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Removal of Zn(II) and Cr(VI) ions from aqueous medium by anionic plant-based surfactant impregnated activated carbon

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ABSTRACT

Herein, anionic plant-based surfactant (sodium lauryl sulfoacetate, SLSA) was introduced for impregnation onto activated carbon (AC). This study was intended to assess its use as a potential adsorbent for removing Zn(II) and Cr(VI) ions from aqueous medium. The virgin and the impregnated AC were characterised using Brunauer–Emmett–Teller analysis and Fourier-transform infrared spectroscopy. Meanwhile, their performances to remove each metal ion were measured through batch adsorption experiments over a range of SLSA concentrations (10–175 mg/L) in 6 h contact time. The results revealed that Zn(II) ion removal was gradually increased as the SLSA concentration increased. The maximum removal was obtained at SLSA concentration of 75 mg/L (SIAC-75) with 31.66% of Zn(II) ion removal. However, an opposite trend of Cr(VI) ion removal was observed as the SLSA concentration increased. It is proposed that electrostatic interaction mechanism may occur during the adsorption process. The introduction of sulfonate group (OSO_3^-) via SLSA impregnation imparted a negatively charged surface on AC, thus resulting in an electrostatic attraction between Zn(II) ion. However, as Cr(VI) ion may dissociate into chromate (CrO_4^{2-}) ion, an electrostatic repulsion would occur. Therefore, in this study, the modified adsorbent prepared would be potentially preferred to remove Zn(II) ion.

Keywords: Activated carbon; Adsorption; Metal ions; Plant-based surfactant; Surface modification

1. Introduction

The importance of water as an essential source of life is seemingly on the verge of unsustainability. The United Nations (UN) annual report revealed that around 3.6 billion people worldwide experienced water scarcity in 2018, and this population is expected to grow to nearly 6 billion by 2050 [1]. This has been driven by the increasing demand for clean water, limiting water resources, and rising water pollution [2]. Moreover, the exponential growth of population and rapid modernisation have also escalated water pollution, which consequently threatens the environment and human health due to the increase of anthropogenic activities [3,4].

In general, various industrial and domestic contaminants are released into water bodies. However, the release of untreated industrial contaminants is regarded as the most significant contributor to water pollution [5]. This includes the release of pesticides, synthetic dyes, radionuclides, heavy

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metals, pharmaceuticals, etc. [5,6], which could lead to both toxicological and esthetical concerns. Among these various contaminants, the release of heavy metals into water bodies has become a prominent concern due to its severe environmental issues [7]. Its persistency in nature, high toxicity, and non-biodegradability had enabled them to accumulate in living organisms [8,9], thereby leading to various diseases and adverse health issues [10].

In particular, chromium is one of the most noxious heavy metal contaminants that intensely constitute health hazards. Its oxidation state of hexavalent chromium Cr(VI) ion is usually regarded as a highly hazardous toxic element due to its solubility, toxicity, and mobility in nature [11]. The pollution of Cr(VI) ion in the environment can cause serious illness to humans and other living organisms. For instance, Cr(VI) ion poisoning could cause dizziness, stomach ulcers, headache, carcinogenic, mutagenic, and teratogenic issues [12,13]. Apart from chromium, zinc contamination is also harmful to the environment. Its ionic form of Zn(II) ion is usually associated with adverse health problems. Long term exposures to Zn(II) ion contaminants could often lead to skin irritations, stomach cramps, vomiting, and anaemia [14]. In concern with their adverse implication towards the living organisms, treating metal-contaminated effluent prior to being discharged into water bodies is thus prudent.

The most commonly used treatment methods for the removal of metal ions from aqueous medium are chemical precipitation, chemical oxidation, reduction, adsorption, ion exchange, reverse osmosis, ultra-filtration and electrochemical removal [8,10,14,15]. Among these various approaches, adsorption process is the most convenient technique for wastewater decontamination. This is due to its simplicity, wide adaptability, versatility, inexpensive and easily operation [16,17].

