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Synthesis and Characterization Membrane Nanofibers Cellulose Acetate-Zeolite for Metal Pb (II) Adsorption

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Abstract

Heavy metal waste, such as lead (Pb), released into the environment negatively affects ecosystems and human health due to its toxic properties. Consequently, it is imperative to develop methods to remove Pb from the environment. One widely used method is adsorption. Synthesizing adsorbents via electrospinning offers several advantages: simplicity, the ability to produce nano-sized fibers, lightness, chemical stability, and reusability. This research aims to determine the optimum conditions for synthesizing cellulose acetate (CA) nanofibers modified with zeolite (CA-Zeolite) by electrospinning to characterize them and evaluate their adsorption capacity. The research results showed that the optimum conditions for the synthesis of cellulose acetate nanofibers were a solution concentration of 14% (w/v), a voltage of 10.5 kV, a flow rate of 0.02 mL/hour, and a tip-to-collector distance of 10 cm. The FTIR spectrum of CA-Zeolite revealed a new peak in the 400-610 cm^{-1} wavelength range, indicative of O-Si-O bonds, characteristic of zeolite functional groups, confirming the successful incorporation of zeolite into the CA nanofibers. From the SEM data, it can be seen that the addition of 6% (w/w) zeolite reduced the average membrane fiber diameter from 662.4 nm to 353.1 nm. EDX results show the presence of Si and Al elements in the CA-Zeolite nanofiber membrane. Incorporating zeolite into CA nanofibers decreased the contact angle from 125.49° to 111.66°, enhancing hydrophilicity. The modified CA nanofibers with 6% (w/v) zeolite demonstrated an adsorption capacity of 1.595 mg/g.

1. Introduction

The worldwide issue of water and air contamination from human activities is a great concern. According to WHO [1], a staggering one billion people currently lack access to safe drinking water, which is projected to rise to four billion by 2050 [2]. In other studies, UNICEF and WHO [3] have reported that 2.2 billion people are unable to access reliable sources of potable water. This escalating water crisis has led to heightened demands for water across residential, industrial, and agricultural sectors in various regions around the globe [4]. Industrial activities [1], transportation and mining contribute to the release of heavy metal waste, posing serious environmental and health risks. Heavy metals are toxic and, when accumulated in the human body, can lead to various diseases, including nervous system disorders, bone diseases, and liver and kidney cancer, particularly from exposure to lead (Pb) and chromium (Cr) [5].

Heavy metals in water can be effectively reduced through various methods, including nanotechnology, oxidation, reduction, bioremediation, reverse osmosis, electrochemical treatment, coagulation, lime softening, precipitation, and ion exchange [6]. Adsorption is a method known for its simplicity, cost-effectiveness, and high efficiency in removing heavy metal content [7]. Besides having high efficiency, this method is also environmentally friendly [8]. The adsorbent must have a specific surface area, porosity, and adsorption capacity. Several materials can be adsorbents, including polymers, inorganic and metal oxides. Many adsorbents, including polymer, have been employed for the adsorption of heavy metals and particularly for the elimination of Pb (II) and

Cu (II) ions [7]. One of the adsorption methods can be done using membrane technology. Membranes began to be recognized and studied in the middle of the 18th century. A membrane is a thin layer separating between 2 phases, the feed and permeate phases [9]. Membranes can be synthesized using several methods, one of which involves the electrospinning process.

Membranes can made from several materials, such as polymer, ceramic, and composite [9]. Cellulose acetate (CA), a low-cost cellulose derivative, offers advantages such as good mechanical properties, biodegradability, and biocompatibility. However, due to its poor adsorption of metals, CA must be combined with additional materials for effective metal removal [10]. Previous research has reported that the diameters of the CA nanofibers increased with increasing CA concentration in the range of 404 nm to 1346 nm [11]. In our study, we incorporated zeolite to reduce the diameter of CA fibers. Zeolite is characterized by its regular pore structure, large specific surface area, strong ion exchange capacity, high thermal stability, and adjustable hydrophilicity and hydrophobicity. Additionally, zeolite can enhance the performance of polymers for adsorption and catalysis [12].

