



# Polystyrene Sulfonate–Chitosan Membrane from Styrofoam Waste as The Adsorbent for Fe (III) Metals from Ex-Tin Mining

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## Abstract

Styrofoam waste is difficult to degrade and decompose because it is included in inorganic waste, so it will negatively impact human health and the environment. A good and efficient method for chemical modifications that can convert Styrofoam into an appropriate material is needed. The heavy metal content in pond water is considered not good if used as drinking water because the heavy metal content in some ponds exceeds the quality standard for clean water, which is not good for health. Therefore, this research utilized Styrofoam waste in the environment with chemical modification into a sulfonated polystyrene–chitosan polyelectrolyte complex membrane as a heavy metal adsorbent in wastewater from a former tin mine. The PSS–chitosan membrane composition of 10:10 shows better adsorption capabilities than that of 12:8 and 8:12 on standard Fe metal. The adsorption efficiency of the PSS–chitosan membrane on Fe metal in underground water was  $(39.1 \pm 3.1)\%$ .

## 1. Introduction

Styrofoam is currently one of the most widely used food packaging materials. Styrofoam is made from polystyrene, a naturally lightweight chemical that can be liquid or processed into solid foam (extruded polystyrene foam) [1]. Styrofoam waste is difficult to degrade and decompose because this waste is classified as polymer-type inorganic waste whose residues contain carcinogenic hazardous substances [2]. Methods of dealing with Styrofoam waste are mostly done physically through combustion and landfill, which is considered less effective because burning Styrofoam will produce CO<sub>2</sub> gas and toxic dioxin PAHs. Therefore, a recycling process is needed for Styrofoam waste, namely by converting Styrofoam waste into sulfonated polystyrene through a sulfonation process with a modification of the formation of complex polyelectrolyte (KPE) [3].

Chemical modification of Styrofoam waste in previous studies has been modified through sulfonation [4], modification as a supporting solid adsorbent silica gel to remove heavy metals in solution [5], and modification through nitration [6]. Modification by sulfonation is considered more effective because it can increase adsorption capacity, has good thermal stability, and the

sulfonate group acts as an ion exchange resin. Through such modifications, it produces a negatively charged surface, which can then be modified into a polyelectrolyte complex to enrich the active site and increase its adsorption ability from Styrofoam so that it can be applied as an adsorbent of heavy metal ions [3]. The process of forming complex polyelectrolytes requires the combination of a polycationic such as chitosan.

Chitosan is a biopolymer derived from chitin processing from the outer shell of crabs, shellfish, and shrimp. Chitosan is a cationic polyelectrolyte biopolymer with the potential for high adsorption ability, especially for metal ions [7]. Chitosan can bind metal ions, especially transition metals, through the formation of coordination bonds of hydroxy (–OH) and amine (NH<sub>2</sub>) groups, as well as the presence of amide groups (–NHCOCH<sub>3</sub>) in chitin which act as ligands when interacting with metals [8].

Methods for removing heavy metals have been widely developed, including electrolysis, membrane separation, reverse osmosis, precipitation chemistry, flocculation, ion exchange, and adsorption [9]. One widely used method is adsorption because it is the most effective, economical, and environmentally friendly [10].

The principle of adsorption is based on the interaction of metal ions with functional groups present on the surface of the adsorbent through the interaction of complex formation. Polystyrene sulfonate-chitosan membrane can adsorb Ni(II) and Cu(II) metals with an adsorption capacity of  $0.805 \text{ mmol g}^{-1}$  for Cu(II) metal and  $0.741 \text{ mmol g}^{-1}$  for Ni(II) metal [3]. Therefore, in this research, the polystyrene sulfonate-chitosan membrane was applied to adsorb Fe (III) metal in the water under the former tin mine due to the large amount of water under the abandoned tin mine in Bangka Belitung and the community used some for toilet washing baths. However, it also has many negative impacts, including containing heavy metal content, such as Fe, As, Cr, Cu, Pb, Zn, and Sn.

