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In situ preparation of silver–polyacrylonitrile nanocomposite fibres

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ABSTRACT

In the article a new method of volume modification of polyacrylonitrile (PAN) fibres with silver nanoparticles was presented. Nanoparticles were synthesized in situ in the polymer spinning mass. Fibres are formed in the wet spinning method. The precursor of silver ions and reductant was added directly to the spinning solution containing polyacrylonitrile and dimethylformamide (DMF) as a solvent. Tests performed by dynamic light scattering method showed that the average size of the resulting silver nanoparticles is approx. 10 nm. Polyacrylonitrile molecules were used as a polymer stabilising agent. Silver nanoparticles interact with nitrile groups from PAN molecules causing the formation polymeric micelles, which prevent their further growth and agglomeration. Observations performed by TEM confirmed the presence of the stabilising polymeric coating on silver nanoparticles formed in situ in spinning mass.

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1. Introduction

Currently, there is a very large demand for functional textile materials with different characteristics, for example with a high electrical conductivity, semiconductor properties, high antibacterial activity, self-cleaning properties or high photocatalytic activity [1–3]. Therefore, studies are being conducted on the modification of various polymer fibres in order to obtain materials with desired characteristics. One way to modify fibres is their doping with different kinds of additions.

Polymeric fibrous materials are mostly surface modified due to their properties and the specificity of manufacture [4–6]. This solution, however, has a significant drawback. Materials modified in this way lose their functional properties during washing or use [7]. For this reason, methods of volume modification rather than surface modification of fibres are desired.

Due to the very large fibre length-to-diameter relationship (usually fibre diameter is from several to more than a dozen micrometers), nanoparticles may be excellent materials for their volume modification [8]. Polymers doped with nanoparticles demonstrate better mechanical properties as well as other unique properties that depend on the nature and concentration of the doping agent (nanoparticles). For example, the introduction of carbon nanotubes to polymer modifies its electrical properties [9,10], and the addition of silver or copper nanoparticles contributes to providing the starting material with antibacterial [11] or conductive properties [12].

Due to the very large relationship of surface atoms to volume atoms in nanomaterials, they have unique physical and chemical properties [13] and they are much better fillers than microparticles. However, their high value of surface energy

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is the main cause of agglomeration. Therefore, uniform dispersion of such particles in the polymer matrix is very difficult [14]. The presence of agglomerates not only reduces the strength of nanocomposites, but it also makes the nanoparticles lose their unique properties. The formation of agglomerates is particularly undesirable in the case of fibrous composites whose diameter is slightly larger than the diameter of the agglomerate.

Another factor that hinders the doping of polymer fibres is their production specificity. They are produced by different methods, such as wet spinning, wet-dry spinning or dry spinning [15]. Polyacrylonitrile fibres (PAN), which are the subject of the presented studies, are formed by wet spinning from spinning solutions. In this case, nanoparticles can be introduced to the spinning solution, from which fibres will be formed:

- in the dry form,
- in the form of a solution,
- or they can be directly synthesized in the polymer material.

In each of these cases it is important to remember that the spinning solution must maintain appropriate rheological properties. Doping agents may not result in any changes in those properties, because this hinders, and in extreme cases prevents, the process of fibre spinning.

In the case of volumetric doping the fibres with dry nanopowders, fibres with worse characteristics, in particular mechanical characteristics, are obtained. This is the result of poor dispersion of nanoparticles in the polymer material and the increase in its viscosity. Adding nanoparticles in the form of dry powders to the polymer material is associated with the implementation of a very complicated process of nanoparticle dispersion. Due to the high viscosity of the spinning solutions, the ideal dispersion of the materials is impossible. As a result, agglomerates are present in the polymer material, and the fibres obtained from this material are characterised by unsatisfactory mechanical and electrical parameters [16]. In addition, the mixing of the polymer material with nanopowders leads to an increase in dustiness in the room, which has a negative impact on human health and repeatability of the conducted processes.

Adding the aqueous colloid of nanoparticles having low concentration increases water concentration in the polymer material, which in turn leads to the change in the rheology of the spinning material. In the case of polyacrylonitrile fibres, the presence of water completely prevents fibre spinning [17].

Nowadays, composite polyacrylonitrile fibres are manufactured only by electrospinning. Using this method, Yang et al. were first to have obtained PAN fibres containing silver nanoparticles. These nanoparticles are formed as a result of the reduction in Ag^{+} ions in the PAN/DMF mixture using hydrazine hydroxide [18]. Reduction in the solution may also occur only in the presence of the DMF at room temperature [19]. However, in such conditions the reduction takes place slowly, within 7 days, and only after this time fibres can be produced using the electrospinning method [20]. The reduction of silver ions in the aforementioned system also occurs by subjecting the polymeric solution to the plasma treatment [21]. As a result of a reduction using the plasma, silver nanoparticles with a diameter of 3–6 nm were formed. They were evenly distributed in the fibre matrix.

In the literature, there are a number of other publications that describe the method of nanofibres obtained by electrospinning and doped with silver. There are, however, no reports regarding doping the polyacrylonitrile fibres, which can be employed in the textile industry. These fibres, with varying degrees of the doping agent, can be used not only in the clothing industry, but – which is of the utmost importance – also in textronics [1]. For industrial purposes, fibres are wet spun from the spinning material, which contains 23% PAN in DMF, while a solution used in the electrospinning method contains only 11% polymer. Therefore, the spinning material used in the wet spinning method is much thicker, which effectively prevents dispersion of nanoparticles.

In this article, we present a new method of volume modification of polyacrylonitrile fibres with silver nanoparticles. Nanoparticles were synthesized in situ in the polymer material. Spinning of PAN fibres, using the forming mass in the wet process involves the extrusion of the forming mass through a forming nozzle into a liquid bath consisting of substances that mix with the solvent of the forming mass well, but they do not melt the polymer. In the solidified bath, partial dissolution of the polymer and separation of phases (precipitation of polymer) take place.

The process of PAN fibres spinning is affected by:

- the structure of polymer used for fibre forming mass (percentage of different types of mers or polymer particles having different mass) and the type of the solvent,
- different conditions of fibre spinning process (solidification conditions, configuration of stretching stage, temperature of stabilisation and drying).

2. Methods

2.1. Preparation of PAN fibres with silver nanoparticles

Preparation of PAN fibres with silver nanoparticles. During the implementation phase, polyacrylonitrile terpolymer (t-PAN) was used, which contained:

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