Cotton/polyester textiles as a possible source for viscose and cupro fibers production

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The textile industry is confronted with several types of waste. Regarding this problem, waste towels made of 100 % cotton and waste shirts made of 60:40 % cotton/PES way the possibility of converting cotton and cotton-polyester blends waste into regenerated fibers was estimated. Preparation of viscose under laboratory conditions at the selected viscosity of the spinning solution was possible even if the input raw material was cotton shirts in combination with polyester which is not the case with cupro.

Key words: *waste textile, viscose, cupro, regenerated cellulose*

1. Introduction

The textile industry is confronted with several types of waste that have been a problem for environmentalists for years [1]. There are many items of clothing that are fashionable for a short period of time, so they are soon discarded [2]. The result is the stockpiling of large quantities of textile materials, which soon become waste and are disposed of in landfills. The disposal of textiles is ecologically very controversial [3], since the splitting life of textile material is very long, especially in the case of clothing made of synthetic fiber-forming polymers or their mixture with natural fibers. In the context of this problem, legislation on the re-use and recycling of textile materials is becoming increasingly demanding [4]. Examples are textile combustion, which enables energy to be generated [5], or the production of technical textiles for the needs of various branches of industry. Recently, so-called social enterprises have also been set up to collect various types of waste [1]. The use of old clothes is also possible in the form of so-called second-hand shops [1, 6]. One of the approaches to textile material recycling is the production of regenerated cellulose [7, 8].

Within this research, the cotton waste and that in the blend with polyester (PES) were selected as a possible source for the recycling of textile material to produce viscose and cupro under laboratory conditions. Waste towels made of 100 % cotton and waste shirts made of 60:40 % cotton:PES were used for the preparation of both types of regenerated cellulose. The main purpose of the research was to evaluate the possibility of converting cotton and cotton-polyester blended waste into regenerated fibers.

The possible use of these types of regenerated cellulose is in the manufacture of filters for dry filtration, e.g. air conditioning filters, cigarette filters or, due to the poor mechanical properties of regenerated cellulose when wet. In addition, activated carbon was added to the individual regenerated cellulose in order to test the possibility of adding compounds with good adsorption properties [9] to bind toxic compounds if regenerated celluloses are intended for e.g. filtration purposes.

The rotational viscometer Fungilab was used to optimize the viscosity of the spinning bath. In addition, regenerated celluloses were evaluated using ATR IR FT spectroscopy and scanning electron microscopy (SEM). Based on the results of these methods, information on functional groups of the newly formed regenerated celluloses and the surface topography was obtained.

2. Materials and methods

2.1. Materials

Towels made of 100 % cotton; surface mass of 340 g/m² and shirts made of the blends of cotton and polyester in the ratio 60:40; mass per unit area of 120 g/m²; the weave plain, was used to produce viscose in cupro.

2.2. ATR FT- IR spectroscopy

The measurements were carried out with apparatus Perkin Elmer 1600 under the following conditions: Resolution 4, scan 16; each spectrum was recorded 15 times over the wavelength interval between 4000 cm^{-1} and 650 cm^{-1} with a resolution of 4 cm^{-1}.

2.3. Viscosity measurements

The viscosity of xanthate and a complex of cellulose cuoxam solution was measured with a rotational viscometer (Model Fungilab S. A., Spain).

2.4. Scanning electron spectroscopy (SEM)

Based on SEM (Karl Zeiss Supra, model 35 WP/I) the surface topography of regenerated cellulose was evaluated [10]. The samples were coated with a thin film of conductive material (Au) before scanning.

