

# **THE EFFECT OF INCORPORATING COPPER OXIDE NANOPARTICLES ON THE PROPERTIES OF RECYCLED ACRYLIC FIBERS MADE BY ELECTROSPINNING**

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**ABSTRACT:** Electrospinning is a simple and effective method to obtain nanofibers with distinctive properties such as large surface area to weight and high porosity, which make them attractive for many applications. In this research, electrospinning technology was used to obtain nano non-woven mats using acrylic nanofibers (PAN) with copper oxide nanoparticles to improve the moisture absorption of the acrylonitrile polymer. CuO NPs were prepared and characterized. It was found that the average diameter of the particles is 28.5 nm. 6% polyacrylonitrile solution was prepared in a 50/50 mixture of acetone and dimethylformamide to produce nano non-woven mats. The nanoparticles were added to the polymer solution at several concentrations (0.25, 0.5, 1%). Samples were then prepared using an electrospinning device. In the end, 4 nano mats were obtained. They were characterized using a scanning electron microscope. It was shown that the diameters of nanofibers increased with increasing copper oxide concentration in the polymeric solution. Then, several tests were carried out to measure the ability of the prepared non-woven mats to absorb moisture, including measuring drop contact angle and absorption capacity test. After recording the results and comparing the samples, it was concluded that by increasing the concentration of CuO NPs, the absorbance improves significantly compared to the reference sample. Resulted non-woven mats can be used in applications that benefit the field of textiles and nonwovens by investing in acrylic exhaust, thus achieving an impact in the field of environmental protection with sustainable nanofibers as well as supporting the industry at the same time.

**Keywords:** Copper oxide nanoparticles, Electrospinning, Polyacrylonitrile, Water absorption, Recycling

## **ELEKTRO-EĞİRME İLE ÜRETİLEN GERİ DÖNÜŞTÜRÜLMÜŞ AKRİLİK LİFLERİNİN ÖZELLİKLERİNE BAKIR OKSİT NANOPARTİKÜL KATKISININ ETKİSİ**

**ÖZ:** Elektro-eğirme, ağırlıklarına göre geniş yüzey alanı ve yüksek gözeneklilik gibi ayırt edici özelliklere sahip nano lifler elde etmek için basit ve etkili bir yöntemdir ve bu da nano lifleri birçok uygulama için cazip hale getirir. Bu çalışmada, akrilik polimerinin nem emilimini artırmak amacıyla, elektro-eğirme teknolojisi kullanılarak bakır oksit nanopartikülleri (CuONP) içeren poliakrilonitril (PAN) dokuzuz yüzeyler elde edilmiştir. CuO NP'leri hazırlanmış ve karakterize edilmiştir. Parçacıkların ortalama çapının 28,5 nm olduğu bulunmuştur. Nano dokusuz yüzeyler üretmek için %6 poliakrilonitril çözeltisi, aseton ve dimetilformamidin 50/50 karışımında hazırlanmıştır. Nanopartiküller polimer çözeltisine çeşitli konsantrasyonlarda (%0,25, 0,5, 1) eklenerek ve elektro-eğirme cihazı kullanılarak 4 farklı numune hazırlanmıştır. Nano lifli yüzeyler taramalı elektron mikroskobu kullanılarak karakterize edilmiştir. Nanoliflerin çaplarının bakır oksit konsantrasyonunun artmasıyla arttığı görülmüştür. Ardından, hazırlanan dokusuz yüzeylerin nem emme kabiliyetlerini ölçmek amacıyla, temas açısı ölçümü ve emilim kapasitesi testi de dahil olmak üzere çeşitli testler gerçekleştirilmiştir. Sonuçlar kaydedilip numuneler karşılaştırıldıktan sonra, CuO nanopartikül konsantrasyonunun artırılmasıyla, referans numuneye kıyasla nem emiliminin önemli ölçüde iyileştiği sonucuna varılmıştır. Elde edilen dokusuz yüzeyler, tekstil ve alanına fayda sağlayacak çeşitli uygulamalarda kullanılabilir. Bu sürdürülebilir nanoliflerin değerlendirilmesi sayesinde, bir yandan endüstri desteklenirken diğer yandan da çevre koruma alanında anlamlı bir etki yaratılması hedeflenmektedir.

