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Physiochemical properties of differently pretreated cellulosic fibers

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ABSTRACT

Surface characteristics of differently pretreated flax fibers have been studied using electrokinetic analysis (EKA) and inverse gas chromatography (IGC). Semi-retted and retted flax fibers were scoured and bleached conventionally. All pretreated flax fibers showed negative zeta-potential at all pH values, indicating an acidic fiber surface. The isoelectric point was not reached at pH 3 and can be estimated to be near a pH of 2 or even below, a value where the fibers will be damaged. Inverse gas chromatography measurements showed differences in the surface energy among the differently pretreated flax fibers irrespective of the relative humidity. The group of retted flax fibers in general was found to have higher surface energies than the corresponding semi-retted group. On the other hand, within the same flax fibers group (retted or semi-retted), it was found that the surface energy decreases in the order bleached fibers > scoured fibers > raw fibers.

1. Introduction

Cellulosic fibers are treated to become more absorbent and easily wetted with water or other aqueous solutions, in a process known as scouring. The success of the subsequent wet processing operations like bleaching and dyeing depends on the efficiency of the scouring process (Abdel-Halim & Al-Deyab, 2011a, 2011b; Abdel-Halim, Fahmy, & Fouda, 2008; Abdel-Halim, Emam, & El-Rafie, 2008a; Abdel-Halim, Konczewicz, Zimniewska, Al-Deyab, & El-Newehy, 2010; Abdel-Mohdy, Abdel-Halim, Abu-Ayana, & El-Sawy, 2009; Fahmy & Abdel-Halim, 2010; Fouda & Fahmy, 2011; Hashem, Elshakankery, Abd El-Aziz, Fouda, & Fahmy, 2011). The constituents of cellulosic fibers mainly responsible for their hydrophobicity are localized in the cuticle of the primary wall, where the pectin acts as cementing material including waxes (Abdel-Halim & Al-Deyab, 2011c, 2011d; Abdel-Halim, Emam, & El-Rafie, 2008b; Abdel-Halim, Abdel-Mohdy, Al-Deyab, & El-Newehy, 2010; Abdel-Halim, Fouda, Hamdy, Abdel-Mohdy, & El-Sawy, 2010; Abdel-Halim et al., 2011; Hashem, Sokkar, Abdel-Halim, & Gamal, 2005; Hebeish, El-Rafie, Abdel-Mohdy, Abdel-Halim, & Emam, 2010; Sokkar, Abdel-Halim, Aly, & Hashem, 2004). The major goal of any scouring process is to improve the water absorbency of cellulosic fibers by removing such water repellent components from the fibers, which facilitates uniform dyeing and finishing. Efficient scouring process should result in considerable removal of both pectic and waxy substances.

The physicochemical properties of the fiber surface are very important because they govern the degree of adhesion of the reinforcing fiber to the polymer matrix (Abdel-Halim, El-Rafie, & Kohler, 2008; Asten, Veenendaal, & Koster, 2000; Jokinen, Mikkola, Matisons, & Rosenholm, 1997; Mukhopadhyay & Schreiber, 1995; Nardin, Balard, & Papirer, 1990).

The zeta potential of fiber materials can be determined with the help of the Helmholtz–Smoluchowski equation (Alvarez & Vázquez, 2006; Buschle-Diller, Inglesby, & Wu, 2005). Bismarck, Springer, Mohanty, Hinrichsen, and Khan (2000) investigated the change in time and pH dependence of zeta potentials of modified sisal, coir and of jute fibers.

The principle of IGC consists of characterizing the fiber material as stationary phase in a chromatography column with known probes at infinite dilution. The nature of probe's interaction with the fiber is solely dispersive. Thus, they yield a reference line when plotted according to the following equation (Gadhe, Gupta, & Elder, 2006; Santos & Guthrie, 2005):

$$RT \ln V_n = 2N(\gamma_S^d)^{1/2} a(\gamma_L^d)^{1/2} + C$$

where R is the gas constant, T is the temperature, N is the Avogadro's number, a is the surface area of adsorbate molecule, C is a constant, and γ_S^d, γ_L^d are the dispersive components of the surface energy of solid and liquid, respectively.