Typically, activated carbon (AC) is the most widely used adsorbent for wastewater treatment. The advantages of AC such as having abundant specific surface area and exceptionally high porosity make them dominant in various applications, including the removal of organic chemicals and metal ions from wastewater [14,18]. However, the removal of metal ions through adsorption using commercial AC is less efficient because the functional groups only cover a small portion of the carbon surface [19]. Therefore, surface modification of AC is possible to enhance the surface functional groups and improve its adsorption capacity towards metal ions [18,19].

As for this, the impregnation technique using chemical or surfactant can be employed in modifying the surface of AC. Over the past few years, surface modification of AC via impregnation technique has attracted increasing attention and has been considered for applications such as removal of heavy metals, retrieval of coloured matters and removal of radionuclides from industrial wastewater. These studies have been explored in literature [14,18–23]. However, most of the studies focused on utilising chemical or chemical surfactant for impregnation on AC which is not environmentally friendly since the chemical may be desorbed and contaminated into the water bodies during the adsorption process. This may create adverse effects on aquatic life and human health, such as inhibiting natural aquatic activity, dermatitis illness, respiratory and eye irritations, etc. [24]. Thus, it is high time to replace the chemical surfactant with a plant-based surfactant for surface-modification of AC.

To the best of the authors' knowledge, little work has been carried out on the impregnation of AC using plant-based anionic surfactant for the removal of metal ions. Therefore, a substantial study on the adsorption of metal ions using plant-based anionic surfactant impregnated AC is essential to understand its relative performance in ensuring the technology is safe and environmentally benign. In this study, plant-based anionic surfactant, sodium lauryl sulfoacetate (SLSA), derived from palm and coconut oils will be used as an impregnating agent to modify the activated carbon. The anionic plant-based surfactant is expected to provide additional adsorption sites for the metal cation. This can be attributed to its anionic head groups, which can attract positively charged metal ions. Thus, it may enhance the adsorption process. As it is derived from the plant-based origin, a source of cheaper and safe anionic surfactant would then provide an additional advantage.

The main objective of this research is to study the removal of Zn(II) and Cr(VI) ions from aqueous solution via adsorption process using anionic plant-based surfactant impregnated activated carbon (SIAC). An anionic plant-based surfactant, namely SLSA, was utilised for impregnation on AC, and its effectiveness as a potential adsorbent was assessed.

2. Methodology

2.1. Materials

A coal-based commercial granular activated carbon (AC) was obtained from Soon Ngai Engineering (M) Sdn Bhd. The anionic plant-based surfactant, sodium lauryl sulfoacetate (SLSA, $C_{14}H_{27}NaO_5S$, 65% purity), was obtained from Personal Formula Resources (M) Sdn Bhd. The analytical reagent grade zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, 99.9% purity) and potassium dichromate ($K_2Cr_2O_7$, 99.8% purity) were obtained from Sigma-Aldrich (M) Sdn Bhd. All the materials used in this study were obtained from local commercial sources. The chemical structure of SLSA is presented in Fig. 1.

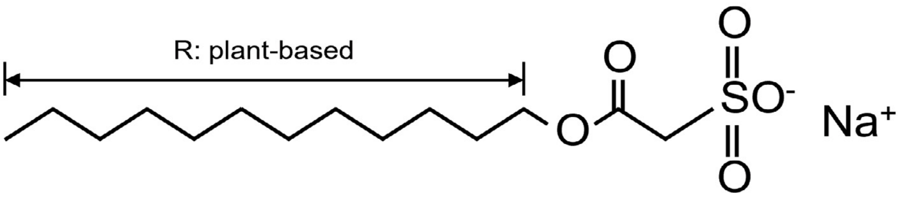

Fig. 1. Chemical configuration of SLSA.