Electrospinning is a simple method employed in the synthesis of membranes. The advantages of utilizing electrospinning include its ability to produce long and continuous fibers or beads [8], its applicability to a wide range of materials such as natural and synthetic polymers, ceramics, and metal oxides, and its capability to produce fibers with diameters ranging from the micro to nanoscale [12]. Additionally, electrospinning is a cost-effective production method [13]. Electrospinning equipment typically consists of a high-voltage generator that generates an electric field, which attracts and transforms the solution into fibers collected on a designated collector [9]. Various researchers have conducted numerous studies on the production of membranes using the electrospinning method.

Choi *et al.* [10] have reported in their research on membrane fabrication of cellulose acetate using the electrospinning method as a metal adsorbent for Cu (II), Cd (II), and Pb (II). The adsorbate forms a homogeneous monolayer with absorption energy distributed on the surface of the nanofibers. Tian *et al.* [14] have also reported their research in making cellulose acetate membranes for heavy metal adsorption in water purification. This research provides good information regarding membrane fabrication with low-cost and high-adsorption capacity. Wang *et al.* [15] also reported research using the electrospinning method for water and oil separation. The adsorption method is one of the most common processes for removing those heavy metals due to its ease, suitability, low cost, and high removal capacity.

Previous research has reported that the highest removal capacities, Pb (II) and Cr (II), use CA/TiO_2 as adsorbent. The adsorption efficiencies of the adsorbents for those heavy metal ions slightly declined with increasing adsorption-desorption phases. The process must repeatedly use CA/TiO_2 adsorbent to increase the adsorption capacities process of adsorption [7]. A cellulose acetate membrane with the addition of zeolite by electrospinning has also been fabricated. The addition of zeolite increases the adsorption capacity for heavy metals removal in water [16].

This study aims to synthesize nanofiber membranes using the electrospinning method, identify the optimum conditions and characterization of the membrane, and determine its application for Pb (II) metal adsorption.

2. Experimental

2.1. Materials

The materials used in this study included cellulose acetate (Aldrich; 39.8 wt%; Mw: 30,000 Da), acetone, DMAc, zeolite 4A (Merck), distilled water, and $Pb(NO_3)_2$ (analytical grade). The instrumentation was electrospinning Dinamo Ex Printer 12-24V serial STD MTR QK1-4677 speed rate of 3200 rpm for the synthesis membrane.

2.2. Methods

Electrospinning consists of several components, as shown in Figure 1. Part A serves as a place to put the syringe. A compressive force, influenced by the chosen flow rate, drives the syringe to dispense the solution through the needle's tip, forming droplets. Droplets are exposed to electric field effects around the needle tip to the collector. Droplets form a Taylor cone and are collected into a drum-shaped collector in part B. Part C regulates the voltage, flow rate, and humidity inside the electrospinning. On the other hand, Part D helps ascertain the state of the solution to be spun, with the results displayed on a monitoring layer. This allows for the assessment of the stability of the jet. Figure 1 (b) shows the results of electrospinning nanofibers membrane.

2.2.1. Synthesis of CA Membranes

CA and CA-Zeolite nanofiber membranes were synthesized using the electrospinning method. Cellulose acetate with a certain concentration was dissolved in a 3:1 acetone/DMAc solution and stirred with a magnetic stirrer for 24 hours. Different polymer solution concentrations were prepared, including 12, 14, 16, and 18% (w/v) [17]. Subsequently, the solution was transferred into a 10 mL syringe. After that, the flow rate, applied voltage, and distance between the needle tip and the collector were adjusted.