## 2. Experimental

### 2.1. Instruments and Materials

The tools used in this research included an atomic absorption spectrophotometer (AAS) (Thermo Fisher Scientific iCE 3000), scanning electron microscope (SEM) (Thermo Fisher Phenom ProX), and infrared spectrophotometer (PerkinElmer Frontier). The materials used in this research were Styrofoam waste, ethyl alcohol (Merck), distilled water, acetic acid 1% (Merck), chloroform (Merck), sulfuric acid 95-98% (Merck), sodium hydroxide (Merck), nitric acid 30% w/v (Merck), dichloromethane (Merck), disodium edetate (Sigma Aldrich), potassium nitrate (Merck), water samples from tin mine in Riding Panjang Village, Merawang District, Bangka Regency, Indonesia.

### 2.2. Styrofoam Waste Preparation

Styrofoam waste (20 g) was washed with distilled water, then mashed and dried in an oven at  $100^\circ\text{C}$  for 30 minutes. Furthermore, Styrofoam waste was dissolved in 467 mL chloroform (Merck), reacted with 276 mL of sulfuric acid 95-97%, and stirred for 3 hours. The mixture was transferred to cold water and continuously stirred until a white precipitate formed. The white powder obtained was filtered and neutralized, then dried at  $50^\circ\text{C}$  for 10 hours [2].

### 2.3. Synthesis of Polystyrene Sulfonate (PSS)-Chitosan Membrane

PSS-chitosan membranes were prepared using PSS: Chitosan with weight ratios of 8:12, 10:10, and 12:8. The PSS powder was dissolved in a mixture of dichloromethane and methanol (30:70) until a homogeneous solution was achieved. This solution was then mixed with a chitosan solution in 1% acetic acid, and the resulting mixture was stirred until homogeneity was attained. The homogeneous mixture was subsequently printed to a Petri dish and dried in an oven at  $55^\circ\text{C}$  for 24 hours. The formed membrane was then characterized using SEM, FTIR spectrophotometer, stability test in acid and base, and swelling test [2].

### 2.4. Optimization of pH

PSS-chitosan membranes with ratios of 8:12, 10:10, and 12:8 were added with 50 mL of 2 ppm standard solution of Fe. The solution was prepared with pH

variations of 3, 5, and 7. Then, the membrane was put into the solution and shaken with a shaker at room temperature for 30 minutes. The membrane was separated from the solution and dried. The solution was measured for absorbance using AAS [2].

### 2.5. Adsorption Kinetics

PSS-chitosan membranes with ratios of 8:12, 10:10, and 12:8 were added with 50 mL of 2 ppm standard solution of Fe. The membrane and solution were shaken with a shaker at room temperature with time variations of 15, 30, 45, 60, and 120 minutes. The membranes were separated from the solution and dried. The solution was measured for absorbance using AAS [2].

### 2.6. Application of PSS-Chitosan Membrane in Ex-Tin Mining

The adsorption process was carried out by inserting the best variation membrane into the water solution based on the results of pH testing and optimal time. Then, the membrane solution was shaken using a shaker at room temperature. After that, the membrane was separated from the solution and then dried. The solution was measured for absorbance using AAS [11].

### 2.7. Sequential Desorption Study

A standard solution of Fe metal with its optimum concentration was made. Then, each solution was reacted with a 20 mg membrane at the optimum pH and time. The adsorption residual membrane was then dried and reused for desorption study. The desorption study, aimed at investigating the disclosure mechanism, was conducted by interacting the residual adsorption membrane with aquablast, employing the exchange mechanism with  $\text{KNO}_3$  0.1 M, utilizing the hydrogen bond formation mechanism with  $\text{HNO}_3$   $5 \times 10^{-4}$  M, and applying the complex bond formation mechanism with  $\text{Na}_2\text{EDTA}$   $10^{-4}$  M. Subsequently, the solution was shaken at the optimum time of each metal. The solution was separated from the membrane, and the metal ion content was analyzed using AAS [2].

## 3. Results and Discussion

### 3.1. Styrofoam Waste Preparation

A chloroform and sulfuric acid mixture can react with Styrofoam and produce polystyrene sulfonate (PSS) (Figure 1), which can be used as an adsorbent for heavy metals in solution. After stirring, the solution changed color from yellow to blackish-brown. After that, the solution was neutralized with distilled water while stirring, and a white precipitate was formed. The white precipitate produced after neutralization formed white lumps. After drying at  $50^\circ\text{C}$  for 10 hours, 49.6 g of white powder was obtained.

