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Removal of thiocyanate from water by using weak base microporous resins of different matrix structure

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ABSTRACT

Thiocyanate (SCN^-) enters into water bodies through several industrial processes. The removal of thiocyanate from water is essential as it is toxic to aquatic organisms and humans. The kinetics and equilibrium study of removing thiocyanate from water was investigated using two microporous anion exchangers having different resin matrices namely Tulsion A-10X (TUL-10) of acrylic matrix and Amberlyst A-21 (AMB-21) of styrene divinyl benzene matrix. The effect of pH, thiocyanate concentration, temperature, contact time and resin dosage was studied on both the resins. Different kinetic models were applied to understand adsorption mechanism. Adsorption of thiocyanate follows pseudo-second-order kinetic model and fits into Langmuir and Freundlich isotherms. The adsorption capacities of the resins from the Langmuir isotherm are 153.19 and 142.87 mg g^{-1} for TUL-10 and AMB-21 resins, respectively. The experimental data indicates that AMB-21 with styrene divinyl benzene matrix has removal efficiency of 90% at adsorption dosage of 150 mg with 350 mg L^{-1} thiocyanate concentration at 303 K, whereas TUL-10 resin having acrylic matrix shows 75% removal efficiency at same conditions. The adsorption is exothermic in nature and does not show significant change in internal structure of the resin. Fourier transform infrared spectroscopy spectra of resins after adsorption confirm the presence of thiocyanate. Desorption study indicates NaOH is more efficient desorbent for AMB-21 showing 99.61% desorption efficiency.

Keywords: Adsorption; Kinetics; Thiocyanate; Weak base anion exchanger

1. Introduction

Thiocyanate gets added into water through several industrial processes like leaching of gold from mining industries, coal coking, fertilizer industries, photofinishing, manufacturing of thiourea, electroplating process. Detoxification of cyanide also results in formation of thiocyanate ions. Higher concentration of thiocyanate in water is a matter of great concern to aquatic life as it acts as an anionic toxin [1] as it converts into cyanide because of oxidants present in water [2]. Likewise, higher concentration of thiocyanate in the human body affects the production of thyroid hormone [3], affects protein dialysis [4], weakens muscles and cause delirium, leads to coma and hyperreflexia [5]. Hence, the removal of thiocyanate from water becomes necessary.

Several methods are used to remove thiocyanate from the water and waste water which comprise degradation of thiocyanate by using environmental friendly oxidants such as ferrates Fe(VI) and Fe(V) [6] and persulfate ($S_2O_8^{2-}$) in the presence of ferric ion [7]. Polymer inclusion membrane [8] is also used to remove thiocyanate from wastewater generated by mining. Adsorption of thiocyanate is carried out using various adsorbents like modified and unmodified hydrotalcite [9–11], ferrihydrite [12], steel slag [13], Fe(III)/Cr(III) hydroxide sludge from industries [14], ion imprinted polymers [15–17], conducting electroactive polymers [18] and ion exchange resins [19,20].

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Nonetheless, ion exchangers are mechanically and chemically stable, economical and can be regenerated easily. Thus, in the present study ion-exchange resins of styrene divinyl benzene and acrylic matrix in chloride form were used to remove thiocyanate from water.

The exchange reaction is represented as,

$$R\text{Cl} + \text{CNS}^- - R\text{CNS} + \text{Cl}^-$$

where R stands for resin matrix.

The adsorption studies of thiocyanate on two resins having different resin matrix were carried out by varying resin quantity, concentration of thiocyanate ions, solution pH and temperature for its effective removal.

2. Experimental

2.1. Materials and methods

The analytical grade chemical reagents such as KSCN, NaOH, HCl, HNO_3, $Fe(NO_3)_3$ were used for batch process. Stock solution of thiocyanate of 1,000 mg L^{-1} was prepared by dissolving appropriate quantity of KSCN in deionised water, which was diluted to get different concentrations.

Anion exchanger Tulsion A-10X (TUL-10) with polyacrylic matrix having polyamine group, supplied by Thermax limited (Pune) and Amberlyst A-21 (AMB-21) with polystyrene having tertiary amine resin fixed ion procured from Himedia Laboratories Pvt. Ltd. (Mumbai) were reconditioned by repeated washing with HCl and NaOH. These reconditioned resins were washed thoroughly with alcohol to prevent hydrolysis and dried to get constant weight. The characteristics of resins are summarized in Table 1.

