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## Electrospun Iron(III)-Montmorillonite/Polycaprolactone Nanomembrane Adsorption Module Design and Testing for Third Level Mining Wastewater Treatment

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### Introduction

Extraction of mineral from ores requires methods that introduce harmful substances that possess threats to the environment and to humans as well. This includes discharge of heavy metal ions like  $\text{Cu}^{2+}$ ,  $\text{As}^{3+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Hg}^{2+}$ , and  $\text{Cd}^{2+}$  to water systems like rivers and canals. Removal of these substances is necessary prior to discharge of the wastewater into water systems (Javier 2011).

One of the ways to remove these ions is through adsorption. Iron(III)-Montmorillonite (Fe-MMT) synthesized from ion exchange between Na-MMT and  $\text{Fe}^{2+}$  has been observed to remove these heavy metal ion by incorporating it in a nanofibrous membrane through electrospinning (Araño et al. 2014). However, direct application of the nanofiber membrane requires incorporation of the technology into a product that can be installed further in mining wastewater treatment facilities.

Module treatment methods utilizes small discrete units that are assembled together (Allgeier 2005). One of the most common type of modules is a spiral wound module, which is composed of various layers of material wrapped around a central holder, with feed spacers to allow fluid flow (Sutherland 2008; Serra et al. 1998).

To apply the Fe-MMT nanofiber membrane in mining waste water treatment, it should be transformed into a product, like a module, that can be attached in a treatment assembly for mining waste water treatment.

### Material and Methods

Fe-MMT was synthesized by ion exchange between  $\text{Na}^+$  and  $\text{Fe}^{3+}$  using Na-MMT Nanofill (Southern Clay Products, Inc.). The Fe-MMT was grinded to particle size less than  $45\text{ }\mu\text{m}$  using a microsieve. 0.1527g Fe-MMT was combined with 0.0763g Cloisite 25A (Southern Clay Products) in vial with 19mL dichloromethane (DCM, J.T. Baker) and was ultrasonicated for 30 minutes. 1.145g Polycaprolactone (PCL,  $M_n = 80\text{ }000$ , Sigma-Aldrich) was added to the solution and was ultrasonicated again for 60 minutes. The solution was electrospun on a #400 stainless steel mesh substrate at 33KV using Inovenso Nanospinner 24. A total of 6 layers was deposited per substrate.

The nanomembrane was rolled onto a 10-inch polyvinyl pipe with 0.125-inch holes bored around its body. The ends of the assembly were sealed using a moulded polyester cap, designed using 3D printing. Parts were attached to one another using a silicone glue. The product was installed into a household water treatment system for testing (Megafresh FA-2-AG).

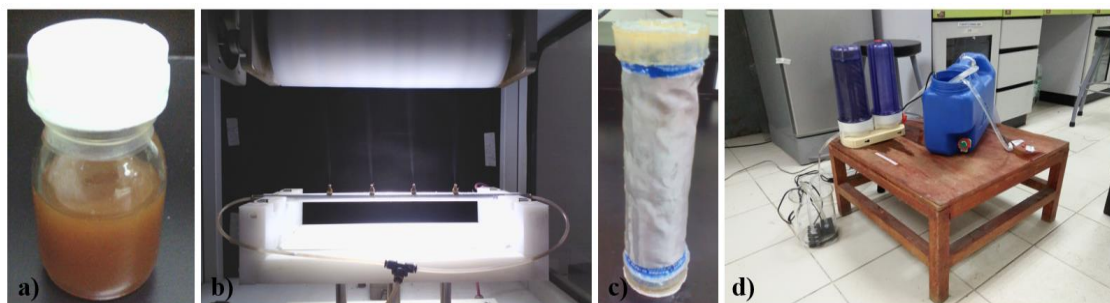
The integrity of the product was tested by allowing water to pass through while covering the body with a plastic film to test possible leakage at the ends of the product. The flowrate and head loss of the product was determined using Bernoulli Equation.



