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Adsorption Profile of Molecular Iodine and Iodine Number of Polyurethane Foam

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ABSTRACT

The sorption of iodine on polyurethane foam (PUF) has been investigated. The maximum sorption of 0.01 N of iodine solution on PUF was found to be >95% from 0.2 N HCl aqueous solution. The maximum equilibration was attained with 30 min agitation time. The iodine number was found to be 436 ± 15 mg g^{-1}. The average pore diameter of PUF was 8.3 nm and total surface area was 67 $m^2 g^{-1}$. The kinetic data indicates that the intraparticle diffusion rate of adsorption (R_d) and rate of mass action (k_1) of iodine sorption on PUF were 0.48 ± 0.03 mmole $g^{-1} min^{-1/2}$ and 52 ± 8 mmole $g^{-1} min^{-1}$, respectively. The sorption data followed both Freundlich and Langmuir adsorption isotherms in the concentration range 0.03 N to 0.15 N of iodine. The Freundlich constants $C_m = 11.2 \pm 1.7$ mmole g^{-1} and $1/n = 0.57 \pm 0.05$, and Langmuir constants $M = 4.9 \pm 0.6$ mmole g^{-1} and $b = 12.9 \pm 1.3$ L g^{-1} were established.

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The sorption free energy (E) $= 5.3 \pm 0.2\,\mathrm{kJ\,mole^{-1}}$ and loading capacity $X_m = 5.15 \pm 0.45\,\mathrm{mmole\,g^{-1}}$ were evaluated using D–R isotherm. The sorption energy reflects the physiosorption of iodine on PUF. The effect of temperature on the sorption yields the thermodynamic parameters $\Delta H = 50 \pm 2\,\mathrm{kJ\,mole^{-1}}$ and $\Delta S = 170 \pm 4\,\mathrm{J\,mole^{-1}\,K^{-1}}$. The Gibbs free energy (ΔG) ranges from 3.72 to $-2.15\,\mathrm{kJ\,mole^{-1}}$, suggesting the endothermic nature of sorption of iodine, but is favorable at higher temperatures (>313K). The effect of different anions and cations on the sorption of iodine on PUF have been evaluated and discussed.

Key Words: Iodine; Polyurethane foam; Adsorption; Kinetics; Thermodynamics; Iodine number.

INTRODUCTION

Iodine is an essential trace element for both mammals and vegetable life. It occurs widely in nature at very low concentrations in iodide and iodate forms. Its deficiency in mammals leads to goiter, a condition in which the thyroid gland becomes enlarged. The antiseptic properties of iodine and its compounds bolster their use for treatment of wounds or sterilization of drinking water. Major industrial uses are in photography and in the dye industry where iodine-containing dyes are produced for food processing.[1,2]

Polyurethane foam (PUF) is a cheaper substitute of diethylether in solvent extraction and has been found to be an effective sorbent for a large number of inorganic and organic species in aqueous solutions and in air. The sorption behavior of iodine-131 on PUF is studied[3,4] both from static and pulse column bedding, in the form of cylindrical shaped packing materials. The separation method of radio Iodine-131 in environment water samples by PUF is much more rapid than any other radiochemical method.[5] The separation is selective in the presence of major fission products. All these studies are of an analytical point of view and are based on the sorbed concentration of iodine on PUF.[6] However, the nature of sorption of iodine on PUF is not clear. A number of different mechanisms have been proposed such as chelation, ion pair formation, ion exchange, absorption, adsorption, clathration, etc., depending upon the nature of the species to be sorbed and active sites of the PUF involved in bonding.[7,8]

Flexible PUF is a three-dimensional network of polyurethane fiber in which the major part (90%) has air holes that act as macropores and the surface area of PUF fiber acts as a micropores surface when mass transfer of solution is slow. In previous studies, the sorption of Au(III),[9] Ag(I),[10] Pd(II),[11] and Fe(III)[12] indicates that the mechanisms of sorption and transfer of the trace-metal ions from bulk aqueous solution to the solid surface of foam and from the outer surface to the interior surface of the foam using batch shaking technique is the adsorption phenomena that takes place at the active sites of PUF through physical or chemical interaction. This paper reports the results of the sorption behavior of molecular iodine on unloaded PUF in detail.