2.5. Samples preparation

2.5.1. Regenerated cellulose trough viscose process

Towels and shirts (input cellulose) were cut into small pieces. 1.5 g of these pieces were transferred to a 100 mL Erlenmeyer flask, where they were treated with 7.5 mL of an 18 % aqueous solution of sodium hydroxide (NaOH) at room temperature to swell the (input) cellulose and convert it to alkali cellulose (Fig.1) according to the following chemical reaction eq. (1)

$$(C_6H_{10}O_5)_n + nNaOH \rightarrow \rightarrow C_6H_9O_4ONa)_n + nH_2O \quad (1)$$

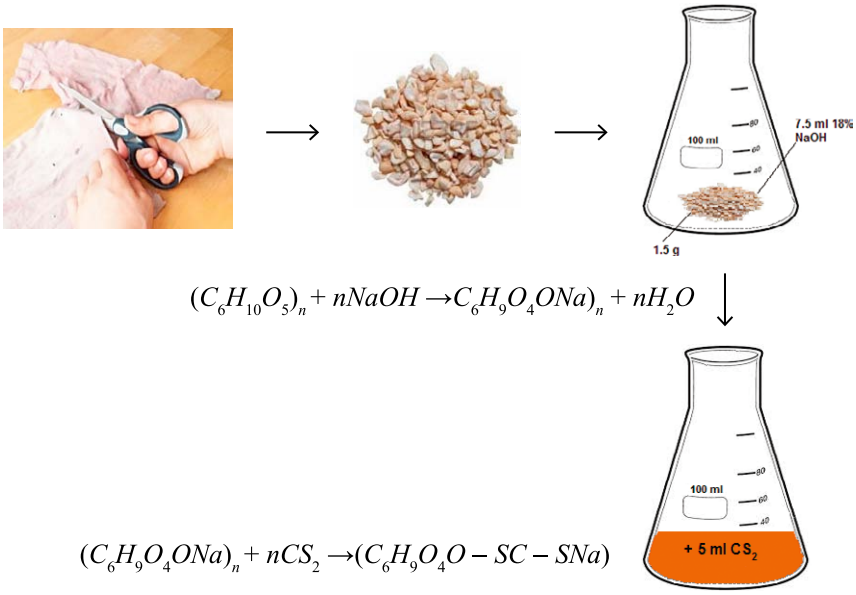

Fig.1 Scheme of preparation regenerated cellulose trough viscose process: a) cutting of waste textiles into b) small pieces, c) converting celllose to alkali cellulose by 18 % NaOH and d) xanthation with carbon disulphide (CS_2)

Than the so-called pre-ageing process was abandoned, and the next step was xanthation (Fig.1d). In this step of viscose preparation, the alkali cellulose was poured with 5 mL carbon disulphide (CS_2) at room temperature and mixed well with the help of a glass rod. In this way, cellulose xanthate was formed according to chemical reaction eq. (2).

$$(C_6H_9O_4ONa)_n + nCS_2 \rightarrow \rightarrow (C_6H_9O_4O - SC - SNa) \quad (2)$$

The orange cellulose xanthate (Fig1d) was then dissolved in dilute sodium hydroxide ($w_{(NaOH)}$=2.5 %) at room temperature under high shear mixing conditions to obtain a viscous orange solution, called 'viscose', which is the basis for the manufacturing process. The orange color of the xanthate appears to be due to side reactions that occur together with the conversion of alkali cellulose to cellulose xanthate. This dyed solution was then subjected to the so-called aging phase, i.e. resting at room temperature for 24 hours, to depolymerize the cellulose, resulting in a reduction in the average molecular weight of the original pulp. The reduction of the cellulose results in a viscous solution

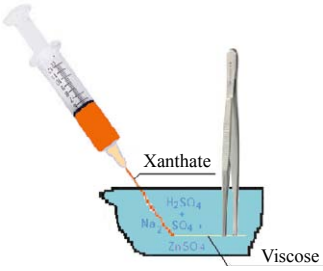

Fig.2 Scheme of viscose wet spinning by pressing xanthate through a hypodermic needle into a spinning bath

with the right viscosity. Any air bubbles trapped in the solution must be removed before extrusion to avoid voids or weak spots in the fine viscose filaments, which is called degassing.