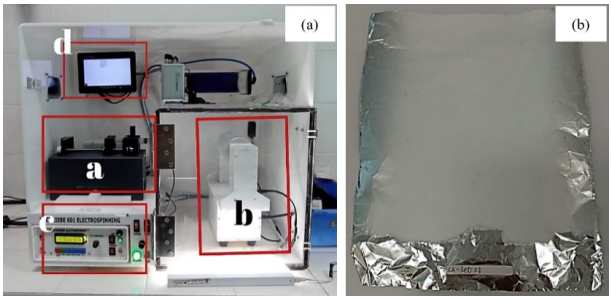

Figure 1. (a) Electrospinning tools and (b) CA-Zeolite nanofiber membrane

Table 1. Variation in parameters of the electrospinning process

Concentration of CA solution w/v (%)	Applied voltage (kV)	Flow rate (mL/H)	Distance between needle tip to collector (cm)
12	7, 7.5, 8, 8.5, 9, 9.5, 10, 10.5, 11	0.02, 0.04, 0.06	10, 11, 12, 13
14	7, 7.5, 8, 8.5, 9, 9.5, 10, 10.5, 11	0.02, 0.04, 0.06	10, 11, 12, 13
16	7, 7.5, 8, 8.5, 9, 9.5, 10, 10.5, 11	0.02, 0.04, 0.06	10, 11, 12, 13
18	7, 7.5, 8, 8.5, 9, 9.5, 10, 10.5, 11	0.02, 0.04, 0.06	10, 11, 12, 13

The variations of applied voltage start from 7, 7.5, 8, 8.5, 9, 9.5, 10, 10.5, and 11 kV. The flow rate variations used 0.02, 0.04, and 0.06 mL/H, and the distance variations used 10, 11, 12, and 13 cm. The CA solution in the syringe was put into an electrospinning tool. In the collector part, preparate glass was added to collect the spun fibers of the CA solution. The electrospinning process was conducted for 5 minutes for each sample variation to collect fiber samples for characterization. Table 1 shows the variation of CA solution samples synthesized using the electrospinning method.

2.2.2. Determination of Optimal Conditions

CA membrane samples with variations in concentration applied voltage, flow rate, and distance were observed on the surface using a binocular optical microscope with a magnification of 40×, as previously reported [18]. This step aims to determine the optimum condition of the CA membrane. Membranes with the most optimum conditions were applied to adsorb Pb (II) metal.

2.2.3. Synthesis of CA-Zeolite Membrane

CA concentration with the most optimum condition was added with variations of 2, 4, and 6% (w/w) zeolite and stirred using a magnetic stirrer for 24 hours. After that, the CA-Zeolite membrane was made similarly by adding aluminum foil to the collector to collect the spun fibers.

2.2.4. SEM-EDX (Scanning Electron Microscope Energy Dispersive X-ray)

The structure and elemental compositions of CA and CA-Zeolite membranes were analyzed using SEM-EDX (Phenom ProX) with 10,000× magnification. Furthermore, the test results data were analyzed using Photoshop software to determine the average diameter of the membrane fibers, following a methodology similar to previous research [18].

2.2.5. FTIR (Fourier Transform Infra-Red)

FTIR analysis was employed to identify the functional groups within the sample. The absorbance spectrum was obtained in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1}. The sample was cut into 2 × 2 cm size for the analysis. This analysis primarily focused on identifying O-Si-O bonds indicative of zeolite's incorporation into the CA solution. The presence of O- Si- O bonds serves as evidence that zeolite has been uniformly dispersed within the CA solution [16].

2.2.6. Contact Angle

Contact angle testing was carried out to determine the hydrophilicity and hydrophobicity of the membranes, as previously done by Wang *et al.* [15]. CA and CA-Zeolite nanofiber membranes were cut into 3 × 3 cm widths and then tested using Optical Contact Angle OCA 25.

