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Electrokinetic Properties of Nanocomposite Fibres

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1. Introduction

Nanocomposite fibres are becoming of great importance since organic–inorganic nanoscale composites frequently exhibit unexpected hybrid properties synergistically derived from the two components.

The incorporation of one-, two- and three-dimensional nano particles, e.g. layered clays, nano-tubes, nano-fibres, metal containing nano-particles, carbon black, etc. is used to prepare nanocomposite fibres. However, the preparation of nanocomposite fibres offers several possibilities, i.e. creation of nanocomposite fibres by dispersing of nanoparticles into polymer solutions, the polymer melt blending of nanoparticles, in situ prepared nanoparticles within a fibre, the intercalative polymerization of the monomer, introduction of nanoparticles from dispersion into a porous polymer, preparation of nanocoatings by different techniques, e.g. sol-gel process, etc. With the respect to nanoparticles organization in / on fibres, respectively nanocoated fibres and fibres with nanoinclusions can be divided.

Nanocomposite fibres demonstrate improved applicable properties, e.g. mechanical, flammability, electromagnetic, electro-conductive, sorption properties, and/ or obtain additional functional properties, like antimicrobial, self-cleaning, shielding, UV protective properties, etc. Combining polymer fibre and nanopaticles of different structures and characteristics influences also fibre's surface properties, i.e. morphological properties and electrokinetic behaviour.

In order to analyse fibre's surface charge, the zeta potential is often determined through streaming potential measurements as a function of pH. These properties are generated by the electrochemical double layer (EDL), which exists at the phase boundary between a solid and a solution containing ionic moieties. By variation of the solution's pH value and electrolyte content it is possible to estimate the acidic / basic and polar / nonpolar character of the solid surface from zeta potential data.

Some examples of nanocomposite fibres (e.g. nanofilled PP fibres, SiO_2 – nanocoated different types of fibres) regarding their electrokinetic properties are presented in the contribution. In addition to, the influence of nanocomposite fibres preparation procedure conditions on their zeta potential values is demonstrated.

2. Nanocomposite fibres

Functional textiles are recently produced by nanotechnology that can significantly improve properties of materials compared with those of conventional ones, and which employs physical or chemical techniques to construct materials on a nanometre scale. This remarkable properties improvement is attributed to the significant increase in the fibre's boundary area or its functional surface. By nanotechnology new materials such as nanofibres, nanocomposites and nanocoated textiles are created (Gowri et al., 2010; Lin &Wang, 2009).

2.1 Nanofibres

Polymer nanofibres are produced by the electrospinning process, which uses electrical forces for creation of polymer fibres with nanometre-scale diameters. Electrospinning is a simple and relatively inexpensive method of producing nanofibres by solidification of a polymer solution or melt. By the fibre process formation the electrical forces at the surface of a polymer solution or melt overcome the surface tension and this phenomenon gives rise to the charged polymer jet to be released. Dried or solidified polymer jet is stretched by an electric field. In comparison to conventional fibre production methods electrospun fibres are much finer and fibre assemblies can form different nonwoven structures (Teo & Ramakrishna, 2006).

This technique enables production of various nanofibres from polymers, polymer blends and polymer composites to ceramics. Various synthetic and natural biodegradable polymers have been electrospun into fibres with diameters in the nanometre range. The nanofibre diameter, structure and physical properties can be effectively modified by controlling various parameters of the electrospinning process. Therefore a lot of research has been performed to study the influence of production parameters, e.g. solution concentration, tip-to-collector distance, and the feed rate in producing nonwovens composed of different size fibres (Zhang et al., 2009).

Processing parameter effects on the internal molecular structure of electrospun semi-crystalline PLLA fibres were investigated by Inai et al. It was found that take-up velocity is a dominant parameter to induce a highly ordered molecular structure in fibres compared to solution conductivity and polymer concentration. The last two parameters were important in controlling the fibre diameter. Due to the ordered structure of the fibres produced by higher take-up velocity their tensile modulus and strength were higher (Inai et al., 2005).

In spite of intensive research activities on interdependences between electrospinning production parameters and fibres finenesses and diameter variations which are the two most important nanofibres properties, the physical and mathematical descriptions of the electrospinning process are still not unambiguously defined therefore additional techniques , e.g. multiple regression analysis are used to study the process. From the results it is evident that polymer concentration and feed rate have significant and controlled impacts on creation of fibres with diameters in the nano-range and by these two parameters fibres diameter could be foreseen (Patra et al., 2010).

Despite the fact that the most of the studies and manufacturing processes were performed for electrospinning procedure of polymer solutions, spinning from polymer melts has a number of advantages and is also possible. Different polymers melts and solutions are appropriate for nanospinning, e.g. polyethylene oxide, polyamide PA, polyamide PA 6

blends, polyimide, DNA, aramid and polyaniline, etc (Reneker & Chun, 1996; Malakhov et al., 2009).

In addition to, PAN nanofibres were prepared via an electrospinning process. Fibres were not obtained in the electrospinning process when the polymer concentration was very low, however as the concentration increased, the fibre morphology changed from a beads structure to the fibre structure with increasing fibre diameters. The addition of a cationic surfactant positively influenced the fibre formation. The effect of polymer concentration on the fibre morphology was studied by Lin et al. (Lin et al., 2005).

The same observations were made for Poly(vinyl alcohol) (PVA) nanofibres which were obtained by electrospinning of PVA in water solutions. Formation of beaded structures combined with decreased fibre diameters was observed for low PVA concentrations. Fibres with higher fineness and more uniform diameter were obtained by spinning blends of PVA of different molecular weight when compared to the fibres spun from homopolymers of similar viscosity (Zhang et al., 2005).

The process of electrospinning of ultra fine PVA fibres which have potential applications in filtration and biomedical engineering was optimised by investigating the effects of various variables such as electric voltage, tip to target distance, flow rate and solution parameters on the morphology of nanofibres (Samani et al., 2009).

Additionally a theoretical analysis of the distribution of the electrostatic field forming around spinning points in the area where polymer streams are formed and stretched was performed by Krucinska et al. (Krucinska et al. 2009).

Due to some problems related to electrospinning technique such as low production rate, capillary clogging, etc. in recent years, several new electrospinning methods have been developed to solve these problems, e.g. by using multiple jets the production is increased.

Morphology of poly(vinyl alcohol) (PVA) electrospun fibres with inserted drugs, like Aspirin etc. was investigated by Zhang with co-workers. Fibres were prepared by electrospinning of PVA aqueous solutions. The researchers found out that the morphology of fibres was conditioned by the amount of drug, i.e. irregularly shaped fibres were formed with increasing drug amounts. However when comparing drug release from PVA nanofibres and PVA film, the release form nanofibres is more efficient due to the large surface area and high porosity of the electrospun fibre mats (Zhang et al., 2005).

Furthermore, it was detected that the morphology of the drug-loaded electrospun PVA mats depended on the nature of the incorporated drug. Drugs molecular weight has influenced inverse proportionally both, the rate and the total amount of drugs released from the drug-loaded electrospun PVA material (Taepaiboon et al., 2006).

Excellent physical properties such as high surface area, high porosity, interconnective pores of the nanofibre matrices and appropriate mechanical properties, well-controlled degradation rates and biocompatibility of the base polymer, make biodegradable polymeric nanofibre matrices ideal materials for developing scaffolds for tissue engineering (Nair et al., 2004).

