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Kinetics of Fluoride Removal from Groundwater Using MgO

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Abstract: Using MgO (light) the adsorption of fluoride from fluoride contaminated groundwater samples from three tube wells of the villages of Pilaniyon Ki Dhani and Kumaron Ki Dhani, a sub-village of Shola in Lakshmangargh Tehsil of the Sikar District of Rajasthan, India has been studied. The pseudo first order adsorption, pseudo second order adsorption, Elovich and intraparticle diffusion models have been used to fit the fluoride adsorption capacity (q_t) data. A very high correlation coefficient for all the tube wells (R^2 = 0.997, 0.996, 0.997) were found using the pseudo second order rate equation. Using the constants derived from the least square regression plots, the modeled fluoride adsorption capacity (q_t) has been calculated from the various equations. The sum of square error calculations of the calculated q_t values is minimum for the Elovich equations. In the initial period of contact of MgO with the fluoride contaminated ground water, the fluoride removal is mainly by intraparticle diffusion. The percentage of fluoride removal increases with increasing dosage of MgO while at the same time the adsorption capacity at equilibrium decreases. The minimum dosage of MgO which causes the maximum percentage removal of fluoride from water, while at the same time has the highest equilibrium adsorption capacity has been determined.

Keywords: MgO, adsorption, kinetics, groundwater.

1. Introduction

Drinking water in India is mainly sourced from rivers and from underground sources [1]. In the arid state of Rajasthan in India, fluoride is one of the major contaminants of ground water due to the presence of various fluoride bearing minerals underground [2]. The WHO recommends the permissible limit of fluoride in drinking water as 1.5 ppm [3]. Fluorosis is disease caused by drinking water contaminated with fluoride for prolonged periods [4]. Fluorosis is a disease of the muskulo-skeletal system in which fluoride attacks the calcium and magnesium rich tissues. The ground water of the Sikar District of Rajasthan contains fluoride beyond the permissible limits [5-7]. The majority of the population suffers from fluorosis with dark yellow streaks on their teeth. It has been observed that many local people suffer from pain in ankle, spine and general lethargy.

MgO is known to absorb fluoride from water [8-10]. Defluoridation filters using $MgO-CaO-CaCl_2$ have also been reported [11-14]. A project sponsored by the Department of Science and Technology, New Delhi, GOI involves adaption of the $MgO-CaO-CaCl_2$ fluoride filter [15-17], and in this context the kinetics of fluoride removal from local fluoride contaminated ground water using MgO has been studied.

In this chapter we report the case study of kinetics of fluoride adsorption using MgO from the ground water from three tube wells (T.W's) of a nearby fluorosis affected sub-villages of Pilaniyon Ki Dhani and Kumaron Ki Dhani in the village of Shola, Lakshmangarh Tehsil of Sikar District in Rajasthan, India.

2. Experimental

Fluoride Ion Selective Electrode and meter (Orion Thermo Scientific, USA) was used to measure the concentration of fluoride after calibration using 1, 10 ppm standard fluoride solution. To control the ionic strength and to de-complex the fluoride TISAB-III was used. pH and total dissolved solids meter were used to measure the pH and total dissolved solids respectively. The total dissolved solids meter was calibrated by using 1000 ppm KCl solution and the pH meter was calibrated using buffer solution of pH = 7. Titration with silver nitrate (0.0268 N) was used to determine the chloride ion concentration. The determination of Ca^{2+} and Mg^{2+} hardness was done by 0.02 N EDTA titration using Eriochrome Black-T and Patton and Reeder indicators (AR, CDH India Ltd). Titration (0.02 N H_2SO_4) using phenolphthalein and methyl orange indicators were used to determine the carbonate and bicarbonate concentration respectively. Shimadzu 1800 UV-VIS Spectrophotometer was used to analyze nitrate an absorbance at two wavelengths (220 nm and 270 nm). Flame Photometer (ESICO, India) was used to determine sodium and potassium. The instrument was calibrated by using 20 ppm K^+, for potassium and 20, 50 ppm Na^+ solution for sodium. Turbidity method using $BaCl_2.2H_2O$ (Qualigens India Ltd.) was used to determine the sulphate concentration using a photo-colorimeter (Instrument India Ltd.) and measuring the absorbance at 420 nm.

All shaker experiments were done in duplicate. 100 mL of fluoride contaminated groundwater from three T.W's, Pilaniyon Ki Dhani (Manish T.W.), Pilaniyon Ki Dhani (Sungaram T.W.), Kumaron Ki Dhani T.W. were shaken with fixed quantity of MgO (light) at 180 rpm, at ambient temperature (20°C) for 85 minutes. The MgO was filtered from water using a Whatman 42 filter paper and the residual fluoride was measured immediately.

3. Results and Discussion

Water samples were collected on 28th Jan 2014 from the three T.W.'s in Pillaniyon Ki Dhani and Kumaron Ki Dhani (Vill. Shola) and analyzed using standard procedures. The results of the ground water analysis are given in Table 1. Notice that the fluoride contaminated ground water samples have very high conc. of bicarbonate nitrate and sulphate.

It has been earlier observed by Aravind and Elango [18] that anions like Cl^-, SO_3^{2-}, NO_3^-, CO_3^{2-} have no appreciable effect on the amount of F^- removal up to 500 ppm. However, the presence of HCO_3^- conc. at 500 ppm slightly decrease the F^- adsorption efficiency due to competitive effect between HCO_3^- and F^-. The fluoride gets incorporated into the lattice of the $Mg(OH)_2$ replacing OH^- ions as $Mg(OH)_{2-x}F_y$. [19]

J.J. Singano et. al. have reported the kinetics of fluoride removal by MgO, however they observed that fluoride removal does not begin before half an hour of contact time [20]. In our case however we did not observe any lag time in our experiments. Aravind and Elango [18] have also studied the adsorption of fluoride by MgO and have used the Natarajan and Khalaf's model [21] to describe the kinetics of adsorption.

