Applied Surface Science xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Full Length Article

Optical properties of thin fibrous PVP/SiO_2 composite mats prepared via the sol-gel and electrospinning methods

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ARTICLE INFO

Article history:
Received 7 November 2016
Received in revised form 23 February 2017
Accepted 28 February 2017
Available online xxx

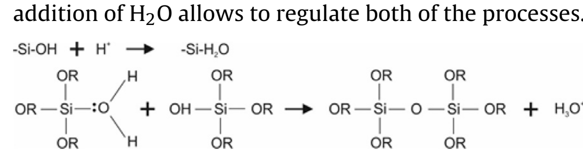
Keywords:
Sol-gel method
SiO_2 nanoparticles
Electrospinning method
Composite nanofibers
Polyvinylpyrrolidone
Optical properties

ABSTRACT

The aim of the research was to create thin, nanofibrous composite mats with a polyvinylpyrrolidone (PVP) matrix, with the reinforcing phase in the form of silicon oxide (SiO_2) nanoparticles. SiO_2 nanopowder was obtained using the zol-gel method with a mixture of tetraethyl orthosilicate (TEOS, Si (OC2H5)), hydrochloric acid (HCl), ethanol (C3H5OH) and distilled water. The produced colloidal suspension was subjected to a drying process and a calcination process at 550 °C, resulting in an amorphous silica nanopowder with an average particle diameter of 20 nm. The morphology and structure of the manufactured SiO_2 nanoparticles was tested using transmission electron microscopy (TEM) and X-ray diffraction analysis (XRD). Then, using the electrospinning method with a 15% (weight) solution of PVP in ethanol and a 15% solution of PVP/EtOH containing the produced nanoparticles equivalent to 5% of the mass concentration relative to the polymer matrix, polymer PVP nanofibres and PVP/SiO_2 composite nanofibres/SiO_2 nanoparticles were produced. The morphology and chemical composition of the produced polymer and composite nanofibres were tested using a scanning electron microscope (SEM) with an energy dispersive spectrometer (EDS). The analysis of the impact of the reinforcing phase on the absorption of electromagnetic radiation was conducted on the basis of UV–vis spectra, based on which the rated values of band gaps of the produced thin fibrous mats were assessed.

1. Introduction

The sol-gel method is widely used for the preparation of a large variety of ceramic materials in the form of various morphologies, including fibers, coatings, monoliths, particles as well as dimensions ranging from nanometers to millimeters. The nanoparticles exhibit unique physicochemical properties due to their nanoscale and large specific surface area [1,2]. Nanoparticles of silica are used in biomedical applications because of their large surface area, the ability to functionalize, electrical application as solar cells [2–4], photocatalytic application, including application in combination with TiO_2, or Ag nanoparticles [5–8]. The sol-gel process is mainly defined as a process of producing inorganic oxides from metal alkoxides. Sol is a stable colloidal suspension of colloidal solid particles in a liquid. Sol can be transformed to gel using the gelation process through the gel-point where the sol solution suddenly changes from fluid liquid, through a viscous state, to a solid state called the gel [9,10]. Compounds with the chemical formula $M(OR)_z$ are also called alkoxides. These compounds are usually in the form of a liquid solution formed through direct or indirect reactions between metal M and alcohol ROH. In general, the sol-gel process consists of hydrolysis and condensation processes. The reaction of hydrolysis occurs in the presence of water, where a controlled addition of H_2O allows to regulate both of the processes.



(1)

The result of the hydrolysis of tetraethoxysilane alkoxides in acidic conditions is silanol, which belongs to the Si-OH group, formed through hydroxoligands bonded to central silicon atoms (1). The linking of the silanol group leads to the formation of a polymer chain with a small amount of branching points. The reaction of the condensation proceeds with the formation of a three-dimensional network becomes a solid state [10–12]. Sol-gel is a colloidal and chemistry route to obtain oxide ceramics with an intermediate sol and gel state. The product of the sol-gel process in

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http://dx.doi.org/10.1016/j.apsusc.2017.02.258
0169-4332/© 2017 Published by Elsevier B.V.

Please cite this article in press as: T. Tański, et al., Optical properties of thin fibrous PVP/SiO_2 composite mats prepared via the sol-gel and electrospinning methods, Appl. Surf. Sci. (2017), http://dx.doi.org/10.1016/j.apsusc.2017.02.258