2.2. Methods

2.2.1. Ion exchange equilibrium and kinetic studies

Kinetic study was carried out by taking known amount of resin with 50 cm^3 of the thiocyanate solution of known concentration in a 100 cm^3 volumetric flask. Thermostat mechanical shaker was used at 303 K for agitating the solution. Thiocyanate concentration in the external solution was found out at fixed time intervals. The amount of thiocyanate on the resin is calculated by difference method.

The thiocyanate concentration in the solution before and after adsorption was estimated by forming Iron (III) thiocyanate complex [21] and absorbance was measured at 460 nm by using Varian Carry-50 Bio UV-Visible spectrophotometer. The analysis was completed within 30 min of complex formation.

Thiocyanate concentration was varied from 150–450 mg L^{-1} by taking fixed amount of resin and the flasks were agitated till the equilibrium was attained. At equilibrium, the adsorption capacity of resin q_e, was evaluated by using the equation:

$$q_e = \frac{V\{C_0 - C_e\}}{m} \tag{1}$$

where C_0 and C_e are initial and equilibrium concentration of thiocyanate (mg L^{-1}) respectively, V is the volume of solution (L) and m is the mass of resin (g).

Distribution coefficient (K_d) at equilibrium was calculated using:

$$K_d = \frac{C_0 - C_e}{C_e} \tag{2}$$

% Removal of thiocyanate was calculated using

$$\%R = \frac{(C_0 - C_e)}{C_0} \times 100 \tag{3}$$

2.2.2. Desorption studies

Desorption study was carried out by using different desorbents for reuse of resin. 50 cm^3 of thiocyanate solution of concentration 300 mg L^{-1} was kept in contact with 150 mg of resin in mechanical shaker (Toshiba model reciprocate type) until the equilibrium is reached. The resin was washed with distilled water after sorption of thiocyanate to remove traces of solution adhered. The washed resin was treated with 50 cm^3 of desorbents like, 1N NaOH, 1N HCl and 1N NaCl and kept for shaking. After desorption, thiocyanate concentration in the filtrate was analyzed. Eq. (4) was used to calculate desorption ratio (D_R) [14].

$$D_R = \frac{C_s}{C_r} \times 100 \tag{4}$$

Table 1
Resin characteristics

Resin	TUL-10	AMB-21
Matrix structure	Cross linked polyacrylic	Styrene divinylbenzene
Functional group	Polyamine	Tertiary amine
Ionic form	Free base form converted to chloride form	Free base form converted to chloride form
Screen size (US mesh)	16–50	22–30
Maximum operating temperature	80°C	100°C
Total capacity in m.eq./250 mg	0.900	0.720
Moisture content %	52%–55%	56%–62%

where C_s and C_r are amount of thiocyanate desorbed and adsorbed to the external solution and on resin (mg L^{-1}), respectively.

3. Result and discussion

3.1. Effect of the various parameters for thiocyanate removal

3.1.1. Effect of pH

The study was carried by varying pH 1–12 by using 150 mg resins with 50 cm^3 of thiocyanate solution of concentration 350 mg L^{-1}. The samples were kept for agitation to attain equilibrium using mechanical shaker at 303 K and the concentration of thiocyanate in the solution was checked after equilibration by spectrophotometric method. The sorption of thiocyanate on both the resins at different pH is represented in Fig. 1 which indicates efficient removal of thiocyanate from water by using these ion exchangers is possible in pH range of 3–10. However, at lower pH less than 3 the percentage removal efficiency drastically reduces due to presence of competing Cl$^-$ ions. At higher pH more than 10, decrease in removal efficiency is due to conversion of resin to free base which shows less affinity towards thiocyanate ions [22].

Among the resins used for present study, AMB-21 with styrene divinyl structure and tertiary amine group has more affinity towards thiocyanate than TUL-10. Similar results were obtained for thiocyanate removal by using different resin matrices [23].

3.1.2. Effect of thiocyanate concentration and temperature

The of sorption of thiocyanate on both the resins in water was investigated at 303, 323 and 343 K by varying concentration from 150 to 450 mg L^{-1} and taking 150 mg of resin. With increase in concentration of thiocyanate the removal efficiency decreases on both the resins as shown in Figs. 2a and b. This may be due to insufficient active sites available on the resin. The effect of temperature is not significant on AMB-21. The percentage removal efficiency is more on resin with styrene divinyl matrix than resin with acrylic matrix at different concentrations which indicates the effect of resin matrix on adsorption. AMB-21 is having polystyrene resin matrix with higher hydrophobicity than TUL-10X with acrylic matrix. So thiocyanate ion with low charge density shows more affinity towards AMB-21 [24].

