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EFFECTS OF PLASMA TREATMENT ON WATER SORPTION IN VISCOSE FIBRES

UČINKI PLAZEMSKE OBDELAVE NA SORPCIJO VODE V VISKOZNIH VLAKNIH

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We investigated water sorption in viscose nonwoven fibres manufactured by Tosama d.d. with the surface density of 175 g/m^2. A comparison between untreated fibres and by oxygen plasma treated fibres was made using optical polarization microscopy. Plasma treatment was done for 10 minutes at pressure of 75 Pa at current of 250 mA at the power of 500 W. Swelling was characterized by measurements of fibre diameter. Modifications of intensity of the polarized light transmitted through the fibre were measured as a function of time of exposure to water. Characteristic swelling and intensity modification times were resolved for untreated and oxygen plasma treated fibres. The swelling time of oxygen plasma in comparison to untreated plasma is reduced by the factor of 0.54 and intensity change time by the factor of 0.4. From the characteristic swelling and intensity change times it was concluded that oxygen plasma treatment of viscose increases the speed of water sorption.

Keywords: plasma treatment, viscose, swelling, optical polarization microscopy

Proučevali smo sorpcijo vode pri vlaknih v viskozni kopreni, ki jo proizvaja podjetje Tosama d.d., s površinsko gostoto 175 g/m^2. Primerjali smo neobdelana in s kisikovo plazmo obdelana vlakna z uporabo optično-polarizacijske mikroskopije. Plazemska obdelava je trajala 10 minut pri tlaku 75 Pa, toku 250 mA in pri moči 500 W. Nabrekanje viskoznih vlaken smo opazovali preko sprememb premera vlakna. Z optično polarizacijsko mikroskopijo smo merili spremembe intenzitete polarizirane svetlobe, ki potuje skozi vlakno med močenjem. Iz meritev na neobdelanih in s plazmo obdelanih vlaknih smo ocenili karakteristične čase nabrekanj vlaken in spremembe intenzitete polarizirane svetlobe, ki potuje skozi vlakna. Pri s plazmo obdelanih vlaknih se časi nabrekanja zmanjšajo za faktor 0.54 in čas spremembe intenzitete za 0.4. Iz meritev smo zaključili, da obdelava s kisikovo plazmo pohitri proces sorpcije vode.

Ključne besede: plazemska obdelava, viskoza, nabrekanje, optična polarizacijska mikroskopija

1 INTRODUCTION

The importance of viscose fibres in medical, sanitary, textile and construction industry is vastly increasing. Viscose fibres are often chemically treated to improve their wettability. Especially the surface layers of fibre structure have to be modified to achieve higher uptakes of liquids.[1] Chemical treatment of fibres to alter their physical properties can be ecologically questionable and expensive. An environmental friendly alternative to chemical treatment is plasma treatment of viscose veils.[2] Viscose nonwoven exposed to oxygen plasma exhibit modified surface layers in comparison to untreated viscose nonwoven fibres.[3–7] The same applies to numerous other textiles as well as organic materials. [8–13] Surface and internal structure modifications are reflected in altered physical and sorptive properties of material that are very important for production of napkins, tampons, tissues, waddings and other sanitary materials. Viscose contains high level of crystalline structure and some amorphous regions, which are responsible for the accessibility of water binding groups.[14] To increase the performance of viscose fibres a variety of chemical compounds is used to introduce new functional groups or compounds to the fibres.[15]

Current fibre treatment analysis focuses mainly on measurements of properties like water retention, water contact angle, braking force and elongation[3]. We focused mainly on changes of optical properties, which can be related to fibre treatment.[16–21] Optical polarization microscopy (OPM) is a contrast enhancing technique that improves the quality of the images of birefringent materials.[22,23] OPM uses the concept of two polarizers oriented at right angles with respect to each other, commonly termed as crossed polarization. The most notable application of OPM is to examine birefringent or doubly refractive specimens. To determine quantitative aspects of the observed specimen with birefringent properties, the optical axis of the sample should be rotated at the angle of 45° with respect to the analyser to achieve maximum brightness. The surrounding isotropic material remains dark, providing basis for the analysis of birefringent material only.[24]

Our aim was to analyse the light transmission changes trough fibre and swelling using highly sensitive recording camera.