2.2. Surface modification of AC

The AC modification process was carried out using isolate impregnation technique as reported in the previous literature [25]. Briefly, the concentration of SLSA was varied from 10 to 175 mg/L. Then, 2 g of as-received AC (virgin AC) was weighed and added into conical flasks containing 200 mL of SLSA at different concentrations. The mixtures were then placed in an incubator shaker (Model: Infors HT Ecotron) and agitated overnight at 60°C and 130 rpm. Afterwards, the resulting mixtures were filtered without rinsing and were dried in a universal oven (Model: UFE500) for 24 h at 60°C. The preparation condition of the samples was summarised in Table 1, and the modified adsorbents were designated as SIACs. The schematic diagram of the synthesis process is also presented in Fig. 2.

2.3. Characterisation of AC

Fourier-transform infrared (FTIR) spectrometer (Model: PerkinElmer Spectrum One) was used in this study to detect the presence of each functional group on SLSA and SIAC-10 over the wavenumber range of 4,000–500 cm^{-1}. For the textural properties analysis of the virgin AC and SIACs, Brunauer–Emmett–Teller (BET) surface area analyser (Model: Micromeritics 3Flex) was used to determine their specific surface area and pore diameter from the nitrogen gas adsorption–desorption isotherm at 77.4 K.

Table 1
Summary of SIACs preparation in 200 mL of SLSA at different concentrations

Samples	Preparation condition
Virgin AC	As-received AC (without modification with SLSA)
SIAC-10	2 g of virgin AC + 10 mg/L of SLSA
SIAC-25	2 g of virgin AC + 25 mg/L of SLSA
SIAC-50	2 g of virgin AC + 50 mg/L of SLSA
SIAC-75	2 g of virgin AC + 75 mg/L of SLSA
SIAC-100	2 g of virgin AC + 100 mg/L of SLSA
SIAC-125	2 g of virgin AC + 125 mg/L of SLSA
SIAC-150	2 g of virgin AC + 150 mg/L of SLSA
SIAC-175	2 g of virgin AC + 175 mg/L of SLSA

2.4. Metal adsorption experiments

In this study, Zn(II) and Cr(VI) ion solutions were firstly prepared by dissolving a required amount of their respective salts in distilled water. The initial concentrations of both metal ion solutions were kept constant at 150 mg/L. After that, batch adsorption experiments were conducted by adding 2 g of the adsorbents into conical flasks containing 100 mL of Zn(II) or Cr(VI) ion solution. The mixtures were then shaken at 130 rpm for 6 h in an incubator shaker (Model: Infors HT Ecotron) under a constant heat supply of 30°C.

2.5. Analysis of metal ions removal

Flame atomic adsorption spectrophotometer (AAS, Model: Hitachi Z2000) was used to determine the final concentration of Zn(II) and Cr(VI) ions in the treated solution at the wavelength of 213.9 and 359.3 nm, respectively. The removal efficiency (R, %) of each metal ion was then calculated using Eq. (1):

$$R = \left(\frac{C_0 - C_f}{C_0}\right) \times 100\% \tag{1}$$

where C_0 is initial concentration of metal ion (mg/L) and C_f is final concentration of metal ion (mg/L).

3. Results and discussion

3.1. FTIR analysis

Fig. 3 shows the comparison of FTIR spectra between SLSA and SIAC-10. As can be seen, SLSA exhibited several functional groups at varying amounts of vibration bands. The broad vibrating band with a maximum peak of 3,459 cm^{-1} is commonly assigned to the O–H stretching vibration, which indicates the presence of hydroxyl functional

