Influence of the preparation method on selected properties of PLA nanofibers modified with lavender oil *(Rapid Communication)*

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Abstract: PLA nanofibers containing 30 wt% lavender essential oil (LEO) was obtained by electrospinning (ES) and solution blow spinning (SBS) methods. The structure was assessed by scanning electron microscopy (SEM), and thermal properties were determined by thermogravimetric analysis (TGA). The ES method produced thinner nanofibers than the SBS method. The method of obtaining nanofibers did not affect their thermal properties.

Keywords: solution blow spinning, electrospinning, nanofibers, PLA, lavender oil.

Wpływ sposobu otrzymywania na wybrane właściwości nanowłókien PLA modyfikowanych olejkiem lawendowym *(Komunikat szybkiego druku)*

Streszczenie: Metodą elektroprzędzenia (ES) i przędzenia z rozdmuchiwaniem (SBS) otrzymano nanowłókna PLA zawierające 30% mas. olejku lawendowego (LEO). Do oceny struktury stosowano skaningową mikroskopię elektronową (SEM), a do oznaczenia właściwości termicznych analizę termograwimetryczną (TGA). Metodą ES uzyskano cieńsze nanowłókna niż metodą SBS. Sposób otrzymywania nanowłókien nie miał wpływu na ich właściwości termiczne.

Słowa kluczowe: przędzenie z rozdmuchiwaniem, elektroprzędzenie, nanowłókna, PLA, olejek lawendowy.

Electrospinning (ES) method is gaining more attention in the field of material and food technology. The method involves creating a ribbon of micro- or nano-scale fibers using high voltage that is applied to a liquid solution as it is extruded from a nozzle [1]. Electrospinning technology is a continuation of traditional electro spraying technology that has been used for many years [2]. For electrospinning, various voltages can be connected to nozzle, 16 kV, 20 kV and 30 kV are the most used [3-5]. Despite many advantages, this method is time-consuming and can be dangerous, because of a high electrical environment that is used. Solution blow spinning (SBS) has been used as a versatile alternative to electrospinning, because of its safer nature (lack of high voltage) and manual controllability [6]. SBS uses compressed gas flow, and the spray apparatus consists of concentric nozzles. The pump regulates the injection rate, and the compressor regulates the pressure. As the solvent evaporates, the fibers

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are extruded and collected on a disc, which can be made of a variety of materials. SBS is not a new technique, it is a method adapted for drug delivery purposes and used by different research groups [7, 8].

Poly(lactic acid) (PLA) is an environmentally friendly polyester with biocompatibility and obtained by condensation polymerization of lactic acid [9]. Essential oils are incorporated into the nanofiber structure for food applications [10]. Nanofibers with different properties can be produced by adding essential oils to PLA-based nanofibers [11]. Packaging food products in nanocomposite films obtained form biopolymers containing essential oils is common technique that can extend the shelf life of foods by preventing microbial growth [12,13]. For food applications, it is also possible to produce nanofibrous mat loaded with essential oils as flavoring and bioactive compounds [14].

Essential oils of the lavender plant (*Lavandula L.*) are preferred in food, cosmetics, and fragrance production [15]. Lavender plant essential oils, which have antimicrobial, anticholinesterase and antioxidant properties, function as active ingredients for skin treatment in the medical field and as bio-pesticides in the agriculture [16]. Thanks to PLA nanofibers containing essential oils,

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fruit contamination with fungi can be controlled, which results in extending their shelf life [17]. Due to their biocidal properties, these nanofibers can be used in food packaging. Ozcan *et al.* investigated the possibility of using SBS method to obtain PLA modified with essential oils from sausage spices. [18]. When ES and SBS methods were compared in the production of PLA-based nanofibrous membranes, it turned out that the membranes made using these methods allows controlled release for up to 500 hours [19]. Combining poly(vinyl alcohol) nanofibers with aqueous plant extracts and applying them as strawberry coating, nanofibers produced by SBS method have bioactive compounds and were able to extend the shelf life of strawberries [20].

It can be concluded that the ES method is often used in the nanofibers production, but the high voltage used in this method is always an important risk factor. For this reason, the SBS method is becoming more and more popular because it is safe, fast, portable and widely applicable.

Therefore, in this study, the author compared the electrospinning (ES) and solution blow spinning (SBS) methods to produce PLA nanofibers modified with 30 wt% wild lavender (*Lavandula stoechas*) essential oil. The obtained nanofibers were characterized by SEM and TGA.

EXPERIMENTAL PART

Materials

Poly(lactic acid) (PLA) (Mn=160000 g/mol) was delivered by Nature Works LLC (4043 D Nebraska, USA). *N,N*-dimethylformamide (DMF) (anhydrous, 99.8%) and 100% pure wild lavender essential oil (LEO) were purchased from Sigma-Aldrich (Taufkirchen Germany) and BAÇEM (Balıkesir Metropolitan Municipality Rural Services, Türkiye), respectively*.*

Samples preparation

PLA/LEO solution preparation

To obtain a homogeneous mixture 0.4 g PLA was added into 5 ml DMF (v/v) and dissolved in a magnetic stirrer at room temperature. After that, LEO was added (w/v) to obtain 30% solution and was mixed for 1 hour [21].