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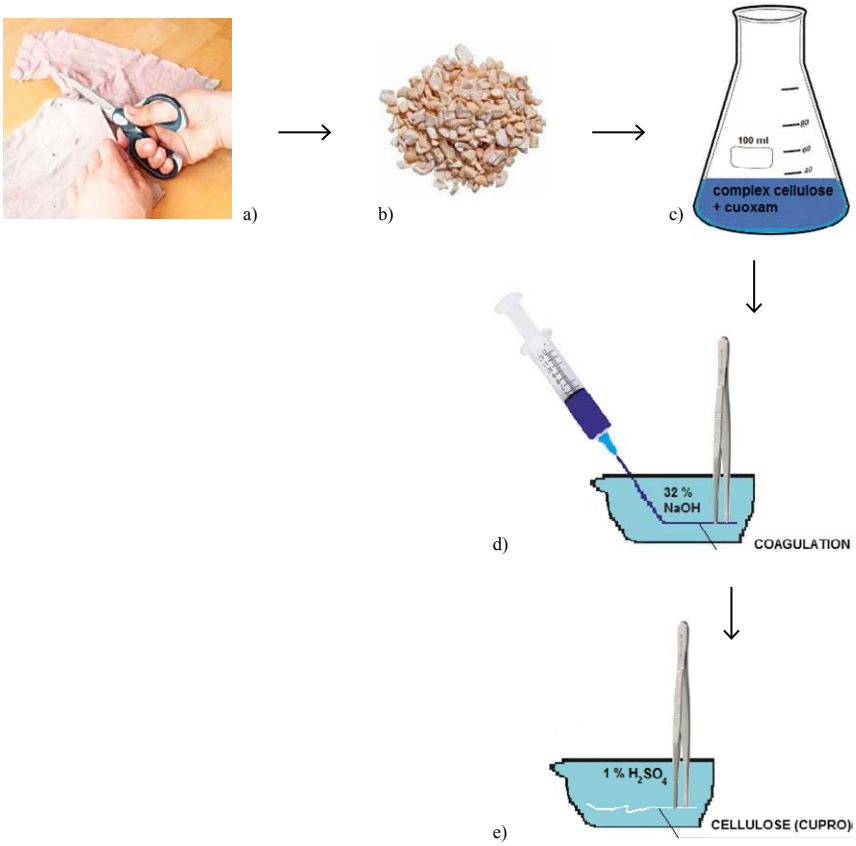
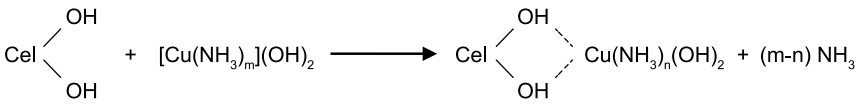
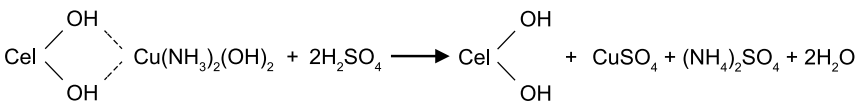


Fig.3 Scheme of preparation regenerated cellulose trough cuprammonium process: a) cutting of waste textiles b) into small pieces, c) treating with Cuoxam, d) coagulation in 32 % NaOH solution and e) treating with 1 % H_2SO_4



(3)



(4)

perature for 24 hours, to depolymerize the cellulose, resulting in a reduction in the average molecular weight of the original pulp. The reduction of the cellulose results in a viscous solution with the right viscosity. Any air bubbles trapped in the solution must be removed before extrusion to avoid voids or weak spots in the fine viscose filaments, which is called degassing.

Wet spinning was carried out by pressing xanthate through a hypodermic needle into a spinning bath (V=50 mL; (Fig.2) heated to 50 °C containing 10 % sulphuric acid (to acidify the sodium cellulose xanthate), 1 g sodium sulphate/ Na_2SO_4 (to give the bath a high salt content necessary for rapid coagulation of the viscose) and 1 g zinc sulphate/$ZnSO_4$ (to form zinc xanthate to cross-link the cellulose molecules).