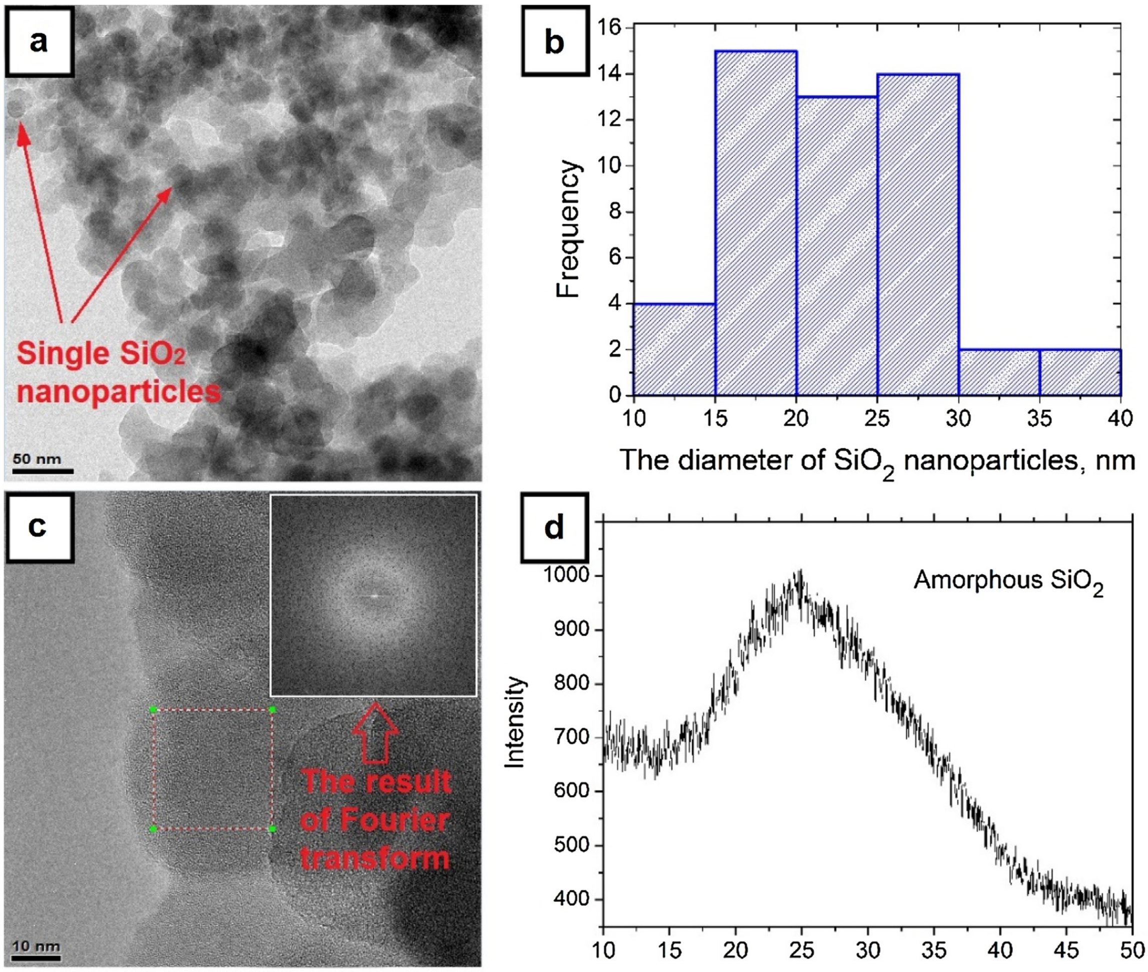

Fig. 1. TEM image of the produced SiO_2 nanoparticles (a), the histogram of distribution of diameters of randomly measured nanoparticles (b), TEM image of a single nanoparticle with the results of the carried out Fourier transformation and the selected area that was used for its calculation (c), XRD spectrum of tested SiO_2 nanopowders.

the form of a viscous liquid can be spun into fibres or even coating materials using the electrospinning technique. A controlled gelation process allows for the formation of monosized droplets and fibres of several dozen or hundreds of nanometres [10,13–17].

In the last fifteen years, a constant interest of the scientific community in the electrospinning method from a solution has been observed. The method allows to obtain polymer and composite nanofibres, while maintaining a low cost of production on both the laboratory as well as the industrial scale. Due to their nanometric size and the inability to obtain them in the case of conventional material physical properties, these types of materials are becoming increasingly recognised due to their wide application possibilities. Composite nanofibres in the polymer matrix with polyvinylpyrrolidone, which are reinforced with nanoparticles, have a number of applications, such as relative humidity sensors [18], fluorescent and clothing applications [19,20], layers to protect from UV radiation and advanced materials intended for applications in the electronics industry [20]. Due to their physical properties, silicon oxide nanoparticles produced by the zol-gel method are used to produce sensors and biosensors [21,22] as well as photonic crystals [23]. Using them as a nanofiller in composite nanofibres contributes to an improvement of the mechanical properties of the obtained fibrous composite layers, meaning that this type of material can be used in the manufacture of lithium-ion batteries [24,25]. In addition, fibrous composite materials based on PVP that are reinforced with SiO_2 nanoparticles can be used as temperature-regulating textiles, solar energy utilisation, building energy efficiency and have potential in building energy conservation [26,27].

This work focuses on an analysis of the morphology and optical properties of composite PVP nanofibres reinforced with SiO_2 nanostructures. In the first place, amorphous SiO_2 nanoparticles were produced using the zol-gel method, the structure and morphology of which were tested using transmission electron microscopy (TEM) and X-ray diffraction analysis (XRD). Then, using the generated SiO_2 nanostructures, the spinning solutions were prepared based on PVP and ethanol containing 0 and 5% mass concentrations of nanopowders used to produce a thin polymer and composite mats using the electrospinning method. The morphology and chemical composition of the produced materials were tested using a scanning electron microscope (SEM) with an energy dispersive spectrometer (EDS). An analysis of the impact of the reinforcing phase on the optical properties of nanofibres was carried out on the basis of UV–vis spectra, based on which the rated values of band gaps of the produced thin fibrous mats were assessed.

2. Experimental section

2.1. Synthesis of SiO_2 nanoparticles

Four components were used for the preparation of silica sols: tetraethyl orthosilicate (TEOS, $Si(OC_2H_5)_4$ with purity of ≥97%), hydrochloric acid (HCl, 38%), ethanol (anhydrous C_3H_5OH, with purity of 99.8%) and distilled water. The silica precursor was mixed