

# Sorption And Kinetics Study Of Potassium Chloride In Polyvinyl Alcohol-Borate Hydrogel

Rehana Saeed, Zain Ul Abdeen

**Abstract:** Isotherms for the adsorption of KCl in PVOH-borate hydrogel was studied at 303 K by determining the amount of potassium and chloride ions adsorbed in PVOH-borate hydrogel using ion exchange chromatography and potentiometric titrations respectively. Experimental data was tested for Freundlich's, Langmuir's, Temkin's and Dubinin–Radushkevich isotherms equations. The adsorption data follows isotherms in order Freundlich's > Dubinin–Radushkevich > Temkin's. Kinetics of adsorption and desorption processes were studied using conductivity method. The applicability of pseudo first order, second order kinetics and intra particle diffusion kinetics were tested. From standard deviation,  $R^2$  and  $\chi^2$  values it can be concluded that sorption of KCl using PVOH-borate hydrogel as an adsorbent obey pseudo first order kinetics. Intra particle diffusion rate constant increases on enhancement in the contents of KCl. Obeying Temkin's isotherm and intra particle diffusion kinetics confirmed the presence of interaction between potassium ions and PVOH-borate hydrogel during sorption phenomenon.

**Index Terms:** Adsorption Isotherm, Conductometric studies, Desorption, Hydrogel, Kinetics, PVOH-borate

## 1 INTRODUCTION

POLYMERS bear magnificent quality to be modified into various materials possessing entirely new, modified and enhanced properties. Because of its feasible properties it plays a decisive role in the selection as an alternative for various materials [1], [2], [3], [4]. Polymers possesses ions diffusion properties which was subjected to its application as an ion diffusing membrane as well as separating membrane in solid state batteries and fuel cells [5], [6]. This indicates the presence of interactions for ions in the polymer moiety which facilitates the storage of charge within its structure. The interactions of charged particles can also be developed in polymer substrate using plasticizers and cross linkers. Cross-linking occurs in between polymer chains without polymerization process because of the functional reactivity between polymer and cross linking agent which is responsible for formation of cross-linked junction. Cross-linking in Polyvinyl alcohol (PVOH) chains using borax was studied by several researchers from different aspects which lead to understand the presence of various types of attractions for ionic substances [7], [8], [9], [10].

PVOH-borate form a hydrogel which contains certain amount of water and so the PVOH-borate hydrogel system has also implementation in the treatment of wound based on its adsorption & desorption characteristics from which drug delivery was carried out [11]. The interactions for ions with cross-linked units in hydrogel can be studied by adsorption isotherms and diffusion kinetics of sorption processes occurring in PVOH-borate hydrogel system. Collective study of Langmuir's, Freundlich's, Temkin's and Dubinin–Radushkevich adsorption isotherms are used to determine the nature of adsorption processes such as physisorption and chemisorptions, homogeneity and heterogeneity of adsorbent surface and various types of interactions responsible for the process of adsorption [12], [13], [14], [15]. Similarly sorption kinetics decides the dependency of sorption processes on adsorbent or adsorbate or both, and to conclude the mechanism of sorption kinetics [16], [17], [18], [19]. The objective of the present work is to investigate the presence of interaction for potassium and chloride ions in cross linked cage of PVOH-borate hydrogel through isotherms and kinetics using conductivity method, different adsorption isotherms are used for the adsorption of KCl in PVOH-borate hydrogel to investigate the mechanism of sorption for adsorption and desorption of KCl through PVOH-borate hydrogel.

## 2 MATERIAL AND METHODS:

All the glassware of Pyrex A grade quality were used. Double distilled water was used having conductivity  $0.06 \mu\text{S}\cdot\text{cm}^{-1}$ . PVOH E. Merck, (< 98 % hydrolyzed), having average molecular weight 65075.11 Da. Sodium tetra borate decahydrate borax ( $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) E. Merck, 98 % pure was used as cross linking agent. Potassium chloride (KCl), Silver nitrate ( $\text{AgNO}_3$ ), Sodium hydroxide (NaOH), Oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ) and Hydrochloric acid (HCl) E. Merck, (99 % pure), were used without further purification. Stock solution of PVOH was prepared by dissolving required amount  $\text{g}\cdot\text{dL}^{-1}$  of PVOH in double distilled water at  $(353 \pm 1 \text{ K})$ . Stock solution of potassium chloride is prepared and dilutions in the range of  $20 \text{ g}\cdot\text{L}^{-1}$  –  $80 \text{ g}\cdot\text{L}^{-1}$  was prepared using double distilled water. Hydrogel was prepared by addition of calculated volume of sodium borate in particular volume of PVOH in beaker. The contents were than stirred using glass rod. Thick and adhesive hydrogel was obtained; it is allowed to settle down for 30 minutes so that air bubbles would be removed. Similarly different composition of hydrogel was prepared by varying