EXPERIMENTAL

All reagents used in this work were of analar grade. Buffer solutions of pH 4, 7, and 10 where prepared by mixing the appropriate amounts of 0.2 M CH_3COONa and CH_3COOH, Na_2HPO_4, and NaH_2PO_4, and H_3BO_3, KCl, and NaOH, respectively. The ionic strength of all buffer solutions was kept at 0.1 M by diluting with deionized water and stability was checked periodically on Metrohm pH type 605, Metrohm Ltd., Switzerland. The surface area and average pore size of the foam were determined by absorbing nitrogen and laser microsizer, respectively.[13,14] The total intrusion volume was measured by mercury porosimeter.

The iodine solution was prepared by appropriate known weight of iodine followed by double weight of KI in deionized water and known volume of standard 6 M HCl to make acid strength 0.2 M HCl of the resultant solution in volumetric flask. The 0.02N sodium thiosulfate solution was prepared by dissolving known weight of sodium thiosulfate in deionized water in 500 ml volumetric flask.

Standardization of Sodium Thiosulfate Solution

The accurate known weight of K_2CrO_4 (10–25 mg) was dissolved in 50 ml of deionized water in a 250-ml conical flask followed by 1 g of KI and 1 ml concentration H_2SO_4. The liberated iodine was titrated with sodium thiosulfate solution, and when solution color came to pale yellow, 1 ml starch solution was added and again titrated, with the disappearance of the blue iodine–starch complex. The normality of the sodium thiosulfate solution was determined using the following reactions[15]:

$$Cr_2O_7{}^{-2} + 6I^- + 14H^+ = 3I_2 + 2Cr^{+3} + 7H_2O$$

$$I_2 + 2S_2O_3^{-2} = 2I^- + S_4O_6{}^{-2}$$

$$\text{Normality of sodium thiosulfate} = W(20.39)/V$$

where W is the amount of potassium dichromate in grams and V is the volume of sodium thiosulfate solution required for titration in milliliters.

Titration of Iodine Solution

The known volume of the iodine solution was taken in conical flask followed by 2 ml of concentrated HCl and titrated with standard sodium thiosulfate solution. When iodine color becomes light yellow, 2 ml starch solution was added and again titrated untill the disappearance of the blue starch–iodine complex. The concentration of the iodine solution was determined using the following reaction[16]:

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$$

Sorption Procedure

The 5 ml of iodine solution was taken in a 25 × 125 mm Pyrex culture tube. One ml was taken in conical flask and titrated with sodium thiosulfate solution using above procedure to determine the initial concentration of iodine (A_o). The remaining 4 ml solution was equilibrated with known weight (29 mg) of PUF on wrist-action shaker. After shaking, 1 ml aliquot was taken and again titrated with standard sodium thiosulfate solution and the concentration of iodine was determined (A_e).

For thermodynamics and kinetic studies, the iodine solution was taken in a culture tube and dipped in the Gallenkamp thermostirrer-100 model BKL 200, Fisons Scientific Equipments, England. water bath at constant temperature for at least 30 min to attain the required temperature and the same procedure was adopted as described above. During shaking, about 70% of the length of the culture tube remained dipped in the water bath so that solid–liquid contents were immersed completely in water at the controlled temperature. The percentage sorption and distribution coefficient was calculated using the following equations.

$$\%\text{Sorption} = \frac{\mathbf{A}_o - \mathbf{A}_e}{\mathbf{A}_0} \times 100$$

and

$$\mathbf{K}_d = \frac{\text{amount of iodine in foam}}{\text{amount of iodine in solution}} \times \frac{\text{volume of solution (V)}}{\text{weight of dry foam (W)}} = (\text{cm}^3/\text{g})$$

Iodine Number

Ten milliliters of 0.1 N iodine solution was taken in the 25 × 125 mm Pyrex culture tube and known weight of PUF was added and shaken for 1 min on wrist-action shaker. After shaking, the contents were filtered. The 5 ml of the filtrate was titrated against the 0.1 N sodium thiosulfate solution as described above. The weight of the foam varied till the normality of iodine in filtrate was in the range from 0.008 N to 0.033 N in order to apply the correction factor as described in the Wijs method.[17] The iodine number is calculated from the following equation:

$$\text{Iodine number} = F\left[\frac{(12.69)(10) - \frac{(\text{ml of } 0.1\,\text{N}\,Na_2S_2O_3)(12.69)(10\,\text{ml})}{5}}{\text{weight of foam, in gram}}\right]$$

where F is the correction factors for the iodine number test.