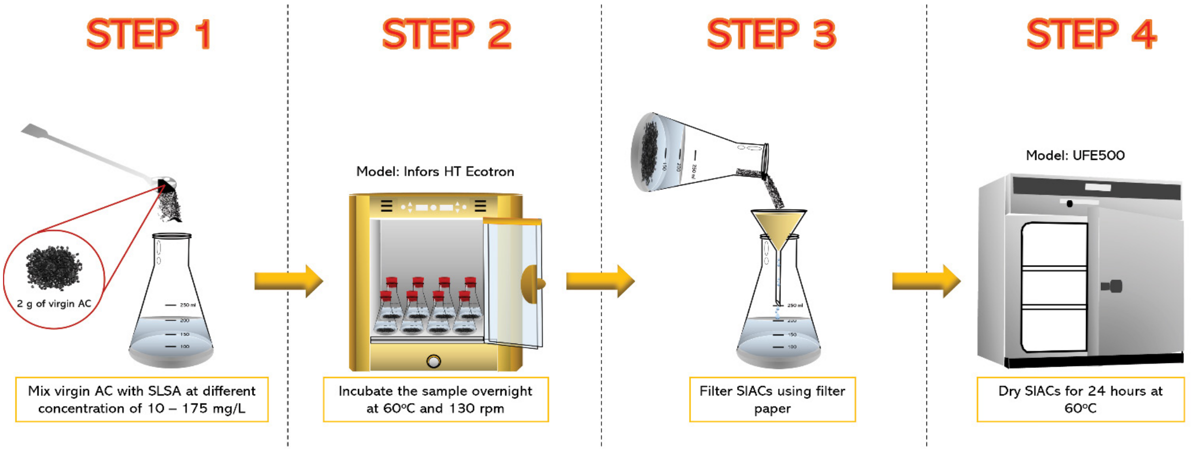

Fig. 2. Synthesis process of SIACs.

groups [20]. The higher intensity peaks in the range of 2,851–2,955 cm^{-1} are usually attributed to the C–H stretching vibration [26], which is assigned to the alkane functional groups. The peaks around 1,718–1,739 cm^{-1} can be regarded to the C=O stretching vibration [27], while the peaks in the range of 1,196–1,217 cm^{-1} can be ascribed to the C–O stretching vibration [28]. The presence of sulfonate functional groups of the surfactant could be assigned to the S=O stretching vibration, which is located around 1,366–1,386 cm^{-1}.

In this analysis, SIAC-10 with a minimal amount of SLSA concentration was chosen to depict the functional groups after the modification process. It is noted that the stretching vibrations of C=O, S=O, and C–O were observed on SIAC-10 in the form of weak absorption peaks at their respective vibration ranges. The presence of carboxyl, sulfonate, and esters functional groups, respectively, are believed to occur due to the hydrophobic interaction between the surfactant and the surface of AC [28,29]. Therefore, it affected the surface characteristics of the adsorbents. Previous researchers have also found that modifying the surface of AC with a surfactant promotes the chemical changes of the functional groups [28,30]. Therefore, it indicates that SLSA was successfully adsorbed on the surface of AC even at a minimal amount of concentration.

3.2. Surface textural properties of AC

Nitrogen gas adsorption–desorption isotherms of virgin AC and SIACs are presented in Fig. 4. According to the classification by the International Union of Pure and

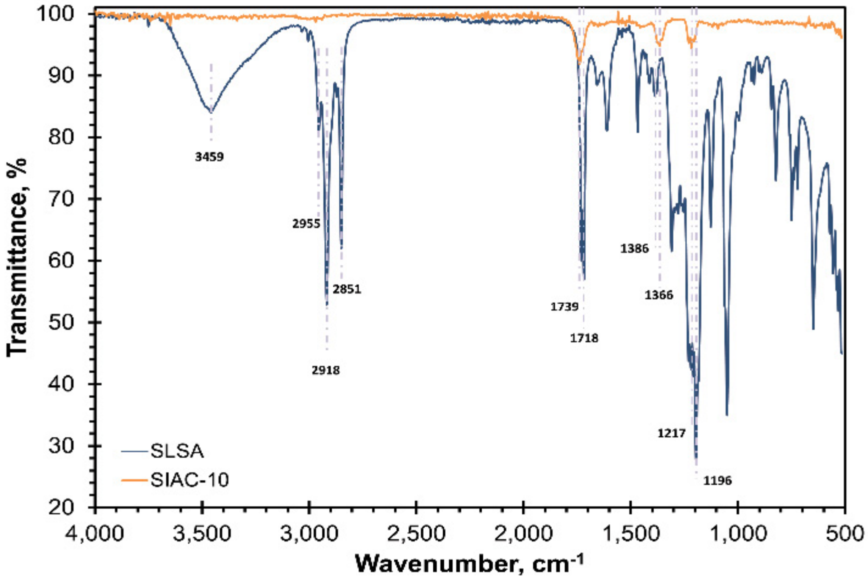

Fig. 3. FTIR spectra of SLSA and SIAC-10.