**Anahtar Kelimeler:** Bakır oksit nano-partiküller, Elektro-eğirme, Poliakrilonitril, Su emilimi, Geri dönüşüm.

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## 1. INTRODUCTION

Electrospinning is a fiber production method based on the use of electrostatic force to draw charged threads of polymer solutions. Therefore, the electrospinning process is entirely physical. Today, electrospinning has gained widespread popularity in both research laboratories and chemical industries, where the main goal is the continuous production of nanoscale fibers [1]. It is among the most accessible nanofabrication techniques over the past three decades [2]. This simple method produces polymer-based fibers with diameters ranging from nano to micron scale [3]. Nanofibers have a high surface-to-volume ratio and a porous structure [4]. Nanofiber membranes (NFMs) made through electrospinning offer advantages such as a large specific surface area, no secondary pollution, and ease of recycling [5].

CuO is a p-type semiconductor with a narrow bandgap that exhibits a wide range of light absorption [5]. The methods used to synthesize CuO nanoparticles include electrochemical techniques, sonochemistry, sol-gel, green synthesis, hydrothermal processes, and biogenic approaches [6]. Copper oxide also has good antimicrobial properties, and its production is more economical compared to other metallic nanoparticles. Additionally, it has applications in magnetic storage [7]. Adding CuO nanoparticles to polymers before electrospinning imparts two distinctive properties to the nanofibers. First, the surface area of the nanofibers increases significantly, and second, copper (II) oxide is a hydrophilic material that imparts hydrophilicity to the base polymer [8].

Polyacrylonitrile (PAN) is a synthetic polymer that has a white color, is hydrophobic, and possesses a semi-crystalline structure. The chemical formula of PAN is  $C_3H_3N$ . PAN is a thermally stable polymer that generally degrades above 300 °C [7]. This polymer has found extensive applications in textile fibers and membranes. PAN nanofibers have also been used in combination with other materials to produce water treatment membranes [9-10]. PAN concentrations significantly affect the wettability of the nanofiber mats, as shown by contact angle measurements which was found to be 99.5°, 100.3°, 105°, and 115°, respectively, for 6, 8, 10, and 12 % PAN nanofiber mats. The electrospun mats become increasingly more hydrophobic as PAN concentration increases. The average fiber diameter grows from 208 nm to 881 nm when PAN concentration rises from 6 wt% to 12 wt% [11]. Compared to other common commercial polymeric membrane materials, PAN is considered relatively more hydrophilic due to its higher molecular polarity, which results from the polar nitrile groups ( $-C\equiv N$ ) in PAN molecules [12]. Water contact or hydrophilicity is an important factor that affects the final applications of developed products to an extent. Correspondingly, water holding capacity becomes essential for hygiene applications.

The traditional recycling method focuses on eliminating plastic waste rather than promoting its beneficial, value-added reuse. Modern recycling offers a better way to turn plastic waste into valuable products, supporting the principles of the circular

economy. Therefore, developing cost-effective recycling technologies and higher-value applications is essential. Electrospinning can recycle and utilize plastic waste thanks to its versatility with various polymer sources. Fortunately, most polymeric waste consists of synthetic polymers such as PET, PS, PA, and others, which can be easily electrospun into micro- and nanofibers under proper processing conditions. [13]. Using inorganic micro- and nanoparticles to create inorganic/organic hybrid materials is among the simplest methods to produce hydrophobic surfaces and control surface wettability [14].

Despite the studies on PAN/CuO nanofibers, this electrospun nanocomposite has not been prepared using recycled acrylic yarns from an old sweater. In our research, we produce nanocomposite PAN/CuO NPs made from recycled acrylic yarns. Copper oxide is suitable for transforming hydrophobic nanofibers into superhydrophilic nanofibers. For this purpose, CuO nanoparticles were prepared using sol gel method and then incorporated into electrospun PAN fibers in many concentrations (0.25, 0.5, and 1%). In addition to studying the effect of concentration of nano-CuO on the fiber diameter of PAN. Water holding capacity was studied for all prepared mats.