2 EXPERIMENTAL METHODS

The material used was a Tosama d.d. viscose nonwoven fibre with the average nonwoven sample surface density of 175 g/m^2.[3,4] The fibres were stored in a sealed environment.

2.1 Oxygen plasma treatment

Viscose non-woven samples were mounted into the discharge chamber of a plasma system. The chamber is pumped with a two stage rotary pump with a pumping speed of 60 m^3/h. The chamber was pumped down to the ultimate pressure which was about 10 Pa. Weakly ionized plasma was created in the discharge chamber by an inductively coupled radiofrequency (RF) discharge.[25–27] The RF generator operates at a frequency of 27.12 MHz and nominal power up to 5000 W. The power absorbed by plasma is adjustable by a matching unit. At these particular experimental conditions the absorbed power was about 500 W. The plasma parameters were estimated by electrical and catalytic probes.[28–32] When oxygen is applied as the working gas and the pressure in the system is 75 Pa, the electron temperature is about 3 eV, the density of charged particles (predominantly O_2^+) is about 1×10^{15} m^{-3} and the dissociation fraction of oxygen molecules is about 10%. Samples were kept in plasma for 10 min. The nonwoven sample was then sealed and stored in plastic containers at room temperature until used in experiments. An untreated viscose nonwoven was also sealed and stored under the same condition until used in experiments.

2.2 Optical polarization microscopy imaging and analysis

Optical polarization imaging was done with a Nikon Polarizing Microscope Optiphot2-pol. Viscose fibres were fixed between object and cover microscopy glass to enable stable focusing of a specific part of the fibre or the entire fibre depending on the magnification. The glass plates were glued by epoxy two component glue leaving small holes for water injection. For long term measurements these holes were sealed with a silicon lubricant to prevent water leakage and evaporation. High resolution video recordings of the time evolution of wetting process were captured by the high resolution video camera (PixeLINK Monochrome Machine Vision Camera) that enables recording 8 frames per second. The recordings were made in two regimes: cross polarizer-analyser configuration and parallel polarizer-analyser arrangement. The fibre axis was oriented at 45° with respect to the polarizer direction to get the maximal intensity output even during cross-polarization measurements. When the fibre was put in contact with water, the intensity of the transmitted light changed, which was ideal for quantitative analysis of kinetics of the water sorption process. The recorded high resolution movies were decomposed into frames, followed by frame to frame analysis of the transmitted intensity values of a selected region of the fibre. To enable statistical analysis several fibres were analysed and for each fibre several regions were investigated to avoid any anomalies that can occur due to the morphological inhomogeneity in some fibre parts. Frame by frame intensity analysis was made by using Matlab software environment. The exponential growth function was used in fitting procedures to obtain characteristic time of the wetting process. Swelling changes were determined by usage of ImageJ programme.

3 RESULTS

In **Figure 1** an example of the time evolution of the swelling process of an untreated viscose fibre is presented from 8 selected frames. Fibre diameter was determined and a relative radius was plotted versus time the fibre was exposed to water. **Figure 2** shows time evolution curves of the relative radius values for the treated and untreated viscose fibres.

Figure 3 shows the wetting process of a viscose fibre. The selection squares indicate the image area where the normalized intensity of transmitted light was calculated from.

Frame by frame intensity values provide information on kinetics of the sorption process. In **Figure 4** the normalized intensity versus wetting time is presented for untreated and plasma treated viscose fibres.