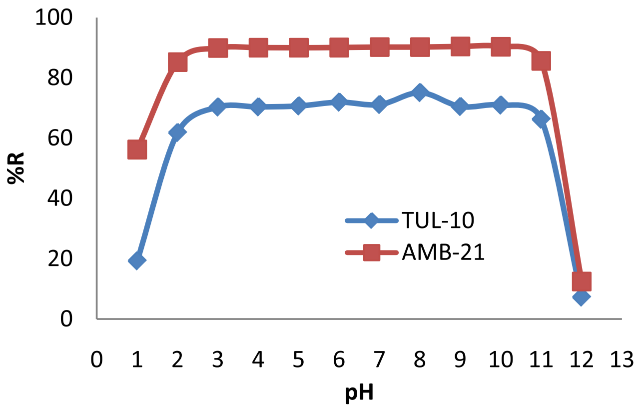

Fig. 1. Effect of pH on removal of thiocyanate using resins.

3.1.3. Effect of contact time

Contact time was varied from 10 to 110 min by keeping fixed thiocyanate concentration of 400 mg L^{-1} with 250 mg of resin. The results are presented in Fig. 3. Thiocyanate removal on both the resins progresses rapidly in the initial stages and reaches equilibrium in 80 min. However, AMB-21 shows maximum removal of thiocyanate.

3.1.4. Effect of resin dosage

The effect of resin dosage was carried out by varying the quantity of resin with fixed concentration of thiocyanate at 303 K. Fig. 4 represents effect of resin dosage as the function of percentage removal. It is seen that, with increase in resin dosage the percentage removal efficiency of thiocyanate increases on both resins as surface active groups available are more at higher resin dosage. The percentage removal efficiency of thiocyanate is more on resin with styrene divinyl benzene matrix than that of acrylic matrix at same resin dosage.

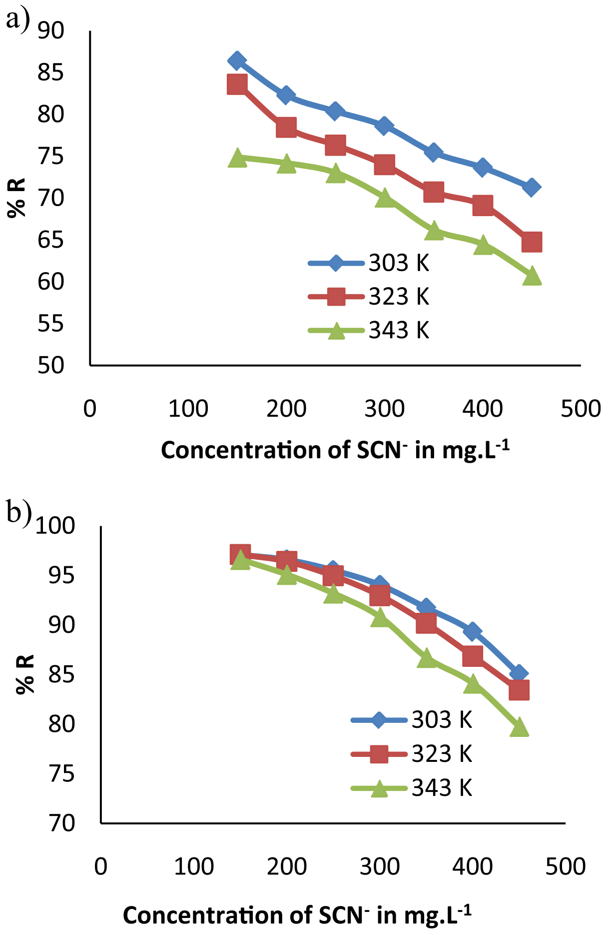

Fig. 2. Effect of thiocyanate concentration and temperature on removal of thiocyanate (a) On TUL-10 and (b) On AMB-21.

3.2. Adsorption isotherms

Adsorption of thiocyanate on both the resins was studied by applying Langmuir and Freundlich isotherm. The obtained isotherm data for three temperatures 303, 323 and 343 K is represented in Table 2.

