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Synthesis Nanofiber PVA/Chitosan Using Electrospinning Method and Application for Gold Recovery

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ABSTRACT

This paper introduces a new process of gold recovery using nanofiber PVA/Chitosan from a gold-cyanide solution. Gold recovery in cyanide solution is made using nanofiber PVA/Chitosan produced with electrospinning technique. This research was conducted through several stages, 1) The designing of electrospinning tool, 2) Synthesis of nanofiber PVA/Chitosan with electrospinning technique, and 3) Gold recovery experiment using nanofiber PVA/Chitosan biosorption with the variations of initial concentration and time. The results showed that nanofiber PVA/Chitosan could be used as a gold ion absorber. The occuration of the isotherm process follows the Freundlich isotherm model, which is advantageous and occurs on a heterogeneous surface. From the results, it was agreed that nanofiber PVA/Chitosan is potential for gold recovery.

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I. Introduction

Nanofibers have become a fascinating topic for researchers due to their application in various research fields such as biotechnology, biomedical, electrical & electronics, environmental and energy resources due to their advanced nature and high potential. The method for the manufacture of nanofiber, which is currently still being developed, is the electrospinning method. Electrospinning is a method for producing nanofibers that utilizes an electric force applied to attract charged threads in a polymer solution to the diameter of the fiber in nanometers. The working principle of electrospinning is that when sufficient voltage is supplied to the liquid droplet, it becomes charged. An electrostatic repulsion occurs against the surface tension. The droplet is stretched at the critical point of flow where the liquid droplet erupts from the surface and becomes a fiber, as shown in Figure 1. [1], [2].

This article shows the work of an electrospinning unit that was designed by utilizing a flyback transformer. The electrospinning unit was tested to synthesize PVA/Chitosan nanofibers applied as gold ion absorbers. The test results are compared with the results of previous studies to indicate whether the tool from this study is stable or not [3].

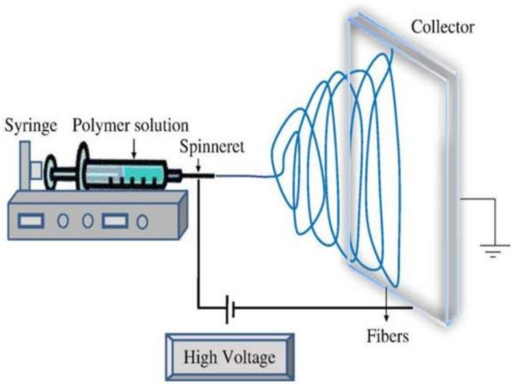

Figure 1. The scheme of the electrospinning tool

This research focused on the functional test of electrospinning tool to synthesize nanofiber PVA/chitosan, which will be applied as gold recovery material in cyanide solution. The application of PVA/Chitosan nanofiber as biosorption is a novelty that is excelled in this research. Previous studies have successfully performed gold recovery in cyanide solution using chitosan and nanofiber PVA/chitosan as biosorption [3]–[5]. Based on the results of some previous studies, the testing indicators in this research will be analyzed using the Freundlich isotherm model [5].

II. Theory

Electrospinning is the most powerful, efficient, and easy technique for fabricating very thin fibers from polymer solutions [6], [7]. Electrospinning techniques are capable of producing continuous fibers and homogeneous diameters. The size diameters produced can be micrometers up to nanometers. It depends on the parameters during the electrospinning process. Those size diameters provide an exceptional value as a superior material that has been applied as bone tissue engineering, wound dressing [8], [9], nanomedical [10], nanocomposite [6], and nanoparticle filtration membrane [11].

Electrospinning has three main components: a high voltage DC source, a syringe pump, and a collector plate or collector drum. The electrospinning scheme, which consists of three main components, can be seen in Figure 1. The high voltage DC (HVDC) source in the electrospinning process has a significant role because the high voltage can produce coulomb force in the solution released from the syringe pump to produce nanofibers and collected to the collector [12].

The electrospinning process requires a high voltage between 15-20 kV, so it is essential to design an HVDC to support this research. Previous researchers have done it a lot with many different methods [13]–[17].