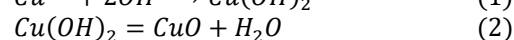
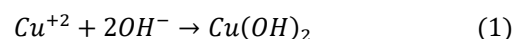
## 2. EXPERIMENTAL PROCEDURE

Acrylic yarns (Polyacrylonitrile) used in this research are commonly used in knitting. The yarn used here was taken from an old sweater meant for destruction. Dimethylformamide (DMF, density 0.95 g/cm<sup>3</sup>) and Acetone (Ac, density 0.79 g/cm<sup>3</sup>) were purchased from POCH Sowińskiego, Poland. Copper sulfate ( $CuSO_4 \cdot 5H_2O$ ) and sodium hydroxide (NaOH) were purchased from Lobachemie.

An electrospinning device was used to create the nanofiber mats. The device had a high-voltage supply of 19-28 kV and a speed of the collector of up to 6000rpm. A 20 mL syringe and 22-gauge needles were used. A trial-and-error method was used to determine the best spinning conditions, which were found to be approximately as in Table 2. The fiber mats were collected on the surface of an aluminum foil. Several fiber mats were produced using a mixture of PAN and a CuO nanoparticle solution.

### 2.1. Preparing Copper Oxide Nanoparticles

Nano-copper oxide is prepared by the sol-gel method, using hydrated copper sulfate ( $CuSO_4 \cdot 5H_2O$ ) as a precursor and sodium hydroxide as a reducing agent in an aqueous medium at 90°C. The reaction takes place according to the Eq. 1- 2:



Then oxide is left to settle and washed three times with water. After that, it was placed in the oven at a temperature of 150°C for several hours until it dried completely. Then, ground it using a ceramic mortar mill until it was ready to use in the next stage.

## 2.2. Preparing the Acrylic Polymer Solution and Processing It on The Electro Spinning Device

6% wt. PAN solution was prepared using a mixture of dimethyl formamide (DMF) and acetone in a ratio of 50:50. The solution was heated to 90°C and stirred for 30 minutes using a magnetic stirrer. After stirring, the solution was left to cool down to room temperature, and any bubbles that could interfere with the electrospinning process were removed.

Once the polymeric solution was prepared, nano-copper oxide was added at the concentrations indicated in Table 1. The solution was stirred for another 30 minutes to ensure that all components were thoroughly mixed.

**Table 1.** Nano-CuO concentration in each sample

Sample No.	Nano-CuO concentration (%)
1	0
2	0.25
3	0.50
4	1

## 2.3. Producing CuO NPs/PAN Fibers

Recycled acrylic is the material used in this research. Poly acrylic is a hydrophobic substance, but copper oxide nanoparticles were added to improve its water absorption capabilities. The electrospinning process was carried out using the parameters shown in Table 2. These parameters were chosen after conducting multiple experiments to create conditions where the polymer solution drop elongates, forms a microfiber, and then is collected. In some cases, atomization occurred, while in others, the drop broke before reaching the collector.

**Table 2.** The Used Electrospinning Parameters

Voltage (kV)	The distance between needle and collector (cm)	Flow rate (ml/h)	Temperature (°C)	Humidity (%)
11	5	0.5	25	65

## 3. CHARACTERIZATION TECHNIQUES

### 3.1. Structural Analysis

Structural analysis was performed for the prepared CuO powder, then for PAN and PAN-CuO mats. The morphology of the prepared particles and fibers was examined using a scanning electron microscope (SEM), with a 7.5K magnification; model VEGA II XMU, Czech. And the diameters were determined through ImageJ software.

### 3.2. Water Contact Angle

The contact angle is measured using the Sessile drop method according to the standard specification (ASTM D5946). The electrospun non-woven mesh sample is placed on the special holder, and the water drop is left to fall vertically on the surface

of the sample with a size of 0.05 ml. The position of a water droplet on the surface of the nonwoven sample is photographed. The contact angle is measured by processing the image using ImageJ software. To avoid possible errors that may occur during measurement. Three readings are taken for each sample, and then the average of the readings is calculated. The test is performed at a temperature of  $23 \pm 2^\circ\text{C}$  and a relative humidity of  $(50 \pm 10\%)$ .