Natural fibers, especially bast fibers are considered as good candidate for replacing synthetic fibers in reinforcing plastics. This is due to the very good tensile strength of such fibers, their economic

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price in addition to their availability as renewable resources. The work presented in this paper aims at evaluating and comparing the surface energy of differently pretreated retted and semi-retted flax fibers and showing to what extent the chemical pretreatments (scouring and bleaching) improve the surface properties of such fibers. This evaluation is to be done using the streaming potential technique (zeta potential) and inverse gas chromatography (IGC).

2. Experimental

2.1. Flax fiber pretreatment

2.1.1. Materials

Semi-retted and retted flax fibers were taken from standard stocks of Institute of Applied Research (IAF) FH-Reutlingen. Rongal HT (reducing agent) and Alboron MBG 100 (non-ionic wetting agent) were supplied by Albon-Chemie Germany; Lavotan DSU (non-ionic wetting agent) and Contavan (H_2O_2-stabilizer) were supplied by CHTR. Beitlich GmbH Germany; sodium hydroxide, sodium carbonate and hydrogen peroxide were all laboratory grade reagents.

2.1.2. Scouring of flax fiber

Retted or semi-retted flax fiber was scoured in an aqueous solution containing 5 g/l NaOH, 3 g/l Na_2CO_3, 2 g/l Regonal HT (reducing agent) and 1.5 ml/l Alboron MBG 100 (wetting agent) using a material to liquor ratio of 1:10 (Tanapongpipat, Khamman, Pruksathorm, & Hunsom, 2008). The scouring was done using Ahiba Turbocolor Apparatus equipped with Ahiba Multiprocess controller mpc 600. The temperature was raised to 98 °C in 10 min and kept at this limit for 60 min. At the end of the scouring process, the fiber was given two boiling washings (5 min each) followed by one hot wash (5 min) and finally one cold wash (5 min).

2.1.3. Bleaching of flax fiber

Scoured (retted or semi-retted) flax fiber was bleached in an aqueous solution containing 6 ml/l hydrogen peroxide (35%), 1.25 g/l NaOH, 1.5 ml/l Contavan TIG (H_2O_2-stabilizer) and 1 ml/l Lavotan DSU (wetting agent) using a material to liquor ratio of 1:10 (Fillat, Pepió, Vidal, & Roncero, 2010). The bleaching was also done using the same Ahiba Turbocolor Apparatus. The temperature was raised to 90 °C in 30 min and kept at this limit for 60 min. At the end of the bleaching process, the fiber was given one boiling washings (5 min) followed by one hot wash (5 min), one cold wash (5 min), cold souring by acetic acid (5 min), one cold rinse (5 min) and finally air dried.

2.2. Zeta potential measurement

The zeta-potential was measured using the commercial Electrokinetic Analyzer EKA from Anton Paar, Austria. The Ag/AgCl-electrodes are perforated to allow the liquid to be pumped through the plug. Using the standard measurement method, the fibers are introduced into the cell, generally a glass tube. Before the fibers are put into the measuring cell, they are wetted and equilibrated in demineralized water. The fibers are then tightly compressed forming a plug between the two electrodes so that the required pressure difference across the cell (typically 350 mbar) is obtained. With each specimen the pH was varied in the range of 3–10. A reversible flow of electrolyte solution is pumped through the fiber-plug between two porous Ag/AgCl-electrodes.

Test parameters:

Electrolyte:	KCl solution 10^{-4} M
Repetitions:	4 measurements for each pH
pH control:	0.1 M HCl or 0.1 M KOH
Pressure:	350 mbar
Sampling time:	120 s

2.3. Inverse gas chromatography measurements

Gadgets: Inverse gas chromatography experiments were carried out on an SMS-IGC 2000 with FID & TCD Detectors. Columns used were SMS standard Glass columns 4 mm in diameter and 300 mm in length.