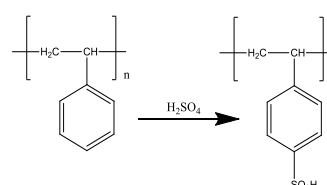


Figure 1. Polystyrene sulfonation reaction [2]

### 3.2. Synthesis of Polystyrene Sulfonate (PSS)-Chitosan Membrane

Chitosan was dissolved in a 1% acetic acid solution, leading to an acidification reaction that generated positively charged acetate ions. In this process, the NH<sub>2</sub> group of chitosan transformed into the positively charged NH<sub>3</sub><sup>+</sup> ion. Chitosan ions form hydrogen bonds with acetate ions, forming a more stable complex polyelectrolyte membrane (Figure 2). Then, the membrane was printed on a petri dish and dried at 55°C for 24 hours. The resulting PSS-chitosan membrane was in the form of white plates.

### 3.3. Functional Group Identification

The functional groups on PSS-Chitosan membranes were characterized using FTIR spectrophotometry, as a comparison analysis was performed on polystyrene, polystyrene sulfonate (PSS) membranes, PSS-Chitosan membranes, and chitosan. The FTIR spectra are presented in Figure 3.

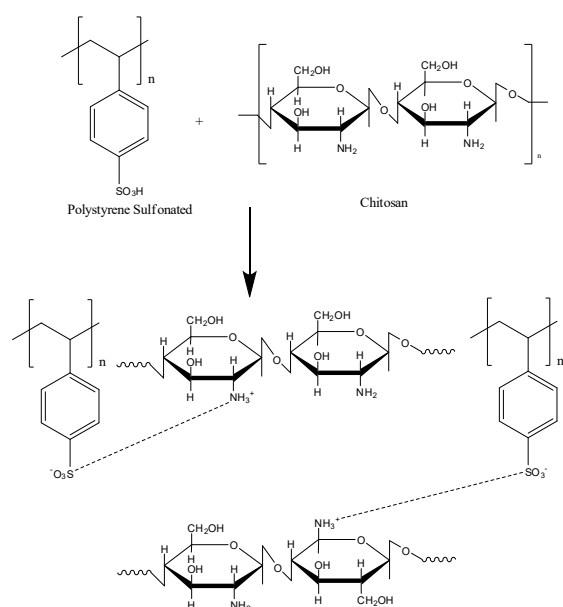


Figure 2. Reaction mechanism of polystyrene sulfonate and chitosan

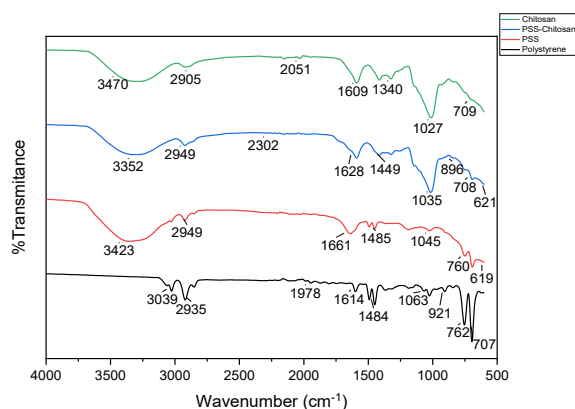


Figure 3. FTIR spectra of chitosan, PSS-chitosan membrane, PSS, and polystyrene

Figure 3 shows the results of the FTIR spectra of polystyrene found in the PSS spectra [3, 12]. Polystyrene absorption in region 1614 cm<sup>-1</sup> indicates an aromatic CH<sub>2</sub> extended vibration. The extended vibration C<sub>sp</sub><sup>2</sup>-H is shown at wavenumber 1484 cm<sup>-1</sup>, and the outstretched vibration C<sub>sp</sub><sup>3</sup>-H is shown at wavenumber 2935 cm<sup>-1</sup>. Chitosan absorption is also found in the PSS-chitosan and chitosan membrane spectra [3]. The absorption appears at wavenumbers 3470, 1610, 1340, and 1027 cm<sup>-1</sup>, respectively, indicating an elongation vibration of -OH, an outstretched vibration of C=O in the amide group (RHN-C=O), a symmetrical bending vibration of -CH in -CHOH, and an outstretched vibration of -CO in -COH, respectively. The characteristic absorption of PSS also appears in the PSS-chitosan membrane spectra. The absorption appears at wavenumbers 3423, 2949, 1485, and 1045 cm<sup>-1</sup>, indicating an aromatic -OH, -CH extended vibration, aromatic C=C bond vibration, and an asymmetric extended vibration O=S=O.