### 3.3. Absorption Capacities Test

Absorption capacity is one of the main properties of polymers, and this property is linked to the presence of hydrophilic groups such as hydroxyl (-OH). and amide (-CONH<sub>2</sub>). The absorption capacity is defined as the amount of water absorbed by the absorbent material relative to one weight of it and is given as In Eq.3 [15]:

$$\text{Moisture content (\%)} = \frac{W_{\text{wet}} - W_{\text{dry}}}{W_{\text{dry}}} \times 100 \quad (3)$$

Where:  $W_{\text{wet}}$  Weight of wet sample (mg)

$W_{\text{dry}}$  Weight of dry sample (mg)

### 3.4. Absorption Test by Capillary Property (Wickability)

The rate of water transmission is measured vertically in the nonwoven mat by immersing a strip (25\*170 mm) vertically in distilled water, so that only 3 mm of the strip is immersed in water. The water transfer rate is measured after (1, 5, 10 min) with length in (cm). Higher absorption values indicate a higher ability to transport water [16].

## 4. RESULTS AND DISCUSSION

### 4.1. Structural Analysis

Scanning electron microscope of the synthesized copper oxide nanoparticles -prepared by the sol-gel method using copper sulfate is presented in Fig. 1. The size of nanoparticles is measured by ImageJ and ranges between 25 nm and 33 nm. By calculating the average diameters of nanoparticles, it is found to be equal to 28.5 nm. It is noticed that the nanoparticles of copper oxide powder were assembled and formed agglomerates because of self-assembly in the nanomaterial. To prevent this issue, magnetic stirring was employed to minimize the agglomeration of nanocopper oxide and promote its uniform distribution in the prepared PAN polymer solution.

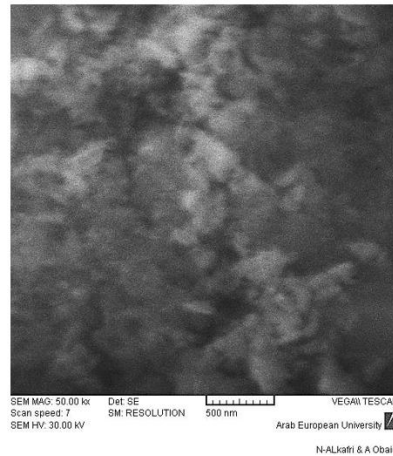
The viscosity of the polymer solutions can be controlled by adjusting the concentration, and it is the most important factor affecting fiber morphology and diameter values. The viscosity of all prepared polymeric solutions was measured before electrospinning. The results are shown in Table 3. Measuring the viscosity of prepared solutions was used by a ViscoEasy R-Wira, and this is a rotational viscometer, at  $25 \pm 0.1^\circ\text{C}$ .

An electron microscopy test was performed for the resulting samples, where the average fiber diameters of the PAN sample were 66.47 nm, as displayed in Fig. 2. Samples 2, 3, and 4 contained nano-CuO in addition to PAN, in Fig. 2. It was noticed

