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## **PREPARATION OF CARBON NANOFIBERS FROM ACRYLIC POLYMER SOLUTION BY ELECTROSPINNING**

***Annotation:** The chemical name of acrylic fiber is polyacrylonitrile .Very thin fibers were made from acrylic yarns as a raw material- containing 85% polyacrylonitrile- in N, N dimethyl formamide (DMF) as a solvent, to obtain acrylic nanofibers by electrospinning technique. Electrospun acrylic nanofibers were stabilized in air at 230°c for 2hours; then carbonizing process at 700°c in nitrogen atmosphere for 1hour was carried out in order to remove almost non-carboneous matters. Morphologies and diameters of stabilized and carbonized nanofibers were investigated by scanning electron microscopy (SEM). And the rate of carbon after carbonizing process was determined by (Energy-dispersive X-ray spectroscopy).*

***Key words:** Carbon nanofibers, Electrospinning, Stabilization, Carbonization, poly acrylonitrile, acrylic.*

## **СИНТЕЗ УГЛЕРОДНЫХ НАНОВОЛОКОН ИЗ РАСТВОРА АКРИЛОВОГО ПОЛИМЕРА МЕТОДОМ ЭЛЕКТРОСПИННИНГА**

***Аннотация:** Правильное химическое название акрилового волокна – полиакрилонитрил.*

*Из (85%-ного раствора полиакрилонитрила в N,N-диметилформамиде) методом электроспиннинга были получены акриловые нановолокна. Как исходный материал полиакрилонитрила в растворе были использованы более тонкие волокнистые нити акриловой пражии.. Нановолокна были стабилизированы на воздухе (230°C, 2 часа). Затем были подвержены процессу карбонизации в атмосфере азота (700°C, 1 час) с целью удаления из них неуглеродных компонентов. После процессов стабилизации и карбонизации были проведены исследования на морфологии и диаметры акриловых нановолокон методом сканирующей электронной микроскопии (СЭМ). Процентное содержание углерода после процесса карбонизации было определено методом энергодисперсионной рентгеновой спектроскопией (ЭДС).*

***Ключевые слова:** углеродные нановолокна, электроспиннинг, стабилизация, карбонизация, полиакрилонитрил, акрил.*

## **1-Introduction:**

Carbon nanofibers have recently attracted considerable attention due to their unique characteristics such as their high length to their diameter ratio and their physical and mechanical properties. [2, с. 54][6, с. 2]

We can produce Carbon nanofibers by stabilizing, carbonizing electrospun PAN Nanofibers. Electrospinning is an easy process to obtain ultrathin fibers from polymers solution and other materials such as composites and ceramics. [3, с. 401][6, с. 3] Electrospinning apparatus consists of the following components: high voltage (DC power), syringe pump, syringe, needle, metal collector. The principle underlying this process is that an electric charge in the range of kilo volt is applied to polymer solution held by its surface tension to produce fiber from liquid. The needle and collector typically serve as electrodes and the distance between them is set in suitable range. A syringe pump is used to control the flow rate. The needle is connected to the positive charge and the collector is attached to the ground.as shown in figure (1). [1, с. 113][4, с. 21]

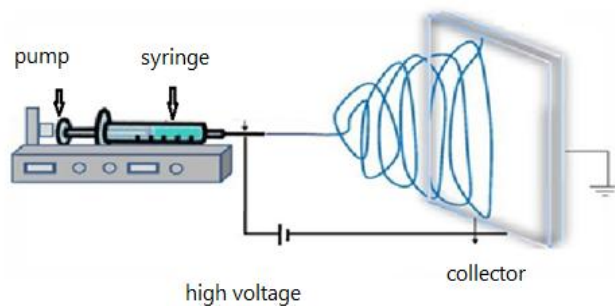


Figure 1. Electrospinning Apparatus

Nanofibers obtained through electrospinning are put through thermal treatment. The thermal treatment processes include stabilization and carbonization of acrylic nanofibers. The changes focused on in this study took place in the structure of polyacrylonitrile

The stabilization process was carried out in air (oxidative stabilization), and this process was very important because it gave a thermally stable structure for PAN molecules before the conversion of the nanofibers to carbon nanofibers. During stabilization, the nanofibers were heated to a temperature in the range of (200 – 230) °c for (2h). The chemical reactions included: cyclization, dehydrogenation, aromatization, oxidation, and crosslinking occurred as a result of the conversion of  $C\equiv N$  bonds to  $C=N$  bonds, and the ladder type structure was formed as shown in figure (2). [2, c. 58] [3, c. 402] [5, c. 1375] [7, c. 157]

