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Full Length Research Paper

Synthesis, Characterization and Adsorption Studies on Cellulose acetate\ Sewage sludge ash Blend Membranes

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Membrane is an alternative technology of water treatment with filtration principle that is being widely developed and used for water treatment. The main objective of this study was to make an asymmetric membrane using cellulose acetate (CA) polymer and sewage sludge ash (SSA) by dry-wet phase inversion method in various proportions of CA/SSA (17.5/0, 15.75/1.75, 14 /3.2, 12.25/5.25 and 8.75/8.75g). Characterization of prepared membranes was performed such as water content, IR analysis and Scanning electron microscope (SEM). Also, the possibility of solute rejection of nickel ions from wastewater using Sludge membrane (SM) was evaluated by different parameters as pH value (2-10) and contact time from (0 to 30 min). Results reported that the best performance of CA/SSA blend membranes at 8.75/8.75g. Adsorption isotherm studies indicated that Freundlich model described the experimental data with R2 values greater than 0.985.

Keywords: Asymmetric membrane, Cellulose acetate\ sewage sludge ash, Phase inversion, Sludge membrane(SM), Characterization and Adsorption isotherm

INTRODUCTION

Water is a precious element that sustains the life over the earth. Water scarcity has a huge impact on environmental, agriculture and industrial development. Therefore, the water shortage showed the most important risks faced by human (Gobarah, M.E., et al, 2015). To protect this precious element from vulnerability we have to consider effective management for water resource.

The treatment of wastewater is one of the most sustainable alternatives to cope with shortage. That includes closing the gap between supply and demand, stopping the pollution of water resources, providing sound solution to water scarcity by removal of pollutant from wastewater for useful uses.

Removal of toxic contaminants and organic pollutants from wastewaters is one of the most important environmental issues. Among these components heavy metal ions such as Ni^{2+} are highly toxic to human bodies even at very low concentration (Han et al. 2009). For this

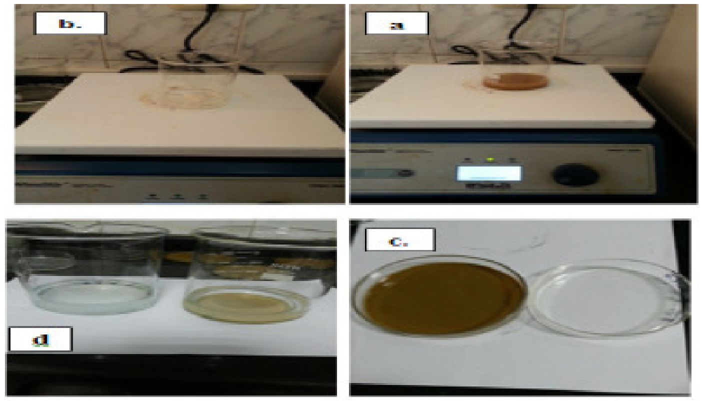


Figure 1. Preparation of Synthesis of Membranes

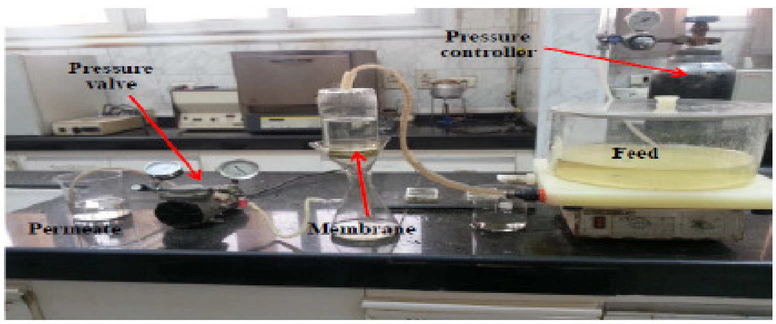


Figure 2. System Used in research

reason the Water Sanitation and Hygiene (WSH) under the World Health Organization (WHO) established the toxic limits of permissible concentrations of nickel at a level of Ni (II) and insoluble compounds of 1.0 mg/l, soluble compounds of 0.1mg/l.

