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DEVELOPMENT AND PERFORMANCE ANALYSIS OF HIGH VOLTAGE GENERATOR FOR ELECTROSPINNING OF NANO FIBRES

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ABSTRACT
This work develops a high voltage generator (50kV) to power polymer melt solution to produce nano fibre. The performance of this single unit high voltage system was evaluated through electrospinning of polylactide (PLA) melt solutions of 0.09, 0.11, 0.13 and 0.14 g/ml concentrations. Composite samples were also produced by reinforcing 0.1 g/ml PLA melt with 20wt. % palm kernel shell particles (PKS). Tensile, fluid absorption (at 32 and 70°C) and morphological evaluations were carried out on unreinforced electrospun PLA and electrospun PLA/PKS composites. The reinforcement (PKS) provides a stiffening effect on the matrix (PLA) as the composite displays the highest magnitude of Young's modulus (150MPa). The ultimate tensile strength (UTS) of 3MPa of the nano composite is higher than other unreinforced PLA nano fibres except for fibres from 0.14g/ml PLA melt (3.5MPa). The water absorption rate of fibres increases with medium temperature and increase in concentration of PLA melt decreases the fibre size.

Keywords: *High Voltage Generator, Electrospinning, Polymer Melt Solution, Tensile Strength, Water Absorption*.

INTRODUCTION
Electrospun fibre production using electrostatic forces is a prominent approach to fibre production and has become important in the last decades. The process uses electrically charged jet of liquid polymers to make fibres of micro and nano meter dimensions. Fibres obtained with this technique possess several unique properties when compared with fibres made from conventional melt, dry or wet spinning

(Jiang *et al.* 2014). Electrospun fibres are smaller in diameter and longer in length with very large surface area to volume ratio. They are placed closer to each other on a mat when compared to fibres produced from dry or wet spinning techniques. However, fibre diameters have been found to reduce by 50% when applied voltage is doubled (Larrondo and John, 1981a; Larrondo and John, 1981b: Seema, 2013). Conventionally, a high voltage source has two electrodes, one is positive and the other is negative. In the electrospinning, positive end is attached to polymer solution / melt and negative end is connected to the collecting ground. By adjusting the voltage, the required electric field for fibre spinning can be created between the positive and negative ends.

When an electrostatic force is applied by a high voltage source, an electric field is formed at the tip of the syringe where polymer liquid is held by its surface tension. The accumulation of charges in the tip causes repulsion, which opposes the surface tension forces and the higher the voltage the stronger the mutual repulsion of the charges at the tip. With increase of the electric field, the pendant polymer drop at the tip of the needle changes its hemispherical shape and takes a conical shape. This is known as Taylor cone (Taylor, 1964). As the voltage is increased, the volume of the drop at the tip decreases, causing the Taylor cone to recede. The jet originates from the liquid surface within the tip, and more beading is seen. As the voltage is further increased, the jet eventually moves around the edge of the tip, with no visible Taylor cone; with these conditions, the presence of many beads can be observed (Zong *et al*. 2002: Deitzel *et al*. 2001).

Taylor cone stability depends on applied voltage. At higher voltage, greater amount of charge causes the jet to accelerate faster leading to smaller and unstable Taylor cone. Higher voltage leads to greater stretching of the solution as fibre with small diameter are formed.

Matthews *et al*. (2002) showed that applied voltage has effects on the morphology of the resultant fibres. As stated earlier, increase in the applied voltage produces fibre with decrease diameter whereas dried fibre can be produced with a high evaporation rate of the solvent; in some cases, when the viscosity of the solution jet has a low value, application of high voltage may lead to multi jet formation. This can lead to formation of very fine fibre. In addition, bead formation tendency is also affected by changing voltage. Wang *et al*. (2003) measured both jet and fibre diameters and investigated the effect of voltage differentials. The results showed that both jet and fibre diameters decreased slightly. Also, better chain orientation within electrospun fibres was seen with increasing applied voltage. Deitzel *et al*.