2.2.7. Application for Adsorption

CA and CA-Zeolite membranes with each variation were cut into 3 × 3 cm widths. Before the adsorption process, an initial step involved establishing a calibration curve. The calibration curve is very important in determining true values [19]. This was accomplished by creating solutions containing Pb (II) metal ions at concentrations of 0.25, 0.5, 1.0, 2.0, and 4.0 ppm, followed by the absorbance measurement using atomic absorption spectroscopy (AAS). A concentration absorbance plot was constructed to derive Equation (1).

$$y = ax + b \quad (1)$$

A Pb (II) solution with a concentration of 2.17 ppm was prepared in four separate containers. CA and CA- Zeolite membranes, each comprising three sheets measuring 3 × 3 cm, were immersed into these containers containing the Pb (II) solution. Subsequently, the membrane-contained solution was stirred using a magnetic stirrer for 4 hours. The solution was filtered and analyzed using AAS. The AAS analysis generated an absorbance value, which was employed to calculate the concentration value using Equation (2).

$$x = \frac{y-b}{a} \quad (2)$$

where x is the concentration of Pb (II) metal ion solution after adsorption, y is the absorbance, a is the slope, and b is the intercept.

3. Results and Discussion

3.1. Determination of Optimum Conditions for CA and CA-Zeolite Nanofiber Membranes

The surface area of the electrospun CA nanofibers membrane was observed under a microscope, and the ideal operational conditions were ascertained. The CA solution was subsequently prepared under optimal conditions by incorporating zeolite. Identifying these optimal conditions for CA nanofiber membranes involves an optimization procedure that depends on several factors, including solution concentration, applied voltage, flow rate, and the distance between the needle tip and the collector. The optimal condition is chosen based on the stability of the droplets that reach the collector. Jet stability will result in fibers with few beads.

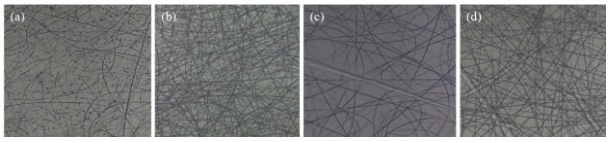

Figure 2. Nanofiber membrane of CA with various concentrations of (a) 12%, (b) 14%, (c) 16%, and (d) 18% with flow rate 0.02 mL/H, voltage 10.5 kV, and a distance of 10 cm

3.1.1. Concentration of Polymer

The polymer concentration plays a significant role in determining the size of the membrane fibers [15]. In this study, various concentrations of CA (12, 14, 16, and 18% w/v) were employed. Figure 2 displays the microscopic examination of the CA solution. At a 12% CA concentration, the resulting fibers exhibited numerous beads. This occurrence can be attributed to the low viscosity of the solution, leading to bead formation on the membrane fibers [16]. Increasing the concentration up to 18% resulted in fibers with fewer beads. However, when the concentration became excessively high, the fibers lost their uniformity, as evidenced in Figure 2 (c) and (d). Excessively high viscosity hindered the formation of a stable jet from the solution [15]. The optimal concentration was 14%, at which point the jet remained stable, the membrane fibers were uniform, and the bead formation was minimal, as depicted in Figure 2 (b).

3.1.2. Applied Voltage

Applied voltage controls the strength of the electric field between the needle tip and the collector. Meanwhile, the intensity of the force was used to spin the membrane fibers [17]. In this study, the applied voltage ranged from 7 kV to 11 kV. A lower voltage results in a weaker electrostatic force, preventing the formation of well-defined fibers, as evident in Figure 3 (a, b, c). As the voltage increases, the diameter of the fibers decreases. This reduction in fiber diameter can be attributed to the augmented electric field strength, which diminishes the fiber's size [16]. Increasing the applied voltage is to achieve jet stability, thus producing thinner fibers. Figure 3 illustrates that the jet indeed stabilizes with increasing voltage. However, at 11 kV, beads appear on the fibers due to the voltage surpassing a critical threshold [18]. The optimal voltage is 10.5 kV, as depicted in Figure 3 (h).