All experiments were performed at least in triplicate at ambient temperature or as specified otherwise. The results are the average of at least triplicate independent measurements and precision in most cases was ± 5%. The linear regression computer program was used for slope analysis and for the statistical treatment of the data. The correlation coefficient (r) for all regression analyses was in the range of 0.95 to 0.99.

RESULTS AND DISCUSSION

The sorption of iodine on PUF[5] from aqueous solution of different composition of different medium containing 0.01 N iodine is shown in Table 1. This table indicates that maximum sorption occurs from the 0.2 N hydrochloric acid solution and decreases with the increase in pH of buffer solutions. This decrease is due to the conversion of iodine to iodide and hypoiodide, which is rapid in alkaline solution [2]:

$$I_2 + 2OH^- = I^- + IO^- + H_2O$$

and was also affected by the presence of different anions. However, in deionized water, the sorption of iodine on PUF is higher as compared to the other buffer solutions used.

Iodine Number

The iodine number of an adsorbent is the weight of iodine in milligrams that can be adsorbed by a gram of adsorbent; when iodine concentration is reduced from 0.1 N to 0.02 N, it is termed the iodine number.[18] It provides a measure of surface area or capacity available to small adsorbents or lower

Table 1. Sorption of 0.01 N iodine from different aqueous solutions onto PUF.

Composition	Distribution coefficient K_d ($cm^3\,g^{-1}$)	Percent adsorption (%E)
pH 4	133	44
pH 7	215	56
pH 10	75	32
H_2O	536	76
0.2 N HCl	3220	95

molecular weight substances. The determined iodine number and other different properties of polyurethane foam are listed in Table 2. The iodine number was found to be 436 ± 15 $mg\,g^{-1}$. Iodine number for different adsorbents range from about 300–1200 $mg\,g^{-1}$ and is also related to the microporisity of the adsorbent.[19] The high value indicates a highly microporous adsorbent. Usually polymeric adsorbents such as Amberlite, Dow XFS, Duolite, etc., have low iodine numbers (ranges 300–600 $mg\,g^{-1}$), while solid adsorbents such as activated carbon have very high iodine numbers (ranges 800–1200 $mg\,g^{-1}$) and sorption of different metal ion depends upon the iodine number of the adsorbents. The average pore diameter of PUF is in the range of the other synthetic polymeric adsorbents, but flexible PUF has low surface area (67 $m^2\,g^{-1}$) and porosity volume (14%) as compared to other polymeric adsorbents surface area ($>100\ m^2\,g^{-1}$) and porosity volume ($>32\%$).[19]

Effect of Shaking Time

The effect of equilibration time for the sorption of 0.01 N iodine solution in 0.2 N HCl on PUF has been evaluated. It was found that sorption increased with the increase in shaking time and maximum equilibration was attained within

Table 2. Different physical properties of polyether-type PUF.

Bulk density	22 $kg\,m^{-3}$
Iodine number	436 ± 15 $mg\,g^{-1}$
Total pore area	67 $m^2\,g^{-1}$
Total intrusion volume	0.14 $cm^3\,g^{-1}$
Average pore diameter	8.3 nm

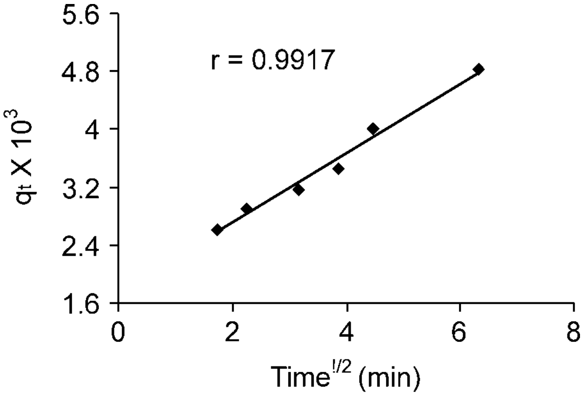

Figure 1. Weber–Morris equation for the sorption of 0.01 N iodine onto PUF from 0.2 N HCl aqueous solution.

