

PRODUCTION OF X-RAY DETECTABLE PADS WITH THE AID OF ELECTROSPINNING

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ABSTRACT

Electrospinning of Poly-L-lactic acid (PLLA) containing various amount of barium sulfate as an X-ray detectable material was the aim of this research project. For this purpose, the feasibility of replacing the electrospun PLLA fiber instead of commercially barium sulfate yarn in medical pads was examined. Electrospinning of PLLA fiber was prepared in the mixture of Chloroform/Dimethylformamide (DMF) (85/15 v/v) solvent. PLLA was solved in chloroform containing 10, 30 and 50 percentages of barium sulfate to polymer weight in the presence of sodium dodecyl sulfate as a surfactant prepared in Dimethylformamide (DMF). Then, the polymer solution was placed in an ultrasonic bath. Optimized electrospinning parameters to obtaining suitable fibers without beads were achieved. All samples were analyzed using SEM, XRD, and, x-ray imaging medical equipments. All results confirmed the presence of barium sulfate in electrospun fibers. Finally, the electrospun fibers as X-ray detectable fibers were examined by X-ray image- detecting device in Mostafa Khomeini hospital (a local hospital). The images completely revealed the PLLA fiber containing barium sulfate as an X-ray detectable pads.

KEYWORDS

medical gauzes; X-ray detectability; surgical gauzes; electrospinning; Poly-L-lactic acid; barium sulfate.

1. INTRODUCTION

The first use of sterilized gauze for medical purposes dates back to the late 1800's (Hall, Ponder, 1992). The remaining of these gauzes in the patient's body has long been the cause of many problems for patients and doctors (Gawande et al., 2003). Because the pads are immobile in the body, as they are not absorbent, they begin to become a composite with a cotton matrix because they do not participate in any biochemical reaction such as hydrolysis (Risher, McKinnon, 1991; Zeltzman, Downs, 2011; Olnick et al., 1955).

The sponge that has been stuffed in the body can lead to two types of reactions: the first is a granuloma that is used to separate the external component of the body (Zeltzman, Downs, 2011; Olnick et al., 1955). The second reaction is the secretion and creation of abscess, which is commonly associated with bacterial invasion (Hyslop, Maull, 1982). These reactions may be at the expense of the patient's life or cause irreparable damage. As a result, finding solutions to the problem is one of the issues of the day.

According to the latest achievements, in order to detect gauzes in the surgical site, a strip called the x-ray sign attaches to them by heating during the process of producing them. This tape absorbs the x-rays and shows the location of the gauze by x-ray imaging (Zeltzman, Downs, 2011).

Poly-lactic acid is a FDA approved thermoplastic aliphatic polyester which has been used for many uses in tissue engineering, food packaging, automotive industry, etc. it is due to its high strength, transparency, biocompatibility and biodegradability. Despite numerous advantages of poly-lactic acid some properties

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like low temperature resistance, inherent fragility, low melting stability and crystalline velocity, limit the applications of this polymer (Zeng et al., 2015).

The X-ray is part of electromagnetic radiation with a very short wavelength of about 0.01 to 10 nm, equivalent to 30 Petahertz to 30 Exahertz, and energy between 100 eV and 100 keV. This beam has a lot of penetration power and passes almost from anything except bone and metal (orbital d), and this is the key characteristic of the beam for exploiting it in proportion to our need. Typically, high atomic elements can reduce the intensity of X-rays through photoelectric effects, reduce radiation damage, or cause discrepancies in the detection of different components of the imaging location, which is why lead is often used as a preventative agent. X-rays are used in radiology (Qu et al., 2015). In this application, the most commonly used radiation reducing agent is barium sulfate. Barium sulfate is approved by FDA and therefore it used directly in many medical imaging equipments.

In this study, nanocomposite fibre containing barium sulfate and other components produced by electrospinning method. Electrospinning is one of the solutions for the production of contrasting fibres, in which barium sulfate particles are used in composite or nanocomposite polymer formations. However, the production of contrasting fibres is also possible in other ways; for example, Que et al. (Qu et al., 2015) produced fibres from a mixture of barium sulfate and regenerated cotton through wet spinning. The results of the X-ray diffraction of the fabric showed that the larger the number of fabric layers, the brighter the x-ray image.

In this research, medical gauze X-ray absorption on the whole surface of the pad was investigated. As a result, Poly-L-lactic acid (PLLA) solution containing various amount of barium sulfate as an X-ray detectable material has been electrospun and analyze properly.

