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# STUDY THE EFFECT OF SPINNERET SPEED AND NEEDLE DIAMETER ON THE DIAMETERS OF PLA FIBERS PRODUCED BY CENTRIFUGAL SPINNING METHOD

***Annotation:** Micro fibers have been widely used in various applications such as tissue engineering and filtration. The large surface area provided by these fibers has paved the way for exploiting these technologies in various innovative applications.*

*So far, electrospinning is the widely used method for producing nanofibers. However, the use of electrospinning greatly restricts the production of nanofibers to its low production rate.*

*Therefore it was necessary to invent and develop the technology of producing nanofibers that could satisfy the mass production of nanofibers required by the industry.*

*Centrifugal spinning is a new technology that uses the centrifugal force to form nanofibers and fine fibers from molten or solution.*

*In this research, Poly lactic acid fibers were produced using centrifugal spinning of the solution using acetone as a solvent, where the spinning solution was prepared at a concentration of 15% wt. The effects of nozzle diameter and rotational speed of the spinneret were evaluated with respect to the morphology and diameter of the fibers.*

*Characterization was performed using electronic microscopy (SEM). The results showed a high production of long fibers with diameters ranging from 398nm to 2.76 $\mu$ m depending on the specific operators.*

*We Found. The decreasing diameter of the spinneret nozzle and the increase in rotational speed of the spinneret have resulted in more uniform fibers of smaller diameters.*

***Keywords:** Electrospinning \_ nanofibers \_ centrifugal spinning \_ nozzle \_ rotational speed.*

# ИЗУЧЕНИЕ ВЛИЯНИЕ СКОРОСТИ ФИЛЬЕРЫ И ДИАМЕТРА ИГЛЫ НА ДИАМЕТРЫ ВОЛОКОН PLA, ПОЛУЧЕННЫХ МЕТОДОМ ЦЕНТРОБЕЖНОГО ПРЯДЕНИЯ

*Аннотация:* Микроволокна широко используются в различных областях, таких как тканевая инженерия и фильтрация. Большая площадь поверхности, обеспечиваемая этими волокнами, проложила путь для использования этих технологий в различных инновационных применениях. На сегодняшний день электро-прядение является широко используемым методом производства нановолокон. Однако использование электропрядения значительно ограничивает производство нановолокон до их низкой производительности. Поэтому было необходимо изобрести и разработать технологию производства нановолокон, которая могла бы удовлетворить массовое производство нановолокон, требуемое промышленностью.

Центробежное прядение - это новая технология, которая использует центробежную силу для формирования нановолокон и тонких волокон из расплава или раствора. В этом исследовании волокна полимолочной кислоты были получены с использованием центробежного прядения раствора с использованием ацетона в качестве растворителя, где прядильный раствор был приготовлен в концентрации 15% мас. Влияние диаметра сопла и скорости вращения фильеры оценивали с точки зрения морфологии и диаметра волокон. Характеристика была выполнена с использованием электронной микроскопии (SEM). Результаты показали высокое производство длинных волокон диаметром от 0,398 мкм - 2,760 мкм в зависимости от конкретных операторов.

Мы нашли. Уменьшение диаметра сопла фильеры и увеличение скорости вращения фильеры привело к получению более однородных волокон меньшего диаметра.

**Ключевые слова:** электропрядение, нановолокна, центробежное вращение, сопло, скорость вращения.

## **1. Introduction:**

### **1.1. biodegradable polymers**

Synthetic polymers include most types of polymers derived from petrochemicals, while natural polymers include most types of polymers that originate in the human body, plant or animal. Pure polymers are rarely directly usable without being subject to a number of technological processes that include adding some materials to make them have the appropriate specifications for the desired applications, and then we get what are called plastics.

One of the most important properties of plastic materials that contributed to its commercial success is its chemical inactivity and its resistance to aging and disintegration due to the attack of living organisms, weather conditions and effects, in addition to its low cost of production, light weight, durability and compatibility with special applications. The resistance of plastics to break-up and disintegration under the influence of various conditions is an important advantage during investment, but this has led to the accumulation of plastic waste as a result of excessive use of such materials and the emergence of a major problem for the environment that requires finding appropriate solutions. In addition, most types of plastic materials used in daily life are derived from non-renewable sources such as crude oil and natural gas. Among the solutions that have been proposed to solve these problems, is to recycle some types of plastic materials in order to be used again, but this method is not a final solution to the problem of plastic waste [1,C.300]. These problems led to an increased interest in developing polymers from renewable and environmentally friendly sources, which necessitated the emergence of new requirements in the specifications of plastics and polymers that are biologically shatterable and disintegrating, and turn into beneficial and non-toxic materials when they are out of investment. Biodegradable polymers and plastics are defined as a class of substances that can degrade and dissolve under the influence of living microscopic organic minutes and their enzymes such as germs, fungi and lichens [2,C.115].