The kinetics of fluoride removal using nano MgO has also been studied by Maliyekkal et al. [22] and Devi et al. [23] Maliyekkal et al. [22] have found that the kinetics of fluoride removal follows the pseudo second order rate equation and the efficiency of fluoride removal is effected most by PO_4^{3-} followed by HCO_3^- and NO_3^- (at up to 200 ppm conc. of each). Pseudo second order rate have also been determined by Devi et al. [23] for fluoride adsorption.

Their studies show that the fluoride removal by nano magnesia is affected by the presence of co-ions (up to 90 ppm conc. OH^-, SO_3^{2-}, HCO_3^- and Cl^- ions).

The absorption capacities at equilibrium and at time *t*, q_e and q_t (mg/g) are defined as:

$$q_e = \frac{(C_0 - C_e)\cdot V}{W} \quad (1)$$

$$q_t = \frac{(C_0 - C_t)\cdot V}{W} \quad (2)$$

$$\%removal = \frac{(C_0 - C_t)}{C_0}\times 100 \quad (3)$$

Where:
C_0 = initial concentration of fluoride in water (mg/L)
C_e = equilibrium concentration of fluoride in water (mg/L)
C_t = concentration of fluoride in water (mg/L) at time t
V = volume of solution (L)
W= weight of nano MgO (g)

Taking C_0 = 3.18 ppm and C_e = 0.446 ppm gives q_e = 0.273 mg g^{-1} (For Manish TW); C_0 = 3.53 ppm and C_e = 0.489 ppm gives q_e = 0.0.3041 mg g^{-1} (For Sungaram TW); C_0 = 7.33 ppm and C_e = 0.788 gives q_e= 0.654 mg g^{-1} (For Kumaron TW).

From the average of two experiments, the time profiles of percentage removal of fluoride from ground water have been determined (fig. 1a, 1b, 1c). Notice that the plot of the fluoride removal versus time is continuous and smooth leading to saturation. Fluoride removal is 86 % at saturation level in Manish (T.W.), 86 % in Sugnaram (T.W.), 89 % in Kumaron (T.W.).

The kinetics of fluoride adsorption from ground water from the three T.W.'s on MgO has been analyzed by using different kinetic adsorption models and are discussed below.

TABLE 1. Water quality parameters for fluoride contaminated ground water from village Shola

No.	Parameter	Pilaniyon Ki Dhani (Manish)	Pilaniyon Ki Dhani (Sungaram)	Kumaron Ki Dhani
1	pH	8.5	8.3	8.1
2	Total dissolved solids (ppm)	1190	1230	1390
3	Cl^- (ppm)	361.0	285.0	285.0
4	CO_3^{2-} (ppm)	90	150	150.0
5	HCO_3^- (ppm)	1647	1342	1738
6	Ca^{2+} Hardness (ppm)	15	15	5.0
7	Mg^{2+} Hardness (ppm)	30	18.0	19.4
8	Total Hardness (ppm)	45	33.0	14.4
9	F^- (ppm)	3.18	3.53	7.33
10	Total Alkalinity (in CaCO3 equiv.)	1500	1350	1675
11	SO_4^{2-} (ppm)	70.0	130.0	170.0
12	NO_3^- (ppm)	132.9	106.3	72.8

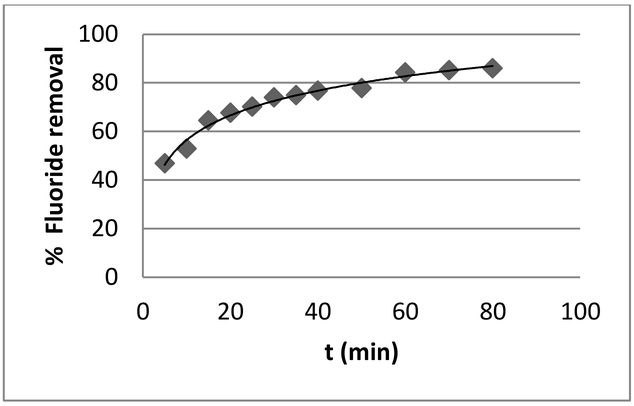

Figure 1(a). Experimental time profile of percentage removal of fluoride by MgO from ground water, Pilaniyon ki Dhani (Manish T.W.)

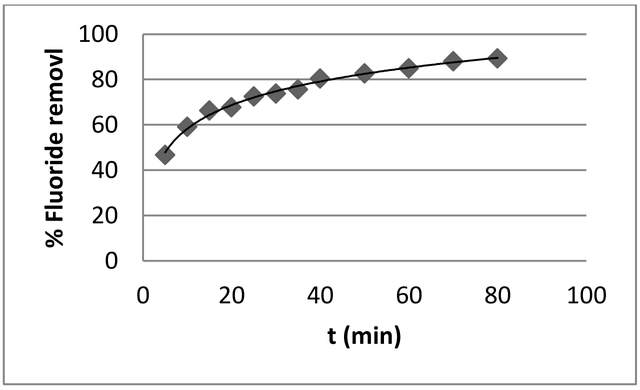

Figure 1(b). Experimental time profile of percentage removal of fluoride by MgO from ground water, Pilaniyon ki Dhani (Sungaram T.W.)

