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ORIGINAL CONTRIBUTION

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Characterization of several modified jute fibers using zeta-potential measurements

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Abstract ζ-potential measurements using the streaming potential method were performed on several differently modified jute fibers. The time dependence of the ζ-potential measured in 1×10^{-3} M KCl solution offers the possibility to characterize the water-uptake behavior and the velocity constant of this process for almost all the jute fibers investigated. All the jute fibers contain, as expected, dissociable acidic surface functional groups as could be verified by measuring the pH dependence of the ζ-potential. Remarkably a peak (increase in the negative ζ-potential values) was detected while measuring the ζ–pH dependency. The origin of this peak is still questionable; however, it could be observed that this peak is a function of the degree of surface coverage of additional components, such as fats, waxes or grafted polymers, i.e. the accessibility of ether functions in the jute fiber surface.

Key words Jute fiber · Surface modification · Zeta-potential

Introduction

Jute, the "golden fiber" from India and Bangladesh is one of the most common agro-fibers, having a high tensile modulus and low elongation at break. If the low density (1.4 g/cm^3) of this fiber is taken into consideration, then its specific stiffness and strength are comparable to those of glass fibers [1–4]. The specific modulus of jute is superior to glass fiber, and on a modulus per cost basis, jute is far superior. The specific strength per unit cost of jute approaches that of glass fiber. On a weight and cost basis, jute fibers have been observed to be superior to glass fibers as composite reinforcements [5]. There are many reports about the use of jute as reinforcing fibers for thermosets and thermoplastics [5]. The structural aspects of jute fiber have also been reviewed [5]. The surface of jute fiber is one of the best instances of hydrophilic behavior induced by the predominance of -OH groups. The moisture content of jute is 12.6 wt% as reported by Bledzki et al. [6]. Due to the presence of hydroxyl and other polar groups in various constituents of jute (in the major component α-cellulose and in lignin and hemicellulose), the moisture regain is high, which leads to poor wettability with resin, weak interfacial bonding between the fibers and relatively more hydrophobic matrices. Surface modification of jute is a logical step to improve the hydrophobicity of the hydrophilic jute surface. Certain surface modifications would not only decrease moisture absorption but would concomitantly increase the wettability of fibers with resin as well as the interfacial bond strength, both critical factors for obtaining better mechanical properties of composites. The surface chemical modifications of jute involving bleaching, dewaxing, alkali treatment, cyanoethylation and vinyl grafting are made in view of their use as reinforcing agents in composites [7, 8]. We have observed better fiber–matrix interactions as a result of suitable surface modifications.

Electrokinetic (ζ-potential) measurements are well known as a useful method to characterize polymer surfaces (natural [9] as well as synthetic [10]). They have also been used to study the influence of different process parameters and surface treatments on the surface properties of textile [11] and synthetic fibers [12]. Since the presence of electrical charges often determines the interaction phenomena at interfaces, such as the reciprocation of dissolved ingredients (dyes, ions, enzymes, etc.) with solid surfaces, ζ-potential measurements can be used to characterize such processes. The variation of the composition of the electrolyte solution used for ζ-potential measurements provides the possibility to determine the surface characteristics, i.e. acidic or basic, polar or nonpolar (hydrophobic). The theoretically expected ζ–pH plots for such solids are shown in Fig. 1. Due to the known hydrophilic character of cellulose materials, ζ-potential measurements can be used to characterize the "wet" state of such materials [13, 14], which is of partial interest for applications such as paper manufacturing [15]. Results obtained from ζ-potential measurements also correlate with adhesive properties of fiber-reinforced polymers [16]. Adsorption processes that might also diminish the adhesive bond strength also affect the maximum ζ-potential (ζ_{max}) measured in KCl electrolyte solutions [17]. This phenomenon can be explained by the assumption that ζ_{max} is mainly influenced by dispersive forces acting at the surface, and these forces are also responsible for adhesion if specific interactions (such as hydrogen bonding) do not occur [17].