### 3.4. Surface Morphology of Chitosan Polystyrene Sulfonate Membrane

Figure 4 shows SEM images of PSS membranes before their transformation into a polyelectrolyte complex membrane and after completing the complexation process with an optimal composition ratio of 10:10. The pre-complexation PSS membranes exhibit relatively smoother surfaces. However, they display unevenness when observed at magnifications of 10,000 times or even 15,000 times. The PSS-chitosan polyelectrolyte complex membrane shows surface morphology characterized by hollowness and unevenness, contributing to forming a porous and uniform structure. Polystyrene sulfonate containing sulfonate groups interacts with -NH<sub>2</sub> from chitosan to form a porous structure, thus enhancing the membrane's capacity for water absorption and material adsorption.

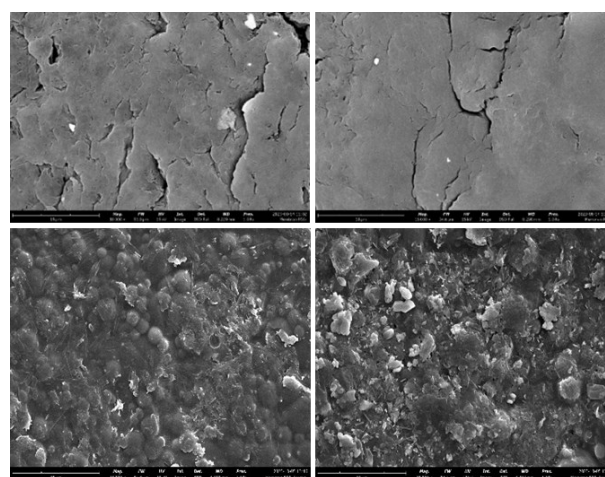


Figure 4. SEM images of a) PSS membrane with magnification of 10,000×, b) PSS membrane with magnification of 15,000×, c) PSS-Chitosan membrane 10:10 with magnification of 10,000×, d) PSS-Chitosan membrane 10:10 with magnification of 15,000×

**Table 1.** Stability test in acids and bases

Membrane variation	Acidic	Neutral	Alkaline
PSS: chitosan (12:8)	Stable	Stable	Decomposed
PSS: chitosan (10:10)	Stable	Stable	Decomposed
PSS: chitosan (8:12)	Stable	Stable	Decomposed

**3.5. Stability Test in Acids and Bases**

Based on Table 1, the stability test of PSS–chitosan membranes against acids and bases shows that the membrane was relatively stable at pH 3–8. Stability tests were performed at pH 3 (acidic), 7 (neutral), and 12 (alkaline) for 24 hours. The membrane becomes unstable at an alkaline pH of  $\geq 10$  due to the ability of the -OH molecule to form hydrogen bonds with amine groups on chitosan, thus weakening the bond between PSS and chitosan and causing the membrane to be unstable [4].

**3.6. Water Absorption Test (Swelling)**

Based on Table 2, the membrane with an 8:12 ratio and the highest chitosan content exhibited the greatest expansion compared to the other variations. This is because chitosan is more hydrophilic than polystyrene sulfonate (PSS). Consequently, the inclusion of chitosan in the PSS–chitosan membrane enhances the membrane’s water absorption capability. When contact with the liquid, there was a progression resulting from the thermodynamic interaction between the polymer chain and the liquid, along with the attraction induced by the crosslinking effect on the polymer chain in water [13].

