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# **ALUMINIUM HYDROXIDE GEL & ITS DERIVATIVES USING REMOVAL OF FLUORIDE FROM DRINKING WATER**

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**Abstract:** The batch sorption system using Aluminium Hydroxide Gel(HAG), HAG impregnated on cross linked guar(HAGG), starch(HASG) and micro crystalline cellulose(HACG) as adsorbents was investigated for removing fluoride ion from aqueous solutions. Effect of initial fluoride concentration, agitation time, adsorbent dose, pH, temperature, surfactants, and added ions, on the percentage fluoride removal, have been reported. The experimental data fitted well in the Freundlich adsorption equation, the values of adsorption capacity, K, and intensity of adsorption, n have been reported. HAG & its derivatives showed promising results in the removal of fluoride from drinking water. The up take of fluoride is rapid and proportionate in initial stages and decreases gradually while approaching equilibrium. The major part of total adsorption, nearly 80 % of uptake at equilibrium takes in less than 15 minutes. The up take of fluoride increases with the decrease in pH of water and the increase in [cationic surfactant], however it decreases with the increase in [anionic surfactant], nonionic surfactants have no effect. The pH of the system in all cases, increases with the removal of fluoride indicating that the exchange of OH<sup>-</sup> with F<sup>-</sup>. The materials also solves waste disposal problem as these may be burnt after the use.

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**Introduction:** Fluoride is an essential dietary constituent for humans as well as animals, depending upon the total amount ingested. The presence of fluoride in drinking water within permissible limits of 0.05- 1.0 mg dm<sup>-3</sup>, is beneficial for the production and maintenance of healthy teeth and bones while an excessive intake of fluoride causes dental & skeletal fluorosis, a chronic disease manifested by mottling of teeth in mild cases and softening of bones , neurological damage in severe cases<sup>1</sup>.

Wang Y. and Reardon E.;J.Applied Geochem 16(2001)531.

People of approximately 177 districts of 20 states in India are suffering with excessive fluoride in ground water<sup>2</sup>. Worst affected states are Andhra Pradesh, Rajasthan, Uttar Pradesh and Tamil Nadu. All the 36 districts of Rajasthan have been declared as fluoride affected. The

fluoride level in the ground water used for drinking purposes of Thar Region<sup>3</sup> is as high as 12-90 mg dm<sup>-3</sup>

Nemode P.D., Rao A. Vamde and Allapat B.J.; Water Sc. Technol: Water Supply 2(2002)311.

The problem of high fluoride concentration in ground water resources has become an important health related geo-environmental issue<sup>1</sup> Epidemiological studies of health risks from drinking water, naturally contaminated with fluoride has been reviewed by Susheela,<sup>2</sup> the cases of endemic fluorensis have been reported from all places of world, particularly China, Japan, Thailand, Persian Gulf, Saudi Arabia, Europe, USA, Canada, Argentina and African Countries<sup>4</sup>

Susheela A

Several defluoridation methods based on the adsorption, precipitation & ion exchange has been developed during last six decades, however none of these could solve this problem at a completely acceptable level<sup>5</sup> The subject has been reviewed recently<sup>5</sup>. The removal of fluoride by oxides, hydroxides and carbonates of multivalent metal ions including Al(III) and Ca(II) ions has been studied<sup>9</sup>. A voluminous work has been reported by several workers using different adsorbents like lime & related compounds<sup>5</sup>, activated alumina<sup>6,7</sup>, trivalent metal oxides<sup>8</sup>, hydroxides & carbonates<sup>8</sup>, activated carbon,<sup>9</sup> coal based adsorbents, bone char<sup>10-12</sup>, ion exchangers<sup>13</sup>, amorphous alumina supported on nano-tubes<sup>14</sup>, Zr(IV) complexes of chelating resins functionalized with N-acetate ligands<sup>15</sup>, Zr(IV) arsenates & vanadates<sup>16</sup>, cationic synthetic resins<sup>17</sup>, D412 resin loaded with Ce(IV)<sup>18</sup>, Ce(IV) poly hydroxamic acid resins, Al(III) complexed amino phosphonic resins<sup>19</sup>, zeolites<sup>20</sup>, amphotites<sup>21</sup>, Magnesia<sup>22</sup> etc.