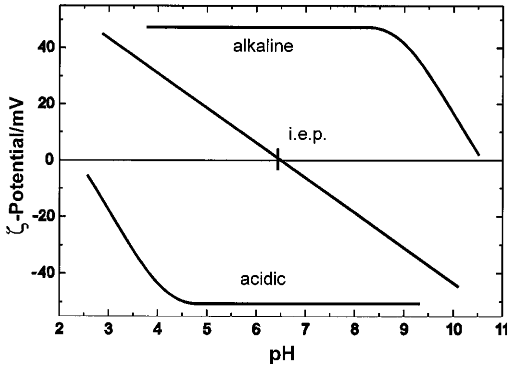


Fig. 1 General pH dependence of the ζ-potential showing the isoelectric point (*i.e.p.*)

Experimental

Materials and surface modification of jute fibers

Jute fibers were supplied by Konark Jute Mills (Dhanmandal, Orissa, India) and the bleached jute fiber was supplied by Bangladesh Jute Research Institute. The jute fibers from India were subjected to various surface modifications, such as dewaxing, alkali treatment, cyanoethylation and vinyl grafting. The chemicals copper sulfate, sodium periodate, sodium hydroxide, ethanol and benzene were of analytical grade and were used without further purification. The monomers acrylonitrile (AN) [18] and methyl methacrylate (MMA) [19] were purified prior to graft polymerization reactions with jute [7, 8].

The Indian jute fibers were washed with 2% Surf (commercially available washing powder, Lever, UK) detergent solution at 70 °C for 1 h, then washed overnight with tap water and finally thoroughly washed with distilled water and dried in a vacuum oven at 70 °C. Different surface modifications of detergent-washed jute were used for the ζ-potential measurements. The surface modification consisted of dewaxing the detergent-washed jute with a 1:2 mixture of ethanol and benzene for 72 h at 50 °C, followed by washing with distilled water. The dewaxed or defatted jute fibers were treated with 5% NaOH solution for 30 min at 30 °C, then washed again thoroughly with distilled water and then dried to obtain alkali-treated jute. The dewaxed jute fibers were subjected to graft polymerization with AN [18] and with MMA [19] as mentioned earlier. Jute samples with different percentages of grafted polymer were obtained by controlling the reactant concentrations, the time and the temperature of the graft polymerization reactions. Cyanoethylated jute samples were obtained by refluxing the dewaxed jute with AN, acetone and pyridine (as a catalyst) at 60 °C for 30 min, then the resulting modified jute was washed with acetic acid and acetone followed by washing with distilled water and finally vacuum drying [20].

ζ-potential measurements

An electrokinetic analyzer (EKA, Anton Paar, Graz, Austria) based on the streaming potential method was used to measure the electrokinetic properties of different modified jute fiber materials. The streaming potential was measured at 20 ± 1 °C as a function of the pressure decay (in the range 30–150 mbar) in a fiber bundle (capillary system) while the electrolyte solution (1×10^{-3} M KCl) was pumped through. The streaming potential, which arises due to the shear off the diffuse part of the electrochemical double layer, was measured using two Ag/AgCl electrodes. More details about this technique have been reported in the literature [21, 22].

Since the reproducibility of electrokinetic measurements depends strongly on the sample quality, every measurement of the pH dependence needs a well-defined starting point. This starting point is fixed by a "long-time" ζ-potential measurement [$\zeta = f(t)$]. In order to assure comparability care must be taken to use samples with similar preparation. Thus, we dried the fibers in a vacuum oven at 60 °C at 1 mbar over phosphorus pentoxide (Sikkapent) for 24 h prior to the measurements. To measure the time dependence of the ζ-potential for the fibers the analyzer was filled with a 10^{-3} M KCl electrolyte solution. The measuring cell, containing about 2 g fiber material, was connected to the analyzer, quickly rinsed with the electrolyte solution, degassed, and finally the measurement was started. The observed decrease in the ζ-potential with time was caused by water adsorption. This effect depends on the hydrophilicity of the solid [23]. One of the reasons for the decrease in the ζ-potential could be the swelling of the solid, which would lead to a transfer of the electrochemical double layer into the solid and, therefore, of the shear plane into the electrolyte solution. The diffuse part of the electric double layer would be withdrawing a mechanical or electrical interaction. Another cause could be the removal of water-soluble components from the surface (and/or bulk): this would also decrease the solid surface potential. According to Kanamaru [23] the quotient $(\zeta_0 - \zeta_\infty)/\zeta_0$ should correspond to the water uptake at a relative humidity of 100% of the solid investigated. The decrease in the ζ-potential as function of time due to the water uptake can be described as follows:

