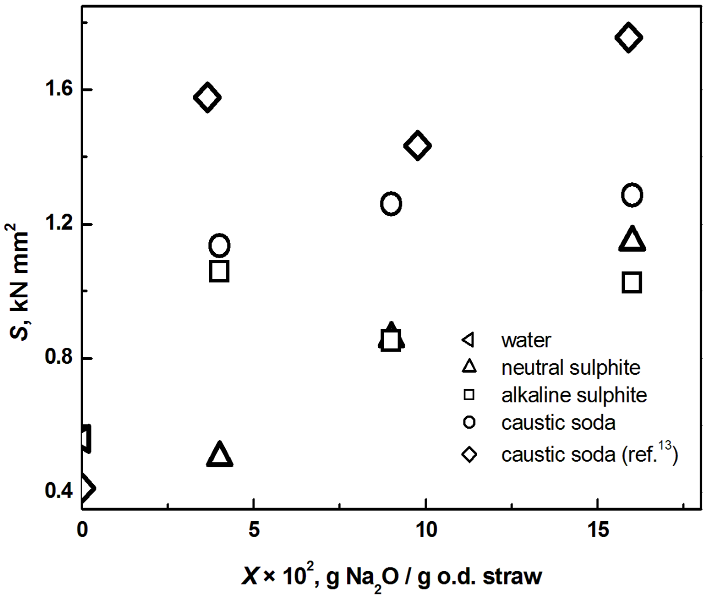

Figure 3: Influence of Na_2O-charge on the bending stiffness, S

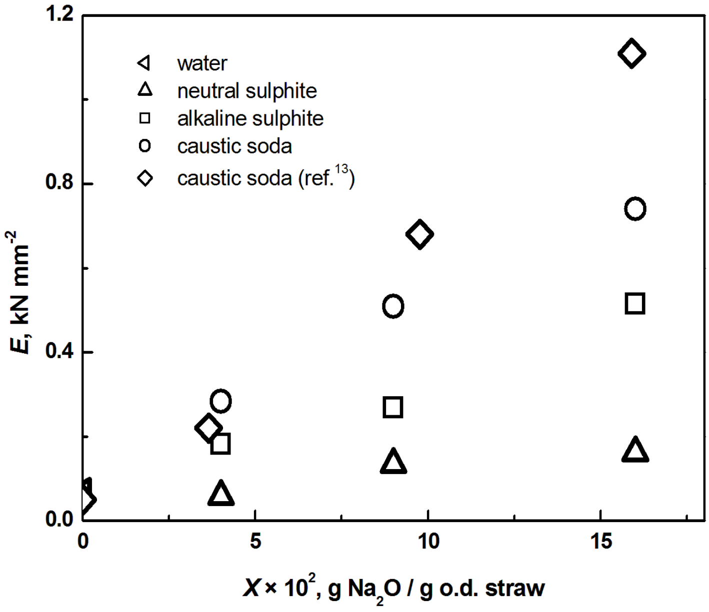

Figure 4: Influence of Na_2O-charge on the bending modulus of elasticity, *E*, in the region of reversible deformation

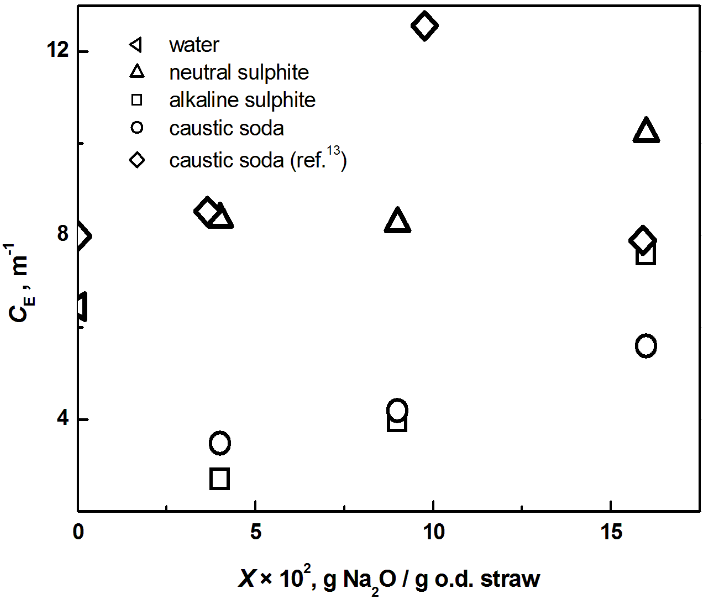

Figure 5: Maximum curvature, C_E, in the region of reversible deformation as a function of Na_2O-charge