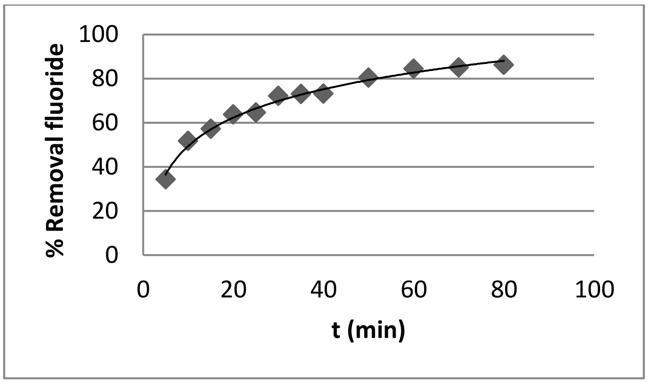

Figure 1(c). Experimental time profile of percentage removal of fluoride by MgO from ground water, Kumaron ki Dhani T.W.

Pseudo First Order Rate Equation

The Lagergren's [24] pseudo first order rate equation was used to fit the experimental data,

$$\log(q_e - q_t) = \log q_e - \frac{K}{2.303} t \quad (4)$$

where K, is the pseudo first order rate constant (min^{-1}).

A plot of log(q_e-q_t) vs. t would be a straight line if the adsorption process follows the Lagergren's equation. It was found that a linear plot was obtained (fig. 2a, 2b, 2c). The corresponding values of R^2 are given in table 2. Notice that after 35 min. there is a considerable deviation of the experimental data from the best fit regression line. The values of pseudo first order rate constant and the theoretical values of q_e were calculated and given in table 2. The first order k values considerably differ from the experimental values.

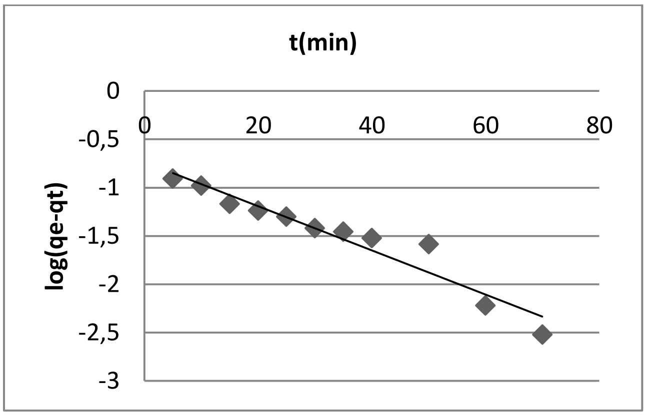

Figure 2(a). Pseudo first order adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Manish T.W.)

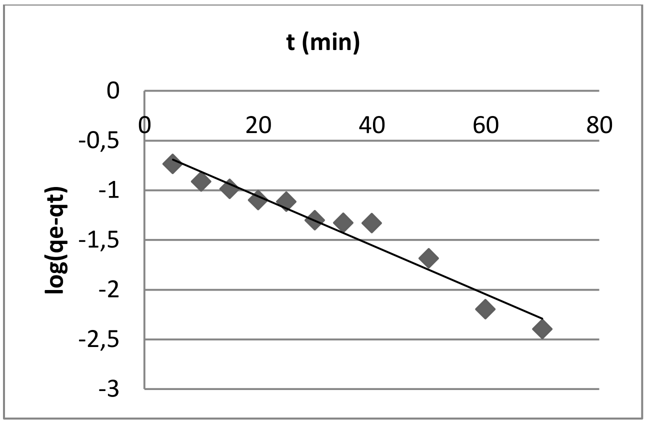

Figure 2(b). Pseudo first order adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Sungaram T.W.)

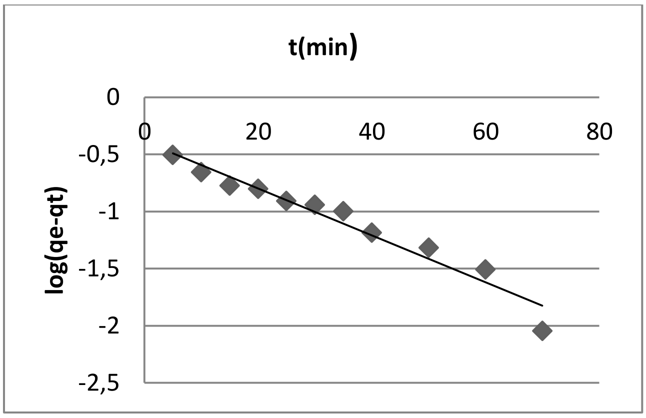

Figure 2(c). Pseudo first order adsorption kinetics of fluoride by MgO from ground water, Kumaron ki Dhani T.W.

TABLE 2. Kinetic parameters for the 1st order rate equation

T.W's	Rate constant (k) (min^{-1})	q_e ($mg\ g^{-1}$) (calc.)	R^2
Pilaniyon ki Dhani (Manish T.W.)	0.0506	0.1840	0.929
Pilaniyon ki Dhani (Sungaram T.W)	0.0552	0.2703	0.953
Kumaron ki Dhani T.W	0.0460	0.4092	0.949

Pseudo Second Order Rate Equation

The experimental data was fitted with the Ho's [25, 26] second order rate equation

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e} \quad (5)$$

Where $h = K(q_e)^2$ (6)

A plot of t/q_t vs. t should yield a straight line if the adsorption process is follow the pseudo second order rate equation. A linear plot was obtained indicating an excellent agreement with experimental data (fig. 3a, 3b, 3c). The values of R^2 and the pseudo second order rate constants are given in table 3. The experimental values are close to the calculated q_e value (table 3).