$$-\frac{d\zeta}{dt} = k(\zeta - \zeta_\infty) \ ,$$

which leads to

$$-\ln \frac{\zeta - \zeta_\infty}{\zeta_0 - \zeta_\infty} = kt \ .$$

k is a constant depending on the structure of the polymer investigated and is smaller below the glass-transition temperature of the polymer than above it [24].

The pH dependence of the ζ-potential of the natural fibers investigated was determined in 10^{-3} M KCl electrolyte solutions. This makes it possible to estimate the acidic and/or basic character of a solid surface. If the solid surface investigated contains acidic functional groups, the negative ζ-potential increases with increasing pH due to increasing dissociation of these groups and remains constant above a given pH due to their complete dissociation (Fig. 1). If basic functions are present at the surface investigated, the curves obtained show inverse behavior. The isoelectric point i.e.p. is also a measure of the acidity or basicity of a solid surface, if the dissociation of surface groups is the predominate mechanism for the formation of the electric double layer. The solid surface displays acidic character, if the value of i.e.p. is low. If i.e.p. is situated in the alkaline range, the solid surface contains basic surface groups [25].

The measurement of the ζ–pH dependence for hydrophobic solids will result in a plot without plateaus due to the absence of dissociating surface functionalities. The increase in negative and positive ζ-potential with increasing or decreasing pH is caused by the enhanced adsorption of hydroxyl ions or protons.

Results and discussion

During the time measurements of the ζ-potential, the negative (in some cases the positive) ζ-potential for all (exemplary) the jute fibers investigated decreases asymptotically from a value of ζ_0 (data point obtained directly after starting the measurement on a dried sample) on different time scales to a constant but smaller value ζ_∞ [ζ_∞ is the "plateau" value of the function $\zeta = f(t)$] as can be seen from Fig. 2 (and Table 1, stressing the main results). All the jute fibers investigated displayed a different ζ-potential time dependence, which should correlate with the different swelling behavior of the modified jute fibers. Another experimental effect on the function $\zeta = f(t)$ can be seen (more or less pronounced) in Fig. 2 (indicated by arrows). This is the influence of the measured ζ-potential on the direction of electrolyte flow (we call it "left–right dependence"). Preferred streaming channels, depending on the swelling of the fibers under investigation, must be formed inside the fiber porous plug placed in the fiber measuring cell due to software limitations (100 single measurements must be performed in one flow direction, indicated by arrows), causing the described effect [21].

On the other hand, in the case of the AN grafted fibers almost no time dependence of the ζ-potential could be observed (see also Fig. 2): the ζ-potential scatters around an average value. However, the "left–right dependence" is more strongly pronounced.

The quotient $(\zeta_0 - \zeta_\infty)/\zeta_0$ [calculated from the values obtained from the function $\zeta = f(t)$] (Table 1), which should correlate with the water uptake at 100% relative humidity, reveals the expected trend that the dewaxed jute fiber adsorbs the most water, followed by the alkali-treated jute fibers, the original jute [protected against water absorption by the natural wax layer consisting of non-water-soluble alcohols as well as several acids (palmic, oleaginous and stearic acids) [26]], and finally the MMA-grafted fibers (the higher the amount of grafted MMA, the lower the water uptake). In contrast