Chemicals: Measurements were undertaken with various non-polar and polar probe molecules, supplied by Aldrich, namely *n*-Hexane, *n*-Heptane, *n*-Octane, *n*-Nonane, *n*-Decane, Ethanol, Ethyl acetate and Acetone.

Test parameters:

Helium gas flow:	25 ml/min
Conditioning temperature:	34 °C

2.3.1. Procedure of analysis

The column was weighed before packing. The fibers were introduced into the column and well packed using a thin smooth-surface metal rod. The fibers ends outside the column were cut off and the column with the fibers inside was weighed to get the weight of the fiber sample. The column was then connected to the inverse gas chromatograph and the experiment parameters were set to condition the column for varying time intervals according to the desired relative humidity and then to inject the probes subsequently in the order of increasing vapor pressure. The probes were taken from the gas phase of each solute and the gas, not the liquid was injected. All surface energy experiments were carried out at 34 °C at 25 ml/min carrier gas flow rate. Calculations were performed using the SMS-IGC analysis software version 1.1. The retention time of nitrogen sets the systems dead time and after the measurement, the dead time was corrected, because the solutes were measured with FID detector while nitrogen was measured with TCD detector.

3. Results and discussion

3.1. Electrokinetic measurements

It has been accepted in literature that cellulosic surfaces generally show bipolar character with prevalent acidic contribution due to the proton of the hydroxyl functional group as well as of present carboxyl groups. The increase of pH lowers the zeta-potential indicating the gradual loss of protonated surface groups (Pothan, Simon, Spange, & Thomas, 2006). It has been further stated that the observed plateau region is the result of complete dissociation of accessible surface groups within a particular pH range. In the case of fibers consisting of pure cellulose to the most part as in scoured and bleached cotton, the plateau was approached by increasing negative potentials, indicating the dissociation of mostly acidic groups (Buschle-Diller et al., 2005). The zeta-potential versus pH curve has a shape that is typical for surfaces with hydrophilic character. All flax fibers (Fig. 1) show negative zeta-potential at all pH values. The negative zeta-potential values suggest the dissociation of Brønsted acid surface sites. The isoelectric point is not reached at pH 3 and can be estimated to be near a pH of 2 or even below, a value where the fibers will be damaged.

Regarding the untreated fibers, the retted material is more negatively charged than the semi-retted fibers, which have a very low charge. This suggests that the raw retted material exhibits more cellulosic surface than the raw semi-retted fibers. The zeta-potential of the retted fibers shows a rather constant value between −9 and