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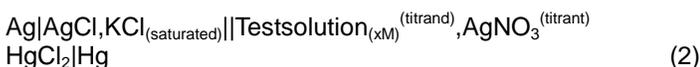
amount of PVOH and sodium borate.  $4.0 \pm 0.05$  g of the prepared hydrogel is weight in Sintered glass cell E. Merck capacity 30 mL, using a weight balance model BL-150s, Sartorius, Germany having least count  $\pm 0.001$  g. hydrogel is allowed to settle for 10 minutes so that smooth and uniform surface is obtained. For studying adsorption kinetics at 303 K,  $25 \pm 0.1$  mL potassium chloride was used, adsorption was allowed for 120 minutes using different concentrations  $20 \text{ g.L}^{-1} - 80 \text{ g.L}^{-1}$  of KCl in different percentages of PVOH in cross linked PVOH-borate hydrogel. Specific conductivity of potassium chloride at different time intervals were measured by using a digital Jenway, 4510 conductivity meter having accuracy  $\pm 0.5$  % was connected with electrode having cell constant  $1.0 \text{ cm}^{-1}$ . For determining the amount of potassium and chloride ions adsorbed in hydrogel, potentiometer Kent EIL 7020, having range 1500mV and least count 20mV is used for chloride ions while for potassium ions; cation exchange chromatography is carried using cation exchanger sodium benzene sulphonate E. Merck was used. For observing desorption kinetics,  $20.0 \pm 0.1$  mL de-ionized water was taken in a glass cell and hydrogel with adsorbed potassium chloride was soaked in water and conductivity of water was observed for 60 minutes with 5 minutes intervals.

### 3 RESULTS AND DISCUSSIONS

A Polyvinyl alcohol-borate has ability to adsorbed ions as cross-linking develops spaces between the cross linked chain and excess charge on boron having the attraction for cations [20]. As there is the possibility of adopting different mechanism for the adsorption of cations and anions, after adsorption remaining concentration of both cations & anions were determine separately

$$C_i = C_{K^+} + C_{Cl^-} \quad (1)$$

The amount of chloride ions " $C_{Cl^-}$ " absorbed was determined by potentiometric titrations by titrating the samples with  $\text{AgNO}_3$  using configuration as shown in (2).



End point of potentiometric titrations was determined by first derivative plot from which concentrations are measured. The amount of potassium ions " $C_{K^+}$ " absorbed was determined by volumetric analysis for which cation exchange chromatography was carried out. The exchange process can be expressed as in equation (3)



The amount of KCl adsorbs in hydrogel at 303 K was determine using relation (4).

$$q_e = \frac{V}{M} \cdot (C_i - C_e) \quad (4)$$

Where, " $q_e$ " is amount absorbed at particular concentration. " $C_i$ " and " $C_e$ " are the concentration of electrolyte initially and at equilibrium respectively. " $M$ " is the mass of hydrogel and " $V$ " is the volume of electrolyte used. It was observed that with the

enhancement in the concentration of KCl in adsorbate solution, the amount of KCl uptake by hydrogel increases. This is due to the fact that more ions were available for the sorption phenomenon. At different contents of PVOH in hydrogel, there was decreased in the amount adsorbed by hydrogel for each concentration of KCl; this can be explained as polymer moieties decreases which results in decrement in the number of cross linked units in hydrogel which also reduces the thickness of hydrogel. Thus, there were lesser interactions for potassium and chloride ions offered by cross linked PVOH-borate hydrogel. The amount of KCl adsorbed from different concentrations of KCl dilutions ion different composition of hydrogel by varying concentration of PVOH was summarized in Table 1.