To achieve additional functionalities of nanofibers, not only pure polymers, but their polymer blends were electrospun as well. By the procedure of bicomponent electrospinning nanoporous polyacrilonitrile fibres can be obtained, therefore polyacrylonitrile (PAN) and poly(ethylene oxide) (PEO) bicomponent fibres were spun and upon removal of the phase-separated PEO domains, became nanoporous. The nanoporous fibres generated from 50/50 PAN/PEO bicomponent precursor contained internal pores of a few nanometres to tens of

nanometres in size and had 50% higher pore volume and 2.5-times higher specific surface (Zhang & Hsieh, 2006).

Several bio-polymers were electrospun recently. Experimental attempts were made to fabricate man-made silk fibres by means of electrospinning and the properties of electrospun fibres were compared to the fibres obtained by wet spinning and dry spinning (Fu et al., 2009).

There were attempts to apply cellulose of different molecular weights from solutions of lithium chloride and N,N-dimethylacetamide (DMAc) and water solution of N-methylmorpholine N-oxide (NMNO), respectively for ellectrospinning procedure. Due to low efficiency some modifications of the process were suggested (heated solvents, etc). However the most efficient way to produce cellulose nanofibers is by electrospinning cellulose acetate (CA) followed by alkaline hydrolysis to revert the acetyl to hydroxyl groups (Du & Hsieh, 2009).

Cellulose acetate (CA) nanofibres mats were produced by dissolving CA in different solvents mixtures (ketones-acetone, methyl ethyl ketone (MEK), and alcohols-benzyl alcohol, propylene glycol and dimethylsulphoxide) and the process was optimized with the aim to produce uniform nanofibres (Haas et al., 2010).

Tissue engineering scaffolds produced by electrospinning are a very interesting research topic; therefore a lot of biocompatible polymers were introduced for their production. Some of them are: cellulose, chitosan, PVA, poly(E-caprolactone) (PCL)/hydroxyapatite (HA), etc. (Zargarian & Haddadi, 2010; Du & Hsieh, 2009; Sajeev et al., 2008).

Chitosan and gelatine as well as PVA have been widely used materials in recent years because of their excellent biocompatibility, biodegradability, bacteriostasis and other excellent characteristics. PVA/chitosan and PVA/gelatin nanofibrous composite membranes were fabricated using the electrospinning technique and the morphology of the fibres was observed to compare the spinnability of PVA/chitosan and PVA/gelatin electrospun fibres. Both materials exhibit good spinnability (Huang et al., 2009).

As chitin is characterised by limited processing possibilities, a chitin derivative has been developed, e.g. dibutyrylchitin, which is an ester chitin derivative and which can be easily dissolved in common solvents. A new method of manufacturing nonwoven products made from dibutyrylchitin for biomaterials was developed by Blasinska (Blasinska et al., 2004).

Nano-fibres containing quaternised chitosan (QCh) have been successfully prepared by electrospinning of QCh solutions mixed with poly(vinyl alcohol) (PVA). The obtained nano-fibrous electrospun mats are promising for wound-heating applications (Ignatova et al., 2006).

Electrospinning process is likewise appropriate for nanocomposites perparation. A lot of research was performed in studying the spinning conditions, the distribution of nanoparticles inside polymer, the polymer crystalline structure formation, interdependence of procedure conditions and structure morphology (Adomaviciene et al., 2010; Kim et al., 2007).

Different polymers and different fillers were used for nanocomposites preparation. Multiwall carbon nanotubes (MWCNT) were introduced into the bamboo cellulose matrix to form composite nanofibres by the liquid crystal electrospinning process (Wan & Ko, 2009); and in addition to, in silk fibroin (Bombyx mori) by dissolving regenerated silk fibroin and MWCNT in formic acid for electrospinning process (Kang et al., 2009).

Electrospun membranes based on nanofibres are used in a variety of applications, including filtration systems, tissue engineering and regenerative medicine, etc. Due to the interdisciplinary fields of their application the characterisation of the material is very

specific. Nisbet with co-authors reviewed the current status of the characterisation of electrospun membranes and pointed out deficiencies (Nisbet et al., 2009).

2.2 Nanofilled fibres

Several different nanoparticles for nanofilled composites, e.g. layered silicates (Benetti et al., 2005; Manias et al., 2001; Filho et al., 2005; Ray& Okamoto, 2003; Alexandre et al., 2002; Han et al., 2003; Tang et al., 2003), silica nanoparticles (Jain et al., 2005; Rong et al., 2001), carbon black (Horrocks et al. 1999; Jakab & Omastova, 2005) carbon nanotubes (Chatterjee & Deopura, 2006; Kearns & Shambaugh, 2002) metal containing nanoparticles (Gubin, 2002), elastomeric nanoparticles (Zhang et al, 2002), TiO_2 (Marcinčin et al., 2009) etc., have been reported.

The incorporation of one-, two- and three-dimensional nanoparticles, e.g. layered clays (Manias 2000; Marcinčin et al., 2008; Sfiligoj et al. 2009) nano-tubes (Chatterjee & Deopura, 2006; Kearns & Shambaugh, 2002), nanofibres (Hine et al., 2005, Li et al., 2004) metal containing nano-particles (Gubin, 2002), carbon black (Horrocks et al. 1999; Jakab & Omastova, 2005), etc. is used to prepare nanocomposite fibres.

For ultraviolet (UV) protection materials PP TiO_2 nanocomposite fibres were developed by Erdem. Filaments were melt spun from master batches of PP and 0.3, 1, and 3% TiO_2 nanoparticles, respectively which were prepared by melt compounding before spinning. Nanocomposites' mechanical properties were slightly affected by the addition of nanoparticles however the filaments exhibited excellent UV protection (Erdem et al., 2010).

By addition of nanoparticles to the spinning polymer melt or solution new fibres functionalities are obtained, however the procedure of spinning is thereby more complicated and it is often difficult to reach suitable spinning conditions. It is sometimes easier and more advantageous to functionalize textiles by subsequent nano-treatment.

2.3 Nanocoated fibres

Comprehensive research activities in the textile nanotechnology science are directed to enhance or/and impart new functionalities to textiles, i.e. improved abrasion and thermal properties (Cireli et al., 2007; Satoh et al., 2004; Hribernik et al., 2007), various repellent properties (water, oil, soil) (Pinto et al., 2008), easy to clean properties, protective properties, e.g. UV and EMR shielding (Xin et al., 2004; Onar et al., 2007; Abidi et al., 2007), electrical conductivity, antimicrobial activity (Gao & Cranston, 2008; Xu et al., 2006; Tarimala et al., 2006), etc. Nanocoatings of fibres offer excellent possibilities therefore (Wei et al., 2004; Mahltig et al., 2005; Sawhney et al., 2008).

To prepare nanocoated textiles there are mainly two distinct methodologies: deposition of morphological well-defined nanoparticles at the fibres surfaces or in situ synthesis of nanoparticles on fibres. For the lattermost procedure the sol–gel method is often used. By the sol-gel processing precursor solutions undergo gelation, aging and drying. By controlling the synthesis conditions carefully, these reactions may lead to a variety of structures, and to different final states of the material.