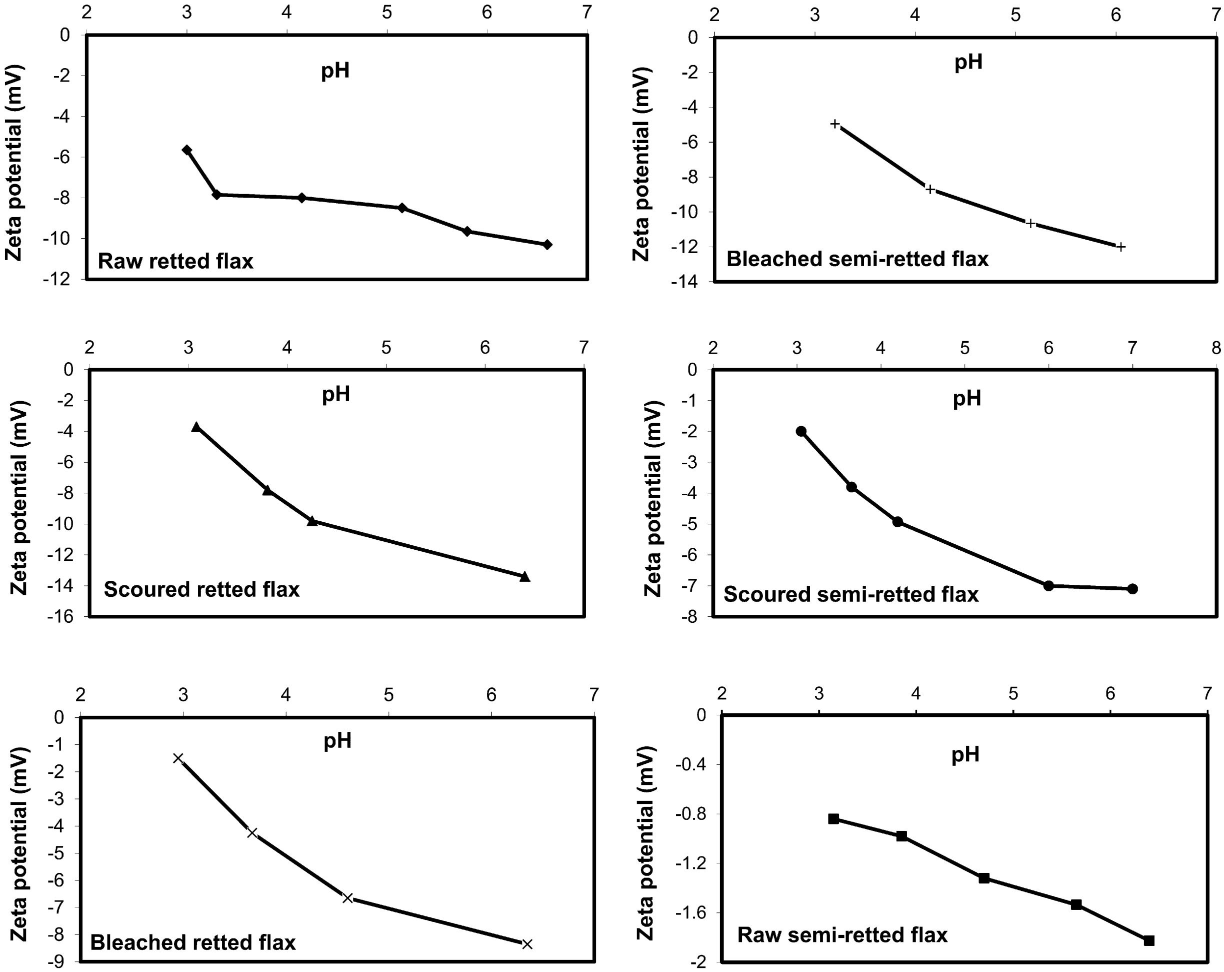

Fig. 1. Zeta potential of differently pretreated flax fibers.

−13.4 mV, independent of the purification. Also the slope of the curve zeta-potential vs. pH (Table 1) is not very different.

The semi-retted flax is quite different. It shows increasing negative charge with each cleaning step, the bleached semi-retted fibers with −12 mV finally ending at the same level as the retted samples. The slope of the curve increases likewise from 0.2 for the raw fibers up to 2.3 for the bleached. By removal of the hemicellulose, pectin and lignin, the surface exhibits more pure cellulose which determines the surface charge. Because of the higher content of the by-products this effect is more pronounced with the semi-retted fibers.

3.2. Inverse gas chromatography

The use of physicochemical parameters is a common and useful way for the description of fiber surface properties. Inverse gas chromatography provides a fast and accurate utility for the determination of these parameters. Due to its high sensitivity, inverse gas chromatography has advantages especially for measurements in the infinite dilution region compared to other techniques like tensiometry.

At these small concentrations only the highest energy sites of a heterogeneous solid surface interact with the probe molecules. Therefore, all parameters obtained in this range show the highest sensitivity to differences between materials. This can be applied to making distinctions between different products obtained from treatment of the same starting material. Typical examples are scouring and bleaching treatments which of course change the surface properties of the fibers due to removal of some fiber ingredients or modifying the whole structure through the blocking of some already existing function groups and/or the creation of new active sites.

In the case of an experiment with non-polar probe molecules, the dispersive surface energy of the fiber surface can be calculated, while using polar probe molecules, acid–base interactions can be investigated. The obtained free energy values even give a direct measure of the strength of interactions for different surface sites.