The investment of these types of polymers is one of the possible solutions in eliminating the problem of increasing plastic waste day after day and reducing

dependence on plastic materials with oil sources. The new directions required research and development work with the aim of obtaining disassembled polymers and plastic materials within a relatively short time after their investment life had been depleted, which led to some research in the industry to develop products compatible with environmental requirements. As the development of biodegradable polymers progressed, new inventions, investment possibilities, and new applications for these novel types of biodegradable and absorbable polymers by living tissues such as surgical sutures and release drugs, continued, and research continued its path in the medical field for the use of biological polymers that resist degradation but are compatible with tissues For use as alternatives to bone and tissue, while some studies and researches have specialized in developing various classes of biodegradable polymers that are similar in terms of mechanical specifications and appearance to conventional polymers with a view to their use in applications as alternatives [3,C.90]. The end of the twentieth century witnessed a significant increase in the production of biodegradable polymers, as the amount of production of this type of polymers increased by about 30% annually [4,C.301].

PLA (Poly lactic acid) is a fibrous biodegradable polyester that is derived from renewable sources such as corn and sugar beet. Because of its high price, PLA has been used in the past decade exclusively in the medical field, such as orthopedic devices, sutures, and drug carriers [5,C.1125]. Because PLA is derived from renewable sources and has relatively good properties and is biodegradable, it is the most promising substance in solving environmental pollution problems associated with the use of plastics with oil sources [7,C.69] [6,C.831].

Poly Lactic Acid (PLA) Figure. (1) is a biodegradable polymer derived from renewable sources (corn, sugar beet). The basic unit of PLA is acid Lactic [8,C.200].

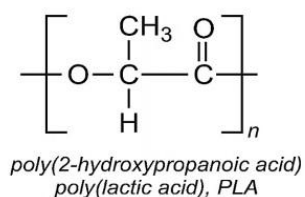


Figure (1): The formula of the Lactic Polymer (PLA).

PLA is used in many applications. In the medical field, it is used in (Delivery Drug) technologies, and in pharmaceutical applications. Attention has also been focused on it recently for use in the bone stents, which are required to be at a rate close to that of the bone tissue. PLA is also used in packaging and canning products because of its relatively good properties, in addition to its use in textile applications where PLA fibers are considered biodegradable.

It is of high performance due to its basic properties that are resistant to bacteria and impede the spread of flame and its stability against weather factors compared to PET fibers. Therefore, it has been introduced in the fields of textile industries, whether traditional ones such as clothing and furnishings, or advanced ones such as synthetic fibers. In the field of electronics, there are several applications of PLA in electronic equipment and devices, and Sony used polylactide (PLA) in the manufacture of structures for portable music players "Walkman" in 2002, where it was produced with extrusion technology.

## **1.2. Centrifugal spinning**

Centrifugal spinning, is a recently developed nanofiber forming technique and it draws extensive attention mainly due to its high production rate, which is 500 times faster than traditional electrospinning.[9,C.13] Instead of using electrostatic force, centrifugal spinning utilizes centrifugal force to realize the high-rate production of nanofibers.[10,C.10] Centrifugal spinning can be used to produce nanofibers by using polymer solutions or polymer melts, without the dielectric constant limitations and the involvement of high voltage electric field.

Figure (2) shows a schematic diagram of a laboratory centrifugal spinning device, which is mainly composed of a nozzle-contained spinneret, a high-speed motor, which is used to rotate the spinneret, a speed controller, which can adjust the rotational speed of the spinneret, and fiber collectors.

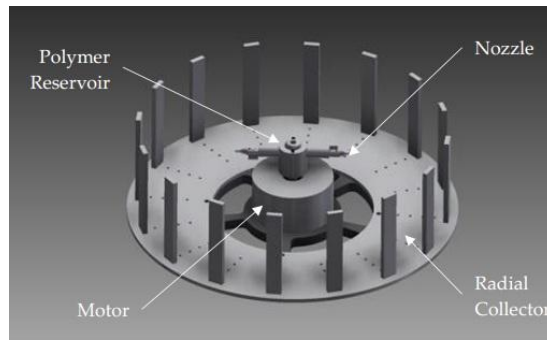


Figure (2): Schematic diagram showing a laboratory setup for centrifugal spinning.[11,C.3831]

The fiber formation process of centrifugal spinning can be separated into three stages: (i) jet-initiation to induce flow of the polymer solution through the orifice, (ii) jet-extension to increase surface area of the propelled polymer stream, and (iii) solvent evaporation to solidify and shrink the polymer jet ,Figure (3).