Conventional treatment for heavy metals from wastewater includes chemical precipitation, ion exchange, electro dialysis (Memarian, R., et al, 2013). These methods are relatively expensive involving either costly equipments or elaborate procedures, while Membrane separation system enjoys numerous advantages (Bahareh, A. E., et al, 2014), over conventional treatment methods (Nagendran, A. ,et al, 2008), including energy saving, environmentally benign, clean technology with operational ease, high quality products, and great flexibility in system design.

Adsorptive membranes have reactive functional groups on the surfaces, by choice suitable polymer as Cellulose acetate is still being successfully used in water treatment with hydrophilic surfaces, easy to manufacture, low cost and good fouling resistance (Hokkanen 2014).

Blending of CA with SSA having capability of adsorbing or removing heavy metals from wastewater by high content of NH, CN groups and Al_2O_3, SiO_2 were the main functional groups of CA\SSA involved in metal binding charged and can be successfully used for the loading of anionic transition metal precursors by chemical interaction (Varma, G., et al, 2013).However, the performance of blending SSA with CA for adsorptive membrane.

The main objective of the present work is to modification of membrane by blending cellulose acetate with sewage sludge ash in various compositions, where the main characteristics effects of membrane preparation on water content (WC), infrared spectroscopy (IR), scanning electron microscope (SEM) and applications on the rejection of nickel ions from wastewater, finally study the adsorption isotherm.

MATERIALS AND METHODS

Materials

Cellulose acetate (CA) purchased from Sigma Aldrich Chemicals Co., sewage sludge ash (SSA) sample used in this study was collected from El-Berka municipal wastewater treatment plant , Poly (ethylene glycol 600) was procured from Merck (I) Ltd., and used as a non-solvent additive; annular grade N, N- dimethylformamide (DMF) and sodium lauryl sulfate (SLS) were obtained from Sigma Co.

Figure (3) Membranes at various compositions

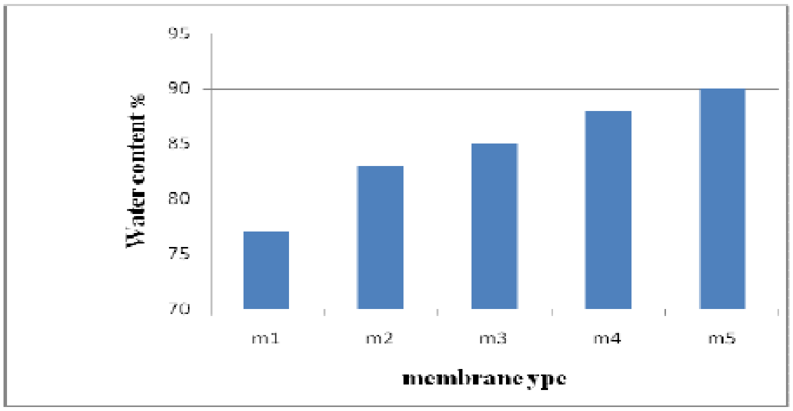


Figure 4. Effect of SSA content on water content using CA\SSA blend membranes

Methods

Preparation of SSA

The sewage sludge (SS) sample was dewatered by drying beds and burned in a modular incinerator at 600°C for 2h to produce sewage sludge ash (SSA) and then ground. The ground SSA was screened by 75μm sieve (El Sheltawy et al., 2016).

Preparation of Synthesis Blending of Membrane

Synthesis of blend membranes were carried out by phase inversion technique (Sudha P. N., et al, 2014) as follows; weight (17.5/0, 15.75/1.75, 14 /3.2, 12.25/5.25 and 8.75/8.75g) from CA\SSA separately, named (M1, M2, M3, M4, M5) respectively were dissolved in 82.5 mL DMF, to achieve total polymer concentration 100%, in 250 ml beaker and stirred well for 1 hour by magnetic stirrer at 200 rpm until complete dissolution was achieved at room temperature ± 25°C as shown in (figure 1a, b) , then stand 1 min for air bubbles then casting blend solution in 8 cm diameter of Petri dishes as shown in (figure1.c) . Also gelation bath of 2L distilled water (non-solvent), containing 2wt% DMF and 0.2wt% SLS was prepared and cooled to 17° C. Then immersed the Petri dish in the gelatin bath to make hardness of solutionin(figure4.d).