In this work, the non-polar probes used were *n*-Hexane, *n*-Heptane, *n*-Octane, *n*-Nonane, and *n*-Decane. Polar probe molecules used were Ethanol as an example for acidic probe molecules and Ethyl acetate and Acetone as examples for basic probe molecules. To address the influence of humidity on the results, these experiments were carried out at different relative humidity values namely 0%, 30%, and 70%. The details of experiments and theoretical background of the measurements are given in the text.

Table 1
Isoelectric point of differently pretreated flax fibers.

Flax samples	Estimated isoelectric point	Slope
Raw retted	0.6	1.1
Scoured retted	2.4	2.6
Bleached retted	2.4	1
Raw semi-retted	0.5	0.21
Scoured semi-retted	2.3	1.25
Bleached semi-retted	1.4	2.6

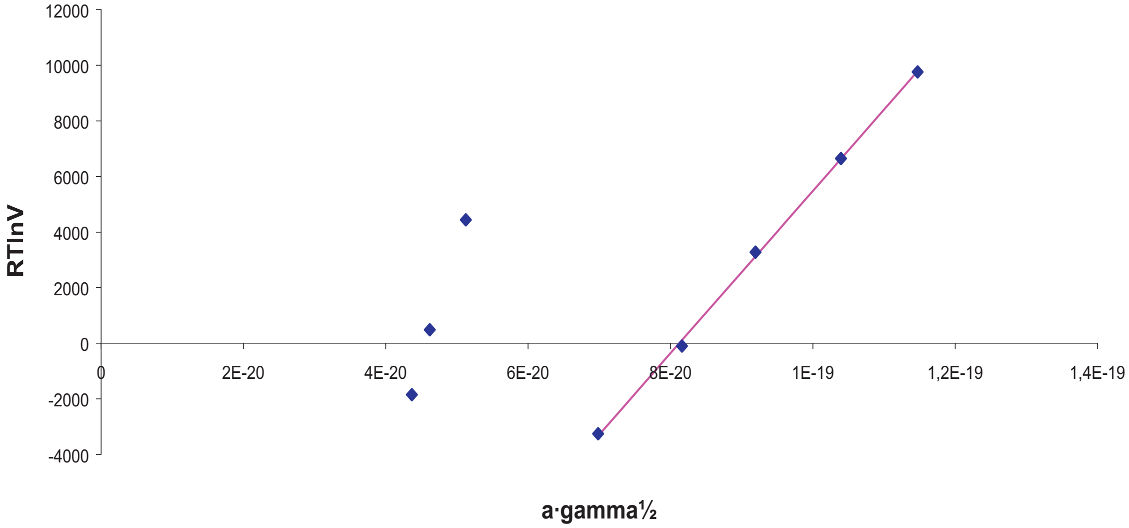

Fig. 2. Surface energy plots of the used polar and non-polar probe molecules.

Fig. 2 shows a general plot of $RT \ln V$ versus the cross sectional area times the square root of the dispersive component of surface tension $(a \times \text{gamma})^{1/2}$ of the used polar and non-polar probe molecules. This plot is computed for each flax fiber sample at a given humidity from which the dispersive surface energy as well as the specific free energy of the polar probe molecules was calculated. Mills, Gardner, and Wimmer in a recent paper measured the dispersive surface energy values for jute, silk floss, coir (Mills, Gardner, & Wimmer, 2008) and Cordeiro, Gouveia, and Jacobi John (2011) measured the dispersive energy of sisal fibers from South Africa. The variation in surface energy can be explained by the particular chemical composition of the fibers, namely the content of the aliphatic components of cellulose, hemicelluloses and lignin. The specific free energy of adsorption was calculated using the difference between the adsorption energy of the polar probe and its dispersive increment.