The linear form of Langmuir isotherm model [25] which is frequently applied for monolayer processes is,

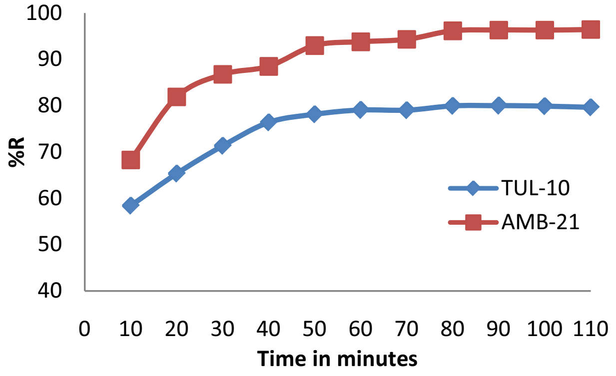

Fig. 3. Effect of contact time on the removal of thiocyanate.

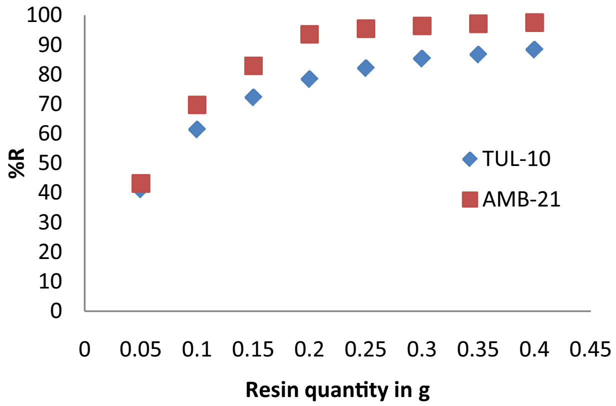

Fig. 4. Effect of resin dosage on the removal of thiocyanate.

$$\frac{C_e}{q_e} = \frac{1}{K_L \times Q_L} + \frac{C_e}{Q_L} \tag{5}$$

where K_L is the Langmuir constant related to the affinity of the bonding sites (L mg^{-1}) and Q_L is the maximum adsorption capacity (mg g^{-1}), C_e is thiocyanate concentration in solution at equilibrium (mg L^{-1}), and q_e is the equilibrium adsorption capacity (mg g^{-1}). K_L and Q_L are obtained by plotting a graph of C_e/q_e vs. C_e from the intercept and slope. Data calculated is represented in Table 2. The R_L is greater than 0 but less than 1 for all the temperatures indicating that adsorption is favourable. R^2 value is ≥0.99 for both the resins which indicates adsorption is monolayer. Adsorption capacities of weak base resins AMB-21 and TUL-10 are 142.87 and 153.19 mg g^{-1} respectively is compared with Purolite A-250, a strong base resin with capacity of 191.20 mg g^{-1} [19]. Although the adsorption capacities are different, the removal efficiency is nearly the same. This implies weak base resins are equally useful in removing thiocyanate.

Multilayer adsorption of adsorbate on the adsorbent with heterogeneous surface is thoroughly explained by Freundlich isotherm model. The linear form of adsorption isotherm [26] is,

$$\log q_e = \log K_F + \frac{1}{n_F} \log C_e \tag{6}$$

where K_F is Freundlich constant (mg g^{-1}) which indicates relative adsorption capacity and n_F is the adsorption intensity. K_F and n_F are obtained from graph of log q_e vs. log C_e. Data calculated is represented in Table 2. n_F values greater than 1 indicate adsorption of thiocyanate on resins take place by chemisorption and decrease in K_F values with temperature indicate the adsorption process is exothermic.

3.3. Adsorption kinetics

The experimental data obtained for kinetic study was analyzed by using three kinetic models. The Lagergren's equation for pseudo-first-order kinetics [27] is,

$$\log\left(q_e - q_t\right) = \log q_e - \frac{k_1 t}{2.303} \tag{7}$$

Table 2
Langmuir and Freundlich isotherm constants for the removal of thiocyanate by using resins

