Abstract

The paper presents results of experiments on formation of nonwoven fabrics from PLA biopolymer. Fibrous structures with parameters advantageous for application as a scaffold for tissue engineering were prepared from polylactide by melt-blown technology. Process optimization enables to obtain ultra-fine fibers. Sizes of pores formed in the material during melt-blown process can be tailored by formation of fibers of defined diameters.

Keywords: melt-blown, fibers, PLA, nonwoven

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Introduction

Fibrous structures due to the advantageous high surface to volume ratio are good materials for tissue scaffolding purposes. When engineering fibrous scaffolds not only fibers diameters but also distances between fibers, forming pores should be taken into account, to ensure cells migration into the fibrous network and cells mobility within this network. In order to do this one need to remember is that the average size of cell can be at the range of 10-50 µm in the case of osteoblast, fibroblast or chondrocyte [1], so that the pores of sufficient diameters are indispensable to avoid shearing of cells during their migration within the fibrous network [2].

Ultra-thin polymer fibers have great potential for applications in a wide variety of fields, including sensors, filtration and separation membranes or biomedical applications, however the final application strongly depends on polymer composition used for fibers spinning. Conventional methods of fiber formation based on fiber drawing allow to obtain fibers of diameters at the level of 10-100 µm. In order to obtain fibers of smaller diameters one has to apply more complicated methods, including bi-component spinning of two different polymers, melt-blown or electrospinning [3-5]. Each of those methods has advantages and drawbacks. Bi-component spinning of two different polymers allows to obtain continuous bunches of parallel, long micro fibers, however it is a multi-step process. The electrospinning is a versatile method of formation on ultra-thin fibers both from the melt and the solutions, however is still a challenge to scale-up of the process up to quantities of kg/h. The third method, melt-blown is a method capable of formation ultra-fine fibers in form of nonwoven in a one stage, but it is limited only to thermoplastic polymers [3,5]. In melt-blown process molten polymer is extruded from the die holes, and then streams of high velocity hot air attenuate the polymer streams to form microfibers, which are subsequently laid randomly on the collecting screen forming self-bonded nonwoven web.

Fibers’ thickness depends on combination of parameters including melt-temperature and viscosity as well as velocity and temperature of hot air. The schematic draw of the process is presented in FIG. 1. Even though polypolyactide has been so far the most popular polymer used in melt-blown processes, the other thermoplastic polymers, including polylactide, can be used in this process as well [6]. In this work we will discuss morphology and selected physical properties of 3D structures obtained by melt-blown spinning of polylactide which indicate that melt-blown spinning of PLA can be potentially useful for fabrication of biodegradable polymeric scaffolds for tissue engineering.

Materials and methods

Nonwoven samples were fabricated from commercial grade NatureWorks®PLA 3051D using lab-scale melt-blown setup WX34 based on single screw extruder (d=25 mm) and flat die head with 80 orifices (diameter 0.5 mm) which enable formation of nonwoven web of 80-100 mm width. The efficiency of WX34 setup is up to 10 kg/h which can be reached in the case polypolyactide with high melt-flow index, but in presented case nonwovens were formed with efficiency of 0.4; 0.6; 0.8 and 1 kg/h. Air velocity which attenuated the molten polymer stream into the fiber was at the range of 50-150 m/s. Other processing parameters such as temperatures, pressure, output and conveyor velocity were registered automatically and are given in TABLE 1. Processing temperatures were evaluated from DSC experiments (TA Instruments 5100). In order to prevent viscosity changes due to hydrothermal degradation nonwovens were formed from polymer dried previously to the water content of 0.025% (250 ppm), according to producer’s recommendation.

Diameters of fibers were estimated based on SEM analysis (Jeol JSM 5500LV). Basis weight of fibrous webs was measured according to standard (PN-83/P-04802). Timlet-73 setup was used to measure the thickness. Density of nonwoven was calculated from samples geometry. Mean pore size and pore sizes distribution were estimated using PMI capillary flow porometer.