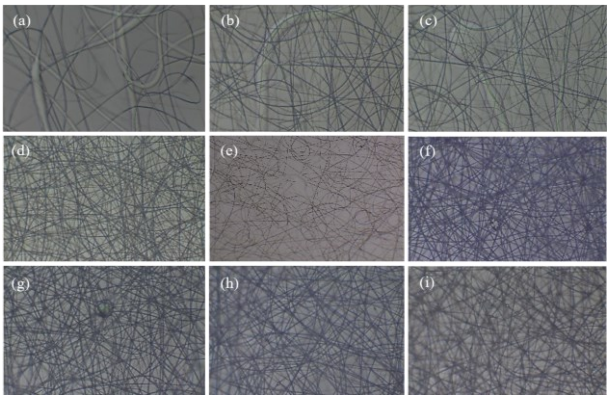

Figure 3. Nanofiber membrane of CA 14% at various voltages (a) 7 kV, (b) 7.5 k, (c) 8 kV, (d) 8.5 kV, (e) 9 kV, (f) 9.5 kV, (g) 10 kV, (h) 10.5 kV, and (i) 11 kV, with flow rate 0.02 mL/H and a distance of 10 cm

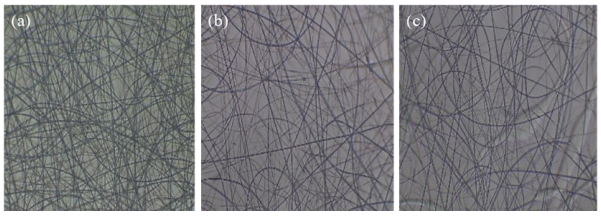

Figure 4. Nanofiber membrane of CA 14% at voltage 10.5 kV with various flow rates (a) 0.02 mL/H, (b) 0.04 mL/H, (c) 0.06 mL/H, and a distance of 10 cm

3.1.3. The Flow Rate of Polymer Concentrations

Flow rate is an essential parameter for determining the speed of the polymer jet rate. A low flow rate is better because the droplets have enough time to form jets before reaching the drum collector [20]. The increasing flow rate results in a faster push of the solution, resulting in fibers with many beads, as there is not enough time for the solution to dry before reaching the collector [21]. The optimum flow rate was 0.02 mL/H, as shown in Figure 4 (a).

3.1.4. The Distance of the Needle Tip to the Collector

The distance between the needle tip and the collector is a pivotal parameter in controlling the size of membrane fibers. This parameter significantly affects the surface morphology due to its connection with deposition time and the rate of evaporation [18]. When the distance between the needle tip and the collector is too close or less than 10 cm, beads develop on the fibers. Conversely, if the distance is excessively far, it results in discontinuous fibers due to the weakening of the electric field strength, causing an increase in fiber diameter. The optimal distance, as illustrated in Figure 5, is 10 cm.

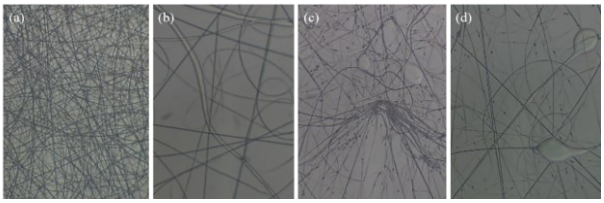

Figure 5. Nanofiber membrane of CA 14% at 10.5 kV voltage and 0.02 mL/H flow rate with various distances of (a) 10 cm, (b) 11 cm, (c) 12 cm, and (d) 13 cm

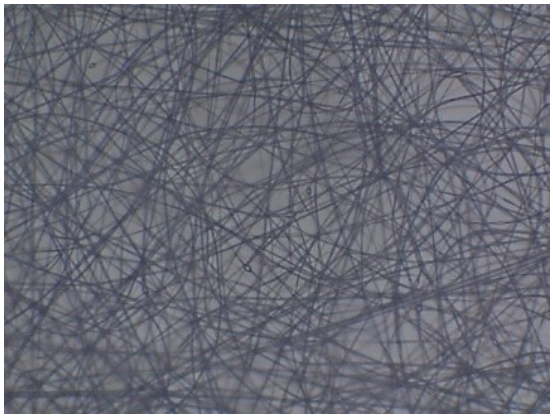

Figure 6. SEM image of CA nanofibers membrane at 14% concentration, 10.5 kV voltage, 0.02 mL/hour flow rate, and 10 cm distance between the needle tip and the collector