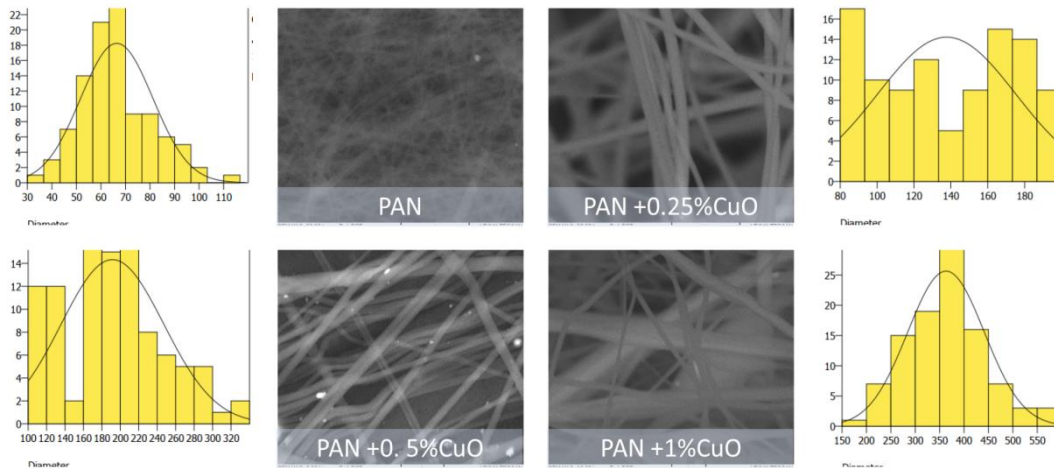
that the average diameter of the fibers ranged between 137-363 nm. There was an increase in the average fiber diameters with the increase in the concentration of nano-CuO. This figure also shows the distribution of nanofiber diameters, where the diameters of one hundred fibers were measured for each sample and then fiber diameter distribution (histograms) using the statistical software (PSPP).

**Table 3.** Viscosity of prepared polymeric solutions

Sample No.	Viscosity (mPa.s)
1	107
2	125
3	134
4	157



**Figure 1.** SEM of CuO Nanoparticles Powder



**Figure 2.** SEM of PAN mats with CuO Nanoparticles

**Table 4** Diameters of fibers in the fabricated mats of the samples

Sample No.	min	max	Average diameters of fibers (nm)	Standard deviation SD	Difference Factor (%)
1	36	110	66.47	14.64	0.22
2	81.27	199.21	137.72	37.43	0.27
3	106.36	335.09	191.44	55.68	0.29
4	180.35	580.66	363.05	78.55	0.22

Table 4 shows the average diameters of fibers in the fabricated mats of the samples. For samples containing nano-CuO, the average fiber diameters ranged between 137-363 nm. In comparison, the PAN sample had an average fiber diameter of 66.47 nm.

#### 4.2. Contact Angle Test

The results for this test are shown in Figure 3. It was noted that the contact angle decreased with the increase in concentration of CuO NPs, i.e., the addition of higher concentrations of CuO nanoparticles led to a decrease in water contact angle, indicating enhanced surface hydrophilicity. The angle reached 31.3 degrees for the sample containing 1% nano-copper oxide, while the angle reached 32.7 degrees for the sample containing 0.5% copper oxide. As for the sample containing 0.25% of copper oxide, the angle reached 34.6 degrees, noting that the angle in the reference sample reached 40.2 degrees. By adding copper oxide nanoparticles to the polymer and embedding them within the electrospun fibers, the nonwoven mesh possesses absorption centers for water molecules, namely the oxide nanoparticles. This led to an enhancement of the hydrophilic property. And this agrees with the literature [17], which notes that an increase in CuONPs concentration in the kefiran-carboxymethyl cellulose films resulted in a smaller contact angle. And the research that said the addition of CuO nanoparticles enhanced the surface hydrophilicity of PAN nanofibers and its antibacterial properties [18].

#### 4.3. Absorption Capacity Test

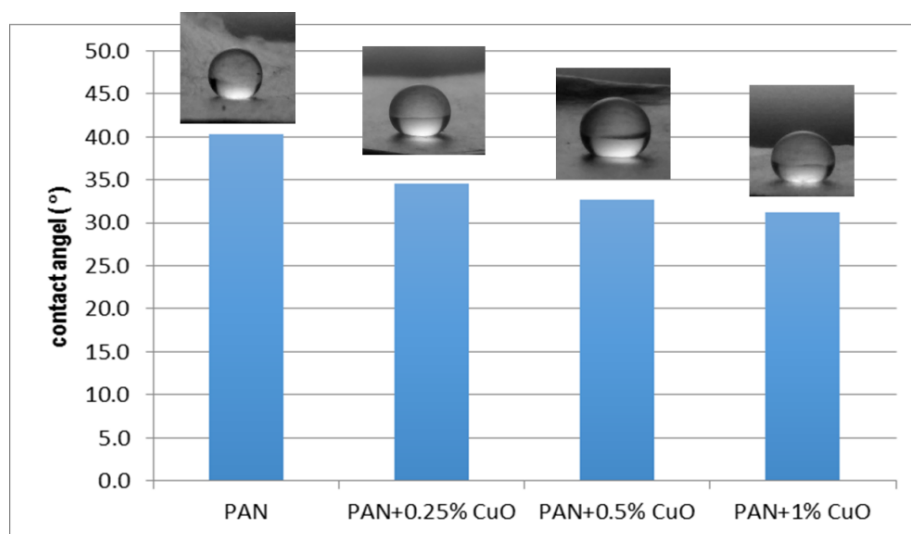
The dry samples were weighed before immersing in water. The samples were then weighed again after being immersed in distilled water for 24 hours. The adsorption capacity was calculated using Equation (3). The results shown in Table 5 were obtained. The table indicates that the reference PAN sample, which did not