**TABLE 1: Amount Of KCl Adsorbed by PVOH-borate Hydrogel having Different Contents of PVOH at 303 K.**

Concentration of KCl ( $\text{g.L}^{-1}$ )	Amount adsorbed ( $\text{mg.g}^{-1}$ )		
	Concentration of PVOH ( $\text{g.dL}^{-1}$ ) + 0.05M sodium borate		
	5.0	4.0	3.0
20	73.948	55.930	40.572
30	110.93	89.675	71.887
40	143.12	124.90	94.950
50	169.98	155.52	111.09
60	221.86	194.89	157.77
70	233.86	216.34	184.90
80	262.90	240.68	206.79

Experimental adsorption data for adsorption process, were analyzed by different adsorption isotherms. Freundlich's, Langmuir's, Temkin and Dubinin-Radushkevich (D-R) adsorption isotherm equations were applied to experimental data. These isotherm equations were respectively shown by the relations (5), (6), (7), (8).

$$\ln q_e = \ln k_f + \frac{1}{n} \cdot \ln C_e \quad (5)$$

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (6)$$

$$q_e = \frac{Rt}{b} \ln K_T + \frac{RT}{b} \ln C_e \quad (7)$$

$$\ln q_e = \ln q_d - \beta R^2 T^2 \ln \left[ 1 + \frac{1}{C_e} \right]^2 \quad (8)$$

Where, " $q_e$ " is amount absorbed at particular concentration. " $C_e$ " is the concentration of electrolyte at equilibrium, " $k_f$ " and  $n$  are Freundlich's constants representing sorption capacity and sorption intensity respectively, " $q_m$ " and " $K_L$ " are langmuir's constants, showing maximum sorption capacity and affinity of binding sites respectively. " $b$ " and " $K_T$ " is Temkin constant for heat of adsorption and Temkin isotherm constant, while " $q_d$ " and " $\beta$ " are D-R constant and free energy constant respectively. On evaluation of data using adsorption isotherms, it was observed that the adsorption process follows

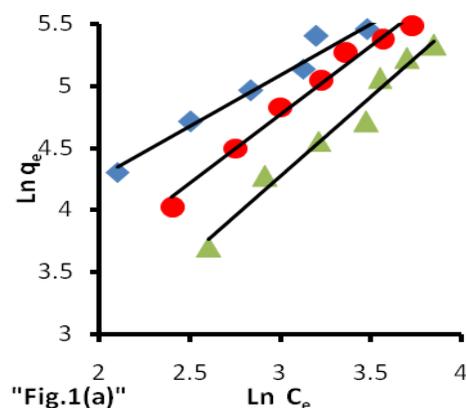
Freundlich's, Temkin's and Dubinin–Radushkevich isotherms as linear plot of these isotherms have linear regression coefficient  $R^2$  nearly 1.00 but adsorption data does not obey Langmuir's isotherm equation. On the basis of  $R^2$  values it can be summarized that adsorption data follows adsorption isotherms in order Freundlich's > Dubinin–Radushkevich > Temkin's, and mostly found fit for Freundlich's adsorption isotherm. The deviation of data from Langmuir's isotherm and following Freundlich's isotherms was attributed due to the fact that the surface of PVOH-borate hydrogel is heterogeneous and there was the possibility of both physisorption and chemisorptions but, as adsorption data also follows Dubinin–Radushkevich isotherm therefore it can be concluded that

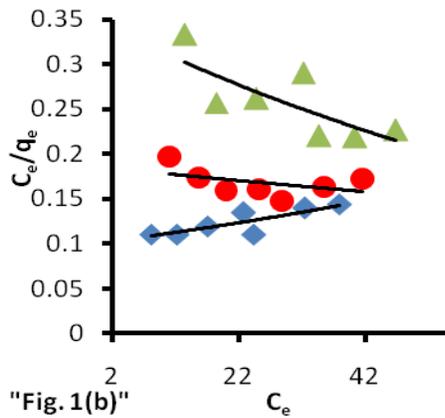
physisorption is most likely to be adsorption process occurring for the adsorption of KCl in PVOH-borate hydrogel. Temkin's isotherm reveals the fact that there was an interaction between KCl and PVOH-borate hydrogel that was responsible for the adsorption of KCl in hydrogel. The effect of concentration of PVOH on adsorption process that be summarized as on decreasing the contents of PVOH in PVOH-borate hydrogel sorption intensity, sorption capacity and heat of adsorption decreases. Thus it focuses on the fact that adsorption process is favorable at higher concentrations of PVOH in PVOH-borate hydrogel. Adsorption isotherms constant for different concentration of PVOH are shown in Table 2.