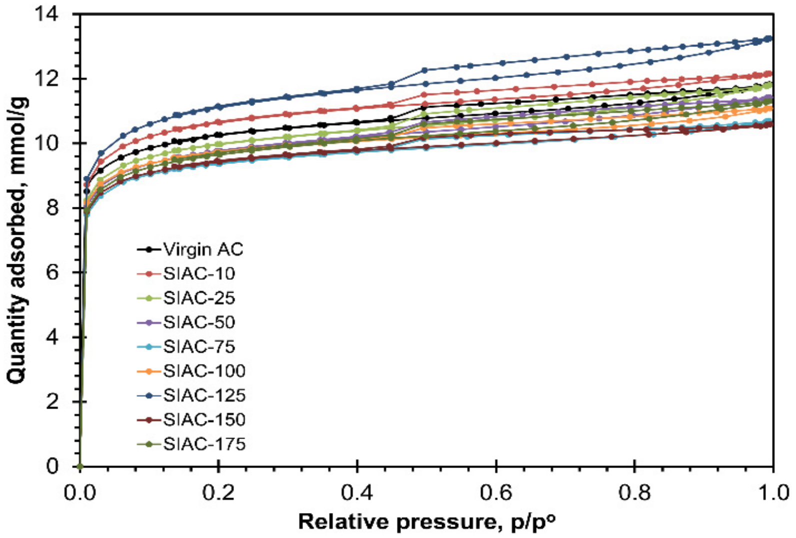

Fig. 4. N_2 adsorption–desorption isotherms at 77.4 K.

Applied Chemistry (IUPAC), the adsorption isotherms of the adsorbents resemble to the type IV isotherm [31]. Type IV isotherm is usually attributed to the presence of micro- and mesopores [32]. From Fig. 4, prominent adsorption of nitrogen gas is observed at low relative pressures of less than 0.1. This phenomenon is believed to occur due to the presence of micropores [33]. As the relative pressures increases, the adsorption of nitrogen gas proceeds into the mesopores of the adsorbents [34]. At this moment, the monolayer-multilayer adsorption is occurring along with the capillary condensation, where the nitrogen gas condenses below its saturation pressure of the bulk liquid [35].

The type IV isotherm also exhibits the formation of hysteresis loops during the desorption of nitrogen gas. A different desorption path than the adsorption isotherms due to the capillary condensation in mesopores has consequently created a hysteresis loop [32]. Based on the IUPAC classification, the hysteresis loops resemble to the type H4 [31]. The type H4 hysteresis loop is usually related to complex materials and is often associated with narrow slit-likes pores [31,34]. As can be seen in Fig. 4, all the samples show a similar type of isotherm. Even so, the trend of nitrogen gas adsorption is observed to be varied for each sample. This indicates that the surface modification of AC with surfactant may influence the micro- or mesoporous structure of the adsorbents.

According to [31], the diameters of micropores can be classified as less than 2 nm, while the diameters of mesopores can be classified over the range of 2–50 nm. As shown in Table 2, the average pore diameter of virgin AC is measured at 2 nm. This indicates that virgin AC primarily consists of mesopores. However, after the modification process with SLSA, a slight reduction of pore diameters has been generally observed. This could be due to the attachment of SLSA onto AC. As can be seen, the BET surface area of SIAC-50, SIAC-75, SIAC-100, and SIAC-150 were found to be lower than the virgin AC (Table 2). This decrease could be due to the pore blockage of AC after the surface modification with surfactant [28].