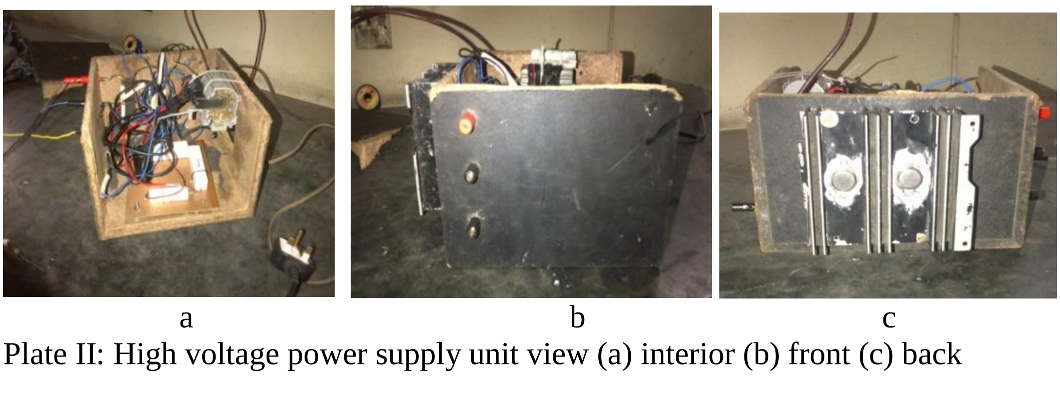
(2001) examined a polyethylene oxide (PEO)/water system and found that increased applied voltage altered the shape of the surface at which the Taylor cone and fibre jet were formed.

MATERIALS AND METHODS

Previous Design

In an earlier work, Adeosun *et al*. (2015) developed an electrospinning machine that has a high voltage supply designed to produce a variable voltage between 20 - 30 kV with positive polarity. Its other features include AC- DC voltage adaptor of 24 V maximum output, digital multimeter for taking the voltage readings, variac for varying the adaptor output voltage (thus varying the high voltage output), AC voltage regulator (AC voltage Stabilizer) and a cooling fan for the High voltage unit (see Plates I and II).

Plate I: Set- up of an earlier Electrospinning high voltage generator.



a b c

Plate II: High voltage power supply unit view (a) interior (b) front (c) back

A New Innovative Design

This electrospinning machine is made up of three components namely: (1) the power supply circuit (0-24V); (2) the real circuit driver unit (oscillator) that uses the principle of zero voltage switching system popularized by Vladimiro Mazzilli with two MOSFETs in a push-pull configuration making the circuit self-oscillates with an LC tank and switches each MOSFET when there is zero voltage across it. This minimizes switching losses and attendant MOSFET heating (3) the flyback transformer, which gives a high voltage output of 50,000 V (50kV). The efficiency of this machine depends on the driver unit and that is what makes the difference. This single unit 50 kv generator with the above components is shown in Plate III.

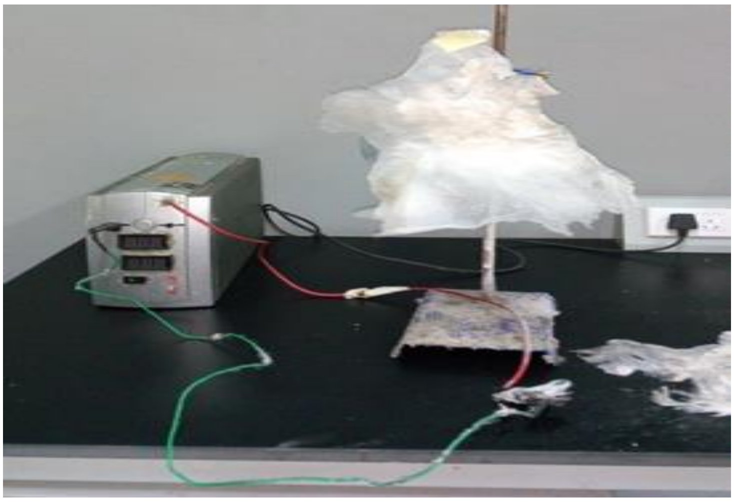


Plate III: A 50 kv voltage generator for electrospinning of nano fibers.

PERFORMANCE EVALUATION

Materials

The Polylactic (PLA) resin used in the fibre production is obtained from Natureworks, China. The Dichloromethane (DCM) of 98.6% purity is a product from France. Also, the Palm kernel shell (PKS) reinforcement was ground at Federal Institute of Industrial Research Oshodi (FIIRO), Lagos, Nigeria. The redesigned Electro spinning equipment was used to spin the unreinforced PLA and reinforced PLA at 90^0 angle position of the spinneret. Different weights of PLA pellets were dissolved in 20 ml of DMI to obtain concentrations 0.09, 0.10, 0.11, 0.13 and 0.14g/ml respectively. For the reinforced PLA, 20 wt. % PKS was introduced and mixed with 0.1 g/ml of PLA. These solutions were electrospun to produce nano fibers that were characterised for tensile, water absorption and morphological responses. The diameter of the fibre with maximum ultimate tensile strength (UTS) was determined using ImageJ software with the scanning electron microscope, SEM image obtained.