**TABLE 2: Adsorption Isotherm Parameters of KCl in Hydrogel having Different Concentration of PVOH At 303 K.**

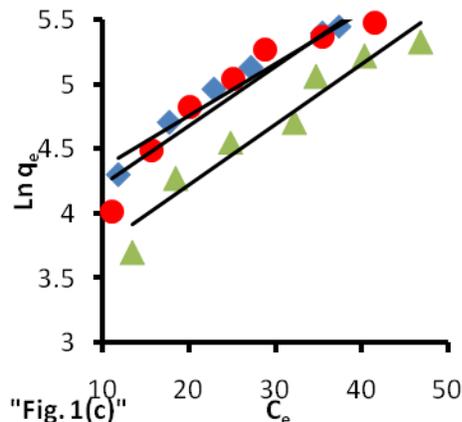
Freundlich's Adsorption Isotherm			
Composition <sup>a</sup>	$K_F$	$N$	$R^2$
5.0	13.57	1.209	0.975
4.0	4.087	0.892	0.981
3.0	1.541	0.781	0.972
Langmuir's Adsorption Isotherm			
Composition <sup>a</sup>	$K_L(\text{L.mg}^{-1})$	$q_m(\text{mg.g}^{-1})$	$R^2$
5.0	0.010	1000	0.771
4.0	-0.005	-1000	0.557
3.0	-0.005	-500	0.580
Temkin's Adsorption isotherm			
Composition <sup>a</sup>	$K_T(\text{L.g}^{-1})$	$b \times 10^{-4}(\text{J.mol}^{-1})$	$R^2$
5.0	11.17	5.778	0.896
4.0	12.16	5.024	0.898
3.0	8.832	5.024	0.936
Dubinin–Radushkevich			
Composition <sup>a</sup>	$q_d(\text{mg.g}^{-1})$	$\beta \times 10^{+6}$	$R^2$
5.0	351.0	1.317	0.957
4.0	412.8	2.218	0.991
3.0	268.8	1.320	0.947
<sup>a</sup> g.dL <sup>-1</sup> + 0.05M sodium borate			

The representative plots for different adsorption isotherms are shown in "Figs." "1(a)", "1(b)", "1(c)" and "1(d)" (a) Plot for Freundlich's Adsorption Isotherms (b) plot for Langmuir's Adsorption Isotherms (c) plot for Temkin's Adsorption Isotherms and (d) Dubinin–Radushkevich for 20 g.L<sup>-1</sup> -80 g.L<sup>-1</sup> KCl in different concentrations of PVOH ◆ 5 g.dL<sup>-1</sup> PVOH ● 4 g.dL<sup>-1</sup> PVOH ▲ 3 g.dL<sup>-1</sup> PVOH in PVOH-borate hydrogel.

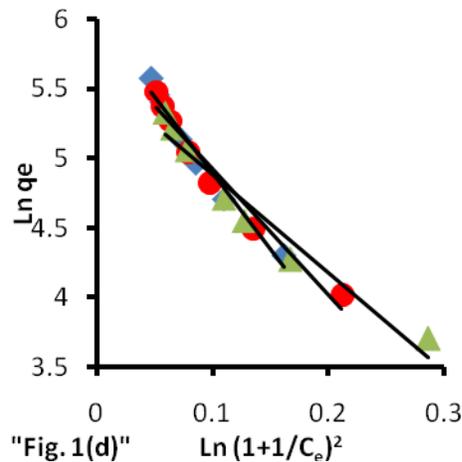




"Fig. 1(b)"

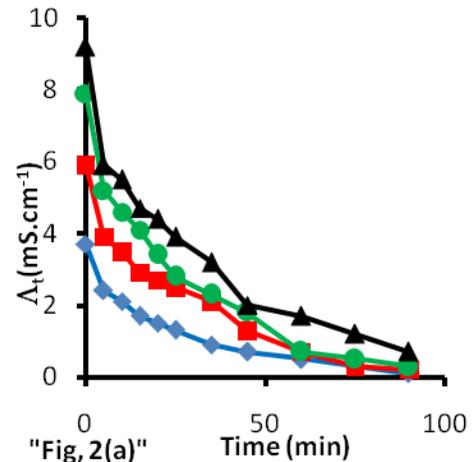


"Fig. 1(c)"