This results in rapid coagulation of the viscose filaments, which is followed by simultaneous stretching and decomposition of cellulose xanthate to regenerated cellulose (viscose). The freshly regenerated viscose contains many salts and other water-soluble impurities, which have been removed by thorough washing in distilled water.

2.5.2. Regenerated cellulose trough cuprammonium process

Towels and shirts (input cellulose) were cut into small pieces (Fig.3a and 3b). 1,0 g of these pieces were transferred to a 100 mL Erlenmeyer flask where they were treated with Cuoxam at room temperature with vigorous shaking to dissolve the input cellulose (Fig.3c).

Cuoxam was prepared by dissolving 6.5 g copper sulphate ($CuSO_4x5H_2O$) in 20 mL distilled water. When cooling the copper sulphate solution to room temperature, 20 mL of a 25 % aqueous solution of ammonium hydroxide (NH_4OH) and 4.3 mL of a 32 % aqueous solution of sodium hydroxide (NaOH) were added. According to this method, a complex of input cellulose and Cuoxam was formed.

The next step was coagulation in a 32 % NaOH solution (Fig.3d) and the treatment with 1 % H_2SO_4 (Fig.3e), by which regenerated cellulose/cupro is formed (4). Water-soluble impurities were removed by thorough washing of the cupro in distilled water.

In some cases, charcoal has been added to the spinning bath when such materials are intended for filtration purposes [11].

3. Results and discussion

3.1. Results of ATR FT-IR spectroscopy

Fig.4 shows ATR FT-IR spectra of different samples.

The ATR FT-IR spectrum of shirt (red spectrum) shows typical signals for

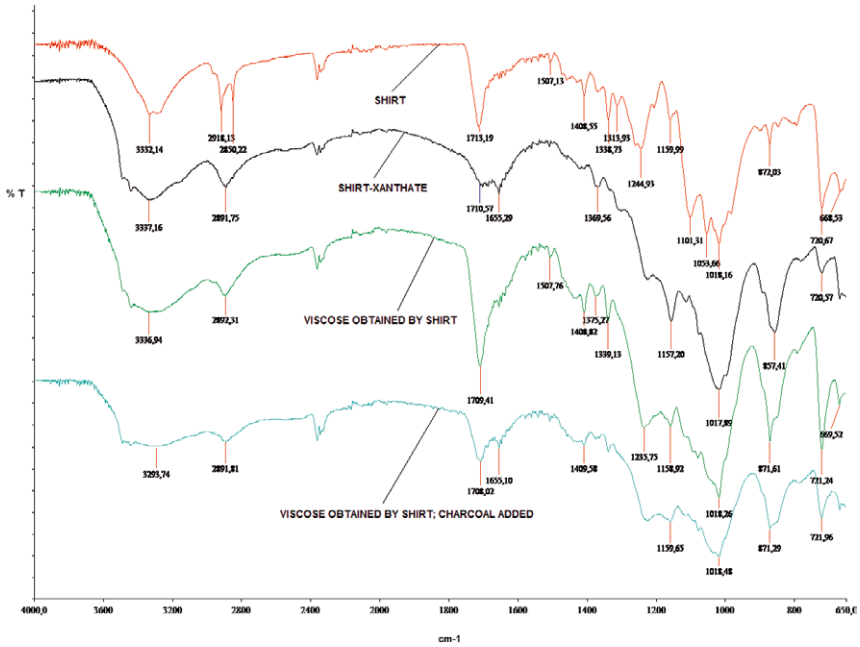


Fig.4 ATR FT-IR spectra of: viscose obtained by dissolving shirt (Co/PES) for the viscose production (green spectrum), viscose obtained by dissolving shirt (Co/PES) for the viscose production with added charcoal (blue spectrum), shirt (red spectrum), shirt-xanthate (black spectrum)