III. Method

Material

Tools and materials prepared in this study: Multimeter, Richmeter, Microcontroller Arduino, DC 12V and 24V power supply, 5V, and 9V Voltage Regulator, Driver Flyback**,** Transformator flyback and step-up circuit, electrical wires, syringes, aluminum foil, preparat, beaker glass, magnetic hot plate stirrer, digital scale, Atomic Absorption Spectrometry (AAS), Polyvinyl Alcohol (PVA) (having molecular weights of 13.000 – 23.000 g/mol) was purchased from the Sigma-Aldrich Corporation, Chitosan (Self-Development from shrimp shells local NTB), acetic acid (Sigma-Aldrich Corporation), gold solution, and aquadest.

Method

The design of the HVDC in this study was carried out to create a high voltage source of 0-20 kV using the Arduino-based PWM technique. Furthermore, electrospinning and performance tools are set up for the manufacture of nanofiber PVA/Chitosan. A general diagram of research completion is shown in Figure 2.

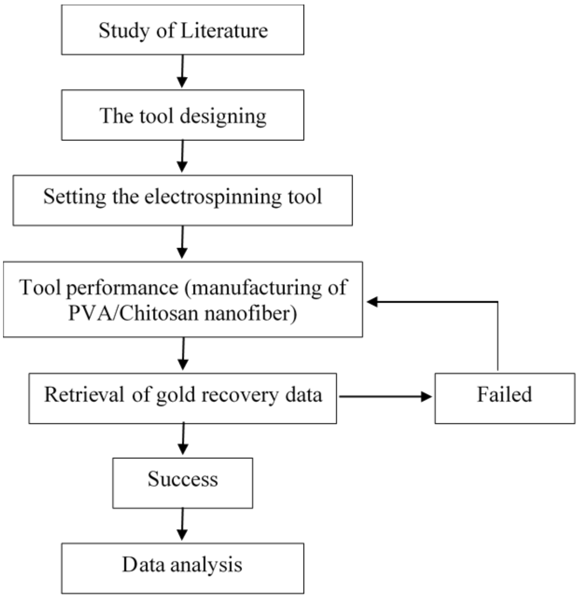

Figure 2. A general diagram of research completion.

After preparing all the hardware, each hardware is assembled into the HVDC system. Furthermore, HVDC is set up with the other main components of electrospinning. Then, a complete electrospinning tool unit as the result of this research can be seen in Figure 3.

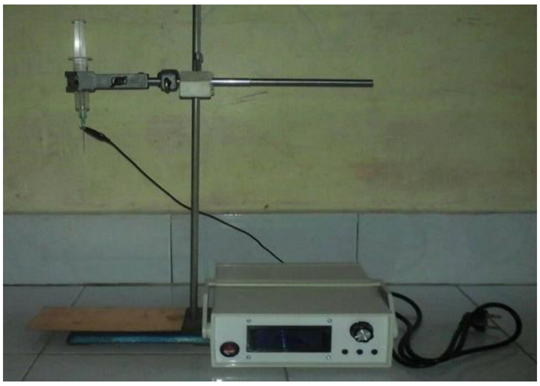

Figure 3. Electrospinning tool unit without syringe pump, solution flowrate is controlled manually by direct observation

The Synthesis Process of Nanofiber PVA/Chitosan

In preparation for the first solution, 10% of PVA (1 g) is added with 10 ml aquadest, and stirred using a magnetic stirrer at the speed of 500 rpm and a temperature of 80 ^{o}C until the solution is homogeneous. Preparation for the second solution, 10 ml acetic acid 1% (0.1 ml) is added with 9.9 ml aquadest, 1% chitosan (0.1 g) is added and then

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stirred using a magnetic stirrer at the speed of 500 rpm until the solution is homogeneous. PVA solution and chitosan solution were mixed with a 9:1 volume/volume (v/v) percentage ratio (Sample 1), stirred using a magnetic stirrer at the speed of 500 rpm until the solution is homogeneous. This process is repeated for 2 PVA and chitosan solutions in a ratio of 5:5 (Sample 2) and 1:9 (Sample 3).

Electrospinning Technique

The mixture of PVA solution and Chitosan is ready to be processed with the electrospinning technique. Two ml of the solution is added to the syringe. Set the distance of the firing needle to the collector of 10 cm with a voltage of 20 kV within 1.5 hours.