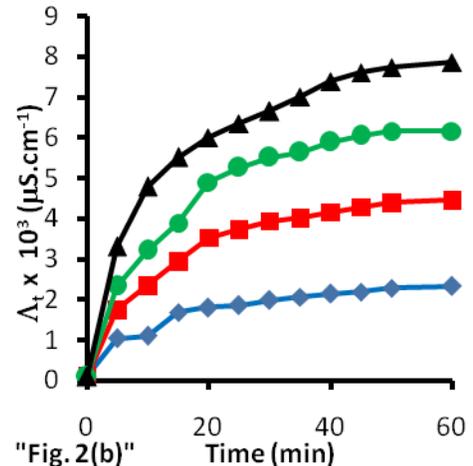


"Fig. 1(d)"

borate hydrogel at 303K in different concentrations  $\blacklozenge$  20 g.L<sup>-1</sup>  $\blacksquare$  40 g.L<sup>-1</sup>  $\bullet$  60 g.L<sup>-1</sup>  $\blacktriangle$  80 g.L<sup>-1</sup> of KCl.



"Fig. 2(a)"



"Fig. 2(b)"

The kinetics of sorption process that is adsorption and desorption, cleaves the way to decide the dependency of adsorption process on adsorbate or adsorbent or both. It also helps to determine the reversible or irreversible nature of sorption phenomenon. The kinetics study of KCl in PVOH-borate hydrogel is carried out using conductometric analysis. For adsorption and desorption data was collected in terms of specific conductance with respect to time intervals. The average rate of reaction for both adsorption and desorption processes increase with increase in concentration of KCl in adsorbate solution. The graphical representation of kinetics data at different concentrations of KCl was as expressed in "Figs." "2(a)" and "2(b)". (a) kinetics of adsorption of KCl and (b) kinetics of desorption of KCl in 5 g.dL<sup>-1</sup> + 0.05M Sodium

The experimental data was analyzed by kinetics equations for determination of order, intra particle diffusion rate equations were applied to data and validity of these equations for sorption data was investigated. For adsorption kinetics the equations utilized were summarized in relation (9), (10), (11).

$$\ln(\Lambda_t - \Lambda_c) = \ln(\Lambda_i - \Lambda_c) - k_A 't \tag{9}$$

$$\frac{1}{(\Lambda_t - \Lambda_c)} = \frac{1}{(\Lambda_i - \Lambda_c)} + k_A ''t \tag{10}$$

$$(\Lambda_t - \Lambda_c) = k_{id} t^{1/2} + c \tag{11}$$

While for studying desorption kinetics following equations were used given in relations (12), (13), (14)

$$\ln(\Lambda_m - \Lambda_t) = \ln(\Lambda_m - \Lambda_o) - k_D 't \tag{12}$$

$$\frac{1}{(\Lambda_m - \Lambda_t)} = \frac{1}{(\Lambda_m - \Lambda_o)} + k_D ''t \tag{13}$$

$$\Lambda_t = k_{id}t^{1/2} + c \tag{14}$$

Where, “ $\Lambda_t$ ” is the conductivity of solvent (KCl in adsorption and double distilled water in desorption) at time “ $t$ ” in which sorption studies is carried out, “ $\Lambda_o$ ” is the initial conductivity of double distilled water before desorption while “ $\Lambda_i$ ” is the initial conductivity of KCl solution before adsorption “ $\Lambda_m$ ” is the final conductivity of water after complete desorption and “ $\Lambda_c$ ” is the saturation conductivity of absorption process at equilibrium.  $k$ ,  $k'$  and  $k_{id}$  are rate constant for pseudo first order, second order and diffusion kinetics. The rate constant obtained by integrated equations and from graphical plots of data. On the basis of standard deviations  $\sigma$ , linear regression constant  $R^2$  and Chi-square test  $\chi^2$  values the applicability of particular model on kinetic data were analyzed.  $\chi^2$  values were obtained using relation (15)