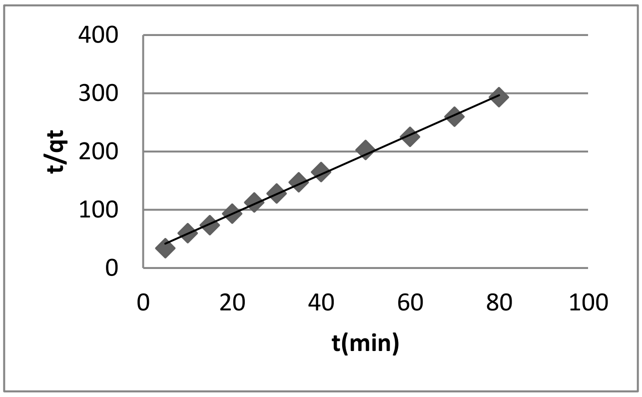

Figure 3(a). Pseudo second order adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Manish T.W.)

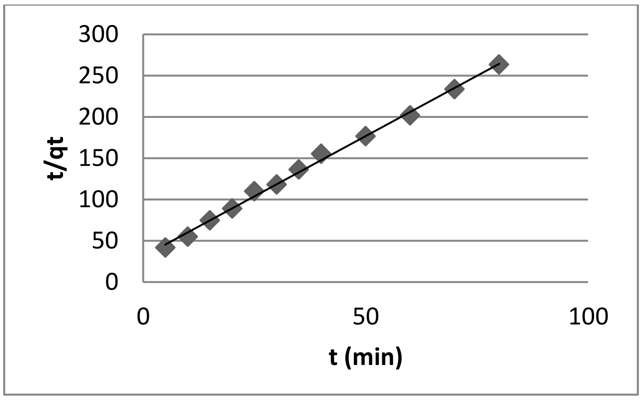

Figure 3(b). Pseudo second order adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Sungaram T.W.).

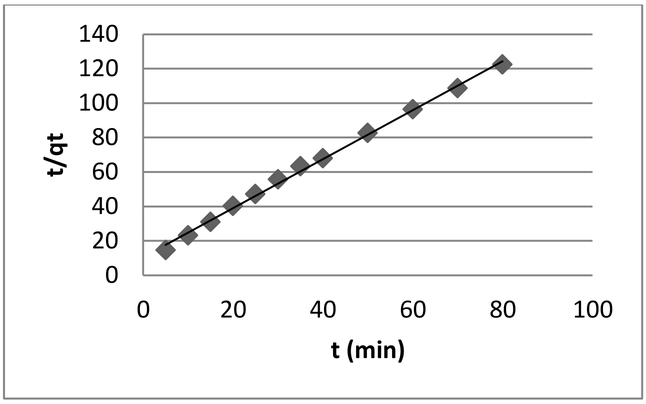

Figure 3(c). Pseudo second order adsorption kinetics of fluoride by MgO from ground water, Kumaron ki Dhani T.W.

TABLE 3. Kinetic parameters for the 2nd order rate equation

T.W's	Rate constant (k) ($g\ mg^{-1} min^{-1}$)	q_e ($mg\ g^{-1}$) (calc.)	R^2
Pilaniyon ki Dhani (Manish T.W.)	0.4705	0.2942	0.997
Pilaniyon ki Dhani (Sungaram T.W)	0.2779	0.3425	0.996
Kumaron ki Dhani T.W	0.1914	0.7037	0.997

Elovich Equation

A simplified version (Chien and Clayton [27]) of the Elovich equation [28] with the boundary conditions $q_t = 0$ at $t = 0$ and $q_t = q_t$ at $t = t$ [29] is

$$q_t = \frac{1}{\beta}(\ln \alpha\beta) + \frac{1}{\beta}(\ln t) \quad (7)$$

where α and β are constants. The constant α is the initial adsorption rate and β is related to the extent of surface coverage and the activation energy for chemisorption. The experimental data equation was to fitted in the Elovich equation and it was observed that a plot of q_t vs. lnt yields a straight line (fig. 4a, 4b, 4c). The Elovich constants α, β and values of R^2 were shown in table 4. The R^2 values in this case are lower than pseudo second order model.

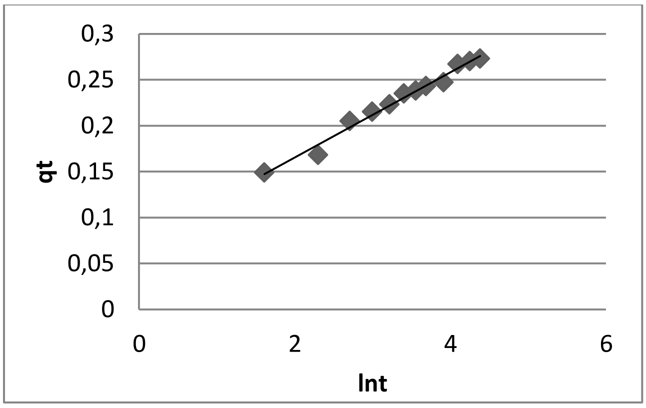

Figure 4(a). Elovich adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Manish T.W.).

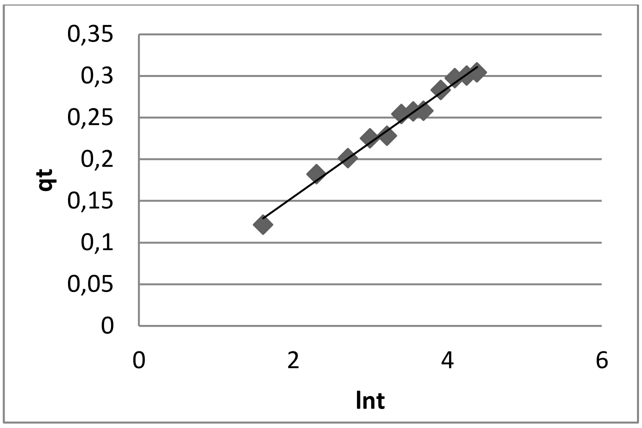

Figure 4(b). Elovich adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Sungaram T.W.)