**3.7. Optimization of pH**

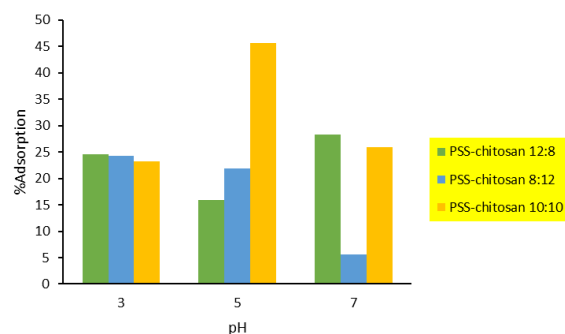
From the conducted tests on different compositions of PSS–chitosan membranes, it was evident that membranes with PSS: chitosan ratio (10:10) showed the highest adsorption efficiency results compared to other variations, namely at pH 5 with an efficiency of 45% (Figure 5). Membranes with a composition of 10:10 showed a higher percentage of adsorption than other membranes at pH 5 due to a more stable membrane structure due to the composition of PSS and chitosan in equal amounts. The increased adsorption efficiency value at pH 5 is due to the neutralization of the charge on the adsorbent surface. At pH conditions  $<5$ ,  $H^+$  ions in solution are abundant, thus blocking the interaction of Fe with the active side on the membrane surface. In contrast, at  $pH >5$ , it causes the membrane to become unstable, and the membrane can decompose.

**3.8. Adsorption Kinetics**

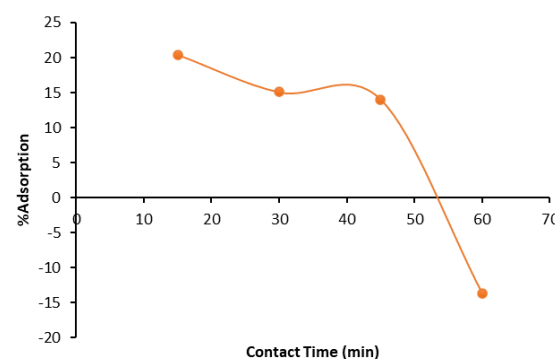
This test was conducted on membranes with a composition of 10:10 due to their highest adsorption efficiency and optimal pH of 5. The effect of contact time on the adsorption of standard Fe solutions on PSS–

chitosan membranes is shown in Figure 6. Based on Figure 6, the highest adsorption efficiency of 20.40% was achieved within 15 minutes.

Rapid adsorption during the early stages can be attributed to the abundant active sites on the surface of the adsorbent. However, these active sites become depleted, leading to saturation as time progresses [14]. The adsorption efficiency value tends to decrease with increasing contact time. The standard adsorption process of Fe metal on PSS–chitosan membranes follows a pseudo–first–order kinetic model (Lagergren model) with a coefficient of determination ( $R^2$ ) value of 0.822. The rate constant value ( $k$ ) obtained is  $-0.0617$  minutes, and the adsorption capacity is 0.725 mg/g (Table 3). The pseudo–first–order adsorption kinetics model shows that adsorption kinetics are influenced by one side of the active site, the amine group ( $-NH_2$ ).



**Figure 5.** Graph of the effect of pH variations on Fe metal adsorption efficiency



**Figure 6.** Graph of variation in the effect of time on the adsorption efficiency of Fe metal

**Table 2.** Swelling test

Membrane variation	Swelling percentage (%)
PSS: chitosan (12:8)	115.40
PSS: chitosan (10:10)	185.01
PSS: chitosan (8:12)	255.12

**Table 3.** Kinetic parameters of Fe metal ion adsorption on PSS-chitosan membrane

Metal ion	Lagergren Model			McKay and Ho Model		
	$k_1$ ( $\text{min}^{-1}$ )	$q_e$ ( $\text{mg.g}^{-1}$ )	$R^2$	$k_2$ ( $\text{mg.g}^{-1}.\text{min}^{-1}$ )	$q_e$ ( $\text{mg.g}^{-1}$ )	$R^2$
Fe	-0.0617	0.725	0.822	0.046	-0.513	0.297

**3.9. Application of PSS-Chitosan Membrane in Ex-Tin Mining**

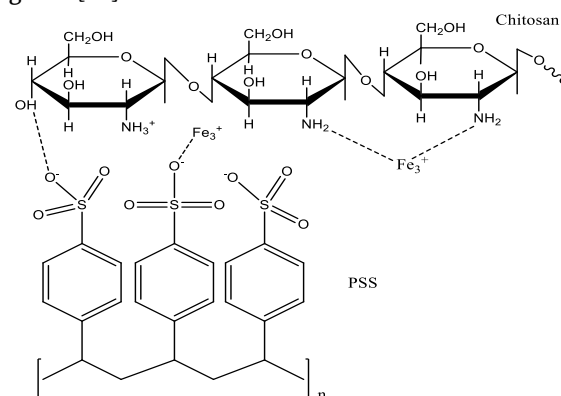
The PSS-chitosan membrane was applied to the water with three repetitions to determine the efficiency of Fe metal adsorption in water from ex-tin mining. The water sample was first filtered using filter paper to remove impurities or sediment. Based on Table 4, the average adsorption efficiency of Fe metal in water from ex-tin mining adsorbed by the PSS-chitosan membrane is  $(39.1 \pm 3.1)\%$ . According to the Regulation of the Minister of Health of the Republic of Indonesia, Number 32 of 2017 concerning environmental health quality standards and water health requirements for sanitary hygiene purposes, swimming pools, solus per aqua, and public baths states that the maximum iron content in clean water is 1 mg/L [15].