In general, as long as the centrifugal force is able to overcome the surface tension of the solution, liquid jet can be ejected from the spinneret orifice. The solution jet then undergoes a stretching process [12,C.3145], accompanied by rapid solvent evaporation, and eventually nanofibers can be collected on the collectors. [13,C.361]

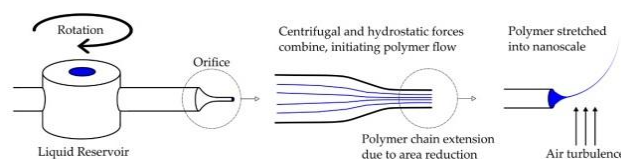


Figure (3): Schematic of nanofiber formation during centrifugal spinning.

## 2. Materials and Methods

**2.1. Material:** The PLA material used for 3D printing has been purchased, which is a 1kg net weight roller, which contains a strip with a diameter of 1.75mm, its color is transparent (CC Transparent), density: 1,25 gr/cm<sup>3</sup>, made. China.

As for the solvent, acetone was chosen because of its high volatility, and it is produced by Atomic Scientific® (Manchester), with a concentration of 99.5%.

All chemicals were used as received.

**2.2. Preparation of the spinning solution:** A polymer solution was prepared at a concentration of 15% wt, as it was heated at 70°C with stirring for half an hour by a magnetic mixer. Figure. (4)

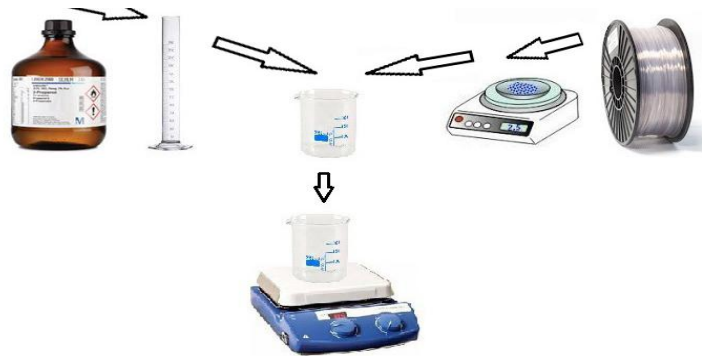


Figure. (4): the solution preparation process of PLA 15% wt

### 2.3. Characteristics of the spinning solution:

The viscosity of the solution was tested by a HAAKE Viscotester 550 from Thermo Fisher Scientific, an SV DIN Sensor was used, Test temperature: 23C°, the viscosity value was  $\mu = 0,90 \text{ mpa.s}$

The test was performed according to ISO 2555, ASTM D 115-72, D789-73, D2196-68.

### 2.4. Centrifugal spinning device settings:

A centrifugal spinning device located in the Textile Engineering Department of the College of Chemical and Petroleum Engineering at Al-Baath University was used to produce fibers. This device enables the spinning of different polymers solutions into continuous fibers that are placed on the collector to form a non-woven fiber web used for special applications, by applying the centrifugal force  $F_{cr}$ .

The spinneret chamber is made of aluminum, outside diameter: 41mm, inner diameter: 32mm, depth: 20mm, capacity: 9ml. Figure. (5)

The motor can reach the speed of up to 17000rpm, and the speed can be changed by a controller, and in this research the machine was operated at speeds starting from 1000rpm to 7000rpm to test the possibility of producing the fibers.



Figure. (5): the spinneret

The distance between the needle tip and the collector can be changed with a range of: 10-30 cm, and in this paper the complex distance is adjusted to 12 cm.

## **2.5. Characterization of fibers**

Fiber morphology was examined using an optika B-192 optical microscope for initial evaluation. SEM was then used to test the fiber samples more accurately.

ImageJ software was used to measure the diameter of the individual fibers and determine the average diameter of the fibers.

## **3. Results and discussion:**

It was found that when using a 25G needle (0.5mm diameter) a very weak web with low throughput at 3000rpm speed was used.

As the centrifugal forces at spinning speeds below 3000rpm were insufficient to remove the solution from the spinneret, resulting in the fibers not forming or producing a very small amount of fibers. Thus, the rotational speed had a direct impact on the production of fibers.