Ultra-filtration process using SM

The ultra-filtration experiments using SM were carried out in a batch mode, dead end cell as shown in Fig. (2), this cell was connected to a compressor with a pressure control valve and gauge through a feed reservoir. The experiments were performed at Chemistry lab for water & wastewater, Housing and Building Research Centre (HBRC).

Characterization of Membranes

Water content

The water content of membrane samples was estimated by removing the membranes from water and weighing immediately after blotting the free surface water, and drying in oven for 1h at 50°C. The percentage of water content of the prepared membranes was calculated from "Eq. (1)" **(**Velu, S., et al, 2014)

$$WC = \frac{W_w - W_d}{W_w} \times 100 \qquad (1)$$

Where, WC: water content of prepared membrane %.
W_w: wet weight of prepared, g.
W_d: dry weight of prepared, g.

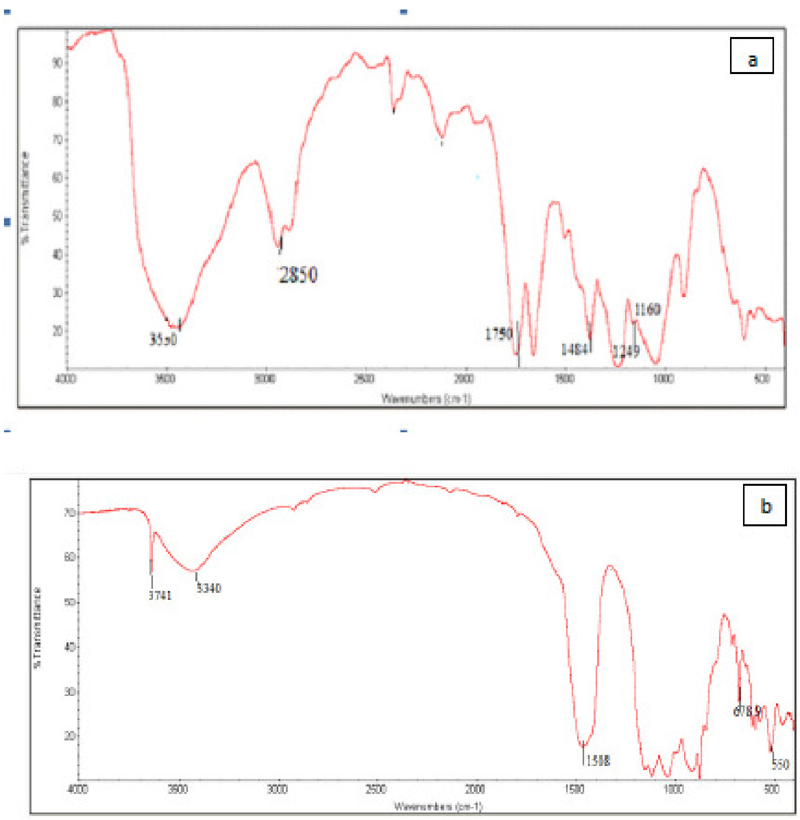
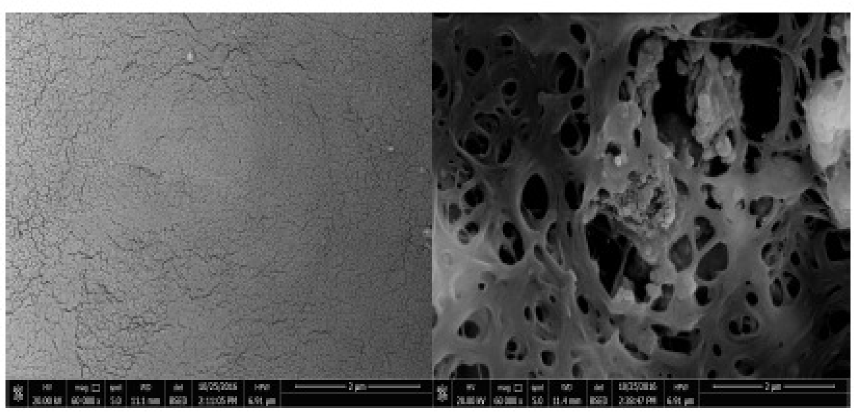


Figure (5) IR of Cellulose Acetate \ Sewage Sludge Ash blend membrane at a) M1, b) M5



(a) (b)

Figure (6) Scanning electron microscopic images of membrane a) M1 b) M5

Infrared spectroscopy

Infrared spectroscopic analysis (IR) was carried out at the Central Lab; Cairo University. In the present study analyzed by Shimadzu S 201 PC spectrophotometer – Japan was used for infrared spectroscopy with range 500-4000 cm^{-1}.