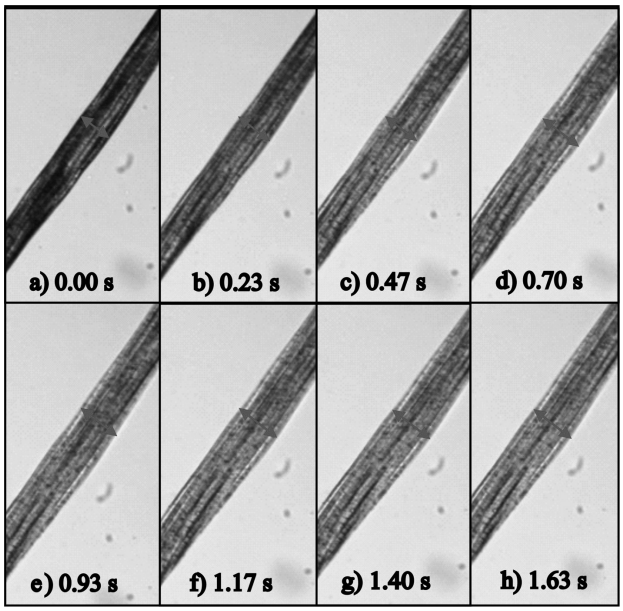

Figure 1: Observation of the wetting process under parallel polarizers. The dependence of fibre diameter versus time, when the untreated fibre is exposed to water. The image sequences a)–h) display the swelling of the fibre after introducing water at t = 0.00 s.

Slika 1: Premer viskoznega vlakna v odvisnosti od časa, ko je vlakno izpostavljeno vodi. Zaporedje slik a)–h) prikazuje nabrekanje vlakna z začetkom močenja ob t = 0.00 s.

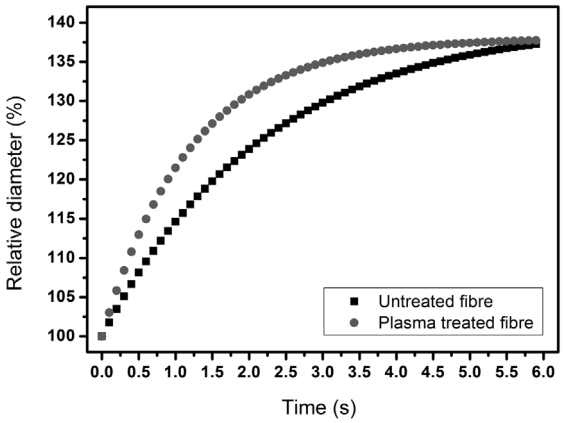

Figure 2: The relative diameter of viscose fibre versus the time, when water was introduced to the fibre for untreated and plasma treated viscose fibres

Slika 2: Relativni premer viskoznega vlakna v odvisnosti od časa, ko je vlakno izpostavljeno vodi za neobdelana in s plazmo obdelana vlakna

To obtain characteristic swelling times for treated and untreated viscose fibres a fitting function of exponential saturation was used:

$$R(t) = R_0 + \Delta R(1 - e^{-t/\tau_R}) \quad (1)$$

where the $R(t)$ represents the relative radius at time t, R_0 = 100 % is the relative radius of a dry fibre, the ΔR represents the relative changes of radius before and after the wetting process takes place and the τ_R represents the time in which the fibre becomes swollen.

To obtain characteristic intensity changing times for treated and untreated viscose fibres a fitting function of exponential saturation was used:

$$I(t) = \Delta I(1 - e^{-t/\tau_I}) \quad (2)$$

where the $I(t)$ represents the normalized intensity at time t, the ΔI represents the normalized changes of intensity before and after the wetting process takes place and the τ_I represents the time in which the intensity changes.

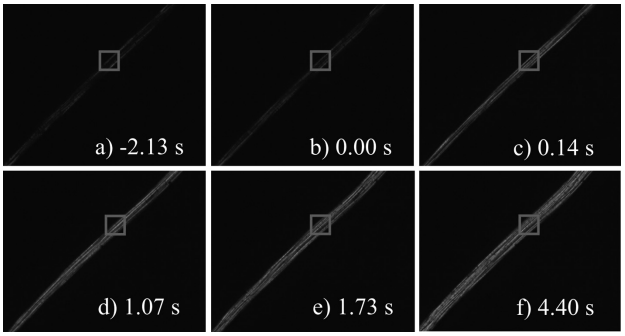

Figure 3: Image sequence a)–f) attained in cross-polarized. At time 0.00 s the water was introduced to the fibre. The squares represent the areas for normalized intensity calculation of the light transmitted through the fibre.

Slika 3: Zaporedje slike viskoznega vlakna a)–f). Polarizator in analizator sta bila prekrižana. Ob času 0.00 s je vlakno bilo izpostavljeno vodi. Okviri na slikah predstavljajo površino, ki je bila izbrana za izračun normalizirane intenzitete svetlobe, ki je potovala skozi vlakno.