When using a 23G needle (0.6mm in diameter), the formation of the fibers starts at the speed of 1000rpm, and with the increase of the speed the formation of the fibers increased to the speed of 4000rpm

When using a 22G needle (0.7mm diameter), fiber formation started at 2000rpm, and with increasing speed the fiber formation increased to 4000rpm speed.

When using a 21G needle (diameter 0.8mm) no fibers were obtained, but a spray of the spinning fluid was obtained.

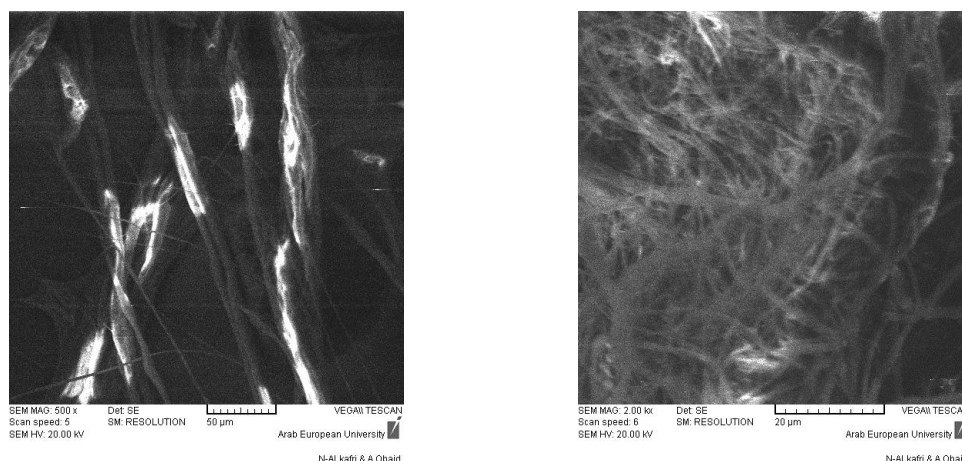
Nozzle diameter is an important factor in determining the flow rate of the polymer solution and the initial diameter of the emission [13,C.390].

When the nozzle diameter is small, the flow rate of the spinning solution will be prevented, which will facilitate the manufacture of nanofibers [14,C.173]. However, reducing the nozzle diameter will increase the viscosity of the polymer solution. Thus, the diameter of the nozzle must necessarily be improved to manufacture high-quality nanofibers.

This is because the increase in the diameter of the nozzle leads to a decrease in the surface tension, and consequently, the ejection force becomes much greater than the surface tension.

Fiber webs were tested and examined by optical microscopy for initial evaluation, and it was noted that the networks formed using the 23G needle were more regular and dense.

The fibers produced by the 23G needle were taken and examined using the SEM scanning electron microscope



B

A

Figure (6): SEM images of fiber networks, (A): fibers produced at a spin speed of 3000rpm, (B): fibers produced at a spin speed of 4000rpm.

SEM images of fibers showed that more uniform fibers were obtained at a rotational speed of 3000rpm, the diameter of the fibers ranging from 0,398 to 2.76  $\mu\text{m}$ , and the average of diameter was 1,088  $\mu\text{m}$ .

This rotational speed enabled the polymer to elongate and elongate in a spiral path towards the collector system while the solvent evaporated to form continuous PLA fibers. Figure (6,A)

Whereas, rotational speeds higher than 3000rpm produced fibers containing many beads with larger diameters due to the shortening time of hardening between ejection of the polymer solution (in the form of a drop) from the spinneret to its deposition on the collector.

There was also a noticeable change in the diameter of the fibers as a function of the spinneret rotational speed. With an increase in rotational speed from 3000rpm and 4000rpm, a large number of fibers formed, Figure (6,B), with a diameter of fibers in the range of 3.9 $\mu$ m and 16.9 $\mu$ m, and the average of diameter was 7,242 $\mu$ m.

We also notice the distribution of the diameter of the fibers at each speed. These changes in the diameter of the fibers are due to the changes in the spinneret rotational speeds, in which case the engine needs time to reach its maximum speed. From the beginning of its rotation until it reaches the maximum speed, the corresponding ejection force changes, and this would affect the efficiency of the resulting fibers.

#### **4. Conclusions:**

PLA fiber webs were produced by centrifugal spinning using a concentration solution of 15% wt, and it was noted that the fibers began to be formed using the same 23G (0.6mm diameter) at the speed of 1000rpm, and with the increase of the speed the fiber formation increased to the speed of 4000rpm, and at the speed of 3000rpm Obtaining fibers with a smaller and more uniform diameter.

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