Biosorption Experiment

The biosorption of gold using nanofiber PVA/Chitosan is done by filtering 10 ml gold solution with the initial concentrations of 10 ppm, 20 ppm, and 30 ppm. Each solution was filtered with time variations of 10, 30, 45, 60, 90, and 120 minutes. The absorption data analyzed using AAS can be seen in Table 1.

Table 1. Results of analysis of AAS biosorption Au by nanofiber PVA/Chitosan with variations in initial concentration of the solution and initial concentration

Time (minute)	10 ppm (g/ton)	20 ppm (g/ton)	30 ppm (g/ton)
10	2.783	2.670	2.924
30	3.045	3.558	3.508
45	3.270	4.096	3.661
60	3.231	3.483	4.150
90	3.217	3.475	3.937
120	3.172	4.111	4.528

Based on the data in Table 1, the graph equilibrium plotting can be obtained from the gold biosorption isotherm process using nanofiber PVA/Chitosan for each solution with the initial concentration of 10 ppm, 20 ppm, and 30 ppm. The equilibrium graph of the gold biosorption isotherm process can be seen in Figure 4.

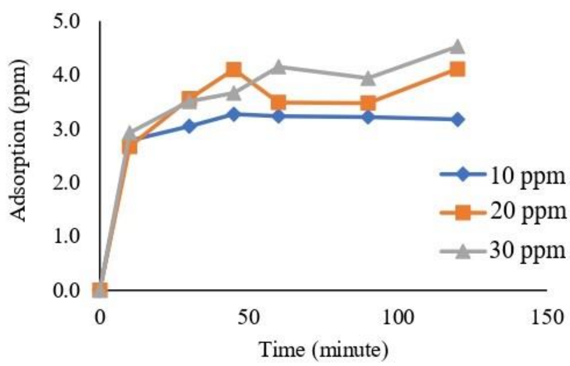

Figure 4. The equilibrium graph of the gold biosorption isotherm process

IV. Results and Discussion

The Designing of Electrospinning Tool

The 24 V positive voltage generated by the 24 V power supply will be fed directly to an inductor with a value of 100uH and is connected directly to the primary winding. The magnetic field in the primary winding changes due to a pulsing current and will produce an induced current on the secondary winding, which has more winding to increase the voltage. In this section, the voltage will be increased from 0-24 V to the voltage in the kV order. The output voltage for TFB in this study can reach voltages in the range of 22 kV. HVDC that is ready and can produce a voltage of 0-22 kV is set up with the other main components, and an electrospinning tool unit is formed, as shown in Figure 2. For the initial test, input voltage (V_i) and output voltage (V_o) are measured. The measurement data can be seen in Figure 5. The graph in Figure 5 shows that the relationship between an input voltage and output voltage is the linear graph. It indicates that the HVDC is stable.

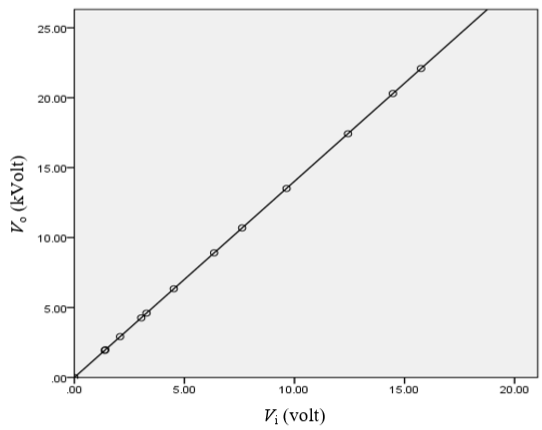

Figure 5. The graph of V_i and V_o

Synthesis of Nanofiber PVA/Chitosan

Synthesis of nanofiber PVA/Chitosan is a direct step used for the electrospinning tool's performance test. Nanofiber PVA/Chitosan is the result of electrospinning that can be seen in Figure 6. The electrospinning process is carried out using a voltage of 20 kV. The distance between the tip of the needle and the collector is 10 cm.

Figure 6. A photo nanofiber PVA/Chitosan from microscope with a size 1000X

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Furthermore, a mechanical test was carried out on the nanofiber to show the mechanical properties of PVA/Chitosan, which are tensile strength and Young's modulus. The result of the nanofiber mechanical test using the Tansilon machine can be seen in Table 2.