In present communication the removal of [F<sup>-</sup>] from water by Aluminium Hydroxide Gel(HAG) & (HAG) impregnated on cross linked guar(HAGG), starch(HASG) and micro crystalline cellulose(HACG), is being reported under varying physical conditions to have an idea of optimum conditions.

The experiments were planned with pure sodium fluoride solutions and also from natural drinking water containing excess fluoride from Jayal in Nagaur district of western Rajasthan, its detailed analysis is given in Table-1.

### **Materials & Methods:**

**Preparation of Aluminium Hydroxide Gel(HAG) :** Aluminium Hydroxide Gel(HAG) was prepared by adding 500 ml of 0.1M AlCl<sub>3</sub> to an excess NH<sub>4</sub>OH solution (35%). The gel was thoroughly washed with double distilled water and dried at 80° C for about 12 h. The product was broken in to smaller particles and stored in desiccator.

The HAG was passed through different sieves to collect particles of 50-100, 100-200 and 200-300 mesh size. The HAG is stable in salt solutions, but starts leaching at pH below 3. It is however not affected by common organic solvents e.g. ethanol, 2-propanol, acetone, benzene, toluene etc.

W U Malik, JS Tyagi, RK Gulati and SP Arora; J. Ind. Chem. Soc. 26(1979)688-691

The HAG was characterized by IR spectro-photometric analysis and x-ray diffraction pattern. The material showed peaks at 1005, 36005, 3570, 3450, 3300 and 1080  $\text{cm}^{-1}$

**Preparation of HAGG, HASG, HACG:** The gel was homogenized with 200 mesh, cross linked guar gum, starch or cellulose powder in the ratio 4:1 by weight for about half an hour, dried for 6 hours, powdered and sieved to get uniform 200 mesh particles. .

All the reagents and chemicals used in this study were commercially available and were of high purity AnalaR grade except epichlorohydrine di & triethylamine(Merck).

All the solutions were prepared in double distilled water.

The stock solution of initial fluoride concentration, 0.1  $\text{g dm}^{-3}$  (100 ppm) was prepared by dissolving 2.2102 g anhydrous NaF in de-ionized water and diluted to 1  $\text{dm}^3$  (1ml = 100 ug F). These solutions were further diluted to suitable concentrations as and when required. The pH of solutions was adjusted by adding dilute HCl or NaOH solution.

Residual  $[\text{F}^-]$  was analyzed by SPADNS method<sup>27</sup> on AIMIL make MK II model spectrophotometer & all pH measurement were made on Naina make pH meter model 303 with combined glass electrode. All the batch experiments and analysis work were carried out at room temperature.

**Distribution Coefficients:** The uptake of fluoride from its aqueous solutions on HAG, HAGG, HASG and HACG was estimated in terms of distribution coefficient,  $K_d$ . The adsorbents (0.05- 0.5 g) were stirred with 100 ml of 0.01  $\text{g dm}^{-3}$   $\text{F}^-$  solution at pH approximately 8, until complete equilibrium was attained. The adsorbent was then removed by filtration through glass wool and the residual  $[\text{F}^-]$  in the solution was computed spectrophotometrically, from the linear optical density (OD) vs. concentration curves by measuring OD at 570 nm, the max absorbance region of the complex, by SPADNS method. The  $K_d$  values were evaluated using the formula<sup>28</sup>

$$K_d = (100 - x) \cdot V / x \cdot m$$

Where V is the volume of solution in ml, m is the weight of the adsorbent in grams and x is the % of  $[\text{F}^-]$  remaining in the solution.