30 min shaking time and remained constant up to 60 min shaking time. In order to evaluate the kinetics of diffusion rate, the Weber Morris[20] equation was applied and is expressed as:

$$q_t = R_d\sqrt{t}$$

where q_t is the amount of iodine sorbed on PUF at time "t" intervals. The plot of q_t vs. $t^{1/2}$, shown in Fig. 1, gives a straight line and the numerical value of R_d was found to be $0.48 \pm 0.03\,\mathrm{mmole\,g^{-1}\,min^{-1/2}}$ from the slope of the graph. However, the slope does not pass through the origin indicating that diffusivity of the solute is concentration-dependent rather than from any intrinsic mobility.

The kinetic data of the rate of mass-action process, which assumes that solute adsorbs on the surface of the particle of the foam according to first-order reaction, has been evaluated using the Lagergren equation in the form[21]:

$$\log(q_e - q_t) = \log q_e + \frac{-k_1 t}{2.303}$$

where q_e is the maximum amount of iodine adsorbed at equilibrium, q_t is the amount adsorbed at time "t," and k_1 is the rate constant of the adsorption process. Plot of log $(q_e - q_t)$ vs. t results in a straight line (Fig. 2), indicating that the sorption process is a first-order reaction with respect to the adsorbed concentration. The value of k_1 was evaluated and found to be $52 \pm 8\,\mathrm{mmole\,g^{-1}\,min^{-1}}$. The kinetic data indicate that the rate of adsorption is much faster than the rate of diffusion so that equilibrium is obtained at each point inside the adsorbent.

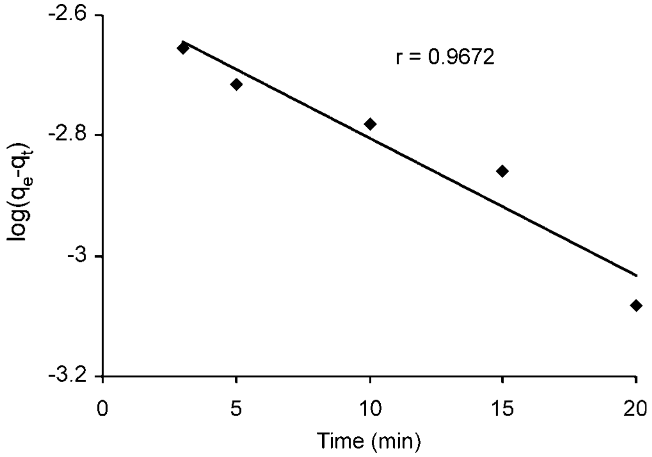

Figure 2. Lagergren equation of the adsorption of iodine onto PUF.

Adsorption Isotherms

The influence of iodine concentration on the sorption under the optimum condition of shaking time (30 min) and PUF weight (29 mg) was measured over the range of 0.01 N to 0.123 N of iodine solution. Figure 3 indicates that distribution coefficient (K_d) is very much affected by the ratio of sorbate concentration. The K_d values decrease from 3220 to 27 $cm^3 g^{-1}$ and percentage sorption is from 95% to 12%. At low concentration, there was a sharp decrease and then becomes almost asymptotic in nature. The decreased in the K_d is attributed to the decrease in the sorption sites and saturation of the active sites with iodine concentration. A similar trend observed for adsorption of Ag(I) on PUF.[10]

Adsorption is usually modeled by isotherms, which relate the relative concentrations of solute adsorbed onto the solid and in solution. The most commonly used isotherm in this regard is the Freundlich isotherm,[22] which gives an empirical expression encompassing the surface heterogeneity and exponential distribution of energy sites was tested in the following form:

$$\log C_{ads} = \log C_m + \frac{1}{n} \log C_e$$

where C_{ads} is the amount of sorbate in mole g^{-1}, C_e is the amount of sorbate in mole L^{-1} at equilibrium, and C_m and 1/n are the constants. The plot of $\log C_{ads}$

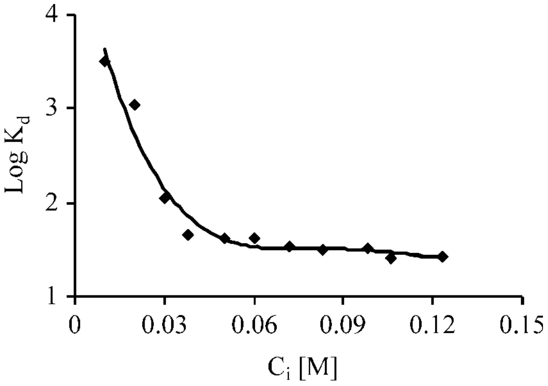

Figure 3. Variation of K_d with iodine concentration.

vs. logC_e is linear as shown in Fig. 4. The numerical values of C_m and 1/n are evaluated from the intercept and the slopes are 11.2 ± 1.7 mmole g^{-1} and 0.57 ± 0.05, respectively. The value of C_m can be taken as a relative indicator of adsorption capacity and 1/n is indication of the energy and intensity of the sorption.