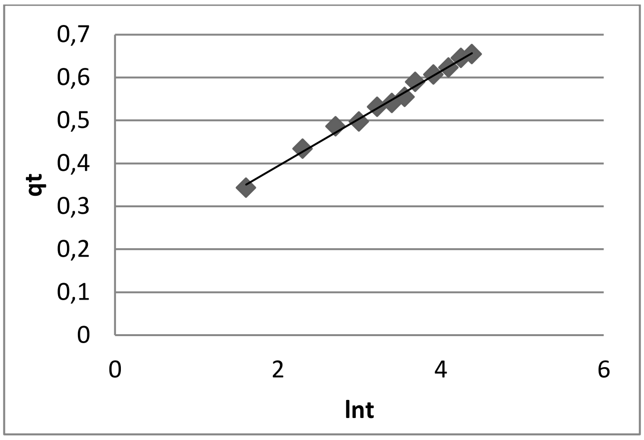

Figure 4(c). Elovich adsorption kinetics of fluoride by MgO from ground water, Kumaron ki Dhani T.W.

TABLE 4. Kinetic parameters for Elovich Equation

T.W's	Elovich constants (α) (mg $g^{-1}min^{-1}$)	Elovich constants (β) (g /mg)	R^2
Pilaniyon ki Dhani (Manish T.W.)	0.2201	21.73	0.982
Pilaniyon ki Dhani (Sungaram T.W)	0.0926	15.38	0.988
Kumaron ki Dhani T.W	0.5298	9.090	0.992

Weber and Morris Intraparticle Diffusion Model

The Weber and Morris Intraparticle Diffusion Model [30] requires that

$$q_t = Kt^{0.5} \qquad (8)$$

where k is the intraparticle diffusion rate constant (mg gm^{-1} $min^{-1/2}$).

If a plot of q_t vs. $t^{0.5}$ passes through the origin then the intraparticle diffusion is the only rate limiting step operating. On plotting the experimental data it was observed that (fig. 5a, 5b, 5c), there are three distinct regions in the plot. There is a linear portion, followed by a curved portion and finally the equilibrium portion. Notice that the linear portion starts from 5 to 30 min. Before this period nearly 45% of fluoride is adsorbed for Manish T.W. (fig. 1a), 34% of fluoride is adsorbed for Sugnaram T.W., (fig. 1b) and 46% of fluoride is adsorbed for Kumaron T.W., (fig. 1c). The second stage (linear portion of the curve) is intraparticle diffusion rate controlled.

We have tabulated in table 5 the values of R^2, C intercepts and the intra particle rate constant values for the initial linear portion of the curve.

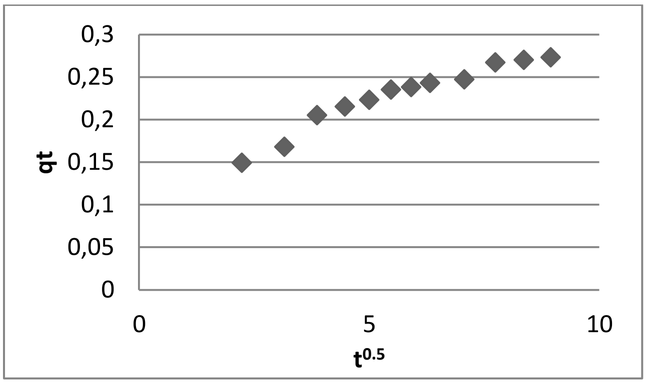

Figure 5(a). Weber and Morris adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Manish T.W.).

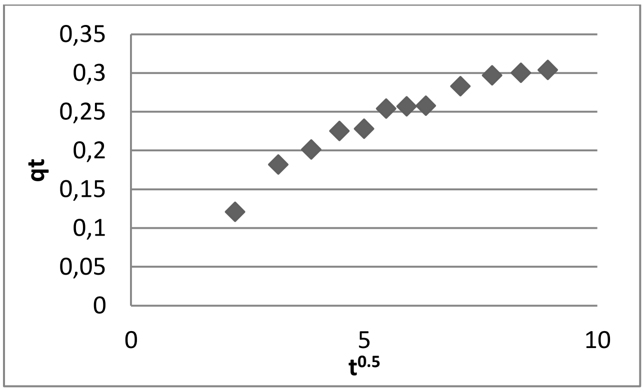

Figure 5(b). Weber and Morris adsorption kinetics of fluoride by MgO from ground water, Pilaniyon ki Dhani (Sungaram T.W.)

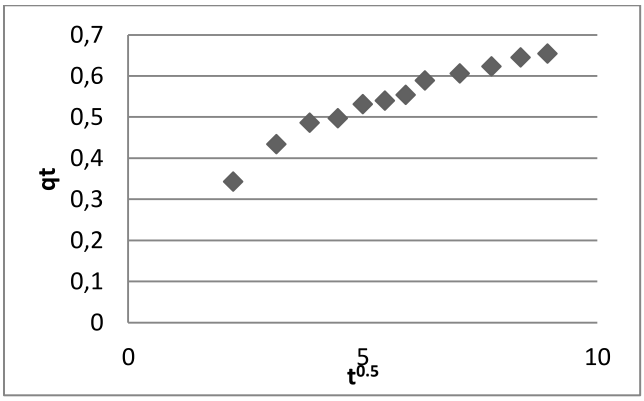

Figure 5(c). Weber and Morris adsorption kinetics of fluoride by MgO from ground water, Kumaron ki Dhani T.W.