Process Setup and Electrospinning

The polymer/fibre solution was fed into the syringe inclined at 30^{o} to the horizontal with the polymer solution and mounted on a retort stand with the aid of a clamp. The diameter of syringe needle was 1.2 mm. A high voltage supply source with 50 KV, 2 mA, 100W was used. The efficiency of the setup is about 90-98% due to the use of zero voltage switching system implementation in the driver unit. This minimizes current loss within the unit. The earlier has an efficiency of 30-40 % owing to the use of the push pull system of driver unit, which leads to large current losses. A stationary rectangular aluminium foil collector of 300 x 300 mm was placed at 40 mm from the tip of the syringe. The process was initiated by connecting the syringe to the voltage source, which initializes the electrospinning process when switched on. The process was repeated for melt solution concentrations ranging from 0.09 to 0.14 g/ml. It was done only for reinforced PLA at 0.14 g/ml with 20 wt. % PKS. A sample of electrospun particle reinforced PLA fibre is shown in Plate IV.

Plate IV: A picture of electrospun reinforced PLA nanofiber composite on the aluminium foil.

Mechanical Testing

Tensile testing of electrospun fibres was performed on 40 x 20 x 0.4 mm tensile specimens using an Instron Model 313 having Bluehill TM Version 1.00 analysis software.

Scanning Electron Microscopy (SEM)

An ASPEX 3020 model variable pressure SEM operated with an electron intensity beam 15kV and equipped with Noran-Voyager energy dispersive spectroscope.

RESULTS AND DICUSSION

Mechanical Responses

The effect of varying amount of neat PLA on the ultimate tensile stress was observed and reported in Fig I. A gradual increase in the UTS from 0.09 g/ml to 0.13 g/ml was observed. There was, however, a sudden increase in the UTS at 0.14 g/ml of neat PLA where the value of the UTS attained the maximum. The UTS value at 0.14 g/ml neat PLA was even higher than the UTS value of 0.1 g/ml of PLA reinforced with 20% weight of palm kernel shell (PKS) by 16.67%. This must have been due to proper electrospinning process enhanced by material homogeneity of the neat PLA. However, the UTS of PKS reinforced PLA was respectively found to be 82.74, 71.68, 69.01% higher than the UTS of 0.09, 0.11 and 0.13 g/ml neat PLA.

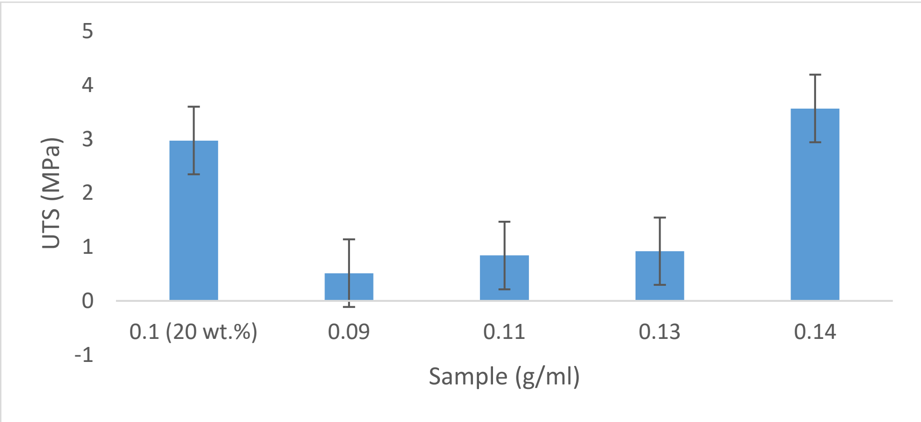


Fig I: UTS of PLA nano-fibre electrospun at 90^0 to the flat plate collector

Fig II presents the Young's modulus of electrospun reinforced and the neat PLA. It was observed that, the PKS reinforced PLA had the highest value of Young's modulus. This high Young's modulus is attributed to the stiffening effect of the PKS filler. This is consistent with micromechanical models (Angelo *et al*. 2006; Garesci and Fliegener, 2013) for compositing and the results got from the work of other researchers (Adeosun *et al* 2016; Oksmana *et al*. 2003). The Young's modulus of the PKS reinforced PLA was found to be 96.02, 94.93, 84.51, 61.08% higher than the Young's modulus of 0.09, 0.11, 0.13 and 0.14 g/ml respectively. It was also observed that increase in g/ml of PLA increases the Young's modulus. This can be attributed to the fact that increased PLA content decreased the tendency of segmental movement when tensile forces were applied during the tensile test.