2. MATERIALS AND METHODS

Poly-L-lactic acid [$M_n = 420\,000$, $M_w = 180\,000$] was purchased from Germany's Böhlinger Co..

Chloroform with purity of 99.5% and dimethylformamide (DMF) with a purity of 99.5% were purchased from Merck and utilized as solvent. Barium sulfate was purchased from Daroopakhsh Co. and obtained in iran. sodium dodecyl sulfate (SDS) used as surfactant with molecular weight of 288.372 g/mol and a density of 1.01 g/cm³ purchased from Merck, Germany.

The imaging was carried out with a Ziehm 8000 X-ray Imaging X-ray machine made in Germany and available at Mostafa Khomeini Hospital.

The x-ray diffractometer used in this investigation was Inel Equinx 3000 made in USA with a voltage of about 40 kV and a current of about 30 MA.

To prepare a polymer solution of Poly-L-lactic acid without added additives, the polymer was first dissolved in the main solvent (chloroform) for two hours under a magnetic stirrer. The surfactant was solved in the second solvent, and after the two solutions were homogenized, the two solutions were added together. Then, using a magnetic stirrer for 30 minutes, homogeneous and single-phase solutions (at ambient temperature) were prepared for the electrospinning process.

In the electrospinning process, polymeric solution of PLLA was used in an electrospinning device equipped with a high voltage source and a power supply with the ability to control the solution rate.

In this process, the polymer solution was transferred into a 1-cc syringe to a needle with its specific internal diameter, and after adjusting the parameters of the electrospinning process referred to in Table 1, the polymer solution was prepared to spin at relative temperature and relative humidity. The most commonly used electrospinning system in this study is a pump, syringe and needle. During the electrospinning, the first drop of the needle is removed, then in the presence of the voltage, a Taylor cone is formed, while in the absence of the field, the polymer will drip from the needle. Ultimately, the surface tension is overcome and the droplet is guided to the collector and the fibre created. The tensile force is very intense (the ratio of the diameter of the fibre to the diameter of the droplet) and the solvent evaporates during the elongation

and production of the fibre. The severity of lowering the diameter of the lip is related to many factors that are not the subject of this research.

Table 1: Electrospinning parameters

Temperature (C°)	Voltage (KV)	Distance (Cm)	Feed rate(ml.h ⁻¹)	δm (Mpa) ^{0.5}	Solvent (CHCl ₃ /DMF)	concentration
30	20	10	0.5	19.28	85/15	4(wt%)

In this study, an innovative idea of the possibility of electrospinning of contrasting layers on Medical cotton pads Common in Surgical Operations was investigated. As the cotton pads were placed on the folding plate and the process of electrospinning continued as before, this process of manufacturing medical pads will greatly improve the application of these pads. The radiological devices image did not differ significantly from the web of poly-lactic acid and the web on the cotton pad which will be fully explained in the results section.

3. RESULTS AND DISCUSSIONS

To evaluate the x-ray detectability of the pad obtained and presence of crystalline structures in the fibre bed, the experiments were carried out.

3.1. SEM

SEM image showed that the Barium sulfate has no significant effect on the uniformity of the fiber diameter. The standard deviation of 0.1120 confirmed this conclusion.