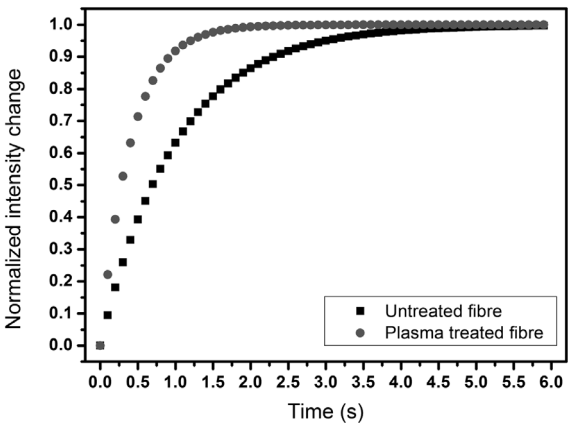

Figure 4: The normalized intensity of the polarized light that was passing through the viscose fibre versus the time, when the water was introduced to the fibre for untreated and plasma treated fibres.

Slika 4: Normalizirana intenziteta polarizirane svetlobe, ki je potovala skozi viskozno vlakno v odvisnosti od časa, ko je bilo vlakno izpostavljeno vodi. Slika prikazuje normalizirano intenziteto za neobdelana in s plazmo obdelana vlakna.

In **Table 1** the average values of τ_R and τ_I are displayed. They were calculated from data obtained in analysis from graphs, such as in **Figure 2** and **Figure 4**.

Table 1: Characteristic response times for untreated and plasma regenerated viscose fibres when exposed to water

Tabela 1: Karakteristični odzivni časi za neobdelana in s plazmo obdelana vlakna, ko jih začne močiti voda

Fibre treatment	Untreated	Plasma
τ_R (s)	2.2 ± 0.8	1.2 ± 0.2
τ_I (s)	1.0 ± 0.3	0.4 ± 0.1

4 DISCUSSIONS

The viscose fibre consists of micro fibrils. The water sorption is dependent on the accessibility of amorphous regions to the water, and the water accessibility to the space between the micro fibrils.[18] The content of carboxyl group increases with plasma treatment which increases the wettability of the fibre.[33] Fibre morphology due to the sub-fibre structure makes the fibres optical properties more complex.[23] The intensity of the light transmitted trough the fibre in crossed-polarized regime changes during wetting, since water sorption changes fibre's optical properties. Experimentally obtained characteristic intensity change times indicate the velocity of internal structure change while the water is introduced to the fibre. The swelling of the fibre is also an indicator for water sorption.[18] As water is absorbed, the water bound into cell wall exerts a swelling pressure, resulting in increase of the volume of the material.[34] The difference between swelling times and intensity change times indicate that we have to differentiate between internal changes of optical properties in the fibre and swelling during water sorption. Namely the sorption process

results in several modifications of fibre mechanical and optical properties.[18] Oxygen plasma treatment modifies the fibre's surface, making it more susceptible to faster water sorption.[33] The changes of properties of modified viscose fibres with chemical treatment like bleaching and slack mercerization can be compared with plasma treatment modifications.[35] Comparable increase of hydrophilicity in comparison to chemical treatment was achieved by plasma modifications of viscose non-woven fibres. Measurements of contact angle, surface energy and polar interactions were performed in previous research to compare plasma treatment effects with different chemical treatment for sorption of water and other fluids.[35] Water retention and improvement of some of the physical properties after oxygen plasma treatment was also studied.[3] On the basis of the experiments described in previous research in addition to this this article it can be stated that oxygen plasma treatment is a viable alternative to chemical treatment of fibres to increase the kinetics of water sorption.[3,18,33,35]

5 CONCLUSIONS

Oxygen plasma treatment of viscose fibres increases the kinetics of water sorption, making it a promising candidate for substitution of chemical treatment. The swelling time of oxygen plasma in comparison to untreated plasma is reduced by the factor of 0.54 and intensity change time by the factor of 0.4. Findings, obtained with the optical polarization microscopy approach, agree with the other research done on comparison between chemical and plasma treatment of viscose fibres, making plasma treatment an alternative procedure of viscose nonwoven modification in sanitary and medical applications, which require faster water sorption kinetics.

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