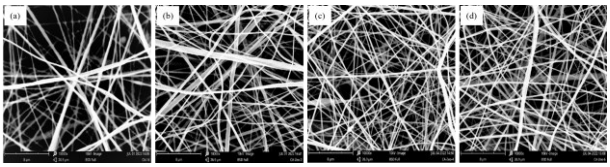

Figure 7. SEM characterization results of (a) CA 14% nanofiber membrane, (b) CA-Zeo 2%, (c) CA-Zeo 4%, (d) CA-Zeo 6% at 10,000× magnification

The optimal electrospinning conditions for the CA nanofiber membrane were determined to be a 14% concentration, a 10.5 kV applied voltage, a 0.02 mL/h flow rate, and a 10 cm distance between the needle tip and the collector, as depicted in Figure 6. This determination was made by assessing the stability of the jet formation and analyzing the image representations obtained using a binocular optical microscope.

3.2. Characterization of CA and CA-Zeolite Nanofiber Membranes

3.2.1. Scanning Electron Microscope

The morphological structure and elemental composition analysis of CA and CA-Zeolite nanofiber membranes were performed using a Phenom Pro X Scanning Electron Microscope (SEM) with 10,000× magnification (Figure 7). The obtained images were also processed using Photoshop and Microsoft Excel to calculate the average diameter of the CA and CA-Zeolite fibers membrane. Notably, the average fiber diameter decreased as the zeolite concentration increased, diminishing from 662.4 nm to 353.1 nm. This observation aligns with findings from previous studies that employed Ultra High Silica Zeolite (UHZS) as an additive to CA [13].

On the other reference, the fiber diameter of zeolite decreases when a high voltage is used [22]. This is why the diameter of CA-Zeo 6% is smaller than that of others. The fiber diameter distribution of CA and CA-Zeolite nanofiber membranes is shown in Figure 8. The higher concentration of zeolite addition results in a more uniform fiber distribution. EDX analysis is intended to verify the presence of zeolite in the CA solution. Figure 9 displays the spectrum of the EDX test results, with Si, Al, Na, and Mg elements as evidence that the zeolite addition was effectively distributed within the CA solution.

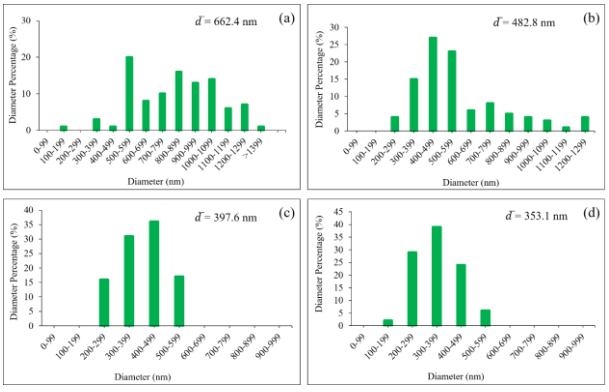

Figure 8. The distribution of fiber diameters of (a) CA 14% nanofiber, (b) CA-Zeo 2%, (c) CA-Zeo 4%, and (d) CA-Zeo 6%