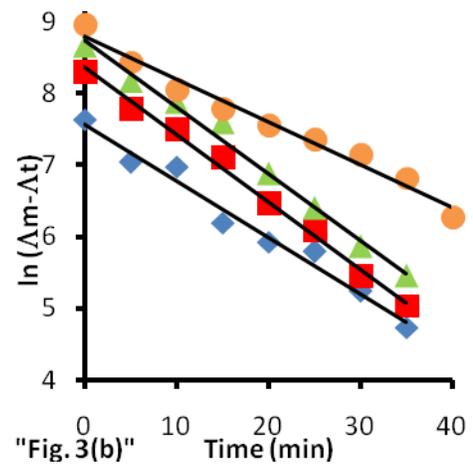
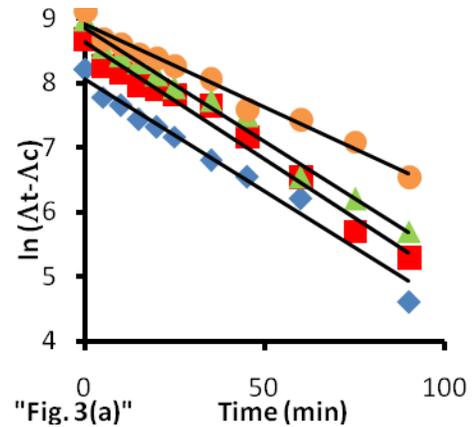
$$\chi^2 = \sum \frac{(K_{gra} - k_{int})^2}{K_{gra}} \tag{15}$$

It was concluded that adsorption and desorption process both obeys pseudo first order kinetics as  $\sigma$  and  $\chi^2$  values of data were small and  $R^2$  was approaching unity. The second order kinetic model was not completely applicable as standard deviations and  $\chi^2$  values was high for second order kinetic fittings. Thus, adsorption process directly depends upon the concentration of KCl present in adsorbate solution. Similarly, desorption of KCl from PVOH-borate hydrogel has direct relation with the concentration of KCl adsorbed in it. Experimental and model values along with  $\sigma$ ,  $R^2$  and  $\chi^2$  values of pseudo first order and second order rate constant were reported in Table 3.

**TABLE 3: Amount Of KCl Adsorbed by PVOH-borate Hydrogel having Different Contents of PVOH at 303 K.**

Proces s	$K_{exp} (\text{min}^{-1}) \pm \sigma$	$K_{mod} (\text{min}^{-1}) \pm \sigma$	$R^2$	$\chi^2 \times 10^3$
Pseudo First Order Kinetic Equation				
Adsorption	0.0365±0.0027	0.0321±0.0048	0.976	4.85
Desorption	0.083 ± 0.0106	0.080 ± 0.0206	0.980	0.76
Proces s	$K_{exp} (\text{cm mS}^{-1}\text{min}^{-1}) \pm \sigma$	$K_{mod} (\text{cm mS}^{-1}\text{min}^{-1}) \pm \sigma$	$R^2$	$\chi^2$
Second Order Kinetic Equation				
Adsorption	0.0178 ± 0.088	0.0377 ± 0.022	0.840	0.075
Desorption	0.082 ± 0.058	0.107 ± 0.072	0.865	0.010

For adsorption and desorption process, representative linear fittings for pseudo first order kinetics is represented in “Figs.” “3(a)” and “3(b)” respectively. (a) Adsorption (b) Desorption in different concentrations  $\blacklozenge$  20 g.L<sup>-1</sup>  $\blacksquare$  40 g.L<sup>-1</sup>  $\blacktriangle$  60 g.L<sup>-1</sup>  $\bullet$  80 g.L<sup>-1</sup> of KCl

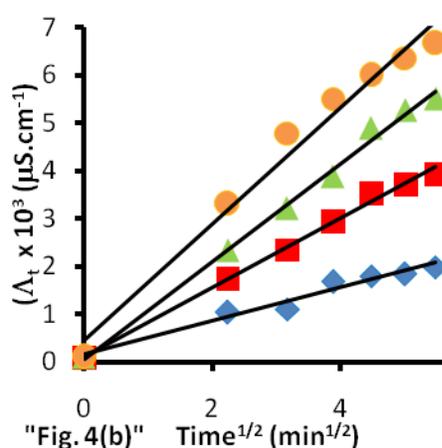
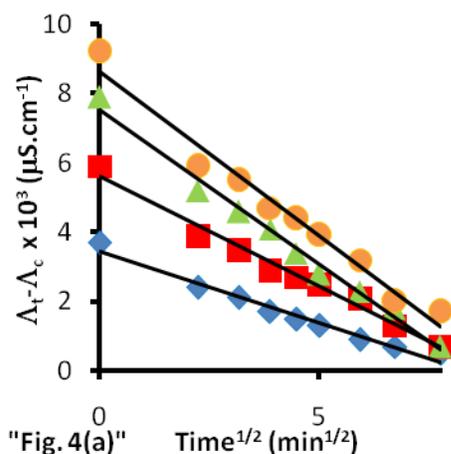