Several authors reported of SiO_2 nanocoated textiles obtained via sol-gel process according to the method developed by Stöber (Stöber et al., 1968). By this method spherical and monodisperse silica nano particles are grown however the coating morphology is determined by the reaction conditions, e.g. alkaline or acid catalysis, etc. Different precursors for the sol-gel processing of silicates are used, e.g. TMOS (tetramethoxysilane; Si

(OCH$_3$)$_4$), TEOS (tetraethoxysilane; Si (OCH$_2$CH$_3$)$_4$). SiO$_2$ nanocoatings via tetraethoxysilane (TEOS) hydrolysis were prepared on different textile substrates (Li et al., 2008; Hribernik et al., 2007, Pinto et al., 2008; etc.). Silica particles were coated on carbon fabrics for enlargement of fibres surface roughness to enhance hydrophobicity on the basis of lotus leaf effect (Hsieh et al., 2008).

Modified silica nanocoatings make additional functionalities possible. Water repellent textiles via coating with various modified silica sols were prepared by Mahltig. Pure and with 3-glycidoxypropyl triethoxysilane co-condensed sols were modified by addition of alkyltrialkoxysilane, polysiloxane derivatives and a fluorine containing silane. The coatings were applied on polyester and polyamide/cotton blends (Mahltig & Böttcher, 2003). Yu et al. developed a water repellent coating for cotton fibres by applying silica nano particles and perfluorooctylated quaternary ammonium silane coupling agent (PFSC). Oil repellent properties of silica particles/PFSC treated cotton fabrics were also improved (Yu et al., 2007). The same effect was achieved by treating cotton fabrics by a combination of silicon precursor TEOS and fluorocarbon polymer (Yeh et al., 2007). The significant enhancement of superoleophobicity by the deposition of silica nanoparticles with a thin fluorination coating on the surface of the particles is attributed to the following facts: (i) surface fluorination (impact on surface tension), (ii) surface topography (roughness), and (iii) creation of bionic surface hierarchically combined with nano/submicron architecture (Hsieh et al., 2010). Gonçalves has prepared superhydrophobic cellulose nano composites by employing amorphous silica particles as a mean to enhance surface roughness and perfluoro moieties for reduction of surface energy. Resultant water contact angles approached a value of 150° (Gonçalves et al., 2008).

Silane agents which are often used in the nanocoating process for modifying fibres functionalities can form surface films on fibres and silica particles (Vanblaaderen & Vrij, 1993; Tomšič et al., 2007) or can be used as coupling agents between the fibres and silica particles.

Silica has served as well as a carrier for incorporation of dye-stuffs. Cheng et al. have prepared dye-containing silica sol-covered wool fabrics. Photocromic dyes were able to provide a very fast response to UV light (Cheng et al., 2007). Mahltig et al. have incorporated dyes into sol-gel coatings on textile fibres to develop a ''one-type'' recipe for dyeing different kinds of fabrics (Mahltig et al., 2004).

Nano-sized zinc oxide (ZnO) and more frequently titanium dioxide (TiO$_2$) particles are used for surface modification of different types of fibres for imparting the stain-release function. The ZnO nanoparticles were produced at different thermal conditions and media (water or 1,2-ethanediol) by Kathirvelu et al. A high temperature was necessary to obtain small mono-dispersed particles, which were then applied to cotton and polyester – cotton blend samples for achieving self-cleaning activity (Kathirvelu et al., 2010).

Numerous literature reports on the fabrication of TiO$_2$ thin films by sol-gel dip coating technique using many types of titanium alkoxides as precursors (Hamid & Rahman, 2003; Chrysicopoulou et al., 1998]; titanium tetraethoxide (Harizanov & Harizanova, 2000); titanium ethoxide (Ozer et al., 1995); titanium propoxide [Bell et al., 1994]; tetraisopropyl-orthotitanate (Su&Lu, 1998; Kajihara et al., 1998; Zaharescu et al., 1998; Avellaneda &Pawlicka., 1998); titanium tetra-n-butoxide (Imao et al., 2006). Others (Phani et al., 2002; Wang et al., 2002) have used titanium butoxide as starting materials for making TiO$_2$ thin films.

Much work has been carried out depositing TiO_2 on heat resistant surfaces like glass, ceramics and silica by sol-gel methods, where temperatures up to 500 °C can be used. Several reports on TiO_2 application in the textile industry can be found as well (e.g. Bozzi et al., 2005a, b,; Daoud & Xin 2004; Veronovski et.al., 2006; Qi et al., 2007; Textor et al., 2007).

To obtain homogeneous TiO_2 nanocoatings and to investigate their self-cleaning effects when applied on cellulose materials Veronovski studied different procedures of nanocoating preparation. Several different approaches for achieving nanocoatings were used. First, coatings were generated in situ through an acid and alkaline catalyzed sol-gel process with or without added water. Titanium isopropoxide, TIP was used as TiO_2 precursor. Another type of coatings was prepared by using commercial TiO_2 P25 nanoparticles with good photocatalytic efficiency towards a broad range of organic pollutants and which is containing a mixture of rutile and anatase crystalline forms (Veronovski et al., 2006; Veronovski et al., 2009 a, b; Veronovski et al., 2010 a, b, c, d). TiO_2 P 25 was used for textile nanocoatings preparation by other researchers as well (Bozzi et al., 2005 a, b; Daoud & Xin, 2004; Qi et al., 2007; Texlor et al., 2007).

In addition, nano-TiO_2 added in the wrinkle-resistant treatment of cotton fabric could act as a multi-functional finishing agent to improve the UV protection property (Lam et al., 2010).

Composite titania (TiO_2) and silica (SiO_2) coatings are used to prevent fibres from photo-oxidative damages. Titanium-silica mixed oxides can be produced by different processes (Doolin, et al., 1994; Nakabayashi, 1992; Reddy et al., 1993; Stakheev et al., 1993; Hung & Katz, 1992; Greegor et al., 1983), but the most common way is the sol-gel method (Imamura et al., 1996; Klein et al., 1996; Liu & Davis, 1994; Dagan et al., 1995; Dutoit et al., 1996; 1995; Walther et al., 1991). The conditions of the sol-gel process have the major influence on the properties of titanium-silica mixed oxides.

A TiO_2-SiO_2 coating on regenerated cellulose fibers was prepared using TiO_2 P25 nanoparticles and the sol-gel process for SiO_2 formation from tetraethoxysilane (TEOS) as the precursor (Veronovski et al., 2010 d). In addition to, pure TiO_2 P25 nanocoated fibres were prepared and the properties of modified fibres were compared. Both TiO_2 nanocoated surfaces demonstrated photocatalytic activity, however, better results were obtained when the surface was treated in TiO_2 P25 nanoparticles dispersion only. In the case of TiO_2-SiO_2 coating, TiO_2 activity was reduced due to SiO_2 film partially covering TiO_2 particles (Veronovski et al., 2010 d).

In addition to, TiO_2 was used to prepare ultraviolet resistant cotton fabrics. UV Protection Factor UPF 50+ values were measured for the TiO_2-coated samples. Further it was found that the rutile phase was better than anatase phase in blocking UV rays. Modified textiles were compared to ZnO nanocoated cotton yarns, were the modification efficiency was not so high (Paul et al., 2010).

Photocatalytic self-cleaning TiO_2 nanocoatings were used for wool fibers modification by Wing. The process of sol-gel TiO_2 nanocoating formation was studied to prevent chemical damages and photo–degradation of wool fibres during the process. The sols were prepared using different catalysts, and media, for the hydrolysis and condensation reactions of the titanium dioxide precursor (Wing & Walid, 2009).