Isotherm model	Isotherm parameters	TUL-10			AMB-21		
		Temperature					
		303 K	323 K	343 K	303 K	323 K	343 K
Langmuir	K_L	0.017	0.016	0.010	0.133	0.110	0.089
	Q_L	153.19	135.92	144.51	142.87	137.99	132.03
	R_L	0.461	0.430	1.000	0.241	0.237	0.231
	R^2	0.985	0.980	0.989	0.998	0.998	0.997
Freundlich	n_F	1.992	2.099	1.780	3.066	3.123	3.212
	K_F	9.436	9.032	5.301	35.943	33.255	30.819
	R^2	0.996	0.992	0.968	0.961	0.973	0.984

where q_t is the amount of thiocyanate on resin (mg g^{-1}) at time t, k_1 is the rate constant (min^{-1}). The Lagergren's first order rate constant (k_1) and q_e are calculated from the intercept and slope of the plot log(q_e–q_t) vs. t as shown in Fig. 5 and the values are listed in Table 3 along with the correlation coefficients. Fig. 5 and the data imply that the adsorption of thiocyanate on the resins is not an ideal pseudo-first-order reaction.

Pseudo-second-order model [27] is represented as,

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{8}$$

where k_2 is second-order rate constant (g mg^{-1} min^{-1}) is calculated experimentally from the slope and intercept of plot t/q_t vs. t as shown in Fig. 6. The k_2, q_e and R^2 calculated are listed in Table 3 which indicate the adsorption of thiocyanate on resins follows pseudo-second-order reaction.

The modified Ritchie's-second-order kinetic model [28] is represented as,

$$\frac{1}{q_t} = \frac{1}{k_R q_e t} + \frac{1}{q_e} \tag{9}$$

where k_R is the modified Ritchie's rate constant (min^{-1}) and is obtained by plotting $1/q_t$ vs. $1/t$. The values of correlation coefficient obtained are listed in Table 3 which suggests adsorption follows second-order kinetic model.

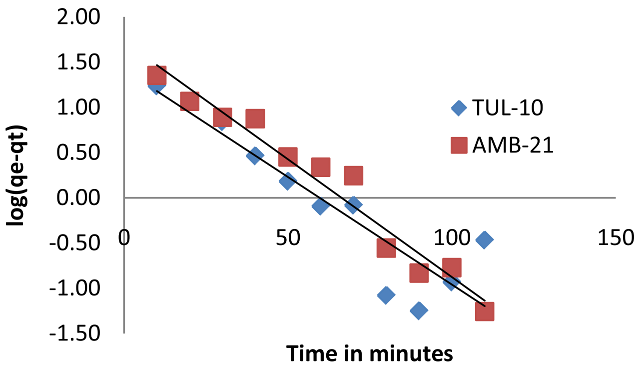

Fig. 5. Pseudo-first-order kinetics of thiocyanate removal.

Table 3
Adsorption kinetic parameters for adsorption of thiocyanate on different resins

Kinetic models	Parameters	TUL-10	AMB-21
Pseudo-first-order	K_1	0.055	0.060
	q_e	26.18	53.35
	R^2	0.8337	0.9577
Pseudo-second-order	K_2	0.0034	0.0025
	q_e	66.93	81.01
	h	15.27	16.21
	R^2	0.9995	0.9997
Ritchie's-second-order	K_R	981.41	1,360.06
	q_e	67.07	80.67
	R^2	0.9683	0.9892

3.4. Adsorption mechanism

To understand the adsorption mechanism and the rate controlling step Weber-Morris intraparticle diffusion model [29] is used. It is represented as,

$$q_t = k_{\text{id}} t^{0.5} + a \tag{10}$$

where k_{id} is the intraparticle rate constant (mg g^{-1} min$^{0.5}$) and a is gradient of linear plot.

If adsorption takes place through intra particle diffusion the plot of q_t vs. $t^{0.5}$ must be linear and should pass through origin. As per Fig. 7 plot of q_t vs. $t^{0.5}$ is multilinear which implies the adsorption of thiocyanate on both the resins is governed by more than two steps. Therefore the rate limiting steps are surface and intra particle diffusion. Larger intercept for AMB-21 suggest surface or film diffusion plays an important role in controlling rate limiting step. This indeed indicates that thiocyanate gets adsorbed more readily on resin AMB-21 with styrene divinyl benzene matrix than TUL-10 with acrylic matrix.