contain copper oxide, had the lowest adsorption capacity. PAN's hydrophobicity mainly derives from its chemical structure, as nitrile groups  $-C\equiv N$  are polar, but the polymer backbone is non-polar and lacks strong hydrogen-bonding sites for water. However, the adsorption capacity of the samples increased with the addition of nano-copper oxide. Copper oxide (CuO) is notably more hydrophilic than PAN because its surface contains oxygen atoms capable of forming hydrogen bonds with water molecules. Consequently, the adsorption capacity increased as the concentration of nano-copper oxide increased. The increased adsorption capacity with added nano-copper oxide likely occurs because the hydrophilic nano-CuO particles introduce polar surfaces that can form hydrogen bonds with water molecules. Additionally, the nanoparticles increase the surface area and roughness of the composite, potentially creating micro- or nano-pores or interfacial regions that enhance water retention compared to the relatively smooth and hydrophobic pure PAN fiber surface.

**Table 5.** Results of Absorption capacity test

Sample No.	Weight of dry sample (g)	Weight of sample after immersing (g)	Absorption capacity (g/g)
1	0.0104	0.2463	15.699
2	0.0213	0.3557	22.682
3	0.0091	0.232	24.495
4	0.0125	0.4158	32.264

The diagram in Figure 4 shows the relationship between the concentration of added nano-copper oxide and the absorption capacity of the electrospun mesh. We note that the polyacrylonitrile sample, which contains 1 g nano-CuO, has the highest absorption capacity (32.3 g/g), while the polyacrylic sample is the least, as its absorption capacity reached 15.6 g/g.



**Figure 3.** Contact angles for prepares samples

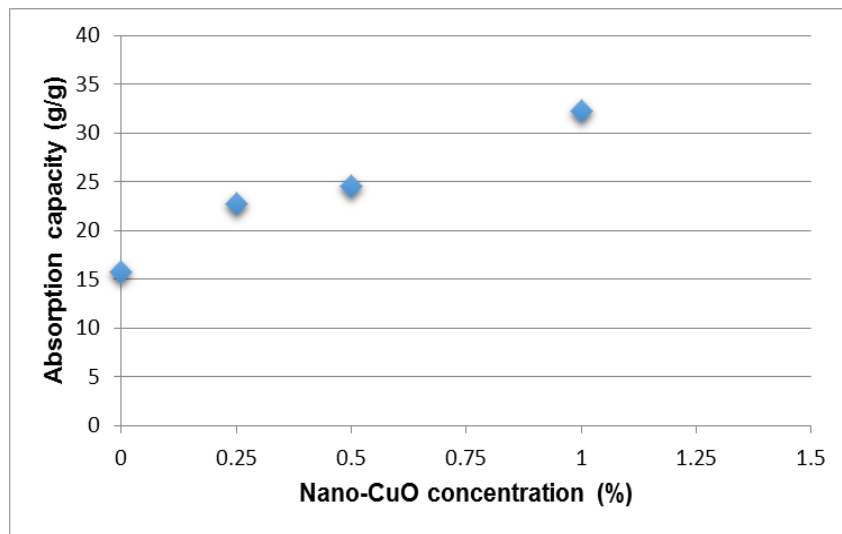


Figure 4. The relationship between nano-CuO concentration and Absorption capacity

#### 4.4. Wickability Test

The samples were cut into strips measuring (25\*170 mm), and then 3 mm of them was immersed in distilled water. The height of the water in the samples was measured at specific times, and the results were recorded in Table 6.