Solution blow spinning of PLA/LEO nanofibers

In the case of blow spinning, the formulation given by Zhang *et al.* [22] has been modified. The PLA/LEO 70/30 solution was added to the prepared portable air injector (fine needle atomizing spray 0.3 mm; SKY-4 – Automatic) shown in Fig. 1a. The solution was sprayed at a pressure of 0.3 MPa and 170 mm to the collector [22].

Electrospinning of PLA/LEO nanofibers

To obtain PLA/LEO 70/30 nanofibers by electrospinning, the prepared solution was placed in a 5 ml syringe in the NE 100 electrospinning device (Inovenso LLC. Istanbul, Türkiye). The process was carried out at a voltage of 16.5 kV and a flow rate of 0.67 mL/h. The distance of the nozzle from the collector tray was 170 mm (Fig. 1b) [23].

Methods

The morphology of PLA/LEO nanofibers was examined using a SEM-EDX scanning electron microscope (JEOL JSM-7100-F, Tokyo, Japan) at an operating voltage of 15 kV. The conductivity was increased by coating the samples (Quarum Coated Device) with Au–Pd (80/20%) [24]. Thermogravimetric analysis (TGA) was performed in a nitrogen atmosphere using a thermogravimetric

Fig. 1. Equipment for obtaining PLA/LEO nanofibers: a) SBS method, b) ES method

 $10 \mu m$

Fig. 2. SEM images of nanofibers: a) PLA obtained by SBS, b) PLA obtained by ES, c) PLA/LEO obtained by SBS, d) PLA/LEO obtained by ES

 $\frac{1}{1} \mu m$

Fig. 3. TGA and DTG curves: a) ES–PLA/LEO, b) SBS–PLA/LEO

analyzer (SDT Q600 V20.9 Structure 20, TA Instruments, New Castle, DE, USA) at a heating rate of 10°C/min and a temperature range of 30–600°C [25].

RESULTS AND DISCUSSION

Morphology

SEM shows that the diameter of the nanofibers obtained by SBS from pure PLA and PLA/LEO is 0.55–0.65 µm and 0.40–0.92 µm, respectively. The thickness of pure PLA nanofibers obtained by the ES method is $0.21-0.86 \mu m$ and 0.54–0.88 µm in the case of PLA/LEO nanofibers (Fig. 2). Thicker nanofibers were obtained by SBS method. The action of high voltage electric current leads to obtaining nanofibers with a more even arrangement and smaller diameter. However, the lack of an electric field results in an irregular arrangement of the nanofibers, increasing their diameter.

Thermal properties

TGA results show that PLA/LEO nanofibers obtained by ES method, LEO degraded at a temperature of 144°C, while PLA started to decompose at a temperature of about 340°C. In the case of the same nanofibers produced using the SBS method, the degradation of LEO and PLA occurs at temperatures of 114°C and 336°C, respectively (Fig. 3). These results showed that the thermal properties of LEOmodified PLA nanofibers are similar and both techniques produced nanofibers with similar thermal resistance.

Oliveira *et al*. observed that the thickness of pure PLA nanofibers is approximately 289 nm for the SBS method and 159 nm for the ES method [26]. Scaffaro *et al.* observed that both techniques allow the production of nanometric fibers with similar mechanical parameters [27]. The diameters of electrospun and blown nanofibers were similar and ranged from 176 to 240 nm [28]. On the other hand, when comparative studies of ES and SBS processes were carried out to produce poly(L-lactic acid) nanofibrous materials for biomedical engineering, the materials produced by the SBS method showed a similar structure to those produced by the ES method [29]. An innovative drug delivery system in nanofiber membranes produced by slow blow spinning compared to electrospinning has been proven to be effective and efficient in encapsulating both processes in nanofiber membranes [30].

CONCLUSIONS

PLA/LEO nanofibers were obtained by ES and SBS methods. It was found that the diameter of the nanofibers depended on the method used, with thinner fibers obtained using the ES method, which was due to a more uniform arrangement of the fibers caused by the presence of the electric field. TGA results showed that both ES and SBS methods produced nanofibers with similar thermal properties. The results indicate that the SBS method may be a useful alternative method for nanofiber production, especially in the food, medical and cosmetics industries, in terms of ease of use, occupational health and safety, and cost-effectiveness.

Author contribution

T.G.E. – conceptualization, methodology, validation, investigation, writing-original draft, writing-review and editing, visualization.

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Conflict of interest

The authors declare no conflict of interest.

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