The Langmuir model has also been verified. This isotherm represents the localized monolayer model based upon the assumptions that all the active sites are energetically equivalent with no interaction between sorbed species and independent of surface coverage. The Langmuir model was tested

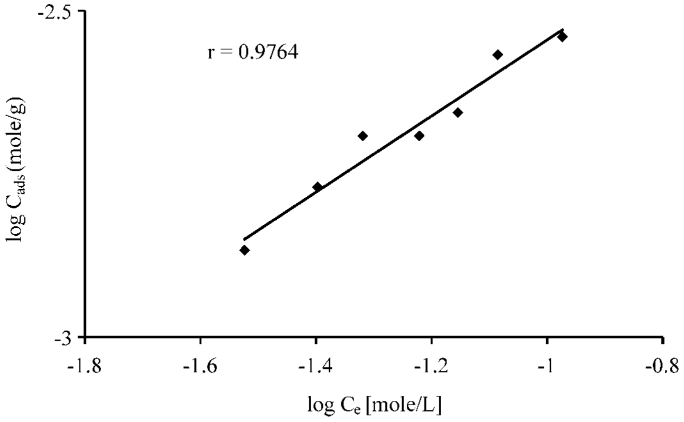

Figure 4. Plot of Freundlich isotherm of the sorption of iodine onto PUF.

in the form[23]:

$$\frac{C_e}{C_{ads}} = \frac{1}{Mb} + \frac{C_e}{M}$$

where C_e is the equilibrium concentration of iodine in solution (mole L^{-1}), C_{ads} is the sorbed concentration of iodine (mole g^{-1}), and M and b are constants related to the maximum amount of solute sorbed and to the binding energy of solute, respectively.

The plot of C_e/C_{ads} vs. C_e is shown in Fig. 5. The straight line indicates the monolayer-type sorption. The numerical value of M and b evaluated from the slope and intercept are 4.9 ± 0.6 mmole g^{-1} and 12.9 ± 1.3 L g^{-1}, respectively. The saturation capacity M corresponds to the monolayer coverage and should, therefore, be substantially independent of temperature. However, the adsorption coefficient b is related to the enthalpy of the adsorption and should vary with temperature.

In order to distinguish between physical and chemical adsorption, the Dubinin–Radushkevich (D–R) isotherm[24] was also checked. This isotherm postulates on "adsorption space" close to the adsorbent's surface where adsorption occurs and relates pore filling to the free energy of adsorption. This model features the surface heterogeneous surface of the adsorbate as in Freundlich isotherm. The linear form of D–R isotherm is:

$$\ln C_{ads} = \ln X_m - B\varepsilon^2$$

where C_{ads} is the amount of solute adsorbed in mole g^{-1}, X_m is the maximum amount of solute adsorbed on PUF and activity coefficient B is a constant with

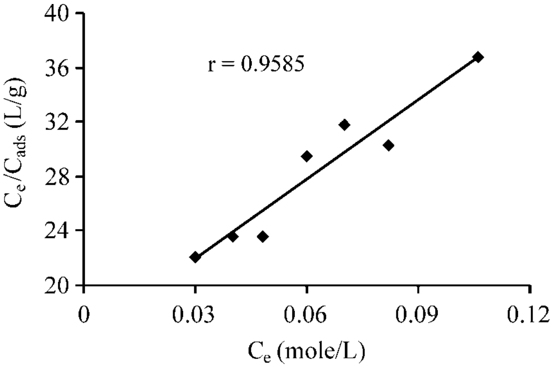

Figure 5. Plot of Langmuir isotherm of the sorption of iodine onto PUF.