Table 6. Results of Absorption test by capillary action

	Sample.1	Sample.2	Sample.3	Sample.4
1 min	2	2	2	1
5 min	5	4	4	4
10min	5.5	7	6	7

It is observed from this test that the reference sample absorbs water according to the capillary property, but at a low rate, while

the absorption property of the polyacrylonitrile samples containing copper oxide improved, as the water column absorption height reached about 7 cm in almost all samples. Which means that all samples are equally absorbing water. Figure 5 also shows a diagram of the relationship between absorption time and water column height. The diagram shows that as time increases, the height of the water column in the sample strip increases, and that the samples containing copper oxide are able to absorb more moisture than the reference sample. The higher the concentration of copper oxide in the sample, the higher the water column, and this is what is shown by sample 4 containing the highest studied concentration. This is due to the increased hydrophilicity of the PAN mat resulting from the addition of CuO [18].

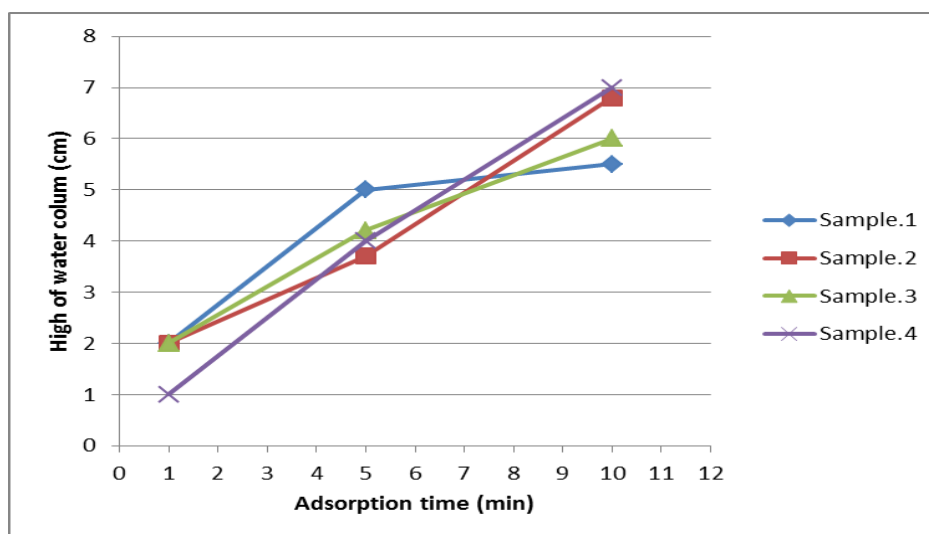


Figure 5. Diagram of the relationship between absorption

## 5. CONCLUSIONS

In this study, electrospun nonwoven mats were produced using recycled polyacrylonitrile. It is known that polyacrylonitrile is a synthetic polymer that does not absorb water very much. In this research, a method was found to improve the moisture absorption of PAN fibers, and they were remanufactured into nonwoven mats, which can be used in various fields, such as wastewater treatment during filtration. 'Nano copper oxide' was added to enhance the fibers' hydrophilicity after dissolving them with acetone and dimethylformamide. Three samples were prepared at concentrations of 0.25, 0.5, and 1% to compare with the reference sample. The resulting samples were examined using SEM, and ImageJ calculated the average fiber diameter for each sample. Tests were then conducted to assess how much the acrylic networks enhanced hydrophilic properties. These tests included measuring the contact angle of water droplets, absorption capacity, and the water column test. After analyzing the results and comparing the samples, it was concluded that increasing the concentration of nano copper oxide significantly improves absorbance compared to the reference sample. In applications benefiting the textile and nonwoven industries, investing in recycled acrylic yarns offer a positive impact on environmental protection while supporting the industry.

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