Intra particle diffusion rate constant describe the mechanism of sorption process that is to investigate the possibility of one of these diffusion mechanisms. (a) Internal diffusion in which sorbate may be penetrating the pores of adsorbent.(b) External diffusion that is sorbate can only reach surface of adsorbent and sorption occurring through the surface of adsorbent. (c) External cum internal diffusion which can be explained that sorbate adsorbed through the surface of adsorbent and reached pores and capillaries as well. An Intra particle diffusion rate constant increase with the enhancement in the concentration of KCl which indicated that adsorption of KCl in hydrogel and desorption of KCl from hydrogel was favorable at higher concentrations of KCl. From the values of  $R^2$  which were closest to unity urged towards the fact that both sorption process obey intra particle diffusion mechanism that is sorption of KCl in PVOH-borate hydrogel obeys mechanism (c). The intercept of linear plot is showed the fact that initially mass transfer is fast which slows down as time proceeds. Thus, rate of sorption mainly depends upon external diffusion that is the adsorption occurring on the surface of PVOH-borate hydrogel. The values intercepts increases as contents of KCl increases thus mass transfer is high at higher concentrations of KCl. Intra particle sorption rate constants for different concentrations 20 g.L<sup>-1</sup> – 80 g.L<sup>-1</sup> KCl in PVOH-borate hydrogel were shown in Table 4.

**TABLE 4: Amount Of KCl Adsorbed by PVOH-borate Hydrogel having Different Contents of PVOH at 303 K.**

Concentration of KCl (g.L <sup>-1</sup> )	Adsorption		Desorption	
	$K_{id}(\mu\text{S}\cdot\text{min}^{-1/2})$	$R^2$	$K_{id}(\mu\text{S}\cdot\text{min}^{-1/2})$	$R^2$
20	413.0	0.975	333.0	0.953
30	512.0	0.960	476.0	0.982
40	636.0	0.983	685.1	0.982
50	737.0	0.976	779.8	0.973
60	867.0	0.978	971.6	0.983
70	944.0	0.992	1072.0	0.979
80	1058.0	0.974	1150.0	0.956

The representative plot for intra particle diffusion rate constant for sorption process at various concentrations of KCl were shown in "Figs." "4(a)" and "4(b)". (a) Intra particle diffusion rate equation plot for adsorption and (b) Intra particle diffusion rate equation plot for desorption in different concentrations  $\blacklozenge$  20 g.L<sup>-1</sup>  $\blacksquare$  40 g.L<sup>-1</sup>  $\blacktriangle$  60 g.L<sup>-1</sup>  $\bullet$  80 g.L<sup>-1</sup> of KCl.



PVOH-borate hydrogel is cross linked cage with excess negative charge on boron as represented [7], [9], [20], [21] which indicates the presence of ionic interactions between electropositive ions and PVOH-borate hydrogel. Thus, the interactions indicated by Temkin's isotherms and applicability of intra particle diffusion mechanism suggested that potassium ions was interacted by weak vanderwaals forces which was responsible for the penetration of potassium ions into inner

surface of PVOH-borate hydrogel.

#### 4 CONCLUSION

Adsorption of KCl in PVOH-borate hydrogel was favorable at higher concentrations of KCl and obeys Freundlich's isotherms more than D.K's Temkin's isotherms which suggested that the adsorption is physisorption which was due to the interaction between adsorbate and adsorbent. Kinetically adsorption and desorption of KCl in PVOH-borate hydrogel follows pseudo first order and intra particle diffusion kinetics. Potassium ions have an interaction for PVOH-borate hydrogel which was responsible for the presence of interactions as indicated by Temkin's isotherms and confirmed the intra particle diffusion kinetics mechanism.

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