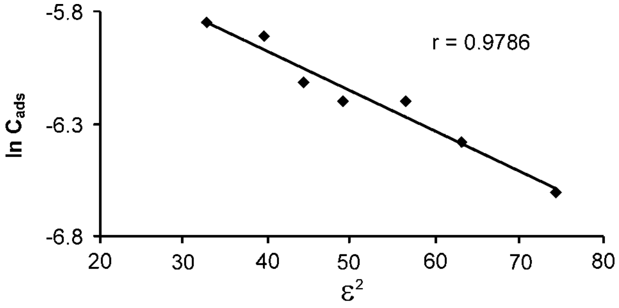

Figure 6. D–R isotherm profile of iodine sorption onto PUF.

dimension of energy and Polanyi potential $\varepsilon = RT\ln(1 + 1/C_e)$, where R is a gas constant in kJ mole^{-1} and T is the temperature in Kelvin.

The graph of $\ln C_{ads}$ vs. ε^2 gives a straight line as shown in Fig. 6. The values of X_m and B were found to be 5.15 ± 0.45 mmole g^{-1} and -0.01775 ± 0.00166 mole^2 k^{-1}J^{-2}, respectively, from the intercept and slope of the graph.

If the surface is heterogeneous and an approximation to a Langmuir isotherm is chosen as the local isotherm,[25] than the affinity coefficient B is related to the mean sorption energy (E) as:

$$E = \frac{1}{\sqrt{-2B}}$$

The sorption energy (E) is defined as the free energy of transfer of one mole of solute from infinity in solution to the surface of PUF. The numerical value of E was evaluated and found to be 5.3 ± 0.2 kJ mole^{-1} and reflects the physiosorption[26] of iodine on PUF.

Thermodynamics Studies

The effect of temperature on the sorption of iodine on PUF has been investigated using the following equations;

$$\log K_c = \frac{-\Delta H}{2.303RT} + \frac{\Delta S}{2.303R}$$

$$\Delta G = -RT\ln K_c$$

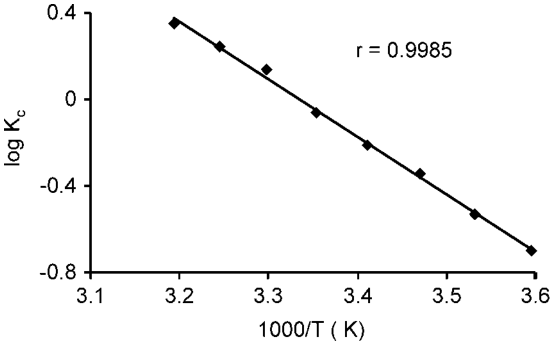

Figure 7. Temperature effect of the sorption of molecular iodine onto PUF.

$$K_c = \frac{F_e}{1 - F_e}$$

where K_c is the equilibrium constant depending upon the fractional attainment of the sorption (F_e) at equilibrium, and ΔH, ΔS, ΔG, R, and T are the enthalpy in $\mathrm{kJ\,mole^{-1}}$, entropy in $\mathrm{J\,mole^{-1}\,K^{-1}}$, Gibbs free energy in $\mathrm{kJ\,mole^{-1}}$, gas constant in $\mathrm{J\,mole^{-1}}$, and temperature in Kelvin, respectively. The linear plot of log K_c vs. 1/T is shown in Fig. 7. The values of ΔH and ΔS evaluated from the slope and intercept are found to be $50 \pm 2\,\mathrm{kJ\,mole^{-1}}$ and $170 \pm 4\,\mathrm{J\,mole^{-1}\,K^{-1}}$, respectively. The positive value of ΔH indicates the endothermic sorption behavior. The Gibbs free energy, ΔG, ranges from 3.72 to $-2.15\,\mathrm{kJ\,mole^{-1}}$ reflect that adsorption is spontaneous at higher temperature. However, it was observed that sorption of iodine on PUF is temperature dependent. The graph is linear in the temperature range 278 K to 313 K, but at higher 313 K, it is independent of temperature.