The water test results demonstrate the PSS-chitosan membrane’s capability to adsorb Fe metal. Specifically, the initial concentration of Fe metal in the water, which was 1.28 mg/L, decreased to 0.7-0.8 mg/L after the adsorption process. This reduction indicates that the post-adsorption water now complies with clean water quality standards. Fe cations interact with sulfonate anions in polymer membranes or can be trapped in membranes that contain many ionic groups from polystyrene sulfonate and chitosan membrane composites. The high degree of sulfonation in polystyrene can increase interactions with Fe cations. However, if PSS has a high degree of sulfonation, it can affect the physical properties of the membrane, which is easily damaged by water. Therefore, the right composition between the degree of sulfonation and chitosan is challenging for optimizing metal ion adsorption (Figure 7).

**3.10. Sequential Desorption Study**

Based on Table 5, the largest percentage of desorption was found in the  $\text{HNO}_3$  solution, followed by  $\text{Na}_2\text{DTA}$ ,  $\text{KNO}_3$ , and aquabidest. Metal ions adsorbed through the trapping mechanism were desorbed by aquabidest, ion exchange mechanisms using  $\text{KNO}_3$ , hydrogen bond formation mechanisms with  $\text{HNO}_3$ , and complex formation mechanisms with  $\text{Na}_2\text{EDTA}$ . According to the desorption results with the sequential desorption method, the chemical adsorption mechanism was dominant over the physical mechanism (trapping).

The result of desorption in the mechanism of hydrogen bond formation is 2.88%. The interaction between metal ions and the  $-\text{NH}_2$  group involves water molecules to form hydrogen bonds. Due to the use of electron pairs, hydrogen bond formation is more covalent than the ion exchange mechanism. A percentage of 1.27% was obtained in the complex formation mechanism. A direct bond occurs between Fe metal ions and  $\text{NH}_2$  groups so that metal ions act as central ions and  $\text{NH}_2$  as ligands [16].



**Figure 7.** Interaction of sulfonate ions with metal ions

**Table 4.** Results of PSS-chitosan membrane application in water from ex-tin mining

Treatment	$C_{\text{initial}}$ (ppm)	Absorbance	$C_{\text{final}}$ (ppm)	Efficiency (%)	Average $\pm$ SD
1	1.28	0.0569	0.8164	36.2	
2	1.28	0.0514	0.7364	42.4	$(39.1 \pm 3.1)\%$
3	1.28	0.0547	0.7844	38.7	

**Table 5.** Percentage of Fe metal desorption based on the used desorption solution

Metal desorption	Solvent	$C_a$ (mg/L)	$C_{\text{desorp}}$ (mg/L)	%desorption
Fe (2 ppm)	Aquabidest	20.51	0.062	0.30
	$\text{KNO}_3$ 0.1 M	19.16	0.006	0.33
	$\text{HNO}_3$ $5 \times 10^{-3}$ M	15.08	0.435	2.88
	$\text{Na}_2\text{EDTA}$ $1 \times 10^{-3}$ M	18.49	0.236	1.27

#### 4. Conclusion

Based on the results obtained, the PSS–chitosan membrane with a composition of 10:10 exhibits better adsorption ability in Fe standard metals compared to compositions of 12:8 and 8:12. The optimal adsorption efficiency of Fe standard metal on the PSS–chitosan membrane with a composition of 10:10 occurs at pH 5, with an optimum time of 15 minutes. The adsorption kinetic follows the pseudo-first-order kinetic model, specifically the Lagergren model, exhibiting an adsorption capacity of 0.725 mg/g. Furthermore, the adsorption efficiency of the PSS–chitosan membrane for Fe metal in water from ex-tin mining is (39.1 ± 3.1)%.

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