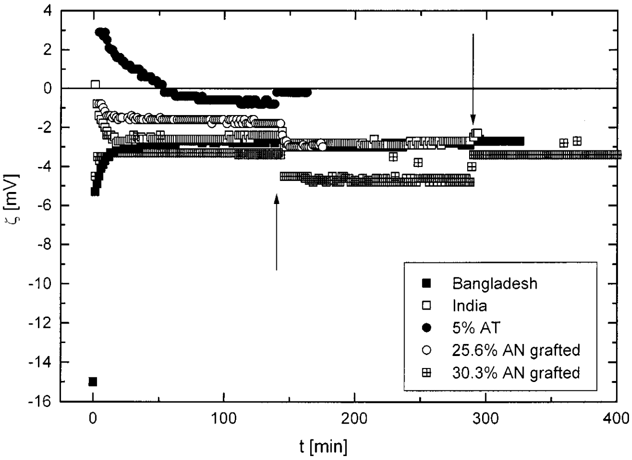


Fig. 2 ζ-potential time dependence of unmodified and several modified jute fibers, NaOH-treated (*AT*) and acrylonitrile (*AN*) grafted, measured in 1×10^{-3} M KCl

to synthetic polymers [27, 28] absorbing water, the velocity constant of the swelling process, k, is higher the more water the jute fibers adsorb.

The $\zeta = f(\mathrm{pH})$ function (Figs. 3–5) for several jute samples generally displays the expected trend, when the water adsorption process is finished, i.e. the material is swollen and therefore the electrical double layer is withdrawn the mechanical interactions, and all natural fibers (except the NaOH-treated jute) display an extremely small (for most of the fibers $\zeta_{\mathrm{plateau}} < -3$ mV) negative ζ-potential plateau (ζ_{plateau}) value. This small negative ζ-potential plateau value of the swollen material is due to the presence of carboxyl and hydroxyl groups. Stronger acidic functionalities are, for instance, present as uronic acid in hemicellulose, whereas weaker acidic groups can be found in the residual lignin [29]. The hydroxyl groups in cellulose were considered to be very weak acids [30]. Only the fibers with a high degree of surface coverage of grafted polyacrylonitrile (30.3%) and poly(methyl methacrylate) (12.5%) display a slightly

Table 1 Main results of the time dependence of the ζ-potential measured in 1×10^{-3} M KCl electrolyte solution. ζ_0 = ζ-potential value measured immediately after starting the measurement. ζ_∞ = ζ-potential value after establishing equilibrium. $(\zeta_0 - \zeta_\infty)/\zeta_0$ value should correspond to the water uptake at 100% relative humidity. k is the velocity constant of the swelling process

Jute fiber	ζ_0 (mV)	ζ_∞ (mV)	$(\zeta_0 - \zeta_\infty)/\zeta_0$	k (min^{-1})
Unmodified, dewaxed[1]	0.2	−2.6	14	0.226
Methyl methacrylate grafted 6.2%	−10.7	−2.5	0.77	0.009
12.5%	−25.5	−9.0	0.65	0.004
Bangladesh[2]	−15	−2.8	0.81	0.123
Alkali-treated 5%	2.9	−0.6	1.21	0.009

[1] Unmodified and dewaxed jute fibers were used as starting material for the (methyl methacrylate as well as the acrylonitrile) grafting reactions

