Synthesis of polyelectrolyte complex (PEC) membrane chitosan-polystyrene sulfonate (PSS) from styrofoam waste as adsorbents of Cd(II) and Pb(II) ions

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Abstract. The adsorption of Cd(II) ions and Pb(II) ions has been studied using a chitosan polystyrene sulfonate (PSS) from styrofoam waste polyelectrolyte complex (PEC) membrane adsorbent. Chitosan-PSS PEC membrane was characterized by FTIR and SEM. The adsorption experiment was performed under various conditions such as weight ration of PSS and chitosan as adsorbent, pH, contact time, and different initial Cd(II) and Pb(II) concentrations. The data showed that the optimum ratio of PSS and chitosan was 60:40 for Cd(II) and Pb(II) ions. The optimum pH value was found to be 4 for Cd(II) ions and 5 for Pb(II) ions. The pseudo-first-order and pseudo-second-order kinetic models were used to describe the kinetic data of Cd(II) and Pb(II) ions. The data fitted well to the pseudo-second order kinetic model. The experimental data of adsorption Cd(II) and Pb(II) ions also were fitted to Freundlich isotherms.

Keywords: styrofoam, chitosan, PEC, Pb(II), Cd(II)

1. Introduction

Contamination of water sources with heavy metal ions is a more serious global concern, because industrial activity is increasing rapidly throughout the world. Cd(II) and Pb(II) are widely used in many industrial activities and are of the most toxic heavy metal ions found in water sources. The maximum allowable levels of Cd(II) and Pb(II) content in drinking water are 0.005 mg L\textsuperscript{-1} and 0.01 mg L\textsuperscript{-1}, respectively\textsuperscript{1,2}. Microorganisms will be absorbed and accumulate toxic elements that are discharged in the effluents. Finally, toxic elements will be transferred to humans through the food chain and accumulate. Cadmium can cause kidney dysfunction, liver damage, bone degeneration, pulmonary insufficiency, and hypertension in humans. Lead is dangerous for children and can cause mental retardation\textsuperscript{3,4}.

Precipitation, ion exchange, and adsorption, etc. are several methods to treat the metal-contaminated effluent, but the selection of the wastewater treatment methods is based on the concentration of waste and the cost of treatment\textsuperscript{5}. However, these techniques like precipitation, ion exchange are associated with problems such as excessive time requirements, high costs and high energy consumption\textsuperscript{6}, and so one of the more popular methods for the removal of heavy metals from the wastewater is adsorption. Adsorption mechanisms such as surface complexion and ion exchange are considered as effective methods in water treatment\textsuperscript{7}. 
Polystyrene or styrofoam (commercial trade name of polystyrene) is hard plastic, structurally; it's a long hydrocarbon chain with a phenyl group attached to every carbon atom and an inexpensive plastic [8]. Polystyrene is produced by polymerizing free radical vinyl from styrene. Most of the polystyrene production is packaged (glass, plates, bowls, trays, etc.) [9]. Asia is the overall leader in the production and consumption of polystyrene, with 53% of total world production and 47% of the total consumption of polystyrene in 2010. North America and Western Europe follow distantly at about 17–19% of the total production and consumption. Polystyrene is non-biodegradable, implying that it persists in the environment and accumulates in landfills thereby reducing the capacity of landfills [10]. Unfortunately, only a few amounts of polystyrene waste is recycled [9]. Therefore, it is desirable to develop an effective method that uses waste materials. There is a need to move towards recycling for environmental, as well as economic reasons [10].

The styrofoam waste, as well as the existence of heavy metals in the environment, is also a big concern a few years back. Recycling of polystyrene (PS) waste by chemical treatment is very important for the environment [11]. The global environmental pollution caused by styrofoam waste material may be resolved and minimized by possible chemical treatment to convert it into useful derivatives [3]. Polystyrene (PS) as a thermoplastic material can be used as a material for making polyelectrolyte complex (PEC) membranes [12] by sulfonation reaction because there are sulfonate groups banded to the benzene ring [8,13–15]. There are much research has been studied about the removal of heavy metal ions with styrofoam waste modification. Ruziwa et al. (2015) have been studied the removal of Zn(II) and Pb(II) ions from aqueous solution using sulfonated polystyrene waste. Mahmoud et al., using sulfonation and nitration to study the removal of Cd(II) and Pb(II) ions. However, a high degree of sulfonation in the aromatic site can lead to excessive development in the water and cause poor physical properties such that the membrane properties need to be increased. Therefore, modification of PSS can be made by forming a complex of polyelectrolytes or PEC. PEC is a complex formed between the contents of opposite particles. In this study, modification is done to form PEC membranes by using chitosan. Chitosan is a cationic polysaccharide having an amine group. The presence of an amine group of chitosan may provide a positive charge which can bind ionically with a sulfonate group of negatively charged PSS. Estimated interactions that occur are illustrated in Figure. 1.