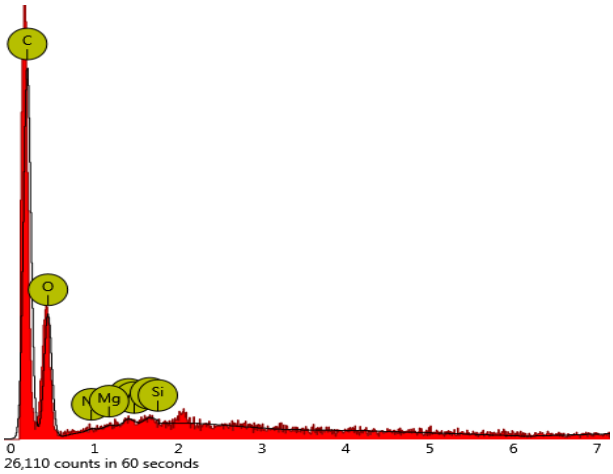

Figure 9. EDX spectrum analysis of CA-Zeo 6% nanofiber membrane

3.2.2. Fourier Transform Infra-Red

FTIR analysis is intended to determine functional groups present in the samples. The spectrum was recorded in the range of 4000-400 cm^{-1} wavenumber, shown in Figure 10. The C-O functional group of cellulose acetate was identified at 1273 cm^{-1} in the pure CA membrane and at 1238.66, 1236.41, and 1236.50 cm^{-1} in the CA-Zeo 2%, CA-Zeo 4%, and CA-Zeo 6% membranes, respectively. C=O functional group appears at 1747.11 cm^{-1} at pure CA, 1748.03 cm^{-1} at CA-Zeo 2%, 1746.79 cm^{-1} at CA-Zeo 4%, and 1745.28 cm^{-1} at CA-Zeo 6%. C-H functional group appears at 2942.37 cm^{-1} at pure CA, 2942.06 cm^{-1} at CA-Zeo 2%, 2942.42 cm^{-1} at CA-Zeo 4%, and 2942.38 cm^{-1} at CA-Zeo 6%.

The wavenumber changes along with the addition of zeolite at 1236.41 cm^{-1}. The functional group O-H appears at 3475.59 cm^{-1} at pure CA, 3468.57 cm^{-1} at CA-Zeo 2%, 3479.89 cm^{-1} at CA-Zeo 4%, 3468.92 cm^{-1} at CA-Zeo 6%. When the zeolite was added, the peak of the functional group of cellulose acetate changed. A small peak appeared in the spectrum of CA-Zeolite in the wavenumber range between 400 and 610 cm^{-1}, indicating the presence of O- Si-O bonds, which are zeolite functional groups. This is in line with previous research [16].

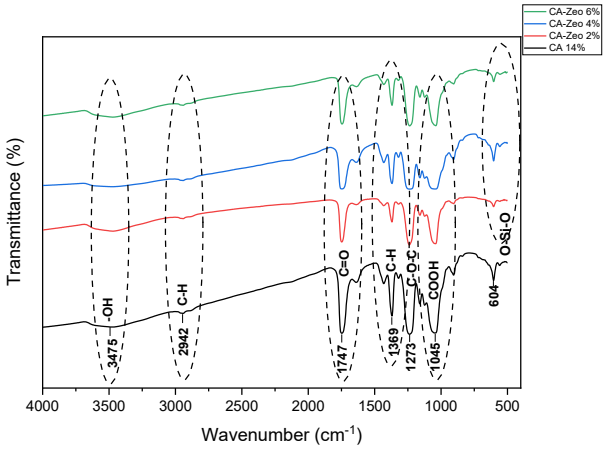

Figure 10. FTIR spectrum of CA dan CA-Zeolite nanofiber membranes

3.2.3. Contact Angle

Contact angle testing aims to determine the hydrophobicity and hydrophilicity of the membrane. Based on the analysis results in Figure 11, both CA and CA- Zeolite nanofiber membranes have contact angle values greater than 90°, indicating their hydrophilic nature [18]. Nevertheless, as detailed in Table 2, the contact angle values decrease with increased zeolite concentration.

3.3. The Adsorption of CA and CA-Zeolite Nanofiber Membranes

The adsorption process commenced with the preparation of several solutions, followed by their analysis using AAS. The results of this test generated a calibration curve, as depicted in Figure 12. This calibration curve was used to derive a linear regression and formulate Equation 3.

$$y = 0.04615x + 0.0003 \quad (3)$$

Previous research has reported the removal of lead ions from the water using cellulose/polycaprolactone (CA/PCL). The adsorption process was set up and placed in a water bath for 24 hours. Then, every 8 hours, the solution was collected and analyzed with an AAS instrument to determine adsorption capacity and kinetics. The capacity of the CA/PCL-doped membrane has improved more than that of the pure CA membrane. The adsorption capacity was increased due to the variation in contact times [23].