3. Electrokinetic effects

When two phases are placed in contact there develops, in general, a difference in potential between them. If one of the phases is a polar liquid, like water, its (dipolar) molecules will

tend to be oriented in a particular direction at the interface and this will generate a potential difference. If the surface of phase I is positively charged, its electrostatic potential will be positive with respect to the bulk of phase II; if phase II is a liquid containing dissolved ions, then as one moves from the surface into phase II, the potential will decrease, more or less regularly, until it becomes constant in the bulk liquid far from the surface of the phase I. The region where the liquid has a positive electrostatic potential will accumulate an excess of negative ions and repel positive ions of the electrolyte. It is this excess of negative ions which gradually lowers the electrostatic potential to zero in the bulk electrolyte. The arrangement of charges on the surface of the phase I and the charges in the liquid phase II is referred to as the electrical double layer at the interface. By the relative motion between an electrolyte and a charged solid surface (phase I, and II) a number of phenomena which are known as electrokinetic effects are observed. There are four distinct effects depending on the way in which motion is induced. They are: (i) electrophoresis, (ii) electro-osmosis, (iii) streaming potential and (iv) sedimentation potential (Hunter 1981; Jacobasch et al. 1995; Delgado et al. 2005; Reischel et al. 2008).

electrokinetic effect	measured quantity	entity, that moves	Movement is caused by
electrophoresis	velocity	moving particles	electrical field
electro-osmosis	velocity	moving liquid through the capillaries	electrical field
streaming potential	potential	moving liquid	pressure difference
sedimentation potential	potential	moving particles	gravitation

Table 1. The principles of electrokinetic effects

Electrophoresis: when a solid or liquid phase is suspended in liquid or gas phase, then a movement of particles can be induced with electrical field in the system. This phenomenon is called electrophoresis. Measurement of the particles' velocity in known external electrical field yields information on total charge of particles or their surface potential, related to the surrounding phase.

Electro-osmosis: the effect is known as electro-osmosis in a case of a non-moving solid phase and a moving liquid phase as a result of an electrical field. It's encountered with solid phases as capillaries or porous plugs, filled with liquid. Electrical field moves the ions of the liquid and therefore the liquid itself. Information is gained on the total surface charge or electrical potential near the surface.

Streaming potential: in this case, movement of liquid is caused by a pressure difference. Excess of charge near the surface is moving along with the liquid phase and the accumulation of charge causes the development of electric field with an opposite direction to that of the liquid. Measurement of the potential difference of the capillary or a porous plug is called streaming potential.

Sedimentation potential: when charged colloidal particles are moving in the liquid as a result of the gravitation or centrifuge field, potential difference occurs. Sedimentation potential is an electrical voltage, which is a result of charged particles' movement. Negative particles create negative field in the direction of movement, while equilibrium is achieved with a flow of positive ions in the opposite direction.

Different models of a charged surface, needed for the evaluation of the electrokinetic surface phenomena, have been developed over the years, however Gouy-Chapman-Stern-Graham model. GCSG is regarded as the most exact one, taking into account various different phenomena occurring in the polymer/electrolyte system and is as such most used in the modern evaluation of the charged surfaces.

3.1 Models of electrically charged surfaces in electrolyte solutions
3.1.1 Helmholtz - Perrin model

Helmholtz and Perrin developed a model which predicts the adsorption of oppositely charged ions from the electrolyte solutions onto a surface. Plane condenser is formed, therefore implying a linear electrical potential (ψ) in the adsorbed layer (Helmholtz layer) on the surface. Charges on the surface itself do not influence the electrolyte solution, since they are completely screened by the adsorbed layer of opposite charges. This model gave rise to the term double layer, which refers to the charges on the surface and charges from the electrolyte solution. Helmholtz - Perrin model does not take into account the structure of water or solvent, only the ions, present in the solution and also neglects their size and adsorption ability – rather, they are treated as point-like species, adsorbed to the surface via electrostatic interactions without taking into account other interactions.

3.1.2 Gouy - Chapman model

Gouy - Chapman model, developed after the Helmholtz - Perrin model, discards the notion of adsorbed charges on the surfaces, but rather predicts the position of ions along the surface according to the Boltzmann distribution. This implies a larger quantity of oppositely charged ions in the vicinity of the surface, while the difference in concentrations between different ions diminishes when moving further away from the surface. In this proposition, the double layer consists of charges on the surface and charges in the electrolyte solution; layer of ions at the surface is called diffuse layer, due to the mobility of the ions. Fall of the electrical potential with the distance from the surface is exponential within the diffuse layer. While Helmholtz - Perrin model neglected the thermal movement of ions in the solution, Gouy - Chapman model takes into account only thermal movement (Boltzmann distribution) and neglects the possibility that some charge may be adsorbed onto the surface – either in a fixed manner or in a form of dynamic equilibrium of constant exchange of electrolyte ions and adsorbed ions.

3.1.3 Stern model

Stern model is a combination of Helmholtz - Perrin model and Gouy - Chapman model. The most important aspects of Stern model are: ions are no longer treated as point-like species but are given some finite dimensions and they can adsorb onto a surface also via non-electrostatic interactions (e.g. disperse interactions between ions and the charged surface). Since ions are not point-like, they find themselves adsorbed farther away from the surface than it was predicted in the Helmholtz - Perrin model. The fall of electric potential within this adsorbed layer is linear, but the surface charge is not completely screened by the adsorbed ions. The remaining part of the charge, needed for the complete screening of charged surface, is positioned in the diffuse layer, as previously postulated by the Gouy - Chapman model: ions in the diffuse layer are subjected to thermal movement and therefore Boltzmann distribution, while electrical potential falls exponentially. Important finding,

derived from the Stern model is, that ions, which screen the surface charge can be situated either within the adsorbed layer (in this case the Stern layer) or in the diffuse layer. Amount of charge in both layers is influenced by the properties of electrolyte solution, that is, valence of ions and their concentration. In diluted solutions, most of the charge will be situated in the diffuse layer, while in concentrated solutions the majority of charge will be found in the Stern layer.

3.1.4 Gouy - Chapman - Stern - Graham model (GCSG model)

This model is considered as one of the most advanced, and is frequently encountered when dealing with charged surfaces in the electrolyte solutions. It differentiates itself from the Stern model by taking into account properties and structure of the water molecules in the solution. Stern layer is therefore divided into two parts, that is, inner Helmholtz plane and outer Helmholtz plane. Inner Helmholtz plane (IHP) is composed of a monolayer of water molecules adjacent to the surface, which are, due to the charged surface, ordered in such a way, that the lowest possible interaction energy exists between them and the surface. Water molecules at the surface are oriented as dipoles, in regard to the sign of the surface charge; when a surface is positively charged, water molecules are oriented toward the surface with their oxygen atoms, while in the case of a negative charge, they position themselves with hydrogen atoms to the surface. IHP also contains ions with a higher degree of mobility, which makes them preferentially adsorbed via dispersion forces. Since GCSG model takes into account the structure of water, the hydration state of ions is predicted – ions are surrounded with water molecules. Cations are more hydrated than anions, which is a result of a steric effect, since water molecules orient themselves with oxygen adjacent to them. Anions are therefore less limited in mobility, since a lesser amount of water molecules is attached to them, while in some cases, they are not hydrated at all. This is the reason for preferential adsorption of anions in the IHP, regardless of the sign of the charge on the surface – this preferential adsorption is also termed hydrophobic interaction.