On the other hand, the BET surface area of SIAC-10, SIAC-25, SIAC-125, and SIAC-175 were also found to be greater than the virgin AC (Table 2). This increase is likely to be due to the removal of material debris from the pores of the adsorbents. A similar finding has also been obtained by [36]. It is suggested that the reduction of material debris from the porous structure of the adsorbents could increase its surface area [36].

Table 2
Surface textural properties of virgin AC and SIACs

Samples	BET surface area (m^2/g)	Average pore diameter (nm)
Virgin AC	820.51	2.00
SIAC-10	880.76	1.91
SIAC-25	822.78	1.99
SIAC-50	807.56	1.96
SIAC-75	749.51	1.98
SIAC-100	804.65	1.91
SIAC-125	920.17	1.99
SIAC-150	782.13	1.88
SIAC-175	834.20	1.88

In view of the results obtained, the variations of the BET surface area may be due to the affinities of surfactant towards the adsorbents. The surfactant properties such as critical micelle concentration and chemical configuration have appeared to be the significant factors that determined the amount of surfactant to be attached onto AC [37]. Moreover, the presence of only a single hydrophobic carbon chain of the surfactant may also affect its binding capability on the adsorbent's surface [19]. This may indicate that SLSA could be partially or completely attached onto AC via hydrophobic interaction.

3.3. Removal of Zn(II) ion from aqueous medium

The removal efficiency of Zn(II) ion from aqueous medium for each adsorbent sample is shown in Fig. 5. Generally, the removal efficiency of Zn(II) ion is observed to slightly increase as the amount of SLSA concentration increases. This indicates that the surface modification of AC with the anionic plant-based surfactant can enhance the adsorption of Zn(II) ion. Without SLSA impregnation, the removal of Zn(II) ion is obtained at 26.44%. After SLSA impregnation, the highest removal of Zn(II) ion was obtained at 31.66% (SIAC-75).This may be due to the presence of high intensity of SLSA on the surface of AC. The result is consistent with the textural properties of SIAC-75 in Table 2, where the lowest BET surface area of 749.51 m^2/g may indicate that the hydrophobic interaction between SLSA and the surface of AC had occurred.

It is believed that the negatively charged functional head groups of the surfactant would provide additional adsorption sites for the Zn(II) ion to be attached on the surface of the adsorbent [20,37]. Hence, this phenomenon resulted in the electrostatic attraction between the adsorbent and Zn(II) ion.

3.4. Removal of Cr(VI) ion from aqueous medium

Fig. 6 shows the removal efficiency of Cr(VI) ion from aqueous medium for each adsorbent sample. As shown, the removal efficiency of Cr(VI) ion decreases as the concentration of SLSA increases. Without SLSA impregnation, the removal of Cr(VI) ion is obtained at 45.76%, which is the highest. However, after SLSA impregnation, the metal removal declined. This indicates that SLSA impregnation would inhibit the adsorption of Cr(VI) ion.

As reported from the previous literature, the hexavalent chromium ion can exists in the form of chromate (CrO_4^{2-}) and dichromate ($Cr_2O_7^{2-}$) ions [38], where both compounds are present as anionic oxyanions in the aqueous medium. Another study also reported that the chromate ion is the primary dissociation form of Cr(VI) ion in the aqueous medium [39]. However, it is believed that the dissociation form of Cr(VI) ion is mainly governed by the pH of the aqueous medium [40]. At the acidic pH, Cr(VI) ion can be mainly dissociated into $Cr_2O_7^{2-}$ and $HCrO_4^-$ ions; meanwhile, at pH above 8, only CrO_4^{2-} ion is stable in the aqueous medium [40].