Meanwhile, in this research, the adsorption process was set up in the beaker and stirred with a magnetic stirrer for 4 hours. Then, the solution is analyzed with an AAS instrument to determine the adsorption capacity. The absorbance data were used to determine the final concentration of the Pb(II) metal solution via Equation 3.

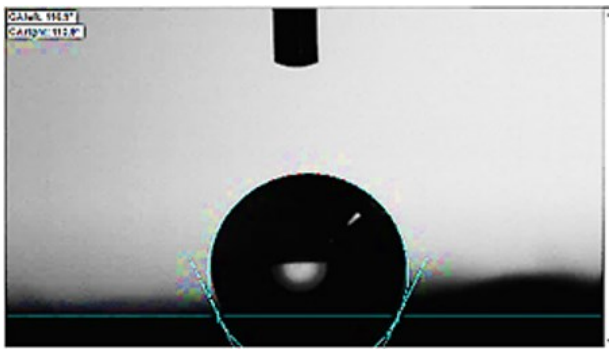

Figure 11. The result of the contact angle for CA-Zeo 6% nanofiber membrane

Table 2. Contact angle measurement of CA-Zeolite membranes

Sample	Contact angle
CA 14%	125.49°
(CA-Zeo 2%)	117.18°
(CA-Zeo 4%)	116.45°
(CA-Zeo 6%)	111.66°

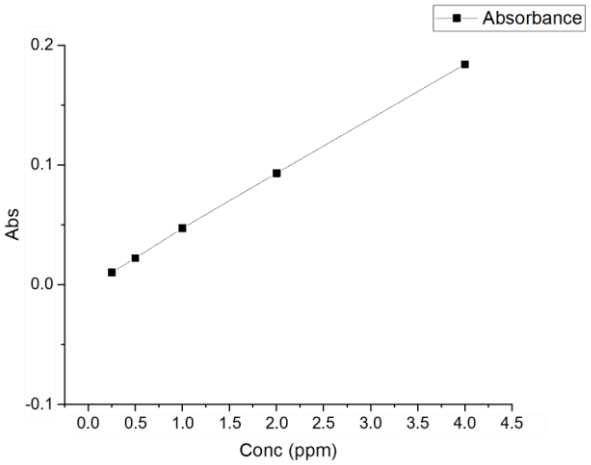

Figure 12. Calibration curve of Pb (II) solution

Table 3. The result of Pb (II) before and after the adsorption process

Sample	Concentration of Pb (II) (ppm)			Q (mg/g)
	First	Final	Adsorbed	
CA 14%	2.1729	1.1635	1.009	1.009
(CA-Zeo 2%)	2.1729	0.708	1.464	1.465
(CA-Zeo 4%)	2.1729	0.578	1.594	1.594
(CA-Zeo 6%)	2.1729	0.578	1.594	1.595

The analysis, employing Equation 3, yielded the value of x, as presented in Table 3. The results demonstrate that increasing zeolite concentration decreases the final concentration of the Pb(II) metal solution, indicating enhanced adsorption of Pb(II) ions. The CA-Zeo 6% nanofiber membrane has the most effective ability to adsorb Pb(II) metal levels. This is also supported by the contact angle value, which is smaller than that of other samples. Hence, the ability to adsorb is more effective than others.

4. Conclusion

Cellulose acetate nanofiber membranes were successfully synthesized using the electrospinning method under optimal conditions: a CA concentration of 14% (w/w), a voltage of 10.5 kV, a flow rate of 0.02 mL/h, and a distance of 10 cm between the needle tip and the collector. The CA-Zeo 6% (w/w) membrane produced nanofibers with an average diameter of 353.1 nm. EDX testing indicated that zeolite had been distributed in the CA solution. The FTIR spectrum showed the presence of O-Si-O bonds as zeolite functional groups. The contact angle value decreased with increasing zeolite concentration. The adsorption capacity of CA-Zeo 6% nanofibers was 1,595 mg/g.

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