[2] Original untreated jute fibers

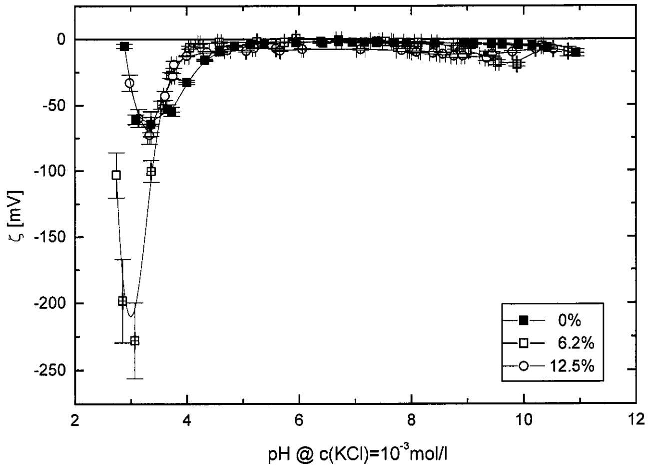
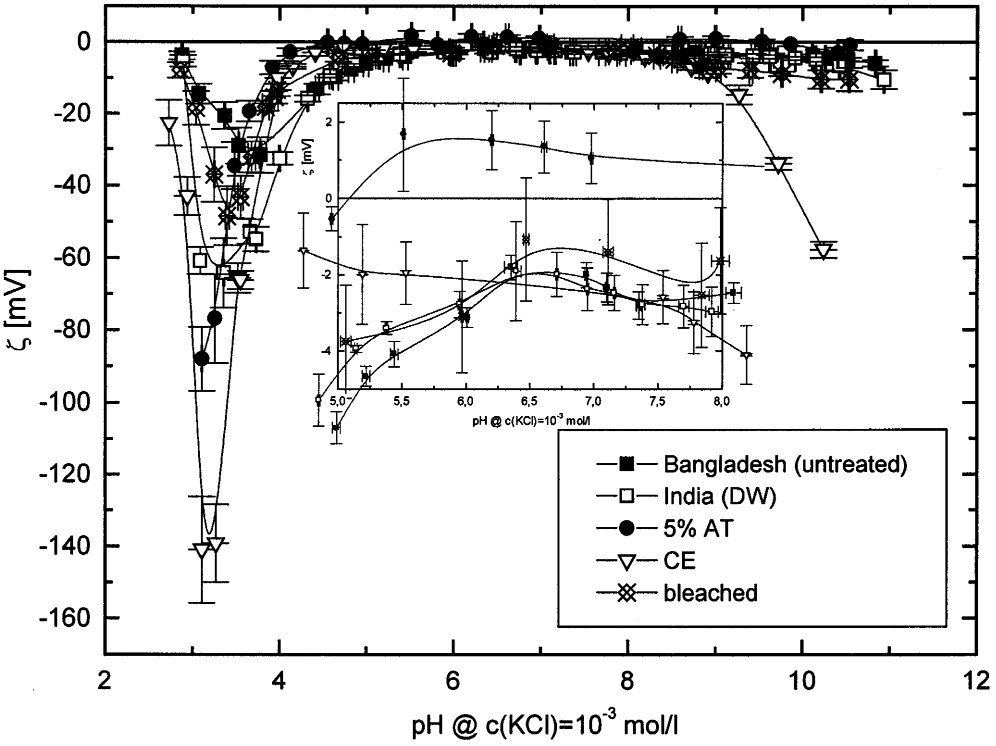


Fig. 4 ζ-potential pH dependence of untreated, DW and methyl methacrylate grafted jute fibers as a function of the percentage of grafted polymer

Fig. 3 ζ-potential pH dependence of untreated, dewaxed (*DW*), AT, cyanoethylated (*CE*) and bleached jute fibers



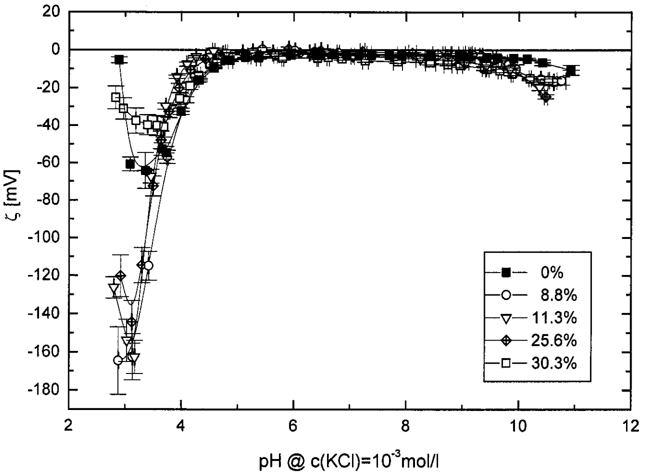


Fig. 5 ζ-potential pH dependence of untreated, DW and AN grafted jute fibers as a function of the percentage of grafted polymer

Table 2 Results of the ζ-potential pH dependence. $\zeta_{plateau}$ = ζ-potential plateau value at pH ≥ 7 and $\zeta_{pH,\ max}$ and $pH_{\zeta,max}$ are the characteristic values of the peaks apparent in the acidic range of the ζ-potential pH dependence