Fig. 3 shows the dispersive surface energy of raw, scoured, and bleached, retted and semi-retted flax fibers measured at different relative humidity. The figure shows differences in the surface energy among the different flax fibers irrespective of the relative humidity. The group of retted flax fibers in general has higher surface energies than the corresponding semi-retted group. On the other hand, within the same flax fibers group (retted or semi-retted), it was found that the surface energy decreases in the order bleached fibers > scoured fibers > raw fibers. This is of course expected because the retting as well as the successive treatments, like scouring and bleaching remove more and more impurities giving rise to an increase in the specific surface exhibiting more pure cellulose molecules. Therefore, more active sites are available for interaction. A comparison with the literature supports this fact. Thielmann and Reutenauer (2001) found that the surface energy of bleached cotton is higher than that of unbleached cotton. Dorris and Gray (1979) reported that the London force component of the surface free energy of cotton cellulose is higher than that of TMP fibers. The figure shows also that the dispersive component of the surface energy is almost independent from the humidity content of the fibers.

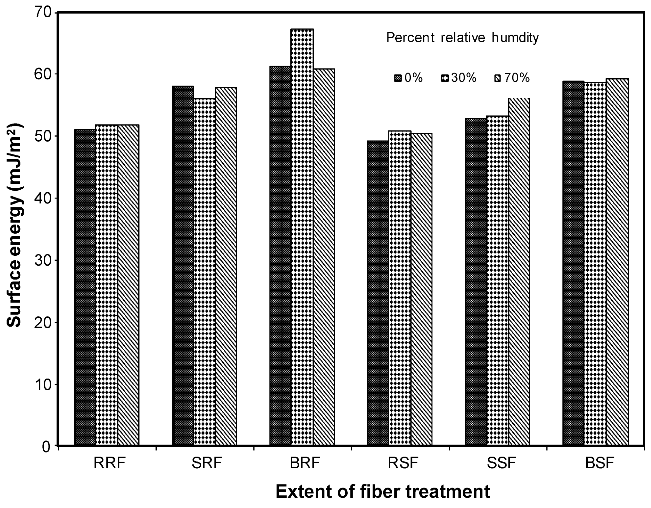

Fig. 3. Dispersive surface energy of differently pretreated flax fibers.

Fig. 4(a–c) shows the specific free energy of interaction for the polar probe molecules ethanol, ethyl acetate, and acetone, respectively. The specific free energies were determined for the differently treated flax samples at different humidity contents. The figures show the dependency of the specific free energy of the polar probe molecules on the percent humidity. In general, the specific free interaction energy increases by increasing the percent humidity for all polar probe molecules. This is quite reasonable because as the humidity increases, there is a greater chance of interaction between the polar probe molecules and the water molecules through hydrogen bonding. On the other hand, by comparing the specific free energy of the three polar probe molecules, it is remarkable that the values for ethanol are always higher than the corresponding values for both ethyl acetate and acetone, irrespective of the percent humidity, the extent of retting (retted or semi-retted flax fibers), and the nature of chemical treatment (raw, scoured, or bleached flax fibers). This can be interpreted in terms of the increasing probabilities of interactions between the hydroxyl group of the alcohol and that of the cellulose backbone of the flax fiber through the hydrogen bonding. This type of interaction is not so strong in the case of ethyl acetate and acetone and its effect can be limited to the week interaction of the lone pairs of electrons of the ester group oxygen. It is also observed from Fig. 4 that for each of the three polar probe molecules at the medium humidity percent (30%) there is no much difference in the specific free energy and it is nearly the same irrespective of the extent of retting and the nature of chemical treatment. It is also remarkable that in general there is only small increase in the specific free energy