Scanning electron microscopy

Scanning electron microscopy analysis was carried out at National research Center to examine the surface morphology and top graphical information of the blend membranes, using Joel JEM-100 SEM**,** The membranes were cut into pieces of varied sizes, mopped with filter

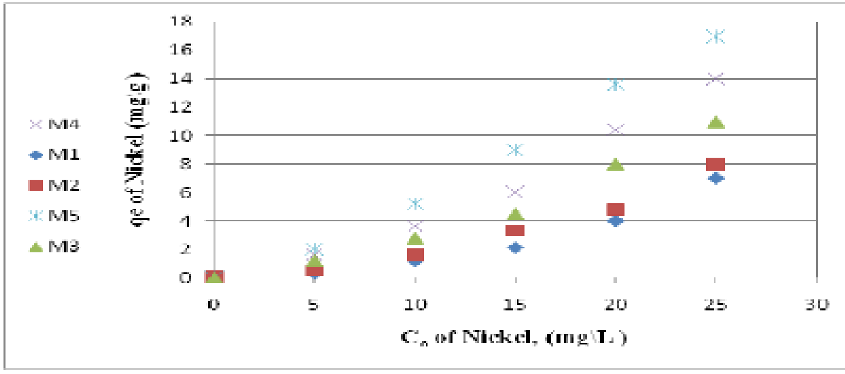


Figure (7) Equilibrium of nickel ion adsorption amounts on CA\SSA blend membranes versus Ce

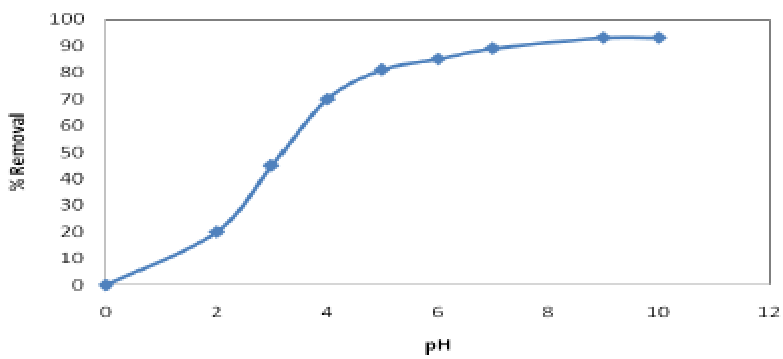


Figure (8) Effect of initial pH value on the % removal of nickel ions using M5 at Ni conc 25mg/L.

paper and immersed in liquid nitrogen for few seconds to fracture the membranes. The dried bits of membranes were stored in desiccators and used for SEM studies.

Adsorption of Nickel Ions

Preparation of nickel ions

According to Standard method for water and wastewater reported in ASTM (2005). The simulated stock of nickel ions (1000mg/L) was prepared by dissolving 4.47g of an annular grade of respective salt in a one liter of distilled water. The salt used is Nickel sulfate $NiSO_4 .6H_2O$. The stock solution was further diluted with distilled water to desired concentration 5-25 mg/L.

Effect of different operating condition on adsorption of Nickel ions

Effect of pH on % removal of nickel ions using M5

The effect of initial solution pH on the equilibrium uptake of nickel ions by CA\SSA membranes was studied at pH range from 2-9, pH was adjusted using 0.5N NaOH or 0.5N HCL solution, 500 mL of feed solution from nickel concentration 25 mg/L pass through the membrane in UF cell at room temperature 25±2ºC, permeate solution was collected in a 500ml beaker and the concentration of Nickel ions was estimated using atomic absorption spectrometer (ASS) (Model ICE 3000 Series - Thermo Scientific, with air acetylene flame at wave length of 231.6 nm). The percentage of Nickel removal was calculated by (Kusworo, T.D., 2014) according to "Eq. (2)"

$$\%NR = (1 - C_p / C_f) \times 100 \quad (2)$$

Where, %NR: the percentage of nickel ions removal.
Cp: Concentration of permeate.
Cf: Concentration of feed.