As far as outer Helmholtz plane (OHP) is concerned, it is actually a Stern layer, composed of adsorbed ions, which are opposite in sign to that on the surface.

The most important aspect of the GCSG model is the recognition of the preferential adsorption of anions into IHP, even when the surface is negatively charged. This clearly implies, that other interactions are present, alongside the electrostatic ones.

3.2 Zeta potential

The zeta potential (ζ) is the term describing the electrokinetic properties at that position of the solid/liquid interface, which is accessible for interactions. This interface is described by chemical and electrochemical potential values different from those of the bulk phases since the molecules in the boundary are subject to interaction forces from both adjacent phases. The resulting net charge of the solid surfaceaqueous solution interfaces is generally attributed to different mechanisms. This is the dissociation of functional surface groups on one hand, and on the other hand the preferential adsorption of potential determining ions and other charged species from solution.

For the calculation of (ζ) potentials from streaming potential measurements, which is a common method for determination of fibres' zeta potential, the approach of Smoluchowski is used. It relates the (ζ) potential to the streaming potential/pressure gradient, the electrical

resistance over the sample, the viscosity of the electrolyte solution, and geometrical factors relating to the streaming channel for the electrolyte solution.

$$\zeta = \frac{U_s}{\Delta p} \frac{\eta}{\varepsilon_r \varepsilon_o} \frac{L}{Q} \frac{1}{R}$$ (1)

Here, U_s is the streaming potential, Δp is the applied pressure difference, η is the viscosity of the solution, $\varepsilon_r \varepsilon_0$ is the permittivity of the liquid medium, L is the streaming channel length, Q is the cross-sectional area of the streaming channel, and R is the electrical resistivity over the sample. This equation holds as long as the curvature of the measured system is sufficiently large ($\kappa a \gg 1$, where κ is the reciprocal Debye length and a is the curvature of the system). In addition, surface conductance and polarization of the electrical double layer is neglected.

Overbeek showed that the considerations for the streaming channel can also be extended to porous sample plugs, forming microchannels for the electrolyte flow. The channel geometry and sample resistivity can then be treated according to Fairbrother and Mastin (Fairbrother & Mastin 1924).

$$\frac{L}{Q} = \frac{R_s \chi_b}{R}$$ (2)

where R_S is the resistivity of the electrolyte solution and χ_b is the electrolyte conductivity. By substitution of Eq. 1 with Eq. 2, the ζ potential can be expressed as follows:

$$\zeta = \frac{U_s \eta \chi_b}{\Delta p \varepsilon_r \varepsilon_o}$$ (3)

A more general approach to the ζ potential equation is given in the following expression (Equation 4). Here, also the surface conductivity of the sample χ_s is accounted for.

$$\zeta = \frac{U_s \eta [\chi_b + f \chi_s]}{\Delta p \varepsilon_r \varepsilon_o}$$ (4)

The term f is called the form factor of the streaming channel. This equation can be used, if the surface conductivity of the sample is nonzero. If the surface conductivity is neglected ($\chi_s = 0$), then Equation 4 reduces to Equation 3.

4. Electrokinetic properties of nanocomposite fibres

4.1 Zeta potential of PP nanocomposite fibres

PP–based nanocomposite fibres were prepared by direct polymer melt intercalation and the electrokinetic properties of nannofilled fibres were measured (Župerl 2004, Sfiligoj et al. 2009). Fibres were spun from an isotactic iPP homopolymer (with MFI = 11g/10min – 16 g/10min and isotactic index 96 % (m/m)) with addition of polypropylene grafted with acrylic acid PPAA as a coupling agent. 10% and 20% of PPAA co-polymer, respectively were added. Montmorillonite (organoclay) MMT modified by N,N-dimethyl-N,N dioctadecylammonium

cations (Nanofil 15 from Südchemie, Germany) was used to prepare nanocomposite fibres with 0.5, 1 and 2 % of MMT, respectively. The melt spinning and in-line drawing of PP filaments was carried out (Župerl et al. 2002). The same spinning conditions were used for neat PP fibres, PP copolymer and MMT modified fibres.

To study electrokinetic properties the streaming potential method was used. The method is in detail described in e.g. Stana & Ribitsch 1998, Ribitsch & Stana 1998, Stana et al. 1999, Stana et al. 2001, Smole et al. 2002, etc.

ζ was calculated from the streaming potential (U_s) data by use of the Smoluchowski equation (1) and Fairbrother and Mastin (FM) approach (2), in which the term (L/Q) is replaced by ($R_s\chi_b$), where R_s is the electrical resistance of the plug when the measurement cell is filled with an electrolyte with accurately known specific conductance χ_b.

Streaming potential measurements of fibres were performed on the Electrokinetic Analyzer EKA, A. Paar KG in the fibre cell using 0.001n KCl as electrolyte solution.

Electrokinetic properties of neat PP and PP fibers with added MMT, PP/ PPAA fibers and PP/ PPAA fibres modified with 0.5, 1 and 2% MMT, respectively were compared. Functional dependence pH/ zeta potential (ZP) for fibres spinned from neat PP homopolymer is demonstaretd in Figure 1.
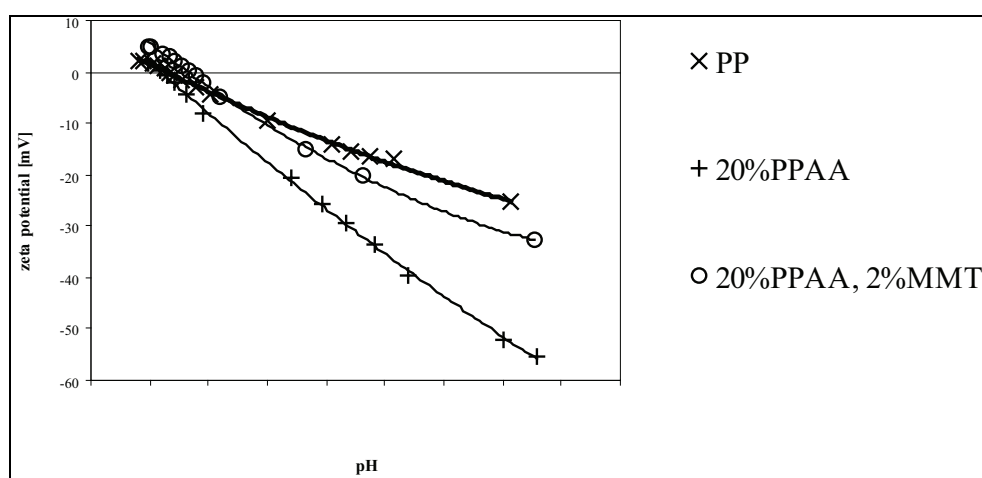
Fig. 1. pH/ζ diagram for PP homopolymer (x), PP/PPAA co-polymer (+) and PP nannocomposite fibres (○), rerspectively

Due to the PP non-polar chemical structure a typical non-polar pH/ζ diagram is obtained. It demonstrates fibres' hydrophobe character with negative zeta potential value ZP at – 25 mV measured at pH=9 and an isoelectric point IE at pH = 3.5. By addition of PPAA (20%) ZP decreases to -51.5mV at pH=9, however only a slight shift of the isoelectric point towards acid region is observed (IE at pH =3.11). For PP nanocomposite fibres containing different amounts of MMT a reduction of zeta potential value and a slight shift of IE point towards neutral region is observed when compared to unmodified PP fibres. For the sample spinned from PP, PPAA (20%) and MMT (2%) the ZP was -32,8 at pH=9 and IE = 3.68. Although electrokinetic properties of nanocomposite fibres are conditioned by both influences; i.e. PPAA and MMT, nevertheless, the ζ -pH function results indicate that the electrokinetic surface properties are dominated by the PPAA containing dissociable carboxyl groups (Sfiligoj et al. 2009).

