Biodegradable Polylactide (PLA) Fiber Mats Containing Al$_2$O$_3$-Ag Nanopowder Prepared by Electrospinning Technique - Antibacterial Properties

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Abstract: This article presents a non-complex method of producing biodegradable polylactide (PLA) fiber mats containing Al$_2$O$_3$-Ag nanopowder which display strong antimicrobial activities against E. coli and S. lutea. The method of preparing fiber mats was electrospinning. This article also gathers the results of the analysis of morphology and mechanical properties of both the Al$_2$O$_3$ nanopowder and the PLA-Al$_2$O$_3$/Ag fiber mats. The examination of the Al$_2$O$_3$ nanopowder was conducted with the use of a scanning electron microscope (SEM) and surface area measurements (BET). The morphology of the PLA-Al$_2$O$_3$/Ag fiber mats was examined using SEM and TEM. The results of the study confirm the great potential of the electrospun PLA-Al$_2$O$_3$/Ag fiber mats for antibacterial application.

Keywords: Antibacterial, PLA, Electrospinning, Fiber mats, Al$_2$O$_3$-Ag

Introduction

In the last decade a great part of research on electrospun fibers was centred on possible biomedical application of these materials [1,2].

Electrospinning has received much interest with regard to fabricating bioengineering materials [3]. This technique is a significant invention in the production of fibers, as it allows manufacturing fibers ranging in diameter from a few nanometres up to 1 mm [4,5]. Such fibers can be applied in various fields, such as aerosol filtration [5], drug and gene delivery [6,7] and many others. Many biocompatible and bioresorbable polymers can be spun with the electrostatic spinning process [8-10]. However, a lot of devices can often cause bacterial infections [11,12]. The presence of infections may pose a serious challenge in the process of tissue engineering.

The incorporation of inorganic particles into electrospun fibers was noted in the literature. A good illustration being the use of hydroxyapatite for bone tissue engineering [13], silver for antibacterial wound dressing [14] or for inhibiting ability to yeast cells [15,16]. Nanofibrous organic and inorganic composite scaffolds containing nano-sized demineralized bone powders with biodegradable PLA by electrospinning may serve as a favorable matrix for the regeneration of bone tissue [17].

The antibiotic properties of silver have been known for ages. Nowadays, researchers are looking for new applications of silver, especially for medical materials [18]. The Ag nanoparticles have the ability to kill a great variety of Gram-positive and Gram-negative bacteria strains and fungi strains [19]. An additional advantage of silver nano-particles over antibiotics is that micro-organisms cannot grow immune against nano-particles, while they can grow immune against antibiotics [20]. Al$_2$O$_3$/Ag nanopowder is biocompatible and shows good bactericidal and fungicidal properties [21,22].

Alumina is one of the most important ceramics for structural applications. It also exhibits excellent biocompatibility, bonding [23-25] and nontoxic properties [26]. Decreasing grain size has shown to improve their hardness and wear resistance as well as strength [27]. Unfortunately, most of the methods to synthesise nanoalumina which are described in the literature of the field are limited in terms of scaling and evaluating [28]. Another problem is the agglomeration process, which occurs during calcinations and leads to the formation of undesired impurities. The use of the innovatory method of nanoalumina and Al$_2$O$_3$/Ag synthesis, described below enables eliminating some of the problems, especially forming the undesired impurities [21,29-31].

Polylactide (PLA) is produced in the lactide ring-opening polymerization process [18]. Due to its high biocompatibility and ability to decompose over a period of time [32], this polymer has been given a great deal of interest and has been widely used in biomedicine e.g. in tissue scaffolds and implants [33-35].

During the research described below, the authors developed a simple method of producing of polylactide (PLA) fiber mats containing Al$_2$O$_3$/Ag nanopowder prepared by electrospinning techniques, which exhibit strong antibacterial activities.

Materials and Methods

Production of Fiber Mats

Al$_2$O$_3$/Ag Synthesis

The organic precursor of aluminum oxide was prepared
obtained was then subjected to reduction in a hydrogen
deposited on aluminum nano-oxide. The intermediary product
process gave the single product-valent silver oxide nanoparticles
triisopropoxide ((i-PrO)$_3$Ag) position in air at 700°C for 24 h, which yielded the final product of each parameter was then estimated. All quantitative analysis
were estimated for their cross-sections and the average value
using stereological methodology [37]. Particles parameters
were estimated for their cross-sections and the average value
of each parameter was then estimated. All quantitative analysis
was performed using the MicroMeter v.086b computer
program.

The Brunauer-Emmet-Teller (BET) specific surface area was measured using the Quadsorb-SI Quantachrome equipment. Before the BET measurements, all samples were degassed in 350°C for 24 h. The process of adsorption was conducted in −195.8°C bath and gaseous nitrogen was used as an adsorbate. Also alumina nanopowder was analysed by an X-ray diffraction method (PHILIPS PW 1830 with Cu K$_\alpha$ radiation).

The PLA-Al$_2$O$_3$/Ag nanocomposite fiber mats were also examined with a transmission electron microscope (TEM, PHILIPS CM 20) operating in a high-resolution mode, chiefly in order to identify the Al$_2$O$_3$/Ag nanoparticles. The sample - single fiber was placed first on the Cu netting and then on a carbon tape. The TEM photographs were taken in both light and dark fields. The PLA-Al$_2$O$_3$/Ag nanocomposite fiber mats were examined by energy dispersive X-ray spectroscopy (EDS unit coupled with the TEM employed). The analysis provided information concerning the local contents of the elements present in the given region.

The average diameters of fibers were measured by a Nikon Eclipse 80i optical microscope and further analyses were performed using the NIS-Elements BR 3.00 SP2, Nikon, computer program.

Mechanical Properties of Fiber Mats
The mechanical properties were analysed with the use of an Instron 5566. The fiber mats were prepared in the form of standard dumbbell shapes (length 30 mm) according to the ASTM Standard D 638 and tensile properties were calculated using the Bluehill 2 programme, version 2.14 provided by Instron. The sample was vertically mounted on two mechanical gripping units of the tensile tester at their ends. Deforming speed was set at 10 mm/min. Cross sections of the fiber and the composite specimens were examined with the use of a micrometer. The average value of tensile strength was tested on the basis of 5 specimens of each composite.

Bactericidal and Fungicidal Activity of the New Nano-
composite PLA-Al$_2$O$_3$/Ag
The bactericidal and fungicidal activity of the PLA-Al$_2$O$_3$/Ag fiber mats were analysed qualitatively using the spread plate method. The biocidal efficiencies of fiber mats were assessed by means of the Kirby-Bauer method [36]. The microorganisms examined in this experiment came from a private collection of the Faculty of Environmental Engineering of Biology Department, Warsaw University of Technology. The microorganisms used in the experiment included bacteria strains such as Gram-negative *Escherichia coli* and Gram-positive *Sarcina lutea*. The bacteria strains were inoculated on a nutritive agar substrate (MERCK nutritive agar for bacteria). The substrate was appropriately prepared and sterilized and then deposited on the surface of a Petri plate. The inoculation was performed using a sterile glass rod. The bacteria were inoculated on the whole substrate surface on which an appropriate fiber mat (specimen - ring shape with