Effect of contact time on % removal of nickel ions using M5

The effect of contact time on removal of Ni(II) ions at 25mg/L was studied using CA\SSA (M5) during the period from 0-60 min at room temperature 25±2ºC for determine the time required for removal of ions.

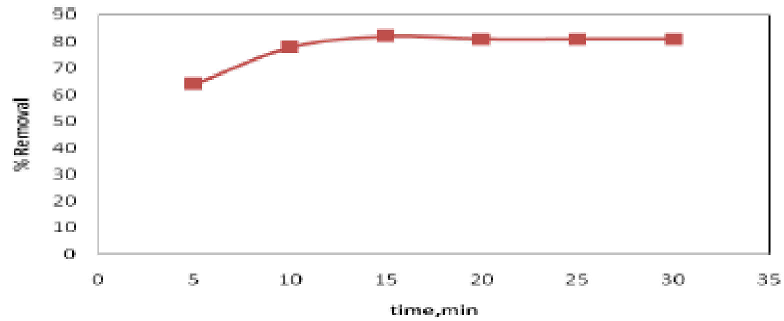


Figure (9) Effect of contact time on the % removal of nickel ions using M5 at con of Ni 25mg/Lat 25±2°C.

Table 1. Isotherm model parameters obtained for removal of nickel ions using CA\SSA.

Membrane CA\SSA	Langmuir parameters			Freundlich parameters			Temkin parameters		
M5	q_{max} mg\g	B L\mg	R^2	K_f mg\g	1\n	R^2	At l\g	B J\mol	R^2
	20.8	0.35	0.973	5.12	0.594	0.985	3.82	4.313	0.983

Adsorption Isotherm Models

Adsorption experiments with CA\SSA at M5 blend membrane for adsorption of nickel ions in solutions with different initial concentrations (5, 10, 15, 20 and 25 mg/L). As for the equilibrium experiments, about 0.69 g of membrane was added into 500 mL of nickel solution then using shaker at 200 rpm for 1 h at room temperature 25± ºC. The amount of nickel adsorbed at equilibrium (q, mg /g) was calculated by (Judith, T. R., et al, 2014) according Eq"(3):

$q = (C_0 - C_e) * V \backslash m$ (3)

Where C_0 : Initial nickel concentrations (mg/l).

C_e: Equilibrium nickel concentrations (mg/l).

V: volume of the solution (L)

m: mass of the (membrane) (g).

Adopted isotherm models were Langmuir, Freundlich and Temkin Isotherm coefficients and correlation coefficients (R^2) were computed from linearized equations of these isotherms in Microsoft Excel.

Langmuir isotherm

Langmuir isotherm has been extensively used for the adsorption of heavy metals, dyes, organic pollutants, etc. It is applicable for monomolecular layer adsorption. The Langmuir isotherm is used to obtain a maximum adsorption capacity produced from the complete monolayer coverage of adsorbent surface (Arivoli, S., Araskumar,A., 2017) . The linear isotherm equation is represented as:

$C_e/q_e = 1/bq_{max} + (1/q_{max})C_e$ (4)

Thus, a plot of C_e /q_e versus C_e should yield a straight line if Langmuir isotherm is obeyed by the adsorption equilibrium. q_{max} and b values will be calculated from the slope and intercept of the graphed line, respectively where b is the adsorption equilibrium constant in L/mg related to the apparent energy of adsorption, q_{max} is the maximum quantity of adsorbate required to form a single monolayer on unit mass of adsorbent in mg/g, A further analysis of the Langmuir equation can be made using a dimensionless equilibrium parameter, the separation factor R_L as given by Eq" (5):

$R_L = 1/ (1 + b\, C_o)$ (5)

For 0 >R_L<1 a favorable adsorption, $R_L > 1$ represents an unfavorable adsorption, $R_L = 1$ represents linear adsorption, whereas $R_L = 0$ translates into irreversible adsorption, (Nechifor, G., et al, 2013).