TABLE 5. Kinetic parameters for Weber and Morris Intraparticle Diffusion Model

T.W's	Rate constant (k) (mg g^{-1} $min^{-0.5}$)	C (intercept)	R^2
Pilaniyon ki Dhani (Manish T.W.)	0.033	0.070	0.934
Pilaniyon ki Dhani (Sungaram T.W)	0.049	0.014	0.953
Kumaron ki Dhani T.W	0.087	0.149	0.993

A linear relationship was obtained only in the initial 5 to 15 minutes (fig. 6a, 6b, 6c). We found that the line does not pass through the origin indicating that intra particle diffusion is not the only mechanism operating in the rate limiting step during the initial time period of 5 to 15 min. The intraparticle diffusion is very slow in the equilibrium portion of the curve as the concentration of fluoride remaining in water was very low. Similar adsorption processes with more than one step have been earlier observed [31].

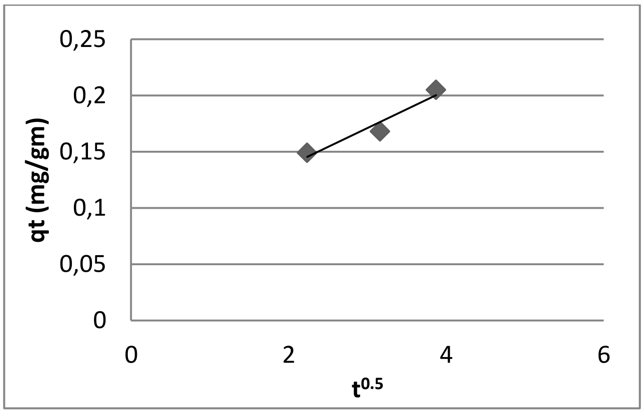

Figure 6(a). Intraparticle adsorption kinetics of fluoride by MgO from ground water (initial part of Weber and Morris plot), Pilaniyon ki Dhani (Manish T.W.)

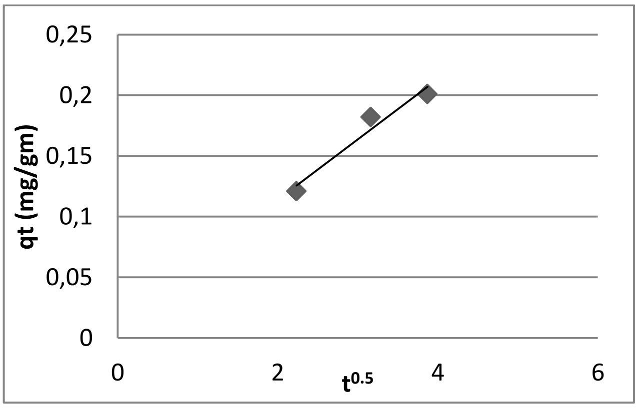

Figure 6(b). Intraparticle adsorption kinetics of fluoride by MgO from ground water (initial part of Weber and Morris plot), Pilaniyon ki Dhani (Sungaram T.W.)

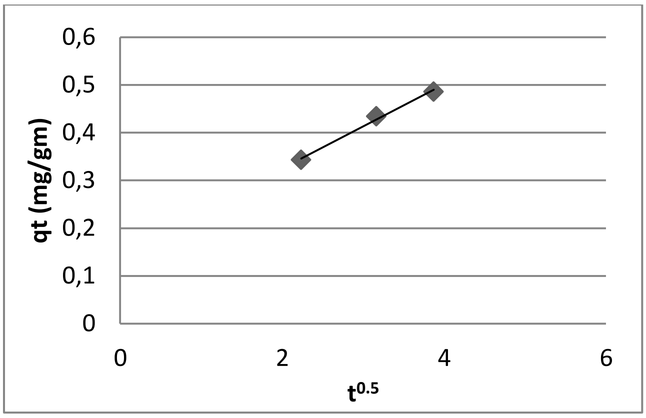

Figure 6(c). Intraparticle adsorption kinetics of fluoride by MgO from ground water (initial part of Weber and Morris plot), Kumaron ki Dhani T.W.

Modeling of Experimental q_t

Using the constants determined from the four different models discussed above, the time profile of fluoride adsorption capacity of MgO from ground water from the three different T.W.'s have been modelled (see fig. 7a, 7b, 7c). Sum of squared errors (eqn. 8)

$$SSE=\sum (qt\ (exp.)-qt\ (calc.))^2 \quad (8)$$

has been calculated for q_t of all the models (table 6). Notice that the modelled q_t values obtained from the Elovich Equation have the least value of the sum of square errors, (table 6).

However, on the basis of R^2 values, the pseudo second order kinetic model is most applicable to describe the kinetics of fluoride adsorption by MgO.

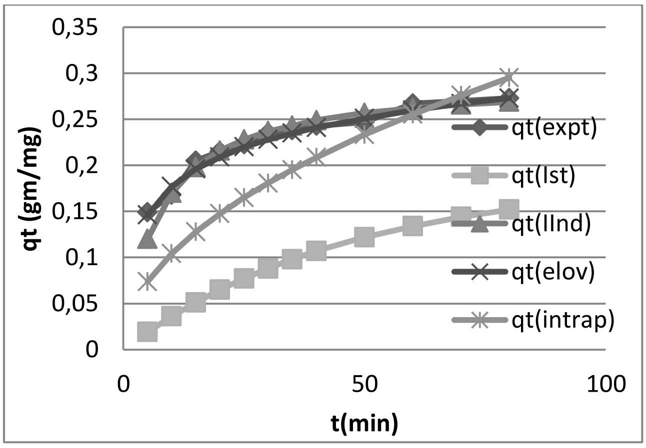

Figure 7(a). Modeled and experimental time profiles of adsorption capacity of MgO for fluoride from ground water, Pilaniyon ki Dhani (Manish T.W.).