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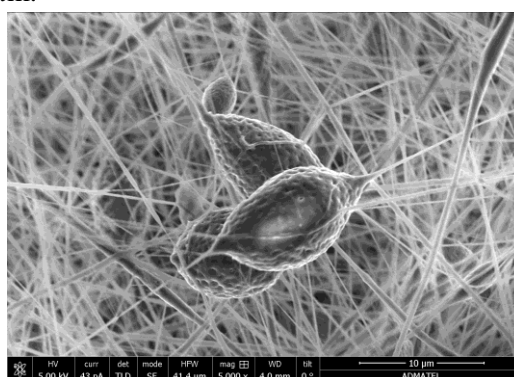
Adsorption capabilities of the design was determined using a laboratory-scale batch adsorption set-up by passing 3L of 40ppm  $\text{Cu}^{2+}$  solution for 180 minutes, with sampling every 5, 10, 20, 30, 60, 120, and 180 minutes. The  $\text{Cu}^{2+}$  concentration was analysed using X-Ray Fluorescence Spectroscopy (XOS HDXRF).



**Figure 1:** a) Fe-MMT/PCL solution, b) electrospinning of Fe-MMT nanomembrane, c) assembled adsorption module, and d) laboratory scale adsorption set-up used in product testing.

## Results and Conclusions

Fe-MMT nano particles was successfully incorporated into the nanofibrous membrane. SEM image of the nanofiber membrane in Figure 2 shows the deposited Fe-MMT nanoparticle in the membrane. Analysis with ImageJ shows that the average fiber diameter was 116.94, and the average nanoparticle size was 5.266 $\mu\text{m}$ .

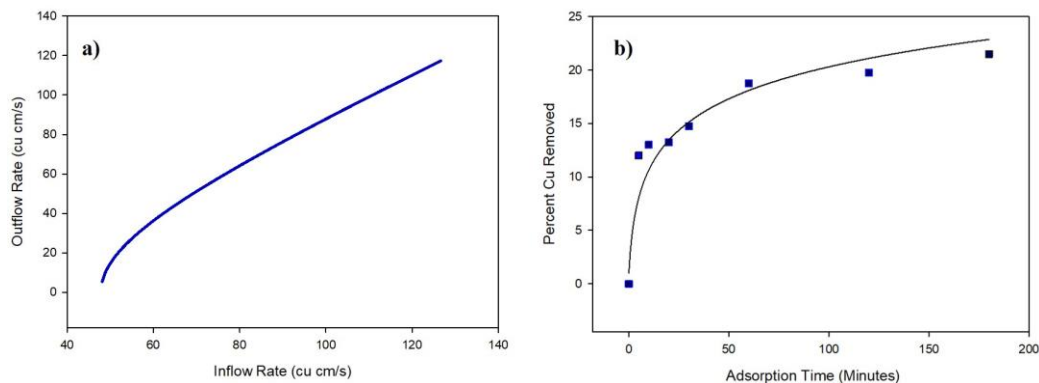


**Figure 2:** SEM image of the Fe-MMT nanofiber membrane at 5 000X

Leakage test of the product shows no possible damage in the cap. Flow rate analysis shows that the module has a head loss of 0.029m, requiring a minimum of 47.8mL/s inflow rate. At inflow rate less than 47.8mL/s, there will be no permeate flowing out of the product. This can be seen in Figure 3a.



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**Figure 3:** a) Inflow rate vs outflow rate of the assembled adsorption module, and b) percent removed  $\text{Cu}^{2+}$  as a function of the adsorption time.

Adsorption of  $\text{Cu}^{2+}$  to the nanofiber membrane shows a drastic removal during the initial stage of the adsorption process. However, as the adsorption time increases, the amount of removed  $\text{Cu}^{2+}$  decreases until there can be no change in the amount removed. This can be associated with the saturation of the nanomembrane with  $\text{Cu}^{2+}$  ions and the equilibria of the ions between the membrane and the feed water. **Error! Reference source not found.** b shows the graph of percent  $\text{Cu}^{2+}$  removed as a function of time.

In conclusion, the product assembly successfully incorporated the Fe-MMT nanofiber membrane in a modular product that can be installed readily in mining waste water treatment facilities. The adsorption capabilities of the Fe-MMT nanoparticles was still preserved even if it was incorporated into a module product. Fe-MMT nanomembrane removes the  $\text{Cu}^{2+}$  in the synthetic waste faster during the initial stage of treatment than in longer adsorption time due to saturation of the membrane. Further development of the product can be done to increase the amount of removed  $\text{Cu}^{2+}$  in the membrane using the product assembly. Also, the adsorption capabilities of the product with other heavy metals should be studied since actual mining waste contains more than one heavy metal contaminant.

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