4.2 Zeta potential of nanocoated fibres

In addition to, we prepared SiO_2 nano coatings on different fibre types and their zeta potential was determined to study the electrokinetic properties and the influence of the coating morphology on zeta potential.
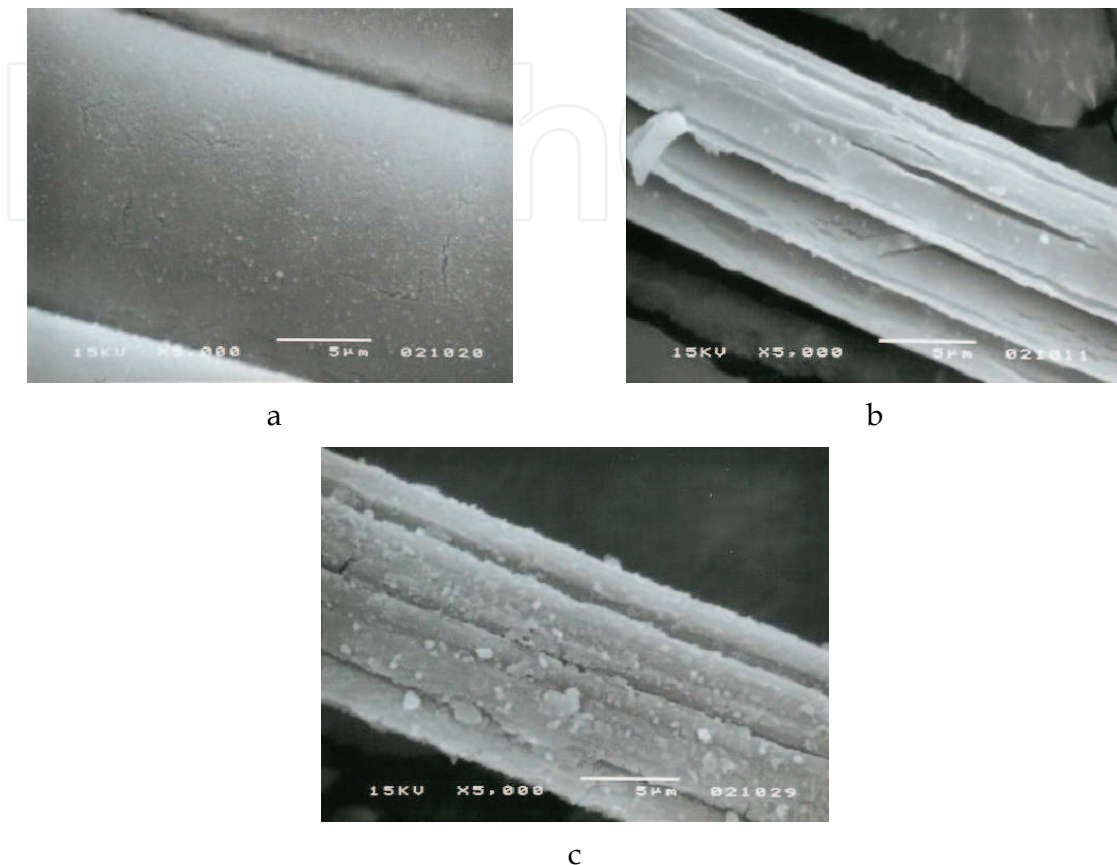

a   b

c

Fig. 2. SiO_2 nanocoated PET fibres (a) and regenerated cellulose fibres Modal (b) and Viscose (c) according to the procedure B

SiO_2 is an important technical material which is often used in pharmaceutical industry and in production of catalysts and pigments, etc. Properties of materials containing SiO_2 particles are above all influenced by particles size and particles size distribution. There are two procedures of sol-gel particles preparation, (i.e. alkaline and acid) known from the literature. They both enable preparation of spherical SiO_2 particles in the range between 10 nm to some μm.

In the research we prepared alkaline catalysed particles. The method is based on controlled hydrolysis of alkyl silicate and alcohol, followed by condensation of the dispersed phase. The procedure is known as the Stöber method. Particles size is conditioned by the type and mass share of alkyl silicate, NH_3 and H_2O, type and mass share of alcohol as the reaction medium and reaction temperature.

Nanocoatings were prepared by the sol-gel process from Tetraethylorthosilicat TEOS as precursor. To obtain different coatings morphologies process conditions were varied, i.e. different concentration of precursor, TEOS (0,2 and 0,5 mol/L), and catalyst NH_3 (0,3 and 0,5 mol/L) and two solvents (ethanol, propanol) were used. Different modification procedures were used and different fibre types were studied; i.e. cotton, regenerated cellulose fibres and polyethylene terephtalate PET. Prior to the modification fibres were pre-treated for 30

minutes in a solution of a non-ionic washing agent (1g/L) and in the case of cellulose fibres in 0.1 M HCl, respectively followed by washing in distilled water to the constant conductivity (≤ 5 μS/cm).

Nanocoatings were prepared in propanol with 0.2 mol/L TEOS (procedure A) and with 0.5 mol/L TEOS (procedure B), in ethanol with 0.2 mol/L TEOS (procedure C) and with 0.5 mol/L TEOS (procedure D), respectively on different fibre types and SEM images of surface morphologies of some modified fibres prepared according to the modification procedure B are demonstrated in Figure 2.

Zeta potential – pH electrolyte functions of cellulose; i.e. natural cellulose cotton fibres (Fig. 3) and regenerated cellulose fibres Modal and Viscose, respectively and of SiO$_2$ modified fibres are demonstrated on Figures 4 -5.
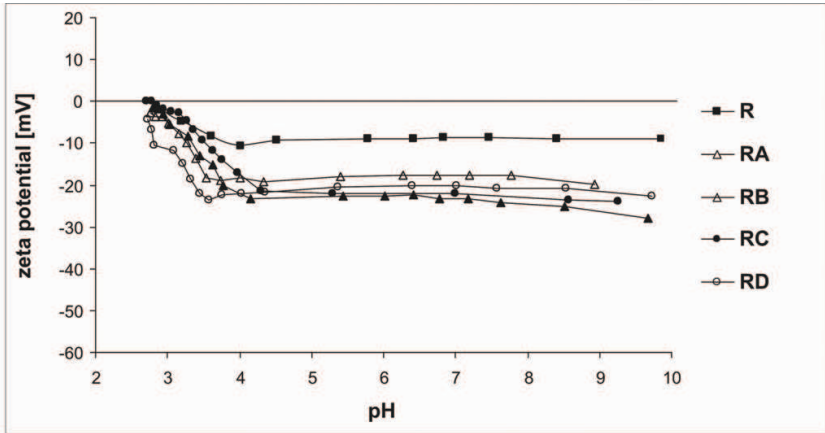
(R-cotton fibre, RA-nanocoated cotton fibres by using procedure A, RB- nanocoated cotton fibres by using procedure B, RC- nanocoated cotton fibres by using procedure C, RD- nanocoated cotton fibres by using procedure D)