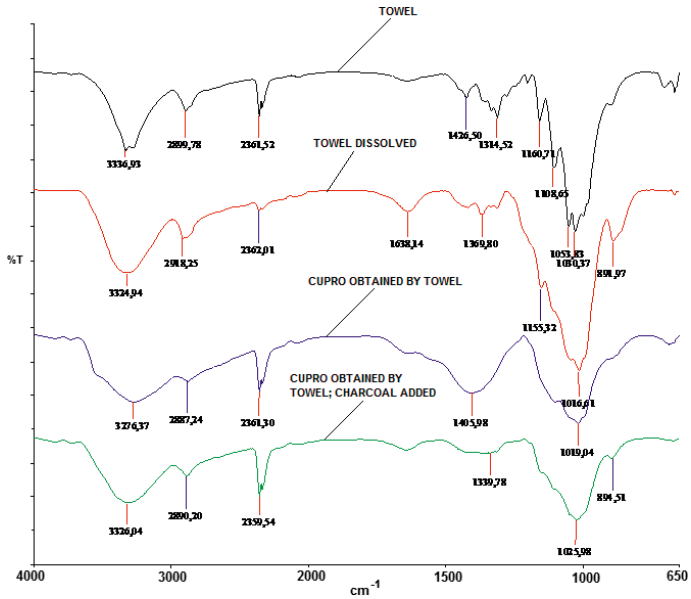


Fig.5 ATR FT-IR spectra of: towel (black spectrum), towel dissolved (red spectrum), cupro obtained by towel (blue spectrum) and cupro obtained by towel with added charcoal (green spectrum)

polyethylene terephthalate and cellulose. In the area between 2918-2850 cm^{-1} signals of -CH groups are presented, and on the other hand at the wave number of 1713 cm^{-1} a signal which is attributed to the C=O group; typical for cellulose is hydroxyl (-OH) groups at the wave number of 3332 cm^{-1}. The ATR-FTIR spectrum of viscose obtained by shirt (green spectrum) also present typical signals for polyethylene terephthalate; signals at the range 2918-2850 cm^{-1} and 1713 cm^{-1}, as well as signals for cellulose at 3332 cm^{-1}. From the ATR FT-IR spectra of the sample »shirt xanthate« (black spectrum) and »viscose (shirt + charcoal) « (blue spectrum) it can be seen the presence of water in the region of 3330 and, 1655 cm^{-1}, respectively.

ATR FT-IR spectrum of towel (black spectrum) present typical signals for cellulose, hydroxyl (-OH) groups at the wave number of 3336 cm^{-1} and signals of -CH groups in the area around 2899 cm^{-1}, Fig.5. Both spectra, spectrum of cupro (blue spectrum), made by dissolving towel from 100 % cotton and spectrum of cupro obtained by towel with added charcoal (green spectrum) presented typical signals for cellulose at 3336 cm^{-1} and signals in the area around 2899 cm^{-1}. In the example of cupro (Fig.5) there was impossible to obtain this type of regenerated cellulose by using textile waste material constructed from mixture of cotton/PES (i.e. shirt) as in case of viscose (Fig.4). It should be noted that PES it has not been removed before spinning, neither in the preparation of viscose fibers nor in the preparation of cupro.

3.2. Optimization of the spinning solutions viscosity

The optimization of the starting material mass together with the solvents was chosen as the main objective of the presented research, since only with the right combination of these compounds a system with suitable viscosity for the optimal spinning of the xanthate through spinnerets before the formation of regenerated viscose can be established. This optimization was also necessary because the starting material used was the textile material consisting of a mixture of cellulose fibers and synthetic fiber-forming polymer, where it is necessary to take into account the fact that the synthetic component cannot be dissolved by using solvents necessary for the production of regenerated cellulose (viscose and cupro). As a measure for the viscosity of the spinning bath, a viscosity was chosen which ensures a good spinning of xanthate through the spinneret (in our case the