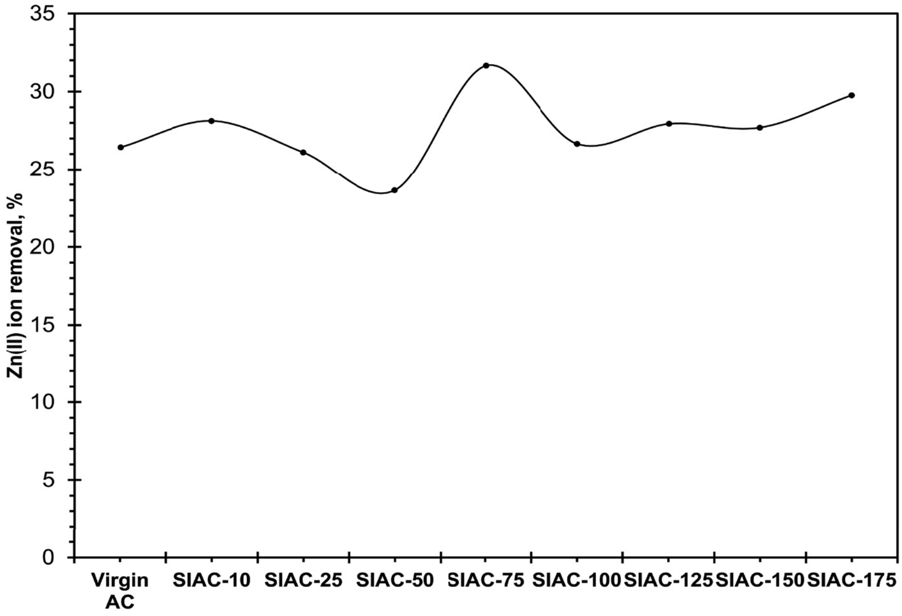

Fig. 5. Removal efficiency of Zn(II) ion for each adsorbents sample.

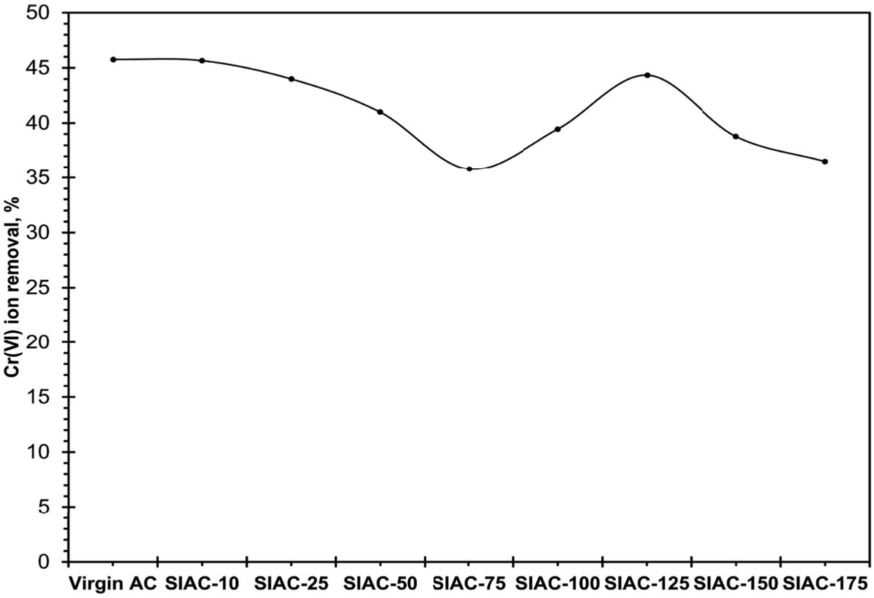

Fig. 6. Removal efficiency of Cr(VI) ion for each adsorbents sample.

In this study, the adsorption experiments were conducted at neutral pH at around 7–8. At this condition, it is believed that only chromate ion exists as the dissociated form of Cr(VI) ion.

As can be seen in Fig. 6, the lowest removal of Cr(VI) ion is observed in SIAC-75 (35.81%). This result is consistent with the previous results obtained from the removal of Zn(II) ion. It is then further proved that a large amount of SLSA has been attached onto SIAC-75. Thus, this would reduce the percentage removal of Cr(VI) ion due to the electrostatic repulsion.