Fig. 3. Zeta potential of unmodified cotton fibre and nanocoated fibres as a function of pH
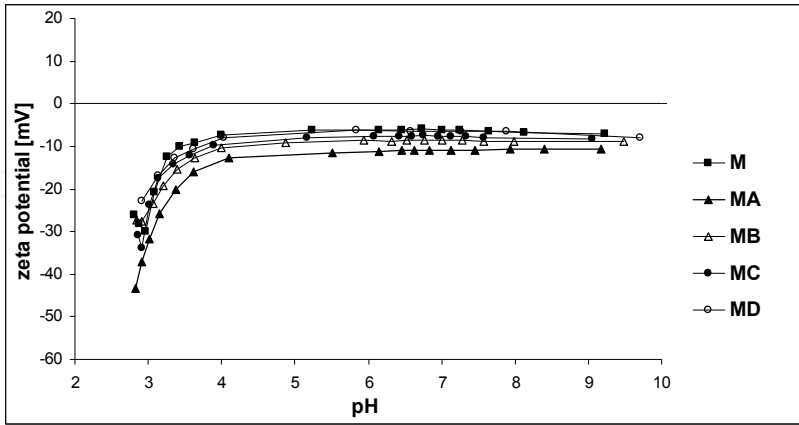
(M-Modal fibre, MA-nanocoated Modal fibres by using procedure A, MB- nanocoated Modal fibres by using procedure B, MC- nanocoated modal fibres by using procedure C, MD- nanocoated Modal fibres by using procedure D)

Fig. 4. Zeta potential of unmodified Modal fibre and nanocoated fibres as a function of pH

In addition to fibres with self-cleaning properties based on photocatalytic nano TiO$_2$ particles were prepared. For the nanomodification regenerated cellulose fibres Lyocell were

involved. Two different coatings types were created, i.e. TiO_2 and TiO_2 - SiO_2 nanocoating by using TiO_2 P25 nanoparticles (Degussa). TiO_2 P25 particles contain anatase and rutile phases in a ratio of about 3: 1. The average sizes of the anatase and rutile elementary particles are 85 and 25 nm, respectively (Ohno et al. 2001). After fibres pre-treatment Lyocell fibres were treated in a dispersion of 5 g/L TiO_2 P25 nanoparticles in deionised water at T = 60°C for 1 hour. For TiO_2–SiO_2 coating preparation, 0.5 mol/L TEOS as a precursor in the presence of ethanol as a solvent and 0.5 mol/L ammonia solution as a catalyst were added to the pre-dispersed aqueous solution of TiO_2 P25 nanoparticles at pH 5.39. Lyocell fibres were treated in the dispersion for 1 hour at elevated temperature (T = 60°C) (Veronovski et al. 2010d). Before measuring zeta potential of the modified fibres, non-adsorbed particles were removed by fibres' rinsing at pH 6.31 and heat treated for 15 minutes and additionally washed in an ultrasound bath for 5 minutes. Coatings morphologies are demonstrated in Fig.6 and zeta potentials as a function of pH for untreated and coated fibre samples in Fig. 7.
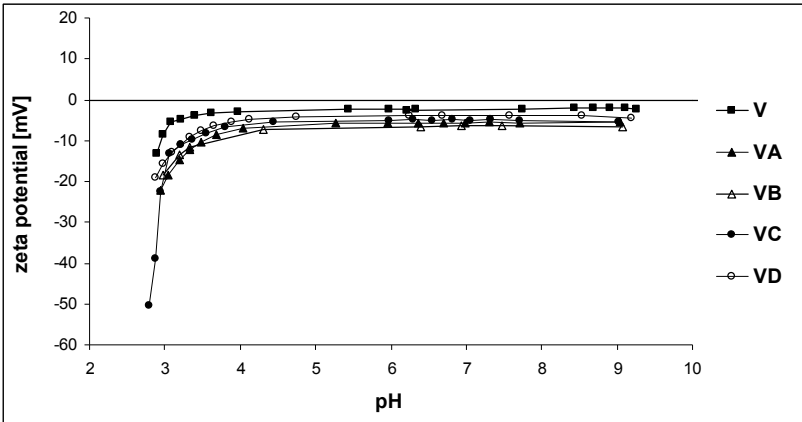
(V-Viscose fibre, VA-nanocoated Viscose fibres by using procedure A, VB- nanocoated Viscose fibres by using procedure B, VC- nanocoated Viscose fibres by using procedure C, VD- nanocoated Viscose fibres by using procedure D)

Fig. 5. Zeta potential of unmodified Viscose fibre and nanocoated fibres as a function of pH
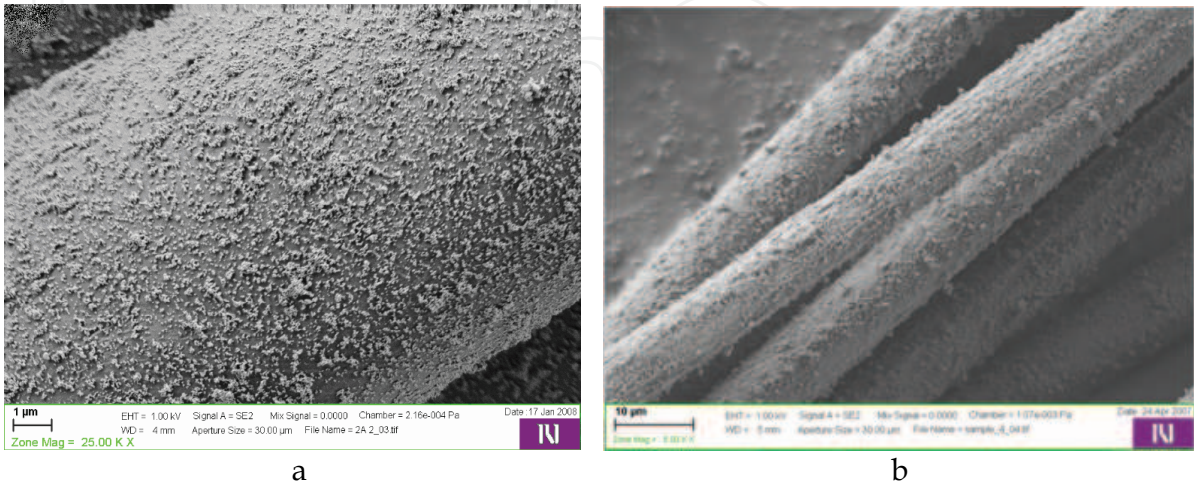

Fig. 6. SEM images of TiO_2 (a) and TiO_2 - SiO_2 (b) nanocoated Lyocell fibres

Natural cellulose fibres are negatively charged (ζpH=9=−11 mV) due to the presence of carboxyl and hydroxyl-groups. In the case of raw (untreated) fibres these groups are covered by noncellulose compounds present in the primary wall of cotton fibre. Regenerated cellulose fibres are also negatively charged and their zeta potential is negative through the whole pH region, however zeta potential value is not the same for different types of regenerated cellulose fibres. It is well known that different production processes for regenerated cellulose fibres (Viscose, Modal, Lyocell) cause differences in their structure and in the reactivity—adsorption character of fibres despite having the same chemical composition. Lyocell fibres posses more ordered structure with higher crystallinity and higher molecular orientation when compared to Viscose and Modal fibres. The pore system of Viscose and Lyocell fibres is very similar and more comprehensive than the pore system in Modal fibres. These differences in the molecular and fine structure between various types of regenerated cellulose fibres cause different reactivity, sorption and electrokinetic properties (Stana-Kleinschek et al 2001). $\zeta_{pH=9}$ = –6.3 mV for untreated Lyocell fibres, $\zeta_{pH=9}$ = -3 mV for Viscose and $\zeta_{pH=9}$ = -9 mV for Modal fibres. We can conclude that the highest negative zeta potential in the alkaline region was determined for Modal fibres and the lowest for Viscose probably due to the distinctions between the accessibility's of reactive groups (OH and COOH).