![Figure 1. Estimated interaction of Cd(II) and Pb(II) ions with chitosan-PSS adsorbent](image-url)
2. Materials and Methods

2.1. Materials

The materials used in this study were Styrofoam waste, aquadest, ethyl alcohol, 1 % acetic acid), 95-98 % sulfuric acid, chloroform, sodium hydroxide, dichloromethane, methanol, acetone, stock standard solution of 1000 mg L\(^{-1}\) of cadmium(II) and lead (II) (All reagents were purchased from Merck). All the chemicals used were of analytical reagent (AR) grade.

2.2. Preparation of chitosan-PSS PEC membrane

20 grams of Styrofoam waste were dried under vacuum at 100 °C, then dissolved in 500 mL of chloroform and added 15 % sulfuric acid at room temperature and stirred for 3 hours. The sulfonation process of the polymer solution is discontinued and the mixed solution is poured into 500 mL of ice water while continuous stirring is carried out. Then the polystyrene sulphonate or PSS produced is filtered and neutralized several times until the pH is neutral. PSS was dried under vacuum for 10 h at 55 °C.

The prepared membrane consisted of three variations of composition ratio, ie, Chitosan-PSS ratio 40:60, 50:50 and 60:40 respectively with a total neutral weight of 0.6 % (w/v). The Chitosan-PSS membrane ratio was prepared with 0.18; 0.15 and 0.12 g of PSS into 60, 50, and 40 mL DCM (30): Methanol (70) and dissolved 0.12: 0.15 and 0.18 g of chitosan into 40, 50, and 60 mL 1 % acetic acid, then the two solutions were stirred for approximately 3 hours. Up to 10 ml of homogeneous stirred membrane solution was printed in a petri dish and then dried under vacuum at 50 to 60 °C for 24 hours. The dried membrane is soaked in a 0.1 M NaOH solution until the membrane is lifted, after which it is washed using aquadest to neutral pH. The membrane is then stored in a desiccator. The results of membrane synthesis of Chitosan-PSS membrane were characterized by using infrared spectrophotometer (Shidamidzu FT-IR 8201PC), SEM (JEOL JSM- 6510LA), also Chitosan-PSS membrane in the ratio of 60:40, 50:50, and 40:60 was tested for water adsorption by weighing dry film to get dry weight (W\(_{\text{dry}}\)), then the film was immersed in distilled water for 5, 10, 15, 30, 60, 90 and 120 minutes. Wet film is weighed to find out the wet weight (W\(_{\text{wet}}\)).

2.3. Adsorption Study

Firstly, the variation of contact time, initial pH, chitosan-PSS dose, initial heavy metal concentration under the aspect of adsorption kinetics were carried out for this adsorption experiment. Adsorption experiments were carried out in 100 mL conical flasks and the total volume of the solution was kept at 20 mL. Initial pH ranged from 3-7 of the solution was controlled by adding 0.1 M NaOH and HCl solutions. For the kinetic studies, the mixture was shaken at room temperature for a time interval ranging from 5, 10, 15, 30, 45, 60, 90, 120, 150, and 180 minutes. At a predetermined time, the flasks were removed from the shaker. The remaining metal ion concentration was measured by using AAS technique (AAS ContrAA 300 Analtik Jena). For equilibrium isotherm studies, 20 mL of heavy metal solutions were shaken for contact time optimum and pH optimum at room temperature and the concentration of heavy metal was 10-100 mg L\(^{-1}\).