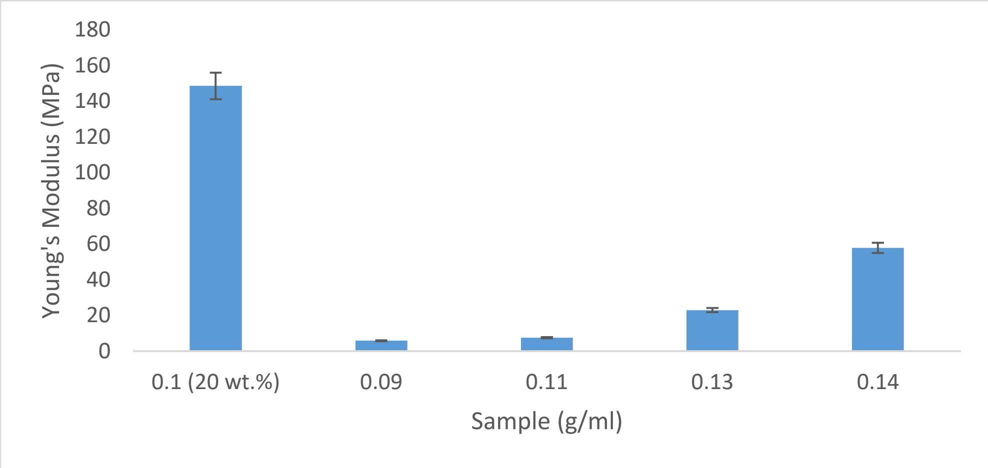


Fig II: Young's Modulus of PLA nano-fibre electrospun at 90^0 to the flat plate collector

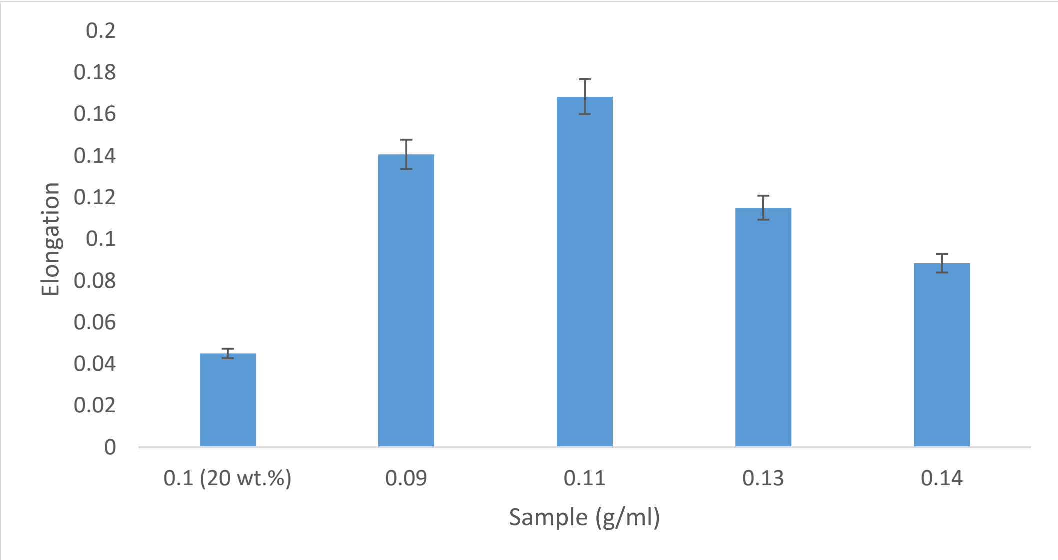


Fig III: Elongation of PLA nano-fibre electrospun at 90^0 to the flat plate collector

The plot on elongation is shown in Fig III. It was observed that 0.11 g/ml gave the highest value of elongation. This can be attributed to the reasonable amount of PLA in the nano fibre. Segmental movement would be prevented, if stiffening is increased. This would reduce elongation during deformation. 0.11 g/ml was found to produced elongation which was 73.27, 16.47, 31.68 and 47.53% higher than the elongation produced by PKS reinforced PLA at 0.09, 0.13 and 0.14 g/ml respectively.