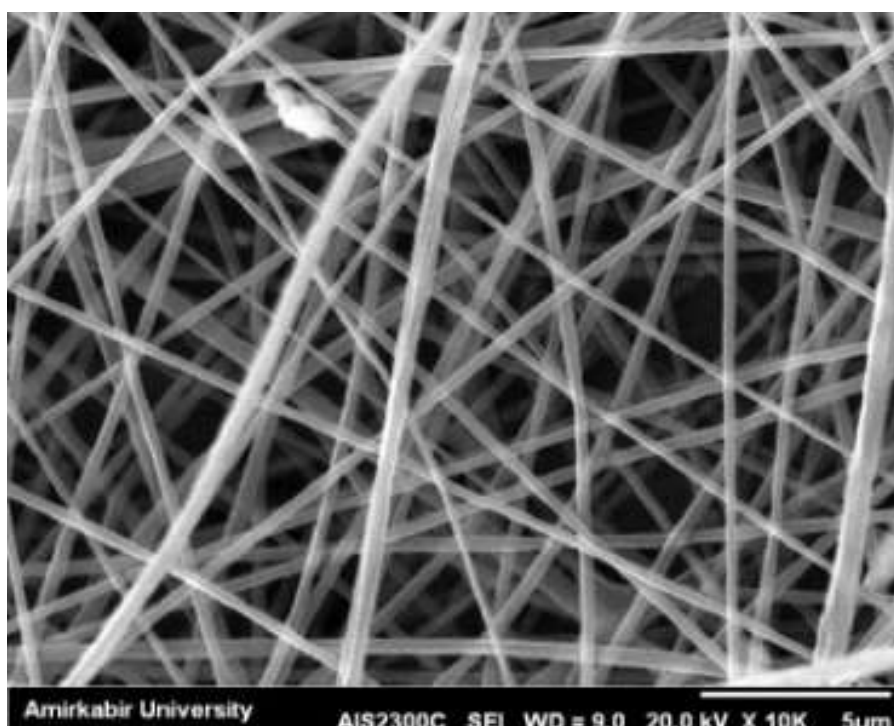


Figure 1: SEM image of PLLA solution containing barium sulfate

3.2. EDS

In the images below EDS test results confirmed the presence of barium sulfate in the context of the resulting fibre.

EDS test results of Poly-lactic acid with surfactant, without barium sulfate, 10% and 30% of barium sulfate are shown in Table 2.

Table 2: EDS test result of Poly-lactic acid with surfactant, without barium sulfate (1), 10% (2) and 30% (3) of barium sulfate

1	<table border="1"> <thead> <tr> <th>A%</th> <th>W%</th> <th>Line</th> <th>Elt</th> </tr> </thead> <tbody> <tr> <td>52.79</td> <td>45.37</td> <td>Ka</td> <td>C</td> </tr> <tr> <td>46.96</td> <td>53.76</td> <td>Ka</td> <td>O</td> </tr> <tr> <td>0.22</td> <td>0.50</td> <td>Ka</td> <td>S</td> </tr> <tr> <td>0.04</td> <td>0.37</td> <td>La</td> <td>Ba</td> </tr> <tr> <td>100.00</td> <td>100.00</td> <td></td> <td></td> </tr> </tbody> </table>	A%	W%	Line	Elt	52.79	45.37	Ka	C	46.96	53.76	Ka	O	0.22	0.50	Ka	S	0.04	0.37	La	Ba	100.00	100.00			<p>EDS spectrum for sample 1. The x-axis is energy in keV (0 to 15) and the y-axis is intensity (0 to 10000). Peaks are labeled: O Kα, S Kβ, S Kα, BaLγ1, BaLα, BaLβ, BaLγ2, BaLγ11.</p>
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According to the results, the amount of barium sulfate in the fibre bed is increasing with increasing of barium sulfate content in the polymer solution, but the increase in slope of barium sulfate from the solution without barium sulfate up to 10% is much higher than the increase in slope of barium sulfate from the solution obtained from sulfate 10% to 30%, which can be justified by the interrupting components.

3.3. XRD

Pure barium sulfate, electrospinning web containing PLLA and surfactant without barium sulfate and webs with different amount of barium sulfate and also, the cotton pads on which the polymeric solution had been electrospun has been studied through XRD analyze.

The results of the XRD test were analyzed in such a way that the wide areas represent the amorphous regions and sharp peaks indicating the crystalline regions and their extent. The amorphous portion is attributed to the polymer, and the crystalline section with a very good approximation represents barium sulfate content.

The results of the test confirm the contents of the polymer solution in the fibre bed in Figure 2.

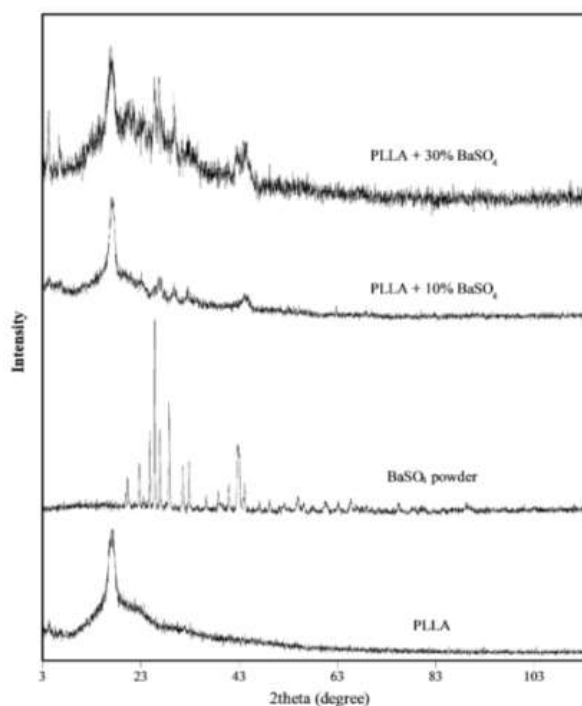


Figure 2: XRD test results for Electrospinning webs

Electrospun polymer solution containing surfactant and 30 wt% barium sulfate on a cotton pad and 12 hours located in distilled water, showed that the immersion in water does not affect the crystalline structures (Figure 3).