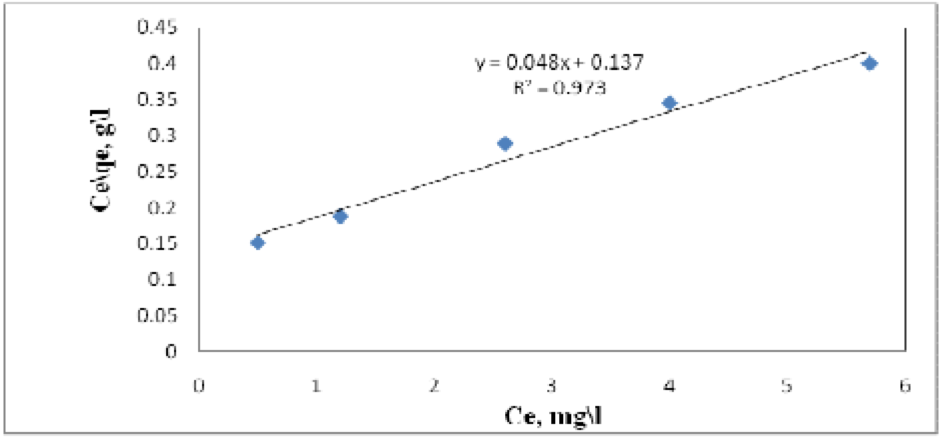


Figure (10) Langmuir Plot for Nickel ion Adsorption on M5 Blend Membrane

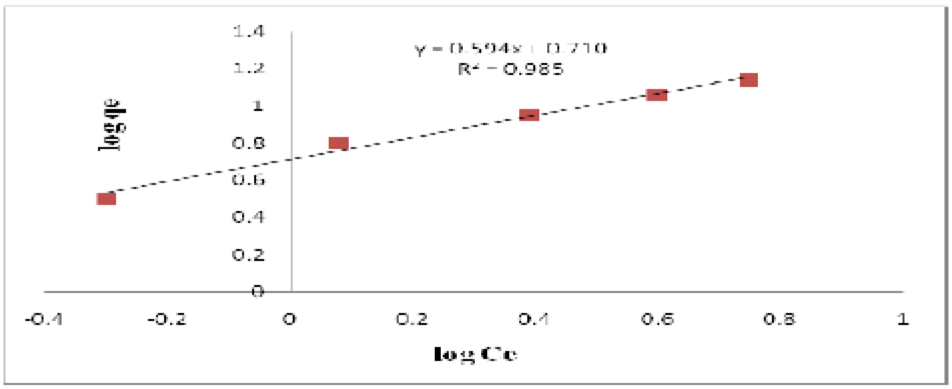


Figure (11) Frendlich Plot for Nickel ion Adsorption on M5 Blend Membrane

Freundlich isotherm

Freundlich expression is an exponential equation and therefore assumes that, the concentration of adsorbate on the membrane surface increases with the adsorbate concentration. Theoretically, using this expression, an infinite amount of adsorption can occur (Freundlich 1907).The equation is widely applied in heterogeneous surfaces with sites that have different energies of adsorption and are not equally available. The Freundlich isotherm can be written in linearized form as Eq (6):

$$\text{Log } (q_e) = \log K_f + 1/n \log (C_e) \quad (6)$$

Where: K_f = experimental constant (Freundlich constant indicative of the adsorption capacity of the adsorbent (l/mg) and n = experimental constant indicative of the adsorption intensity of the adsorbent. By plotting $log\ q_e$ versus log (C_e), values of K_f and n can be determined from the slope and intercept of the plot. (Bartczak, P., et al, 2015).

Temkin isotherm

Temkin isotherm studies the effects of some indirect adsorbate/adsorbate interaction on adsorption isotherms and suggested that because of these interactions the heat of adsorption of all the molecules in the layer would decrease linearly with coverage (Dada, A. O., 2012). The Temkin isotherm has been generally applied in the following form as Eq (7):

$$Q_e = BLinA + BLinC_e \quad (7)$$

Where: B =RT\b

B = Constant related to heat of sorption (J/mol).

A (slope) = Temkin isotherm equilibrium binding constant (L/g).

b (intercept) = Temkin isotherm constant.

R = universal gas constant (8.314J/mol/K).

RESULTS AND DISCUSSIONS

Preparation of blend membrane from CA blended with SSA after immersed blend solution in gelation bath with different weight ratio at (total polymer concentrations 100wt %) and adapted thickness of membranes 0.56 mm are shown in Fig. (3).