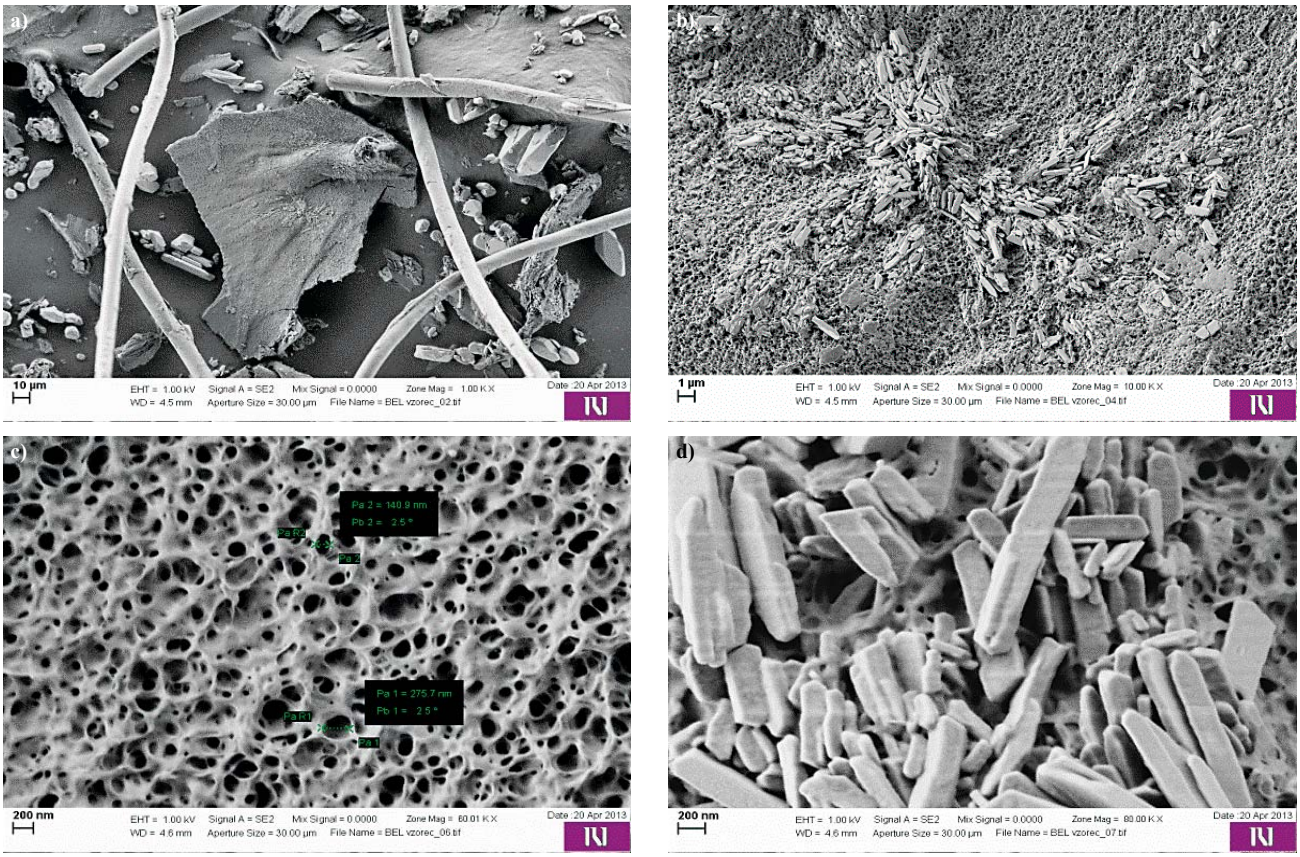
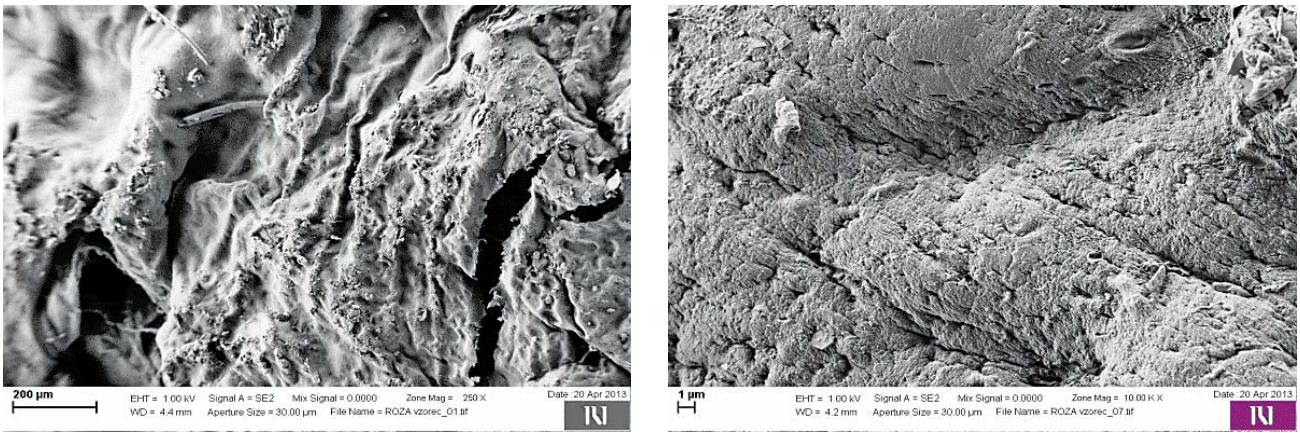