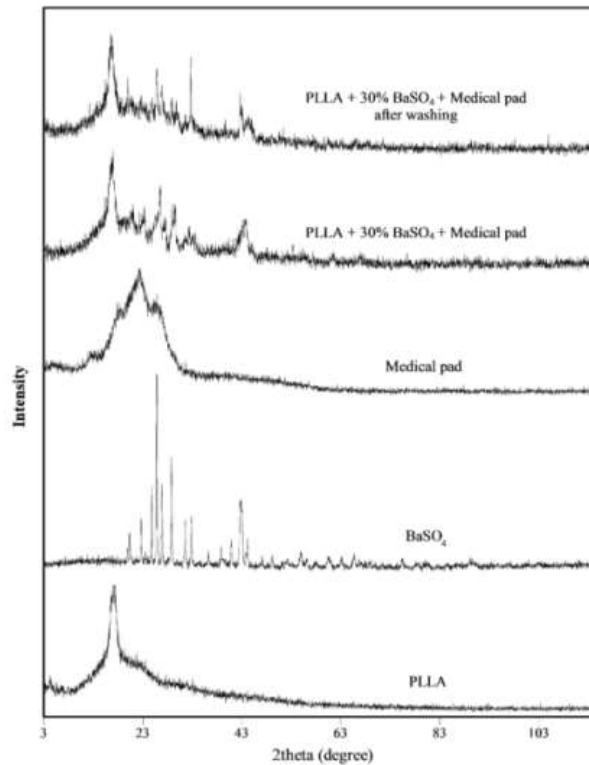


Figure 3: XRD results of cotton web on which the polymeric solution had been electrospun

According to the references in the angles of 23.5, 26.6, 29.5, 32.5, 73.7 degrees orthorhombic crystals of barium sulfate has been observed, the XRD test results in this study showed peaks at 23.25, 26.6, 30 and 32.5 angles as well (Ramaswamy et al., 2010).

3.4. Medical devices x-ray photography

The electrospun samples of PLLA containing 10% and 30% barium sulfate was placed between two pieces of barium sulfate industrial tapes and tested by medical devices x-ray photography. The results showed that 10% barium sulfate is not enough to detect but 30% of barium sulfate make the web detectable and even after 26 hours of exposure to the serum, it was detectable by X-rays, which confirmed the presence of barium sulfate in the sample.

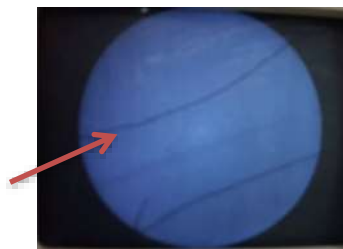


Figure 4: PLLA containing 10% barium sulfate

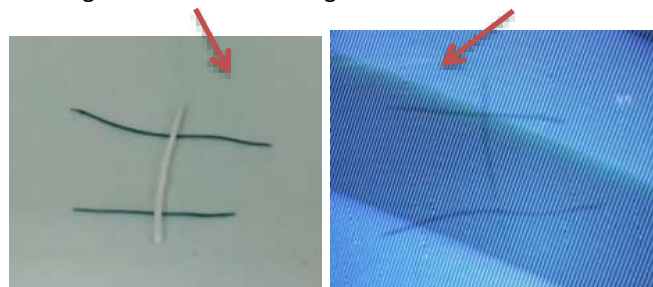


Figure 5: PLLA containing 30% barium sulfate after 26 hours of exposure to the serum

4. CONCLUSION

The results showed that the optimal percentage of barium sulfate, considering the possibility of identification by the radiology devices, is the main factor that improves with increasing barium sulfate content. However, due to the difference in the resolution of the 10% and 30% pad in the radiology device, the 10% pad evaluated Inappropriate. Considering the electrospinning conditions to improve this process and to improve the consistency and non-settling barium sulfate, pad containing 30% barium sulfate can be considered as optimal. The presence of barium sulfate in electrospun fibres bed with different tests was confirmed.

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