Fluid Absorption Rate of PLA Nano Fibre

Certain areas of application of PLA require that its fluid absorption rate be ascertained. Fluid absorption affects the degradation profile of PLA and this may have positive or negative impact on its performance depending on the usage (SuPing and Darrel, 2009). The rate of water and PBS absorption of neat PLA are shown in Figs IV-V. It was discovered that temperature and fluid type affect the amount of fluid absorbed by PLA significantly. Electrospun PLA from 0.13 g/ml solution for 5 days absorbed more water at all temperatures than PBS. PLA absorbed 64.62% more water than PBS. This shows that PLA has more affinity for water than PBS. It also indicates that the degradation of PLA may be more affected by water absorption than by PBS absorption. Of all the samples, 0.13 g/ml absorbed most water and PBS in 5 days and at 70^{o}C. 0.09 g/ml absorbed as much PBS in 5 days at 70^{o}C as 0.13 g/ml within the same time and at the same temperature. This implies that increase in temperature also increases absorption rate.

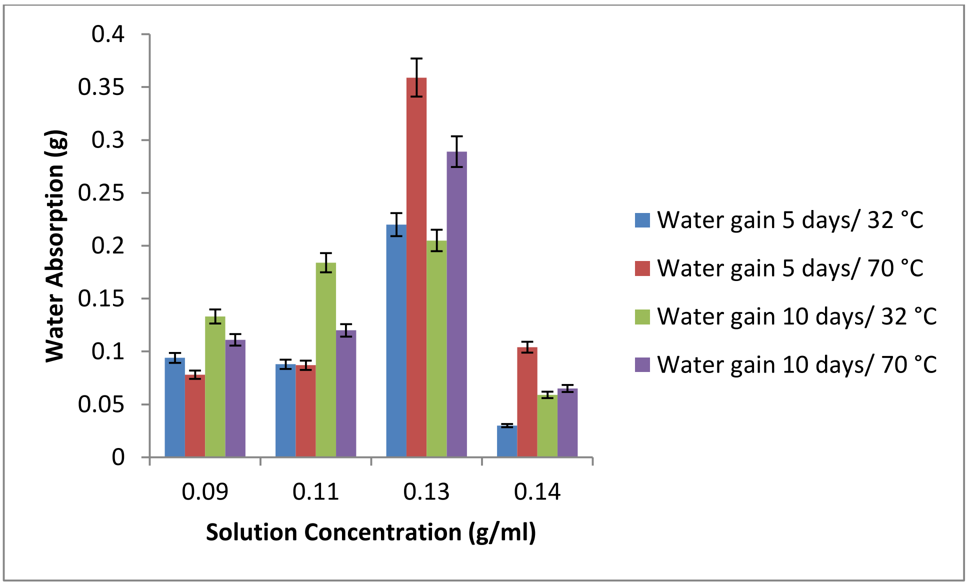


Fig IV: Water absorption at 32 and 70 degrees

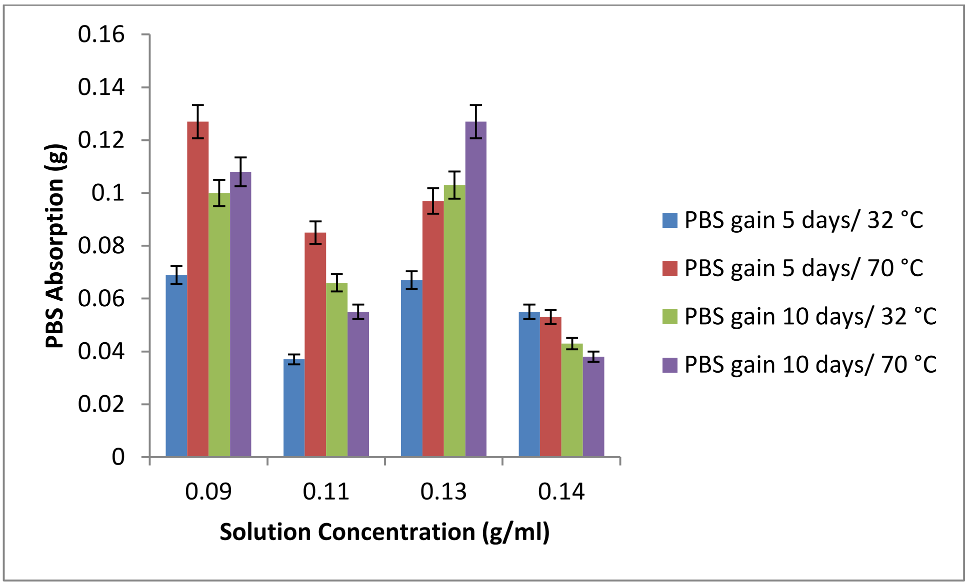


Fig V: PBS absorption at 32 and 70 degrees

Fibre Diameter

In Plate V and Fig VI, the surface morphology and size of electrospun fibres' arrangement are shown. Plate Va is the SEM for fibres from 0.11 g/ml. The image shows irregular sized fibres with relatively smooth surfaces. The morphology of the fibres show the presence of defects (irregular pores and dimples) on the surface of the fibres which could be attributed to the large size of fibres formed during the spinning process. This concentration is found to lead to fibres with average size of 10,090 nm. McCann *et al.* (2006) also reported the presence pores and dimples throughout the cross-section of the fibers electrospun from polyacrylonitirle, poly (vinylidine fluoride) and polycaprolactone. Similarly, Zhu *et al.* (2002), Rnjak-Kovacina *et al.* (2011), as well as Eda and Shivkumar, (2007) reported that pore size is closely related to the fiber diameter of electrospun mats affirming that the pore size increases with the increase of the fiber diameter. In addition, Bognitzki *et al.* (2001) also noted the presence of pores and dimples on electrospun fibres but proposed that phase separation is the main mechanism for the formation of pores and dimples on the surface of the fibres. Srinivasarao *et al.* (2001); Megelski *et al.* (2002) and Casper et al. (2004) proposed that the possible mechanism that may aid in the formation of pores or dimples on the fiber surface is the condensation of moisture in the air on the surface of the electrospun fibres. Casper *et al.* (2004) reported that increasing humidity, led to increase in the number of pores, pore-diameter and pore size distribution of polystyrene electrospun using tetrahydrofuran as the solvent.