Tabel 2. The result data of nanofiber PVA/Chitosan mechanical test

No.	τ (MPa)	E (MPa)
1	22.059	579.16
2	20.260	25.044
3	18.793	5.562

Note: τ = tensile strength and E = Young's modulus

The data of tensile test values in Table 2 shows the highest tensile strength properties possessed by nanofiber PVA/Chitosan sample 1. Sample 1 contains less Chitosan with a tensile strength of 22.059 MPa. Sample 2 has a tensile strength of 20.260 MPa, while the lowest tensile strength is 18.793 MPa, owned by sample 3. The more Chitosan is added to the sample. It turns out to decrease the mechanical properties of PVA/Chitosan nanofibers.

Based on the result above, this study also proves that Polyvinyl alcohol (PVA) is one of the polymers often used as a blending material to improve thermal and mechanical stability. Dissolves easily in water, mechanical stability, flexible, easily formed into thin fibers, and non-toxic are the properties of PVA. Those are why PVA is used for medical, cosmetics, and agriculture [18], [19].

Freundlich Biosorption Isotherm

The Freundlich isotherm model is an empiric equation widely used to explain heterogeneous surface adsorption, active site, and exponentially distributed energy [20]. Empirically stated in equation (1).

$$Q_e = K_f C_e^{\frac{1}{n}} \quad (1)$$

The linearization of equation (1) is obtained:

$$LogQ_e = LogK_f + \frac{1}{n} LogC_e \quad (2)$$

Where C_e is the equilibrium concentration of adsorbate (g.dm^{-3}), Q_e is the amount of metal adsorbed per 1 g of the adsorbent at equilibrium (mg.g^{-1}). The K_f is a constant as an indicator to estimate the absorption capacity. At the same time, $\frac{1}{n}$ is a function of adsorption intensity in the adsorption process [21] or surface heterogeneity [22]. The slope value $0 < \frac{1}{n} < 1$ indicates that the adsorption isotherm is beneficial. If the slop value is $0 \geq \frac{1}{n}$, the surface of the adsorbent is more heterogeneous, and the nonlinear adsorption isotherm and $\frac{1}{n} > 1$ indicates that the adsorption isotherm is not beneficial [23] and indicates a cooperative adsorption process [24].

Figure 7 is a graph plot following the Freundlich isotherm equation and obtained $R^2 = 0.98$, and the linear regression equation for this process is $y = 0.179x + 2.398$. Based on the linear regression equation obtained $\frac{1}{n} = 0.179$, n = 5.593, and $K_f = 250.207$. The graph plot results in Figure 7 indicate that Au biosorption by nanofiber PVA/Chitosan is a beneficial isotherm and occurs on heterogeneous surfaces. The Freundlich isotherm occurs in (1) many layers (multilayer), (2) active sites on heterogeneous surfaces, and (3) involves the Van der Walls force such that the adsorbate can migrate from one area of the surface to another, according to Atkin (1996) in Bedolla et al. [25]. The biosorption of Au by nanofiber PVA/Chitosan follows the Freundlich isotherm, as indicated by an $R^2 = 0.98$.

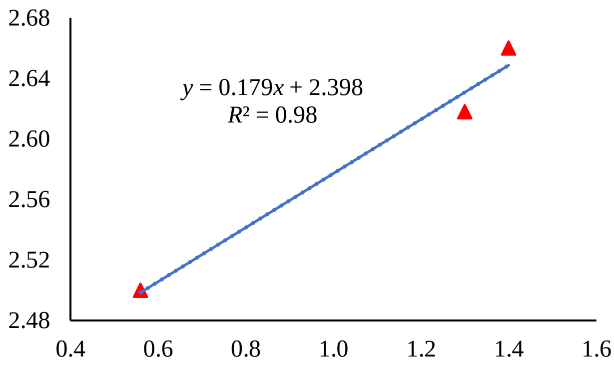

Figure 7. Freundlich biosorption isotherm

V. Conclusion

Based on the results of this research and discussion, it can be concluded that the electrospinning unit as a result of this study is feasible to use. The PVA/Chitosan nanofiber produced has mechanical properties that increase when added with PVA. The application of PVA/Chitosan nanofibers as gold biosorption shows good potential.

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