Jute fiber	$\zeta_{plateau}$ (mV)	$\zeta_{pH,max}$ (mV)	$pH_{\zeta,max}$
Untreated, dewaxed (0%)	−3.0	−64.2	3.4
Methyl methacrylate			
grafted 6.2%	−2.6	−228.0	3.1
12.5%	−8.5	−72.5	3.3
Acrylonitrile grafted			
8.8%	−2.4	−164.5	2.9
11.3%	−3.3	−162.7	3.2
25.6%	−1.5	−144.3	3.1
30.3%	−5.6	−40.8	3.7
Cyanoethylated	−2.6	−141.0	3.1
Bangladesh	−2.6	−31.6	3.8
Bleached	−1.1	−48.4	3.4
Alkali-treated 5%	1.1	−88	3.1

higher negative $\zeta_{plateau}$ value, which corresponds to the "decreased hydrophilicity" as becomes obvious from the ζ-potential time dependence (Fig. 2, Table 1). During the process of alkalization (also called mercerization) "alkali cellulose" is formed, which is a sodium alcoholate; the reaction probably takes place at each hydroxymethyl group [31]. This process leads to the small positive ζ-potential (Fig. 2) of the NaOH-treated fibers caused by the presence of "strongly" adsorbed alkali cations. The increase in the negative ζ-potential at higher pH values (pH > 8) as can be clearly seen for the cyanoethylated jute (Fig. 3) is very probably due to the dissociation of phenolic hydroxyl groups in the residual lignin on the fiber surface [32]; however, in the acidic part of the ζ-potential courses, a more or less pronounced peak becomes apparent and, therefore, no i.e.p. can be measured. It is obvious from these plots (Figs. 3–5), that the peak maximum of the negative ζ-potential ($\zeta_{pH,max}$) increases and shifts to lower pH ($pH_{\zeta,max}$) for the differently treated jute fiber materials compared to the original (Bangladesh) fiber (Table 2). One possible explanation of this peak might be the protonation of the ether groups either present in the lignin (around 12% [26]; however, it could be shown for kraft pulp fibers that the surface coverages of lignin and extractives are much higher than the average of the components in the fibers [29]) or cellulose (about 64.4% [26]) components of the jute fibers creating a positive surface charge, which then might induce the preferential adsorption of chloride ions. The more ether links there are present at the jute fiber surfaces the more pronounced is the peak. Starting from the original untreated jute (Bangladesh) fibers, whose surfaces are "protected" by natural wax layers, with the smallest peak at the highest pH value, all modifications leading to an increase in the maximum negative ζ-potential and a shift towards lower pH values indicate an increased number of protonable ether bonds. Regarding the grafting reactions (with AN and MMA) on dewaxed jute fibers one can observe a higher the degree of grafting, i.e. the higher the degree of surface coverage, the smaller the negative ζ-potential and the higher the pH needed to protonate "all" available ether functions.

However, this assumed "mechanism" is still questionable, since it has not been observed for other protonated surface groups, such as amines. Another possible explanation could be a pH-dependent variation of the surface structure of the fibers in contact with the electrolyte solution.

Only a few reports have been published on the influence of the composition (pH and salt concentration) of an electrolyte solution on the properties of cellulose-like materials [30]. Grignon and Scallan [33] investigated the effect of pH and neutral salts upon the swelling of cellulose gels. They found for sulfite and kraft wood pulps a significant decrease in the degree of swelling for these fibers at low pH (pH < 3) and very high pH (pH > 12) values in pure water, whereas this effect vanishes in 0.25 M NaCl solution. The swelling behavior is attributed to electrolytic effects primarily brought about by the concentration of nondiffusible ionizable groups in the fibers. This causes an exchange of mobile ions between the swelling fibers and the electrolyte solution, but because the groups are covalently bound to the fiber, the mobile ions are unevenly distributed; therefore, the swelling fibers contain a higher ion concentration, and the driving force of the swelling process is the reduction of the osmotic pressure due to the entry of water. Such a pH-dependent swelling process will, of course, also influence the fiber surface structure. On the other hand, the treatment of cellulose materials in dilute mineral acid results in the reduction in chain length of the cellulosic backbone of, for