Based on the above discussions, the proposed adsorption mechanism of Zn(II) and Cr(VI) ions is illustrated in Fig. 7. As can be seen, SLSA is hydrophobically attached to the surface of AC, and at the same time, its hydrophilic head is pointing towards the aqueous medium [20]. In this case, the sulfonate (OSO_3^-) group is the hydrophilic head of the surfactant. With the presence of such a functional group, a new adsorption site for the metal ion has been created. For instance, the electrostatic attraction exists between the opposite positively charged Zn(II) ion and the anionic sulfonate group. This type of interaction would enhance the binding capability of Zn(II) ion onto AC. However, a different adsorption mechanism is observed for Cr(VI) ion. Since it has been dissociated into chromate ion (CrO_4^{2-}), an interaction between both chromate ion and the anionic sulfonate

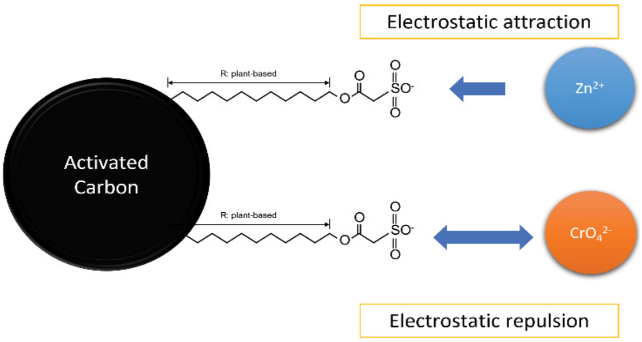

Fig. 7. Adsorption mechanism of metal cations.

group has resulted in the electrostatic repulsion. This type of repulsion would inhibit the adsorption of Cr(VI) ion onto AC, thus reducing its removal efficiency from the aqueous medium.

Apart from that, competitive binding of the natively existing Na(I) ion of SLSA and the respective metal ion may also occur during the adsorption process. As shown in the reversible Eq. (2), Na(I) ion could be re-attached again to its negatively charged moieties [19]. Since Zn(II) ion existed as a positively charged metal ion in the aqueous medium, a competitive binding for the same active site may occur. Hence, it may influence the adsorption of Zn(II) ion.

$$2RSO_3Na + M^{2+} \leftrightarrow \left(RSO_3\right)_2 M + 2Na^{+} \quad (2)$$

4. Conclusions

This work was devoted to assess the capability of the modified adsorbent, SIAC, in removing Zn(II) and Cr(VI) ions from the aqueous medium. Based on the analyses conducted, it was found that SLSA surfactant was hydrophobically attached to activated carbon. As identified by the FTIR analysis, the presence of carboxyl, sulfonate, and esters functional groups proved that the SLSA impregnation process had been successfully conducted. Thereby, enhanced the surface heterogeneity of activated carbon.

In this investigation, the activated carbon impregnated with 75 mg/L of SLSA concentration (SIAC-75) showed the lowest BET surface area (749.51 m^2/g). This may imply that a large amount of SLSA had occupied the porous structure of the adsorbent. Even with the lowest BET surface area, SIAC-75 showed the highest removal efficiency of Zn(II) ion, at 31.66%. This proves that a large amount of SLSA could help Zn(II) ion adsorption. However, the removal efficiency of Cr(VI) ion was found to be the lowest in SIAC-75. This proves that a large amount of SLSA could hinder the adsorption of Cr(VI) ion. It was proposed that electrostatic attraction had been taken place during the adsorption of Zn(II) ion, while electrostatic repulsion had been taken place during the adsorption of Cr(VI) ion. The existence of chromate ion (CrO_4^{2-}) as a dissociation form of Cr(VI) ion may hinder the adsorbents' capability to adsorb Cr(VI) ion.

In conclusion, the modified adsorbent prepared in this study may be applied as a potential adsorbent for the adsorption of Zn(II) ion contaminants as compared to Cr(VI) ion. For further works, it is suggested that pH adjustment and ionic strength analyses be considered. These may help in describing the metal speciation, surface charge, complexation of functional groups, or sorption capability in the adsorption mechanism.

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