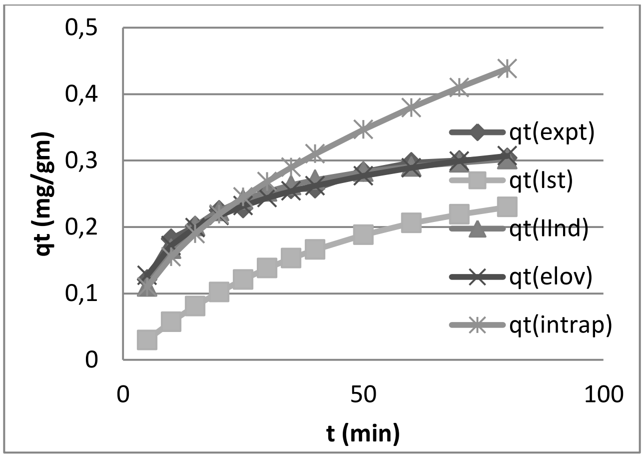

Figure 7(b). Modeled and experimental time profiles of adsorption capacity of MgO for fluoride from ground water, Pilaniyon ki Dhani (Sungaram T.W.)

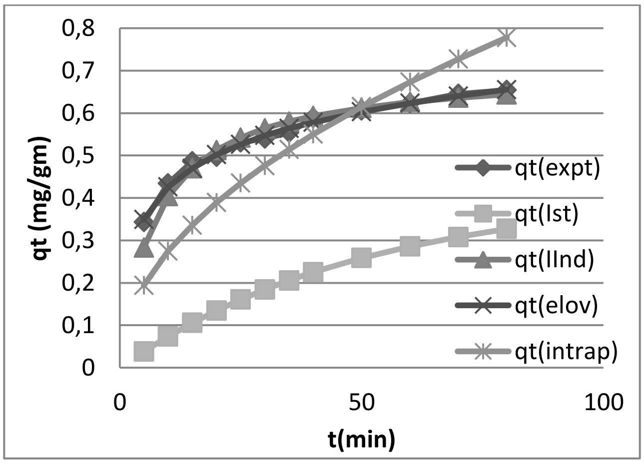

Figure 7(c). Modelled and experimental time profiles of adsorption capacity of MgO for fluoride from ground water, Kumaron ki Dhani T.W.

TABLE 6. Sum of Square Errors of the modelled data from the experimental data

T.W's	1st Order	2nd Order	Intraparticle Diffusion	Elovich Equation
Pilaniyon ki Dhani (Manish T.W.)	0.2228	0.00114	0.0303	0.00036
Pilaniyon ki Dhani (Sungaram T.W.)	0.1396	0.00061	0.0456	0.00054
Kumaron ki Dhani T.W	1.3563	0.00660	0.1222	0.00070

Next, fluoride contaminated ground water was equilibrated with increasing quantities (0.25-1.5 g/L) of MgO to determine the effect of increasing dosage of MgO on fluoride removal. The percentage removal of fluoride increased logarithmically as the dosage of MgO was increased (see fig. 8a, 8b, 8c, with R^2 of 0.983 for Manish T.W., 0.908 for Sungaram T.W., and 0.930 for Kumaron T.W).

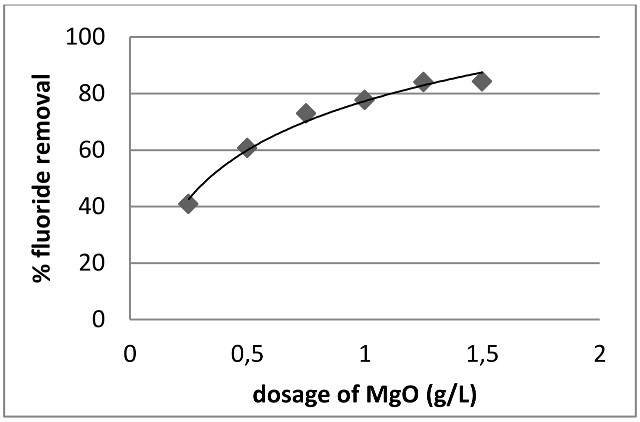

Figure 8(a). Variation of percentage removal of fluoride with increasing dosage of MgO, Pilaniyon ki Dhani (Manish T.W.)

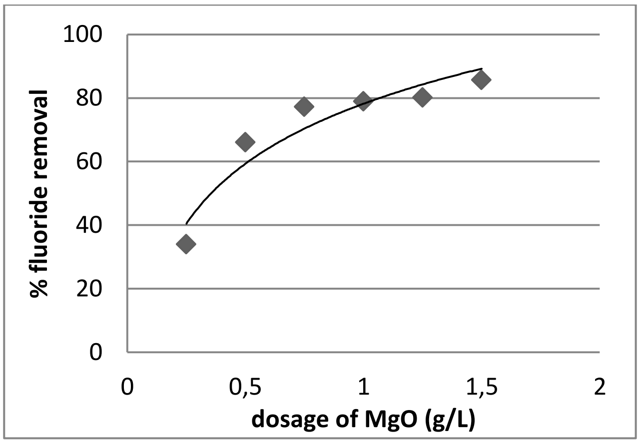

Figure 8(b). Variation of percentage removal of fluoride with increasing dosage of MgO, Pillaniyon ki Dhani (Sungaram T.W.)