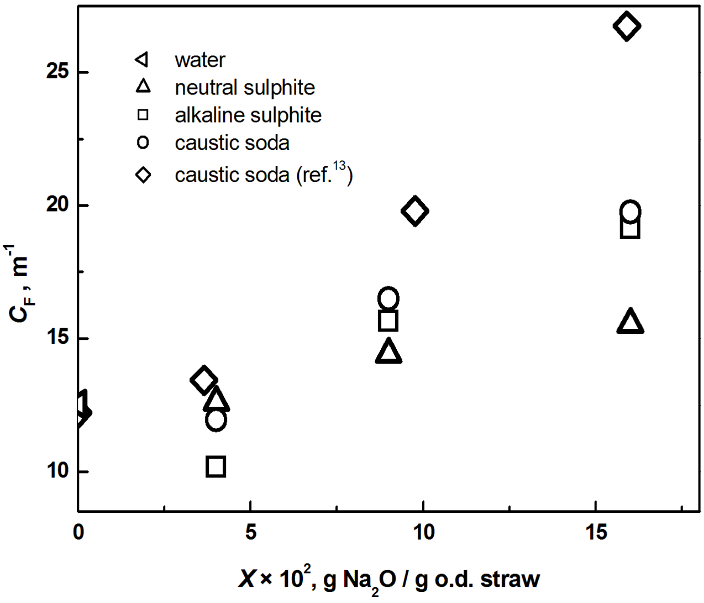

Figure 6: Curvature for tensile crack, C_F, as a function of Na_2O-charge

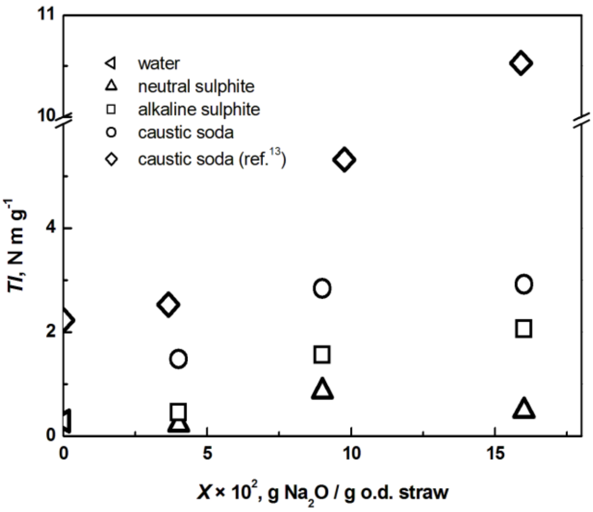

Figure 7: Tensile index, *TI*, as a function of Na_2O-charge

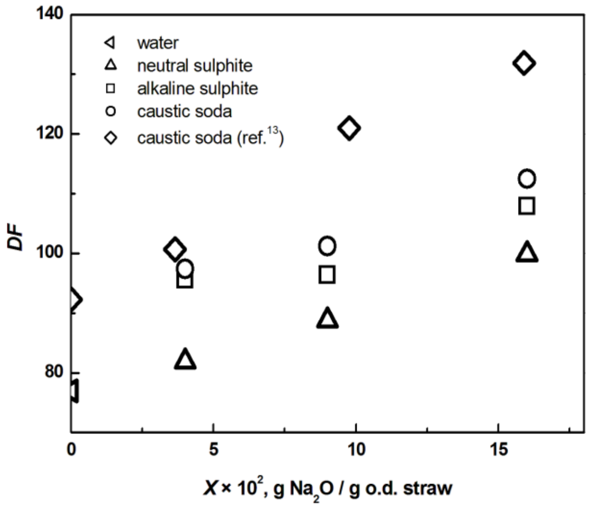

Figure 8: Degree of polymerization, *DF*, as a function of Na_2O-charge