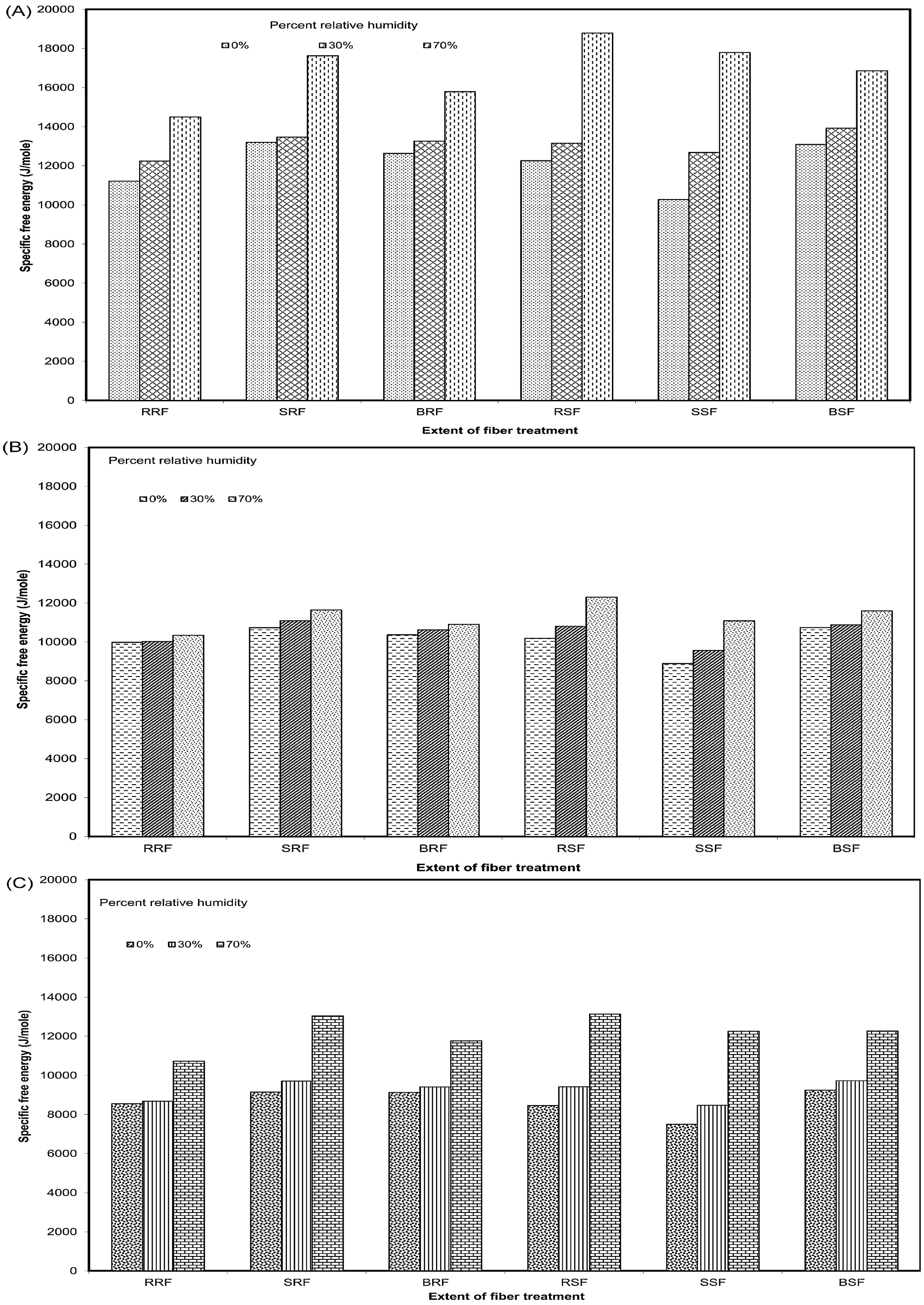

Fig. 4. Specific free energy of interaction for ethanol (A), ethyl acetate (B) and acetone (C).