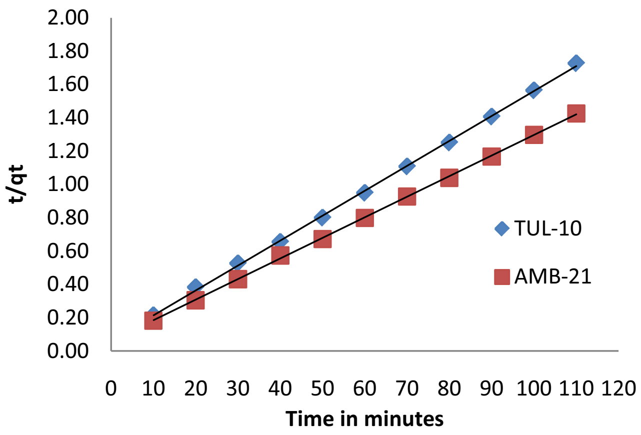

Fig. 6. Pseudo-second-order kinetics of thiocyanate removal.

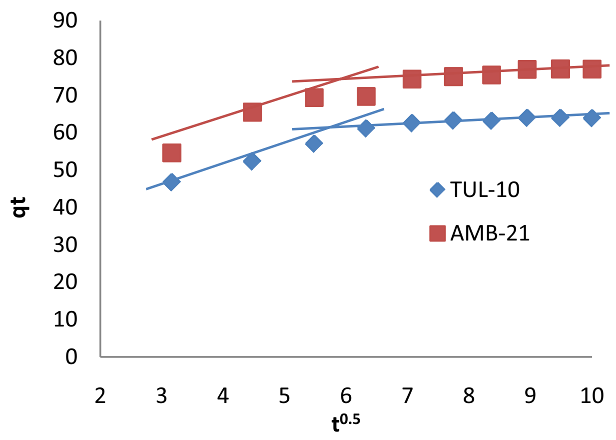

Fig. 7. Intra particle diffusion model of removal of thiocyanate.

3.5. Thermodynamic parameters

As the adsorption process is temperature dependent, adsorption of thiocyanate on resins was carried out at three different temperatures 303, 323 and 343 K to study thermodynamic parameters. The adsorption capacity of thiocyanate decreased with increase in temperature which is due to weakening of the bonds between the thiocyanate and resin.

The free energy change ($\Delta G°$) in kJ mol^{-1} is calculated by using Eq. (11) [30],

$$\Delta G° = -RT \ln K_d \quad (11)$$

where R and K_d are gas constant and equilibrium coefficient.

Enthalpy ($\Delta H°$) in kJ mol^{-1} and entropy ($\Delta S°$) in J mol^{-1} K^{-1} are calculated using Van't Hoff linear equation by plotting a graph of $\ln K_d$ against $1/T$.

$$\ln K_d = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \quad (12)$$

Table 4
Thermodynamic parameters of removal of thiocyanate using resins

	Temperature (K)	$\ln K_d$	$\Delta G°$	$\Delta H°$	$\Delta S°$
TUL-10	303	1.35	–3.41	–11.04	–25.09
	323	1.11	–2.99		
	343	0.84	–2.40		
AMB-21	303	3.04	–7.67	–11.15	–11.43
	323	2.79	–7.79		
	343	2.52	–7.21		

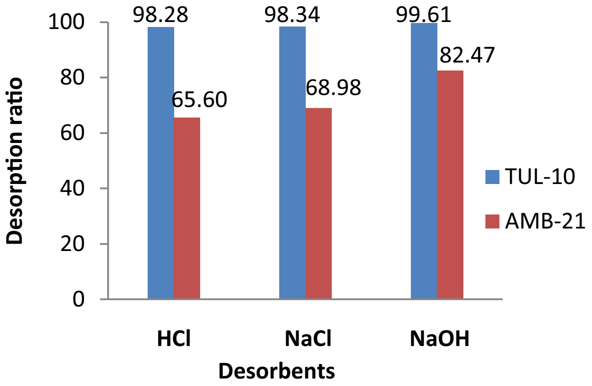

Fig. 8. Desorption of thiocyanate.

The adsorption of thiocyanate on resins is spontaneous and exothermic in nature, as indicated by negative values of $\Delta G°$ and $\Delta H°$ which are listed in Table 4. During adsorption there is no significant change in the internal structure of the resin as $\Delta S°$ values are negative.

3.6. Desorption studies

Desorption studies of thiocyanate on both the resins was carried out by using 1 M NaOH, 1 M HCl and 1 M NaCl as desorbents and results are represented in Fig. 8. The study indicates that NaOH is more efficient desorbent than HCl and NaCl for AMB-21, whereas thiocyanate can be efficiently desorbed from TUL-10 using all the desorbents. However, thiocyanate ions cannot be desorbed completely from AMB-21 as interaction of thiocyanate is more with the resin.