Water Content of Membranes

The influence of SSA content in the casting solution of

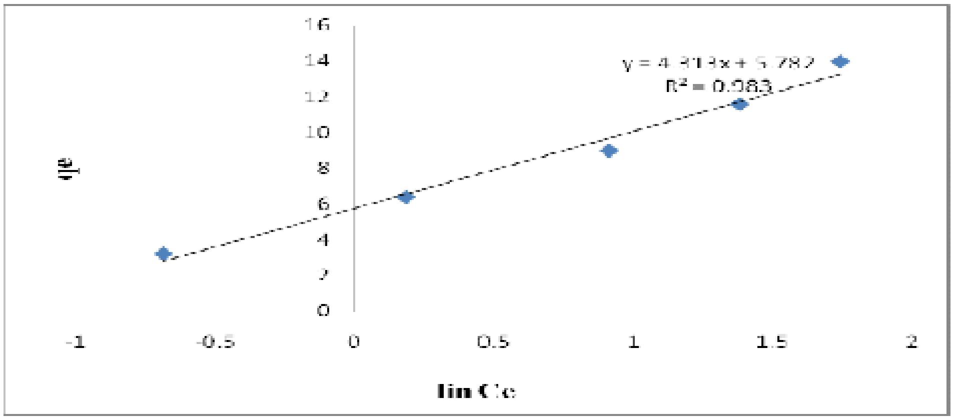


Figure (12) Temkin Plot for Nickel ion Adsorption on M5 Blend Membrane

CA\SSA at, M1, M2, M3, M4 and M5 in solvent DMF (82.5 wt %) on water content of prepared membrane are presented in Fig. (4). It reported the increased of water content from 77 % to 90% by increasing of SSA content (0-8.75g) from m1 to m5 respectively. This may be due to the fact that gelation was produced leading to formation of pores acting as demine of water molecules as hydrophilic source for attracting water molecules inside the membrane blends. This property will increase the service life of the used SM and therefore decreasing the operating cost of wastewater treatment process.

Infrared Spectroscopy

Infrared spectroscopy provides a simple and rapid instrumental technique for the identification of functional groups in organic compound. The IR spectra of CA\SSA (M1) at (17.5\0 g) showed 3580 cm^{-1} corresponding to OH stretching. Aliphatic –CH stretching was observed at 2850 cm^{-1}. Peaks at 1750 cm^{-1} corresponding to OH bonding, 1484 cm^{-1} indicate COOH groups and peaks at 1160 cm^{-1} is due to C-O-C linkage or cyclic ether bond in figure (5.a). The IR of M5 (8.75\8.75g) at SSA content was increased the peak at approximately 3741cm^{-1} corresponding Si-OH stretching, indicate the silicon content of sewage sludge ash was observed with higher intensity shown in figure (5.b), peaks at 3340cm^{-1} corresponding OH stretching of axial OH group, NH stretching and strong polymerization of cellulose acetate and sewage sludge ash .A Peak at 1508 cm^{-1} confirmed the presence of lignin from lingo cellulosic materials. New peak obtained around 678cm-1 for the –NH –Wagging.

Scanning Electron Microscopy

The performance of asymmetric membrane depends on structural and geometrical characteristics of the produced asymmetric cellulose acetate membranes were studied by scanning electron microscopic (SEM) for membranes cellulose acetate \sewage sludge (M1) as shown in figure (6.a) it could be seen that there was uniform distribution and disappear of pores on the skin layer indicates the amorphous nature of
Cellulose acetate. While increase of SSA in blend membrane in figure (6.b) represent a dense skin layer supported by spongy porous substructure with small macro voids, like a finger. Finger like phenomenon commonly occurs in asymmetric membranes preparation due to the high viscosity fluid in the membrane has been replaced by low viscosity fluid in the immersion process. The results were also in agreement with pervious results from (Kusworo, T.D., et al 2014) by preparing membrane from cellulose acetate with polyethylene glycol.