3. Results and Discussions

3.1. Preparation of chitosan-PSS PEC membrane

Pilot plant scale synthesis was carried out after the small-scale laboratory bench synthesis. The preparation chitosan-PSS membranes are done by the first synthesis of polystyrene sulfonate or PSS. PSS is prepared by mixing sulfonation agent of sulphuric acid and chloroform organic solvent. The use of sulphuric acid as sulfonation agent is expected to be a more uniform and homogeneous distribution of sulphonate in polystyrene while chloroform as a solvent because insolvent sulfonation reaction should not react with polymer nor sulfonation agent and chloroform also have good solubility.
for polystyrene. The resulting PSS is a white solid granule. The reaction of PSS formation through sulfonation reaction with a sulfonation agent can be seen in Figure 2.

Figure 2. The reaction of PSS formation [3].

Membranes have been made from converting Styrofoam waste through sulfonation methods to form polystyrene sulfonate or PSS (polyanionic) having a -SO$_3$H group and then binding electrostatically with the -NH$_2$ group on chitosan (polycationic). PSS was reacted with chitosan to form a polyelectrolyte complex or PEC membrane. The synthesis of chitosan-PSS PEC membrane with a neutral weight of 0.6 % (w/v) was made in several variations, such 60:40, 40:60 and 50:50. The result of membrane adsorption test with a variation of chitosan and PSS weight composition can be seen in Figure 3 that membrane can adsorb Cd(II) and Pb(II) ions in the best compression ratio of Chitosan-PSS that is 60:40.

Figure 3. The effect of the weight ratio of chitosan and PSS.

The membrane was characterized to know the ability of water adsorption and stability at medium pH. The results of the water adsorption test are shown in Figure 4 that the water adsorption test results indicate the membrane with more chitosan compositions or the same composition has more water-absorbing capacity than the larger PSS composition. Test of membrane resistance in acid and base medium has also been done by varying pH, i.e. 2, 3, 4, 6, 8, 10, 11 and 12 and immersion time for 1-2 days. The results show that the membrane dissolves at pH 2 conditions for all membrane variations on the first and second day and on the second day the membranes 50:50 and 40:60 are damaged under pH 3 conditions. This can be due to the ability of chitosan that easily dissolves in a very acidic state.

The membrane was characterized by FT-IR to determine the functional groups present in the membrane. The identification of functional groups is influenced by the interaction of functional groups owned by PSS and chitosan. The chitosan- PSS PEC membrane used for FT-IR characterization is a membrane with a ratio of 60:40 because it has the largest value of adsorption capacity compared to the
others. Functional group analysis with FT-IR was also performed on the polystyrene (Styrofoam waste), PSS membrane, and chitosan membrane shown in Figure 4.

![Figure 4. Water adsorption test result for variations of the membrane.](image)

**Figure 4.** Water adsorption test result for variations of the membrane.

![Figure 5. FT-IR spectra of polystyrene (a), PSS membrane (b), chitosan-PSS membrane (c), and chitosan membrane (d).](image)

**Figure 5.** FT-IR polystyrene (a), PSS membrane (b), chitosan-PSS membrane (c), and chitosan membrane (d).

Based on the FTIR spectra of polystyrene in Figure 5, there are specific absorptions in the wavenumbers 1600, 1451, 2924 and 3024 cm⁻¹ appearing on the PSS spectra, each showing a C=C vibration in aromatics, Csp³-H and Csp²-H [3]. The PSS spectra have -SO₃ vibration at 1031 cm⁻¹ and 1180 cm⁻¹. The chitosan spectra showed a bending -NH vibration in the 1590 cm⁻¹ region and the vibration of C-O at 1648 cm⁻¹. Characterization of the chitosan- PSS PEC membrane spectra indicates the presence of PSS as well as chitosan absorption peaks. In the chitosan-PSS PEC membrane spectra, the absorption occurs at 1451 cm⁻¹ is the aromatic C=C vibration of the benzene ring in PSS, the shifting vibration -SO₃ at 1038 cm⁻¹, and in the region of 1061 cm⁻¹, there is a vibration of CO and vibration -NH at 1583 cm⁻¹ from chitosan. PSS membrane and chitosan-PSS PEC membrane also present absorption at 1126 cm⁻¹ indicating the presence of -C-S vibration from benzene ring and sulfonate group. The formation of bonding groups on the Chitosan-PSS PEC membrane of the
The constituent material shown in Figure 4 ensures that the membranes are successfully synthesized and can be used as adsorbents in the adsorption of Cd (II) and Pb (II) ions.