Fig.6 SEM of viscose, made using shirt waste consisting of a blend of 60 % cotton and 40 % polyester, at magnification of: a) 1.000 x, b) 10.000 x, c) 60.010 x and d) 80.000 x



a) b)

Fig.7 SEM of viscose using waste towel made of 100 % cotton at magnification of: a) 250 x and b) 10.000 x

needle attached to the injection piston). Based on the optimization of the starting compounds (existing textile materials in combination with solvents) and the results of the methods used in this study, it was found that, with regard to good spinning of the xanthate through the spinneret under laboratory conditions, the viscosity between 15 and 17 Pa s is most favorable. With this viscosity a good exit of the spinning bath through the spinneret and its immediate conversion into regenerated cellulose could be guaranteed, no matter whether viscose or cupro is produced under laboratory conditions.

3.3. Scanning electron microscopy analysis

Fig.6 shows SEM of the viscose fibers produced from shirt waste (60 % cotton and 40 % polyester) at different magnifications.

From this SEM one can see regenerated viscose in combination with polyester (Fig.6a). At a magnification of 10.000, areas with a layer of adsorbed porous particles are seen, Fig.6b. The porous structure (magnification 60.010, Fig.6c) is relatively homogeneous. In Fig.6c pores of the size of 141 and 276 nm are visible.

SEM at a magnification of 80.000 (Fig.6d) shows adsorbed particles of relatively uniform and similar shape. It is assumed that this is due to the presence of particles because of suspended or precipitated compounds used in the production of viscose.
The SEM image of viscose fibers at different magnifications from recycled towel (Fig.7) shows a different trend, in this case there is no presence of adsorbed particles, as can be seen in the previous example.

4. Conclusion

- Recycling of textile waste is becoming a necessity to avoid the disposal of such waste.
- Particularly problematic are textile materials made up of different fiber mixtures.
- Production of viscose fibers under laboratory conditions at the selected viscosity of the spinning solution was possible, even if the input raw material was shirts made of cotton/PES.
- In the case of regenerated cellulose in cupro fibers, this type of regenerated cellulose was only possible to obtain when towels made of 100 % cotton were used as the raw material. In the case of a mixture of cotton and polyester, the laboratory production of cupro was not successful.
- Laboratory experiments show a successful approach to the recycling of potential garment waste from cotton and a mixture of cotton and polyester with a view to obtaining regenerated cellulose fibers.

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