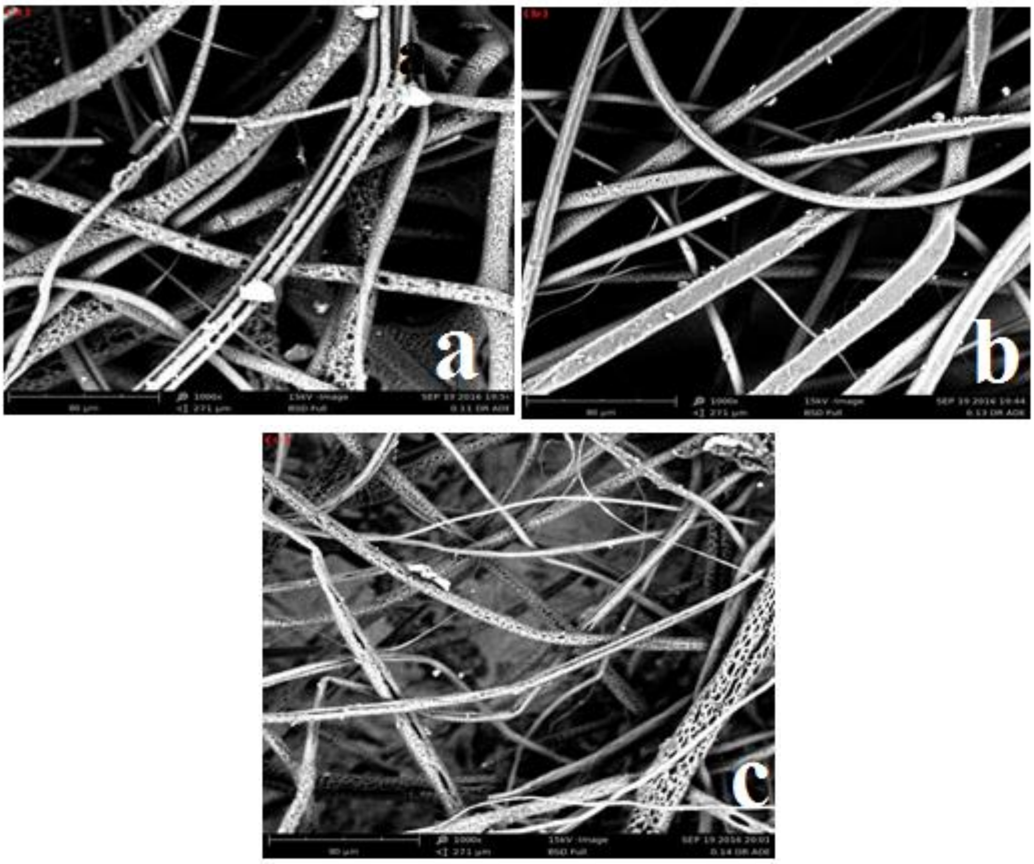


Plate V: SEM Images of electrospun fibres from g/ml PLA: (a) 0.11 (b) 0.13 (c) 0.14

Plate vb shows that these pores and dimples disappear and very smooth fibres are realized. There is also a decrease in average fibre diameter (see Fig. vi). This shows that increase in solution concentration did not affect the average diameter of the fibres. The pores and dimples on the surface of the fibres are quite tiny and almost invisible. This corroborates the fact that decrease in fibre diameter may lead to decrease in number and size of pores and dimples. Boland *et al*. (2005) supported that pore size of electrospun fiber generally increases with increasing fiber diameters. Garg *et al.* (2009) in his study achieved pore size of over 20 µm diameter. Neubert (2010) argues that the pore size of the electrospun membrane is also dependent on the choice of polymer to be electrospun. Moreover, further increase in concentration led to a decrease in fibre size (see Fig VI). Some of these fibres have relatively rough surfaces with large number of dimples (Plate Vc). In this case it will be permissible to say that atmospheric humidity may contribute to the presence of pores and dimples on the surface of