Adsorption of Nickel Ions

Nickel ion adsorption amount

The amount of nickel ions adsorbed at adsorption equilibrium qe, mg\g on CA\SSA at different blend ratio at initial Ni ions concentration C_0, mg\l ranging from 5-25 mg\l, in figure (7) represented the equilibrium adsorption amounts increased with increase of nickel ion concentration. It is also clear that the M5 always had the highest adsorption amounts, the amount of nickel ions uptakes by blend membranes followed the order of M5>M4>M3>M2>M1. This order is related to increase of SSA contents due to increase of pore size in membranes as shown in SEM test.

Effect of pH on % removal of nickel ions using M5

The pH of solution is an important parameter in adsorption process because of pH dictates not only the dissociation of functional groups but also the complexation reactions at the adsorption surface (Ghosh, A., et al, 2012). Figure (8) studied different media of solution as acidic, neutral and alkaline with pH(2-7-9),the removal efficiency of nickel ions 20, 89 and 93% respectively indicate at pH=2,the Ni (II) removal was negligible , where as the removal increased abruptly from 70 % to 89% at pH range from 4 to 7 respectively , indicated of the competition between hydrogen and nickel (II) ions on the sorption sites , above pH 7 occurs high removal efficiency of nickel ions at 93%, for result insoluble nickel (II) hydroxide starts precipitating from the solution ,so adsorption rate increase. This results seems by (Aseem Ali Palliparambil., et al, 2016) using membrane from cellulose acetate mixed nano-zero valent for nickel removal.

Effect of contact time on % removal of nickel ions using M5

Contact time is active agent for the studies of removal process through identification of suitable time to complete the process of adsorption of nickel ions on membrane M5 as shown in figure (9), the % removal is rapid in the first 15 min of contact period, efficiency removal of nickel ions increased from 64% to 82 % at contact time 5 to 15 min respectively indicated % removal increase with time due to diffusion through the fluid film in CA\SSA membrane and diffusion through the pores to internal adsorption sites, then after 20 min , the adsorption decreases to 81% due to slow pore diffusion of the solute ion into the bulk of membrane .

Nickel ion adsorption isotherm

Only M5 membrane was examined for isotherm study. At different concentration 5, 10, 15, 20 and 25 mg/L .The constants and correlation coefficients of the Langmuir Freundlich and Temkin isotherms are presented in Table (1) .The fitting using the Langmuir model is shown in figure (10) represent R^2 value of 0.973, q_{max} indicate the maximum amount of nickel ions that could be adsorbed was calculated to be 20.8 mg\g, R_L represent a favorable adsorption at 0.36, 0.10 at 5, 25 mg\l respectively. Freundlich model in figure (11) shown the R^2 value of 0.985, the values of 1\n and Kf in the freundlich model were calculated to be 0.594 and 5.12 respectively. This indicates that although the binding energy on membrane surface shows a heterogeneous structure. R^2 value of Temkin isotherm 0.983, B, represent of heat sorption 4.313 J/mol, as shown in figure (12).

The results of the experimental model are best described by Freundlich model where the values of the regression coefficients (R^2) are higher than the ones obtained in the case of Langmuir and temkin model as shown table (1). Therefore, the CA\SSA membrane is a potential and active adsorptive membrane for removal of nickel ions from wastewater.

CONCLUSION

The present research that modified of cellulose membrane with sewage sludge ash can be concluded as:

- Asymmetric membranes from CA\SSA can be prepared by dry-wet phase inversion.
- FTIR analyses represent the functional group of blend membranes of CA\SSA which appear the bonded –OH groups, SO_3 stretching, C-O stretching, secondary amine groups and CN stretching were the main functional groups of adsorption of nickel ions.
- The scanning electron microscope (SEM) of CA\SSA it can be seen that the surface morphologies of M5 can be observed a dense skin layer supported by spongy porous related to SSA added in blending membranes.
- The best fit of Frendlich model rather than Langmuir and temkin models were obtained for adsorption of nickel ions using CA\SSA membrane with correlation coefficient, R^2=0.985, 0.973 and 0.983 respectively, the maximum monolayer coverage capacity ($_{Qmax}$) from Langmuir isotherm model was determined to be 20.8 mg/g at 25±2ºC and 1\n 0.594 from Freundlich isotherm indicate that the adsorption of Ni ions using CA\SSA at m5 is favorable.
- This research proved manufacture of an effective adsorptive membranes with high efficiency, good selectivity, low energy requirement and large permeate flux.

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