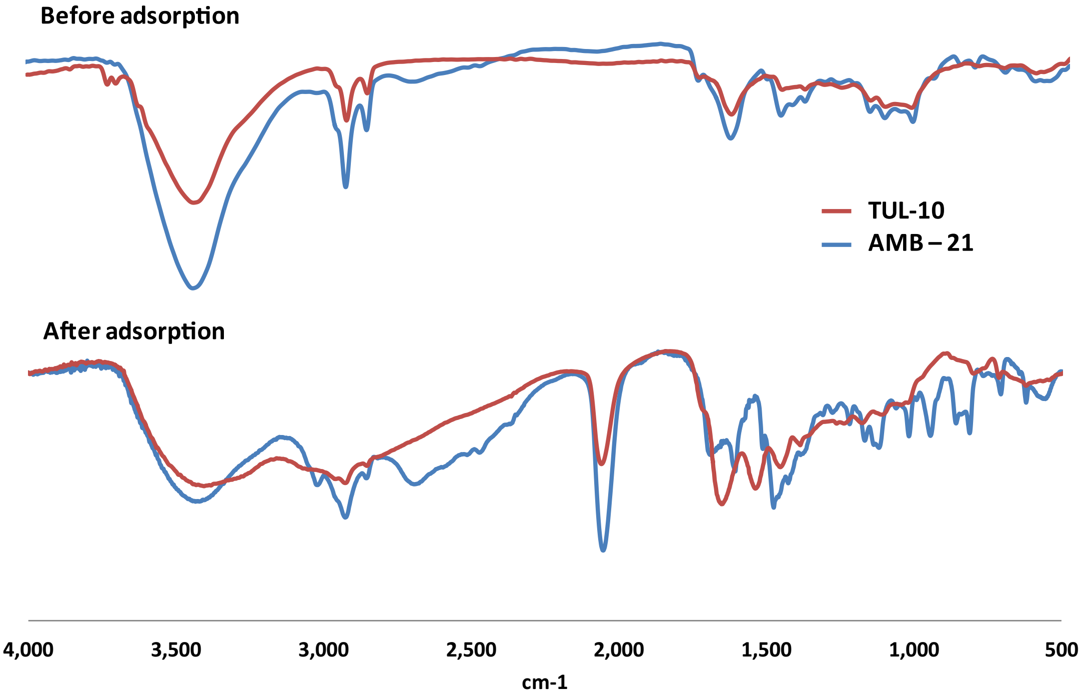

Fig. 9. FTIR spectra of resins.

3.7. Characterization of resins by fourier transform infrared spectroscopy

Nicolet 6700 Fourier transform infrared spectroscopy (FTIR) spectrophotometer was used to take FTIR spectra of the resins by KBr pellet technique and represented in Fig. 9.

FTIR spectrum of the AMB-21 resin before adsorption shows spectral bands centered at around 1,463 cm^{-1} confirms the presence of tertiary amine surface groups. The spectral band observed at 3,434; 2,924; 2,854 and 1,020 cm^{-1} corresponds to stretching vibration of $-OH$ group, $-CH$ from $-CH_3$ or $-CH_2$ groups, $-C-N$ group, respectively. The spectrum of resin after the adsorption studies shows a strong band at 2,052 cm^{-1} which reveals the presence of SCN^- group [31] and thus confirms presence of thiocyanate on the resin.

FTIR Spectrum of TUL-10 shows peak at 3,437 cm^{-1} which indicates the presence of hydroxyl and amine groups. The band at 2,928 cm^{-1} attributes to symmetric stretch vibrations of the CH_2 groups. A band at 2,058 cm^{-1} after adsorption indicates the presence of SCN^- adsorption on the resin.

4. Conclusions

To summarize, the results obtained from the data for resins of different matrices AMB-21 with styrene divinyl benzene is ideal for separation of thiocyanate from water in the pH range from 3 to 10 than TUL-10 with acrylic resin matrix. The adsorption of thiocyanate on both resins is favorable which is supported by both Freundlich and Langmuir isotherms. Kinetic data suggests adsorption of thiocyanate followed pseudo-second-order kinetics and adsorption mechanism is through surface and intra particle diffusion. Thermodynamic data generated reveals that the adsorption is less with increase in temperature and NaOH is efficient desorbent.

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