by increasing the humidity from 0% to 30% while on the other hand, increasing the humidity from 30% to 70% is accompanied by a greater increase in the specific free energy. These phenomena could be understood from the sorption isotherm of flax (Fig. 5). It is clear from Fig. 5 (Dorris & Gray, 1979) that at the normal relative humidity up to (30–40%) the flax fiber reaches its equilibrium moisture content (about 4–7%). This moisture content is enclosed and distributed somewhere in the fiber structure, which means that these water molecules are not available for interaction with the polar probe molecules. At the high relative humidity of 70% the flax fibers are completely saturated and beyond the equilibrium moisture content there are water molecules available for interaction with the polar probe molecules. Referring to Fig. 4(a–c), it is also remarked that the specific free energies of the polar probe molecules at humidity 70% in all fiber samples decrease in the order Ethanol > Acetone > Ethyl acetate. This supports the finding that the specific free energy at the high relative humidity (70%) is mainly due to interaction between the polar probe molecules and the free water molecules and of course, ethanol with its hydroxyl groups has a higher tendency to form hydrogen bonding with the free water molecules than do acetone and ethyl acetate. Finally, having a look at the specific free energy values irrespective of the nature of the polar probe molecule and the percent humidity, one can easily observe that comparing the values for raw and scoured retted flax to the corresponding values for raw and scoured semi-retted flax, there is no correlation. This is simply because considering the case of raw retted and raw semi-retted flax, there is of course difference in the constitution of the two types of the fibers due to difference

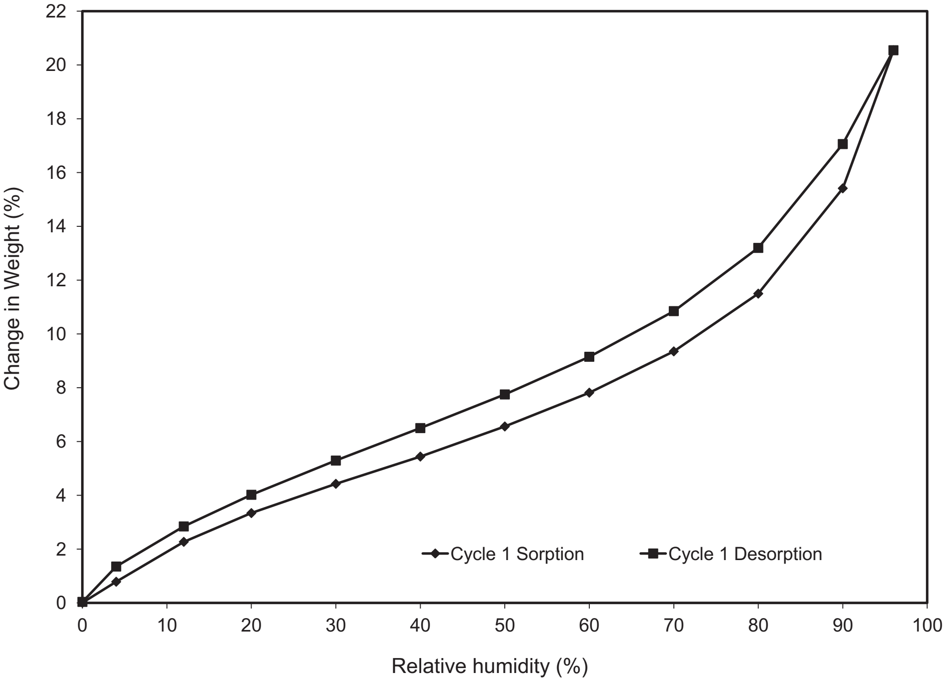

Fig. 5. Dynamic vapor sorption Isotherms of scutched retted flax at 25 °C.

in the extent of retting which leads of course to varying interactions with the probe molecules giving rise to varying specific free energy. Also considering the comparison between the scoured retted and scoured semi-retted flax fibers, one can also expect that the scouring effect will not be the same on the two types of fibers giving rise also to different constitution and accordingly different interactions with the polar probe molecules. What is interesting is that the specific free energy values irrespective of the nature of the polar probe molecule and the percent humidity were almost the same for both bleached retted and bleached semi-retted flax fibers, which indicates that after the bleaching process for both retted and semi-retted flax fibers, most of the non-cellulosic impurities were removed and only pure cellulose was left.

In order to confirm the above results, the specific free energy of the polar probe molecules were calculated once more based on the polarizability factor of the probes instead of the factor ($a \times \text{gamma}^{1/2}$) and the results obtained showed the same trend obtained in case of specific free energy calculated on basis of the factor ($a \times \text{gamma}^{1/2}$).

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