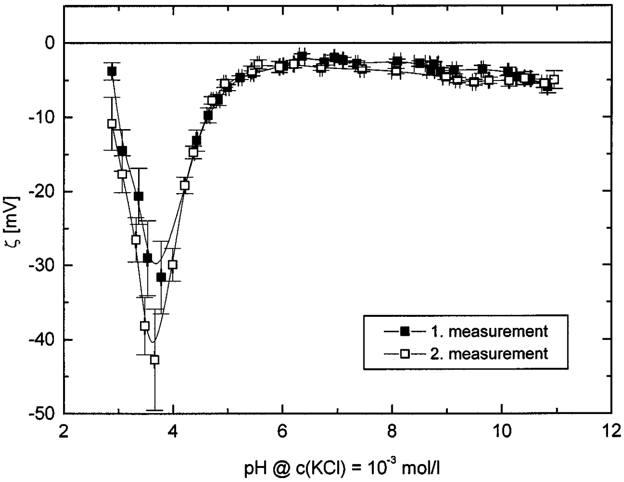


Fig. 6 Reproducibility of the ζ-potential pH dependence of Bangladesh jute fibers measured for different samples

example, jute fibers by random hydrolysis of the 1,4-glycosidic bond and, therefore, also results in a change in the chemical fiber surface structure. The hydrolyzed cellulose retains its fibrous structure but loses its mechanical strength significantly. The degree of surface modification depends upon temperature, the concentration of the acid solution as well as on the duration of contact. Jute fibers particularly remain fibrous even in strong aqueous sulfuric acid solutions and do not reduce to colloidal solutions unless subjected to severe mechanical action [34].

In order to check the reproducibility of the measured ζ-potentials, we decided to repeat the pH-dependent ζ-potential measurement since we only had a limited amount of fiber sample. As can be seen from Fig. 6 the curves obtained for the original jute fibers are the same within the error ranges, therefore, it can be concluded that the measured effects are real.

Comparing these pH-dependent ζ-potentials measured on jute fibers with the results obtained by Ribitsch and coworkers [35, 36] on unmodified, pure and differently treated cellulose fibers (pure cotton) no peak was measured in the acidic range. In contrast to the cellulose fibers, which were subjected to textile finishing processes in order to degrade, remove or complex non-cellulose compounds causing an increase in the negative ζ_{plateau} values due to the improved accessibility of anionic groups, the ζ_{plateau} values for differently modified jute fibers were more or less unaffected by the fiber modifications. However, also in the case of jute fibers the electrokinetic surface properties are mainly influenced by the non-cellulose components, for example, waxes (influencing the ζ-potential time dependence and diminishing the interaction between the fiber surface and the KCl electrolyte solution; therefore, smaller maxima of the negative ζ-potential in the pH dependence at low pH values were measured).

Conclusion

Several modified, such as alkali-treated, polymer-grafted, bleached and cyanoethylated jute fibers were characterized using ζ-potential measurements. The ζ-potentials were measured as function of time and of the pH value of a 1×10^{-3} M KCl solution. Using the approach of Kanamaru it was possible to estimate the relative amount of water uptake and the velocity constant of the swelling process. Removal of the fatty substances or waxes from the fibers increases the relative amount of adsorbed water. Grafting of poly(methyl methacrylate) onto the jute fibers decreases the amount of adsorbed water.

As could be shown from the pH dependence of the ζ-potential almost all the jute fibers, except the NaOH-treated jute, display a relatively small negative ζ_{plateau} value in the alkaline part due to the presence of dissociable acidic surface functional groups. The ζ_{plateau} value increases with "increasing hydrophobicity", which is in accordance with the measured ζ-potential time dependence. However, all the jute fibers show a pronounced peak in the measured acidic range. We assume that this peak is caused by the protonation of the ether linkages contained in the cellulose or lignin component of the fibers followed by preferential chloride ion adsorption. The jute fiber surface modifications influence this process significantly.

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