Sorption Mechanism

The solubility of iodine in aqueous solution is very low, but in the presence of potassium iodide, its solubility increases many times due to the formation of water-soluble I_3^- complex. The nature of the species to be sorbed on PUF is either I_2 or I_3^- and IO_3^- can be identified using

TLC method.[27] The carrier free radioactive solution of iodine-131 in the form of iodide ($*I^-$) was mixed with iodine solution. The radioactive iodide exchanged the molecular iodine immediately by the following reaction:

$$*I^- + I_2 = *I_2 + I^-$$

The active solution was loaded on 10 × 50 mm PUF column. After thorough washing with 0.2 N HCl and deionized water, the loaded radioactive iodine was eluted with acetone ($10\,cm^3$) and run on TLC with standards. After comparing the R_f values, it was observed that loaded iodine is free from any traces of iodide or iodate. This indicates that the sorbed iodine on PUF is in form of molecular iodine (I_2).

The violet color of I_2 is attributed to the absorption of a lone pair of electrons of a vacant orbital of I_2 molecules. Thus, it is readily soluble in the solvents having lone pair, of electrons forming as $S—I_2$ donor acceptors or charge-transfer complexes.[28] The color intensity of iodine depends upon the separation of allowed transition. The I—I bond has been weakened as expected when there is partial donation to the antibonding orbital from any strong electron pair donor such as ROH, R_2O, RNH_2, and so on.[28,29]

The amino group and carbonyl group of PUF are quite strong donor groups that donate lone pairs of electrons to the iodine during sorption process and need some energy, which is observed from thermodynamic studies.

The sorption energy (E), derived from D-R isotherm indicates that physical adsorption is operating during the adsorption process. Moreover, the thermodynamics studies indicate that sorption is independent at higher temperature 313 K. The physical adsorption results from the action of Van der waal's forces comprised on two effects.[30] First, the induction effect, which is caused by polarization of molecules and independent of the temperature. The net magnitude of the dipole–dipole attraction has been correlated to the polarization and magnetic susceptibilities of the molecules involved.[31] The second effect is the orientation effect, which is significant for the mutual interaction of molecules that have permanent dipoles, and magnitude of interaction is temperature-dependent and requires energy. The high values of enthalpy and positive entropy of sorption indicate that the iodine molecules are in a more ordered form and have strong interactions with the active sites of PUF. Iodine is highly polarizable and during sorption, the electron distribution of iodine molecules and nitrogen and oxygen donor groups of PUF undergo some distortion in mutual proximity; however, the electrons maintain their association with the original nuclei.

Table 3. Effect of different anions and cations on the sorption of iodine on PUF from aqueous solution of 0.2 N HCl.

Anions[a]	% sorption	Cations[b]	% sorption
Nil	95	Ni(II)	95
Sulfate	94	Cu(II)	96
Acetate	93	Cd(II)	94
Oxalate	94	Mn(II)	95
Citrate	95	Mg(II)	88
Nitrate	86	Al(III)	81
Tartrate	94	Cr(III)	94
Phosphate	82	Fe(II)	<5
Thiourea	<10	Fe(III)	90

[a] Sodium or potassium salts.

[b] Chloride or sulfate salts.

Diverse Ion Effect

The selectivity and sensitivity of the sorption of iodine on PUF under the optimum condition was also studied. The influence of common anion and cations play a vital role in the removal of particular ion of interest. A study was carried out to investigate the effect of common anions and cations of $10\,\mathrm{mg\,cm^{-3}}$ of salts on the sorption of iodine from 0.2 N HCl onto PUF. The results are listed in Table 3. This table indicates that thiourea completely masked the adsorption due to oxidation of iodine, while phosphate and nitrate slightly reduces the sorption of iodine. Among the common cations, only Fe(II) interference was due to oxidation–reduction reaction. However, Al(III) and Mg(II) slightly reduce the sorption of iodine on PUF.

CONCLUSION

Polyurethane foam is a cheaper and more easily available material and the developed method can be used for the separation and purification of molecular iodine on a large scale from natural brines. The purification and preconcentration of iodine onto PUF is cheaper and has more advantages than vaporization and sublimation process in the production of iodine.

The kinetic data reflect the intraparticle diffusion process involved in adsorption of iodine on PUF. The sorption of iodine follows the classical

Freundlich, Langmuir, and D–R adsorption isotherms. The low value of sorption free energy (E) indicates the physiosorption phenomena is predominant for the accumulation of molecular iodine onto PUF. The temperature study shows that adsorption is endothermic and spontaneous at higher temperature. Moreover, diverse, ion effect indicates that thiourea and Fe(II) reduces the adsorption of elemental iodine significantly.

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