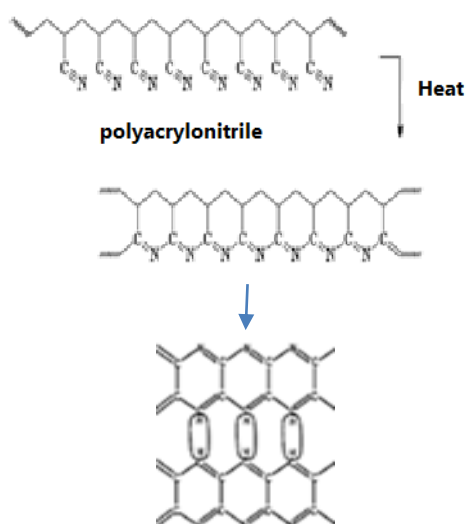


Figure 2. PAN after oxidizing.

Carbonization process carried out in an inert (Nitrogen) atmosphere for (1h) and the range of temperatures was (400-1000)°c. During this stage, a thermally stable rings structure were formed.

Chemical reactions involved reorganization and merging of cyclized sections leading to an increased levels of H<sub>2</sub>O, NH<sub>3</sub>, HCN, CO and CO<sub>2</sub> during the low temperature stage of carbonization. The foundation of the carbon building block is set-up. Cross-linking fixed the structure of the polymer. The cyclized structures underwent dehydrogenation and cross-linking produced a graphite-like structure of three

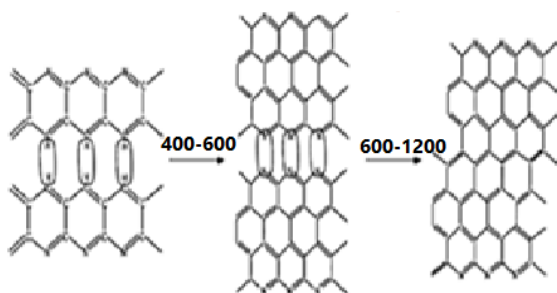


Figure 3. PAN after carbonizing.

hexagons in the lateral direction bounded by nitrogen atoms as shown in figures (3).  
[2, c. 67][3, c. 402][7, c. 244]

## 2-Experimental:

### 2-1-Materials:

Yarns of acrylic were purchased from a local market. The dimethylformamide (DMF) (99.7%) were got from LOBA CHEMIE PVT.LTD

### 2-2- procedure:

#### 2-2-1- Electrospinning of acrylic polymeric solution:

The raw material (acrylic yarns) were dissolved in DMF at (10g/100ml) ratio and was mixed and stirred for (30min) at (80°c). The mixture was then injected into (20ml) syringe. Nanofibers were fabricated using the electrospinning technique with (30kv) at a feed rate of (5ml/h) and the distance between the needle and collector was (10cm) and figure (4) shows a photo of electrospun acrylic sample.



Figure 4. a photo of electrospun acrylic sample

### **2-2-2- stabilization of nanofibers:**

The nanofibers were stabilized in an oxygen atmosphere at (230°C) for (2h) in a drying oven. The sample was given a little stretching by tweezers in two directions. Figure (5) shows a photo of an oxidized electrospun acrylic sample.



figure 5. a photo of oxidized electrospun acrylic sample

### **2-2-3- carbonization of stabilized nanofibers:**

The stabilized nanofibers were carbonated in an inert atmosphere (Nitrogen). We put the sample in an ashtray at (700°C) for (1h) with a little stretching of the sample by tweezers in two directions. Figure (6) shows a photo of carbonized sample.



figure 6. a photo of carbonized electrospun acrylic sample.

The color of the sample was turned to black because of the oxidizing and carbonizing processes.

### 3-Results and discussion:

After preparing nanofibers from (acrylic yarns) by the electrospinning technique, the nanofibers were oxidized and carbonized. The electrospun, oxidized, and carbonized samples were tested using the scanning electron microscope, and the rate of carbon element was determined by (Energy-dispersive X-ray spectroscopy).

#### 3-1-Scanning electron microscope (SEM):

The nanofibers obtained from (acrylic yarns) were identified by the scanning electron microscope (SEM, VEGA II, XMU, and Czech)