TABLE 1. Processing parameters of melt-blown PLA nonwovens formation.

<table>
<thead>
<tr>
<th>Temperature of heating zones [°C]</th>
<th>I</th>
<th>195</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature of pump [°C]</td>
<td>-</td>
<td>255</td>
</tr>
<tr>
<td>Temperature of spinret [°C]</td>
<td>-</td>
<td>250</td>
</tr>
<tr>
<td>Melt temperature [°C]</td>
<td>-</td>
<td>268</td>
</tr>
<tr>
<td>Temperature of air [°C]</td>
<td>at heater</td>
<td>250</td>
</tr>
<tr>
<td>Distance from spinret to conveyor [mm]</td>
<td>at the air outflow</td>
<td>215</td>
</tr>
</tbody>
</table>

FIG. 1. Schematic drawing of melt-blown process.
Results and Discussions

According to DSC analysis (FIG. 2) NatureWorks PLA 3051D should be processed at temperature above 160°C. The polymer is stable at least to 250°C, however TGA (not shown here) confirmed stability up to at least 280°C. The temperature of molten polymer during the process was established experimentally, that is at registered melt temperature of 268°C process was stable and quality of obtained nonwovens was satisfactory (organoleptic estimation).

As observed from SEM microphotographs presented in FIG. 3 diameters of fibers depend on process efficiency and air velocity. The thinnest fibers with diameters at the level of up to 5 µm were obtained in the case of spinning with medium air velocity (100 m/s) and the lowest efficiency. Too high air velocity (150 m/s) resulted in breaking of polymer streams before they were attenuated into thin fibers. As the effect relatively thick entangled fibers were formed. Due to the intensive contraction of the solidifying broken fibers there can be observed characteristic structures on the fibers’ surface (FIG. 3 b1, c1), which were not observed in the case of fibers solidified by air of lowest velocity (50 m/s) (FIG. 3 a1). On the other hand when the air velocity is too low (~50 m/s) the drawing forces are not enough to form thin fibers, however the fibers are relatively straight and smooth. The surface roughness which is the result of uneven radial solidification resulting in shrinkage of polymer can be beneficial as it potentially could enhance cells attachment.

Melt blown process allows to fabricate materials of controlled porosity, dependent mainly on the diameters of entangled fibers, as only thickness of fibers crossing each other limits distances between them [7]. Results of porosimetric studies (TABLE 2) have confirmed that the lower were diameters of fibers the smaller were average pores sizes. The same was observed in the case of pore sizes distribution, which was much more narrow in the case of the thinnest fibers. The knowledge of processing parameters is critical for the sake of construction of 3D structure, which can be formed by subsequent formation of layers composed of fibers of different diameters and porosity. The example of such possible structure is presented in FIG. 4.
Conclusions

Melt-blown technology was applied to obtain microfibrous nonwoven structures from polylactide. Pore sizes of the nonwoven and their distribution suggest that the process can be appropriate to fabricate fibrous scaffolds for tissue engineering. Process parameters enable to tailor diameters of fibers and density of nonwoven structures. Moreover the efficiency of the process is high as it is possible to take advantage of existing textile technologies.

Acknowledgments

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TABLE 2. Parameters of nonwovens obtained by melt-blown process.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Basis weight [g/m²]</th>
<th>Thickness of nonwoven [mm]</th>
<th>Average pore sizes [μm]</th>
<th>Main fraction of pores [μm]</th>
<th>„Bubble point“ (*) [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>270</td>
<td>3.05</td>
<td>45.1</td>
<td>41.8-47.4</td>
<td>412</td>
</tr>
<tr>
<td>b</td>
<td>120</td>
<td>1.25</td>
<td>82.5</td>
<td>79.2-86.2</td>
<td>420</td>
</tr>
<tr>
<td>c</td>
<td>30</td>
<td>0.38</td>
<td>13.8</td>
<td>13.4-14.3</td>
<td>23</td>
</tr>
</tbody>
</table>

(*) Bubble point - material's largest through-pore

References