The adsorption of nanoparticles on cellulose fibres can be qualitatively confirmed by measuring the streaming potential of the samples as the introduction of nano groups significantly influences the ζ_{P}– pH diagram. The negative zeta potential is additionally decreased by nano SiO_2 coating. There are some differences in zeta potential according the coating procedure which was used.

In Figure 7 the zeta potential as a function of pH for untreated Lyocell and TiO_2 and TiO_2-SiO_2 coated fibres is presented. The typical diagram for a cellulose fibre with a negative zeta potential value through the whole pH region is changed by nanomodification; i.e. zeta potential values are increased. For the TiO_2 coated sample the zeta potential at pH =9 is –4.4 mV and for TiO_2–SiO_2 coated sample – 5.3 mV, respectively. The appearance of the isoelectric point in the diagram of nanocoated samples confirms the adsorption of nanoparticles. It is well known that the isoelectric point (IEP) of TiO_2 is in the range of pH 4.5–5, and in the case of SiO_2 particles, at pH 2 (Veronovski et al. 2010). The IEP of TiO_2-coated Lyocell fibre is at pH 3.55 and it shifts to pH = 2.75 when silica particles are covering TiO_2 particles.
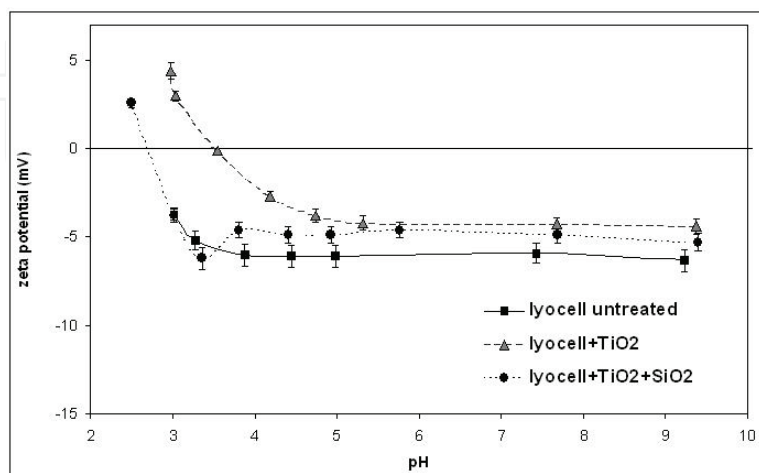
(untreated Lyocell fibres, TiO_2 nanocoated Lyocel fibres and TiO_2 - SiO_2 nanocoated Lyocel fibres)

Fig. 7. Zeta potential of unmodified Lyocell fibre and nanocoated fibres as a function of pH
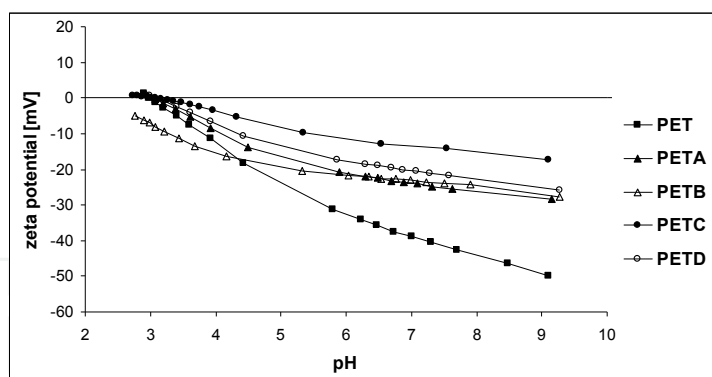
(PET-polyethylene terephthalate fibre, PETA-nanocoated polyethylene terephthalate fibres by using procedure A, PETB- nanocoated polyethylene terephthalate fibres by using procedure B, PETC-nanocoated polyethylene terephthalate fibres by using procedure C, PETD- nanocoated polyethylene terephthalate fibres by using procedure D)

Fig. 8. Zeta potential of unmodified PET fibre and SiO_2 nanocoated fibres as a function of pH

The zeta potential at pH=9 ($\zeta_{pH=9}$) of unmodified PET fibres is app. −50 mV and the IEP at pH =3 (Fig.8) and significant changes of the zeta potential – pH functions are observed by SiO_2 nanocoating. Zeta potential values are changed conditioned by the modification procedure, however the IEP in not influenced by the treatment.

5. Conclusion

Improvement of existing properties and the creation of new material properties are the most important reasons for the functionalization of textiles. To design these additional materials properties nanotechnology is recently used. Nanocoating and inclusion of nanoparticles into fibres represent modification techniques with great potential for synthesis of novel functional materials however these technologies are still inadequately employed in the textile production.

The influence of nanomodification on surface properties of different fibre types can be studied by determination of zeta (ζ) potential as a function of pH. Based on functional dependence ζ=f (pH) we can conclude about acid – alkaline character of functional groups on fibres' surface and accessibility of these groups. ζ-potential of fibres surface is not only a parameter of quantity and type of dissociable species and adsorbed charged molecules or ions; but by measuring ζ-potential the hydrophilic /hydrophobic character of fibres and structural changes on fibres' surface can be valued.

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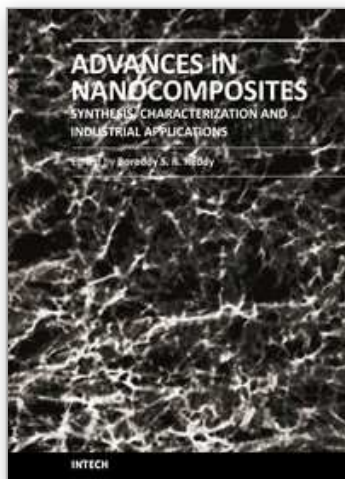
Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications
Edited by Dr. Boreddy Reddy

Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications was conceived as a comprehensive reference volume on various aspects of functional nanocomposites for engineering technologies. The term functional nanocomposites signifies a wide area of polymer/material science and engineering, involving the design, synthesis and study of nanocomposites of increasing structural sophistication and complexity useful for a wide range of chemical, physicochemical and biological/biomedical processes. "Emerging technologies" are also broadly understood to include new technological developments, beginning at the forefront of conventional industrial practices and extending into anticipated and speculative industries of the future. The scope of the present book on nanocomposites and applications extends far beyond emerging technologies. This book presents 40 chapters organized in four parts systematically providing a wealth of new ideas in design, synthesis and study of sophisticated nanocomposite structures.

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