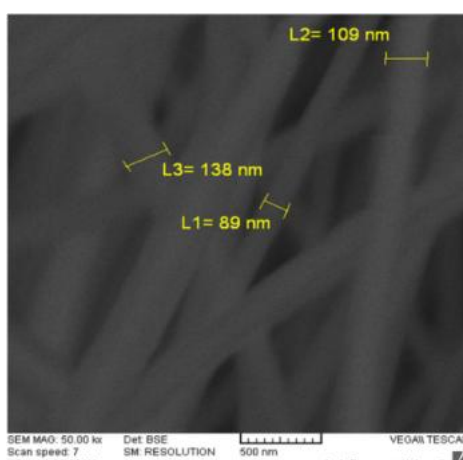


Figure 7. SEM images of the electrospun acrylic

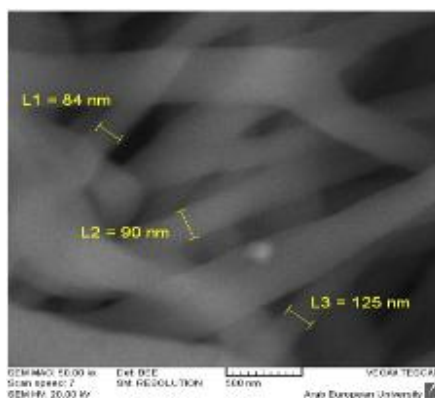


Figure 8. SEM images of Stabilized electrospun acrylic nanofibers

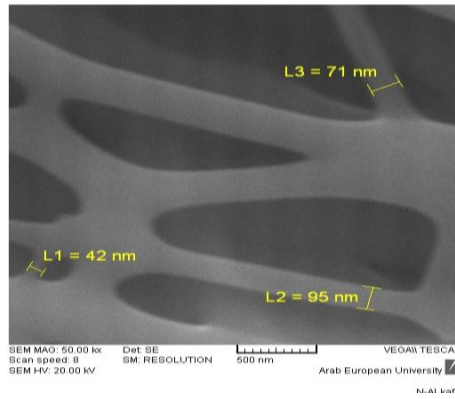
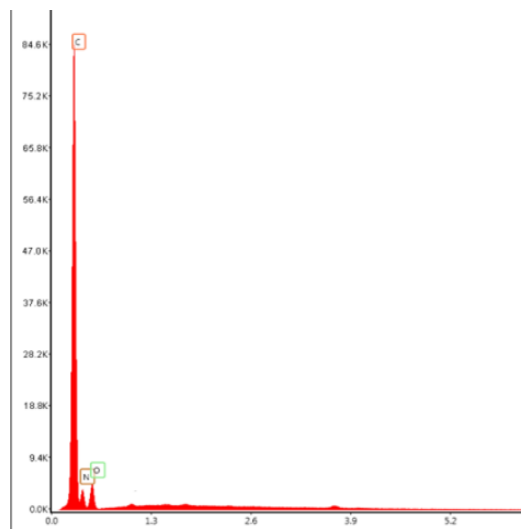


Figure 9. SEM images of carbonized electrospun acrylic nanofibers

The above figures (7,8) show that electrospun acrylic and oxidized acrylic nanofibers have almost the same diameters; while we can see in figure (9) the decrease of diameter of carbonized acrylic nanofibers and the increase in the porosity because of the decrease of nanofibers diameters.

### 3-2-Energy-dispersive X-ray spectroscopy (EDX)

The rate of carbon in the sample at (700°C) was determined by Energy-dispersive X-ray spectroscopy (EDX). Graph (1) shows the rate of carbon element in the sample with the higher peak referring to carbon element. It shows too the rate of residual elements such as oxygen and nitrogen. Further, the carbonization rate was (86.55%) as it appears in table (1)



Graph (1): the rate of carbon element in the sample after carbonizing

eZAF Smart Quant Results									
Creation D #####									
Project D   Area 1   Full Area 1									
Specimen									
Area									
Sampling Region									
kV 20									
Live Time 35.701									
Amp Time 7.68									
Takeoff An 39.18									
Resolution 126.6711									
Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
C K	84.63	86.55	3.27	9443.27	0.5139	1.0107	0.9954	0.7794	1
O K	7.81	8.05	11.07	385.95	0.0198	0.9863	1.005	0.0861	1
N K	7.56	5.4	10.87	486.4	0.0111	0.9653	1.0137	0.1021	1

**Table 1.**

**the rate of carbon, nitrogen, hydrogen elements in the sample after carbonizing**

#### **4-Conclusion:**

Nanofibers were obtained by electrospinning of solution of acrylic polymer as a raw material in DMF as solvent. Then, the nanofibers were stabilized by heat treatment in an oxygen atmosphere at (230°C) for (2h). After that, the carbonization process was carried out for the oxidized nanofibers in nitrogen at (700°C). The scanning electron microscopy images showed the nanofibers after oxidizing and carbonizing. Results showed that the diameters were in the range of nanometers. In addition, it could be noticed there was a decrease in diameters after the carbonizing process and the rate of carbon in the sample at (700°C) - determined by Energy-dispersive X-ray spectroscopy (EDX)- was (86.55%).

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