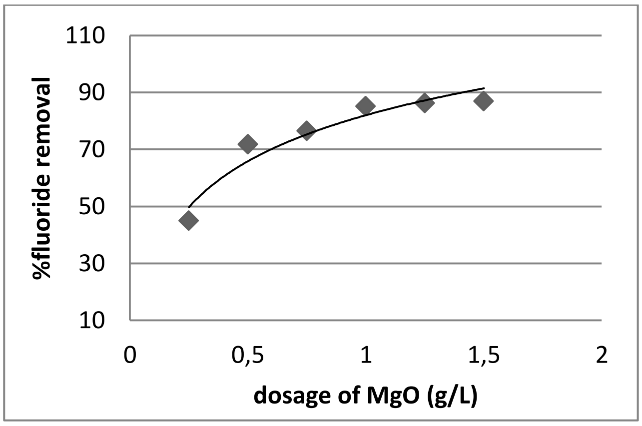

Figure 8(c). Variation of percentage removal of fluoride with increasing dosage of MgO, Kumaron ki Dhani T.W.

The adsorption capacity q_e decreased logarithmically as dosage of MgO was increased, (fig. 9a, 9b, 9c, R^2 = 0.999 for Manish T.W., 0.900 for Sungaram T.W. and 0.990 for Kumaron T.W.). The equations of the fitted lines were solved simultaneously to determine the minimum dosage of MgO that results in the maximum percentage removal of fluoride from water, and at the same time causes its highest equilibrium adsorption capacity.

The minimum dosage of MgO required was as follows, 0.047 g L^{-1} (Manish T.W.), 0.058 g L^{-1} (Sungaram T.W.) and 0.032 g L^{-1} (Kumaron T.W.).

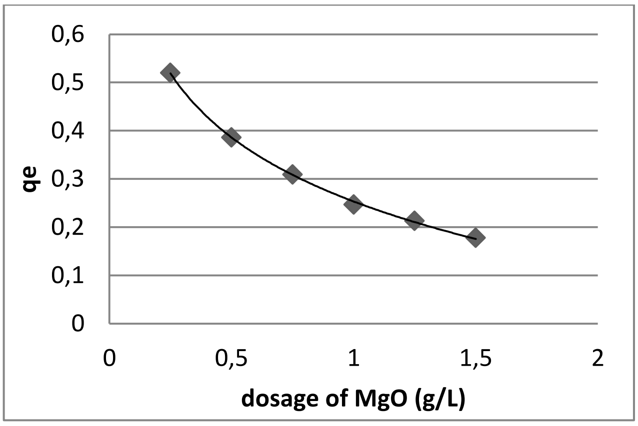

Figure 9(a). Variation of equilibrium absorption capacities of MgO with increasing dosage of MgO, Pilaniyon ki Dhani (Manish T.W.)

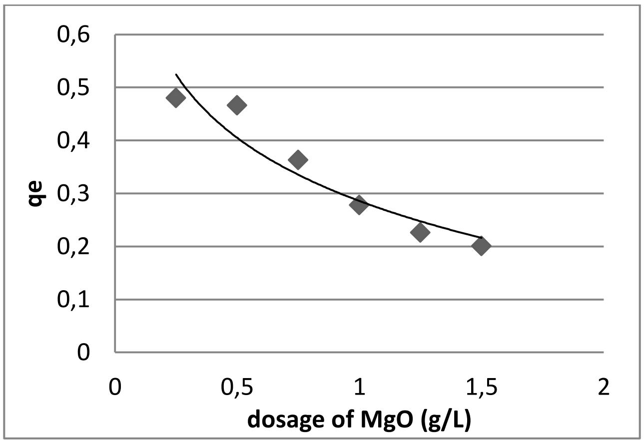

Figure 9(b). Variation of equilibrium absorption capacities of MgO with increasing dosage of MgO, Pilaniyon ki Dhani (Sungaram T.W.)

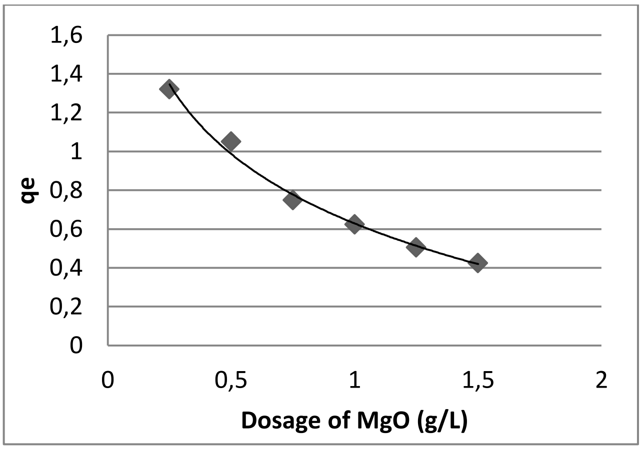

Figure 9(c). Variation of equilibrium absorption capacities of MgO with increasing dosage of MgO, Kumaron ki Dhani T.W.

4. Conclusions

A pseudo second order kinetics is followed for the fluoride removal process by MgO from the groundwater of all three TW's tested. Sum of square error calculations indicates that modelled q_t values closely follow the Elovich equation. In the initial 5 to 15 min of the adsorption process, the intraparticle diffusion is the main mechanism of fluoride removal. As the plot of q_t vs. $t^{0.5}$ does not pass through the origin it is not the sole adsorption mechanism. The adsorption capacity to remove fluoride decreases on increasing the dosage of MgO.

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