The chitosan-PSS PEC membrane was characterized by SEM to determine the morphology of the membrane surface and was also performed on the PSS membrane to see the presence of PSS membrane differences and that have been modified with chitosan to form Chitosan-PSS PEC membranes. The membrane characterization results with SEM are presented in Figure 6.

![Figure 6](image)

**Figure 6.** Morphology of PSS membrane (a), chitosan-PSS PEC membrane (b) with 5000x magnification.

Figure 5 shows the morphology of the PSS membrane without the addition of chitosan having a straight and non-porous surface shape and also shows that there is a difference in the surface of the chitosan-PSS PEC membrane when compared to the surface of the PSS membrane, that means by the addition of chitosan causing the membrane morphology of chitosan-PSS PEC membrane to change and has an irregular and slightly porous surface shape.

### 3.2. Adsorption Study of Cd(II) and Pb(II) Ions on Chitosan-PSS PEC Membrane

#### 3.2.1. Effect of pH

Protonation of metal binding sites is affected by pH so pH is an important process parameter in the adsorption of metal ions from aqueous solutions. It was found that Cd(II) and Pb(II) uptake by chitosan-PSS PEC membrane was a function of solution pH. Cd(II) and Pb(II) 25 mg/L ions solution was varied pH before interaction with 40:60 chitosan-PSS PEC membrane. The variations used are 3, 4, 5, 6, and 7. The adsorption capacity of Cd(II) and Pb(II) ions by the adsorbed chitosan-PSS membrane is shown in Figure 7.

Based on Figure 7 shows that the pH of the solution greatly affects the ability of the membrane adsorption in adsorbing Cd(II) and Pb(II) ions in the solution. Figure 6a shows that the adsorption capacity of Cd (II) metal ions increases from pH 3 to pH 4, then decreases to pH 7, so it is known that the chitosan-PSS membrane adsorbs the Cd(II) optimally at pH 4. Figure 6b also shows that the adsorption capacity of Pb(II) metal ions increases from pH 3 to pH 5 and then decreases to pH 7 so that chitosan-PSS PEC membrane adsorbs the optimum Pb(II) at pH 5. The difference between the optimum pH of these two metals is possible because of the differences in the two metal species in the solution with the effect of pH [16]).
3.2.2. Effect of contact time

The study of the effect of contact time was performed on 25 mg chitosan- PSS membrane with 25 mL of metal ion solution of Cd(II) 25 ppm and Pb(II) 25 ppm. The contact time variations used were 5, 10, 15, 30, 45, 60, 90, 120, 150, and 180 minutes. The Cd(II) or Pb(II) ions that have been trapped on the membrane will increase with increasing contact time and there are conditions at certain contact times where the saturated membrane means that it cannot adsorb metal ions Cd(II) or Pb(II) (optimum adsorption), the number of metal ions no longer increases significantly. The result of the contact time analysis obtained is shown in Figure 8.

Based on Figure 8, the optimum contact time of chitosan-PSS PEC in adsorbing Cd(II) ion is 90 min with adsorption capability of 0.0526 mmol g⁻¹ and for Pb(II) ion that is at minute 60 with adsorption ability 0.0702 mmol g⁻¹. The optimum contact time achieved in the adsorption of these two metal ions indicates the achievement of equilibrium in the interaction between metal ions with the active sites of the adsorbent, whereby at the time of reaching equilibrium the amount of the adsorbed metal ion will be equal to the number of dissolved metal ions.

![Figure 8](image-url)
3.2.3. Adsorption Kinetics

The adsorption kinetics is one of the factors in the adsorption process as it shows the rate of adsorbent adsorption on Cd(II) and Pb(II) ions. Information on the adsorption rate can be obtained from the rate constant of the reaction using data from the variation of contact time. The adsorption kinetics can be determined by comparing the linear correlation quadratic coefficients ($R^2$) of the adsorption kinetics equations of each kinetic model obtained. The value of $R^2$ closest to 1 becomes the reference for selecting the appropriate adsorption kinetics equation. The adsorption kinetics model tested is the first order & second-order kinetics model, the Lagergren (pseudo-first-order) kinetics model and the McKay & Ho (pseudo-second-order) kinetics model.