the fibres. In our scenario it is very possible that the moisture affect some of the fibres spun in this batch. Dimples and pores are particularly needed in electrospun fibres used in drug delivery applications because it may have a direct effect on cell growth (Teo *et al*., 2009). However, the effect of pore diameters and fibre diameter on cell growth must be controlled.

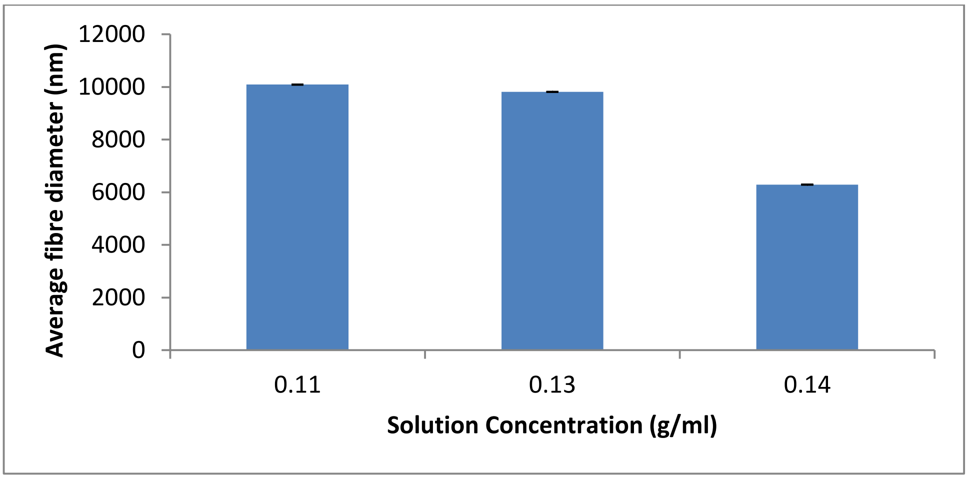


Fig VI: Average fibre diameter of electrospun PLA fibres

CONCLUSION

In this study the performance of a newly developed single unit 50Kv high voltage generator for the production of PLA nano-fibre has been studied. The fibre diameter decreases with concentration of the PLA in DCM solution. The fibres generated when compared to fibres from earlier design were more porous with low beads. At this moderate voltage or field strengths, a drop typically occurred at the needle tip, as a jet originates from the Taylor Cone producing bead-free spinning. With this voltage, low stretching of the solution occurred as fibre with large diameter is formed and the flight time of the fibre to collector plate decreases. At this voltage better crystallinity in the fibre is expected.

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