Table 1. The kinetic equation of metal ion adsorption of Cd(II) and Pb(II) by chitosan-PSS membrane.

| Reaction Order | Equation of kinetic model | $R^2$ Cd(II) | $R^2$ Pb(II) |
|----------------|---------------------------|--------------|--------------|
| Pseudo-1 order | $\ln (q_e - q_t) = \ln(q_e) - k_1 t$ | 0.2341 | 0.4867 |
| Pseudo-2 order | $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}$ | 0.9963 | 0.9975 |

Based on the result of this linear correlation value, it can be concluded that the adsorption kinetics of Cd(II) and Pb(II) on the chitosan-PSS PEC membrane follow the equation of McKay & Ho pseudo-2nd order kinetics. McKay & Ho's pseudo-second-order kinetics model illustrates two stages of the process, the fast and the slower second phase [17]. This statement is following this study, in Figure 7 it is seen that in the first few minutes of adsorption proceeds very quickly, then after reaching the optimum adsorption time, adsorption runs slow (tends to be constant).

3.2.4. Effect of Cd(II) and Pb(II) ions initial concentration

The study of the effect adsorbate concentrations was performed on 25 mg chitosan-PSS membrane with 25 mL of metal ion solution of Cd(II) 25 ppm and Pb(II) 25 ppm. The metal ions variations used were 10-100 mg L$^{-1}$. Figure 9 can be seen that the higher concentration of Cd(II) and Pb(II) ions, the more metal ions are adsorbed on the active site of the membrane. The result of adsorption generally shows that the increase of metal ion concentration will decrease the adsorption percentage of a metal ion at the optimum concentration.

The variation data of the effect of the initial concentration of Cd(II) and Pb(II) ion are also used for the determination of adsorption isotherm. Determination of adsorption isotherm was performed to determine the equilibrium relationship between chitosan-PSS PEC membrane as adsorbent and Cd(II) and Pb(II) ion as the adsorbate. The adsorption isotherm can describe how the adsorbate molecule interacts with the surface of the adsorbent. The determination of this adsorption isotherm is the Langmuir isotherm equation and Freundlich isotherm shown in Table 2.

Based on the result of this linear correlation value (Table 2), it can be concluded that the adsorption kinetics of Cd(II) and Pb(II) on the chitosan-PSS PEC membrane follow the Freundlich isotherm. The earliest relationship describing the adsorption process is Freundlich isotherm model. Adsorption on heterogeneous surfaces with the interaction between adsorbed molecules that apply to this model and the application of the Freundlich equation also suggests that sorption energy exponentially decreases on completion of the sorption centers of an adsorbent [18].
Figure 9. The effect of metal ion concentration on adsorption of Cd(II) (a) and Pb(II) ions.

Table 2. Parameter of adsorption isotherm.

| Ions Adsorption | Langmuir Isotherm | Freundlich Isotherm |
|------------------|-------------------|-------------------|
|                  | $q_{\text{max}}$ (mmol g$^{-1}$) | $K_L$ (L mmol$^{-1}$) | $R^2$ | $K_f$ | $N$ | $R^2$ |
| Cd(II)           | 0.69              | 1.613             | 0.8225 | 2.66  | 0.79 | 0.9688 |
| Pb(II)           | 0.39              | 3.979             | 0.7256 | 5.12  | 0.77 | 0.9947 |

4. Conclusions

Based on the results of research and discussion that has been done, it can be concluded as follows that chitosan-polystyrene sulfonate (PSS) PEC membranes can be used as Cd(II) and Pb(II) ion adsorbent with optimal condition of PSS: chitosan composition 60:40 (w/v). The chitosan-PSS PEC membranes were able to adsorb Cd(II) optimally at pH 4 for 90 min using an initial concentration of Cd(II) 40 ppm and were able to adsorption Pb(II) optimally at pH 5 for 60 min with using an initial concentration of Pb(II) 70 ppm. Chitosan-PSS PEC membranes as adsorbents of Cd (II) and Pb (II) ions follow the pseudo-second-order kinetics model and Freundlich isotherm model.

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