**Batch Adsorption Study:** The batch equilibrium studies were conducted by agitating 0.05- 0.5 g of HAG and its derivatives with 100 ml water containing 0.01  $\text{g dm}^{-3}$  fluoride in skew cap jars. The agitation was continued for 30 min. at 30±0.02° C. The initial pH of the solution was adjusted to 8 by adding requisite amounts of acid or alkali solution. The equilibrated solutions

were centrifuged for 10 min at 10,000 RPM in T 24 model (GDR) centrifuge and analyzed for the residual  $[F^-]$ .

All the experiments were carried out in triplicate and an average of the 3 values is reported. The experimental variables considered in the study were initial fluoride concentration, dose of adsorbent, pH, contact time, temperature and effect of nature and concentration of surfactants & added ions.

**Results and Discussions:** The values of  $K_d$ , obtained for the removal of fluoride ion on HAG, HAGG, HASG & HACG from its aqueous solutions at pH 3, 4, 5, 8 & 9 and at temperature  $30^\circ\text{C}$  are shown in Table-2 & 3. The results show that the adsorption of  $F^-$  on HAG and its derivatives is fairly good, more than 67 % fluoride is removed at an adsorbent dose of 4 g per liter.

The plots of % fluoride removed is not proportional to the amount of gel, Fig-1 shows the variation in the percentage removal of fluoride with different doses of the adsorbents. The adsorption of fluoride increases with the decrease in initial pH of the solution. The  $K_d$  values for fluoride ions on HAG & derivatives are maximum at pH 3 ( $K_d = 1148$ ) and minimum at pH 9 ( $K_d = 184$ )

**Adsorption Isotherms:** In order to determine the adsorption capacity of HAG & derivatives, the study of adsorption isotherms (A.I) were conducted. The experimental data was analyzed in the light of Freundlich, Langmuir and BET equations to predict the nature of adsorption. Adsorption isotherms for fluoride ions in water on HAG & derivatives, when the system is in equilibrium, are shown in the Fig. 2, all the A.I were found regular, positive and convex to the concentration axis. The A I rise proportionately in the initial stages, and then try to become parallel to the concentration axis, forming a plateau, after that adsorption increases sharply showing a multilayer adsorption. The A I do not strictly follow any one model. The plots of  $C_e/(x/m)$  against  $C_e$ , according to Langmuir equation, remain no longer linear at moderate  $[F^-]$ . The plots of  $\log C_e$  against  $\log x/m$  according to Freundlich model were found linear, yielding an intercept on the ordinate at low concentrations of  $F^-$  (1- 10 ppm) as are generally available in the drinking waters.

$$\log(x/m) = \log K + 1/n \log C_e$$

Where  $C_e$  is the equilibrium concentration,  $x/m$  is the amount of  $F^-$  adsorbed per gram of the adsorbent at equilibrium,  $K$  and  $1/n$  are Freundlich constants. The values of  $K$  and  $1/n$  were calculated from the intercepts and slopes of these curves using computerized LRG program and are reported in Table-2. The adsorption capacity of HAG was found maximum. The extent of adsorption, denoted by  $n$ , was found in the range of 1.74-2.10, which is quite satisfactory.

**Effect of Adsorbent Dosage:** The amount of fluoride removed as a function of HAG dose (0.05, 0.1, 0.2, 0.3, 0.4 and 0.5 at  $[F^-]_i$ ,  $0.01 \text{ g dm}^{-3}$  temperature  $30^\circ\text{C}$ , and an equilibrated time 15 min. From the results, it is evident that optimum dosage of adsorbent per 100 ml required

for the maximum removal of fluoride is .08 g. It is interesting to note that the same dose of other adsorbents exhibit comparatively low fluoride removal.

**Effect of pH:** The effect of pH on the % removal of  $F^-$  on to HAG & derivatives was studied at initial pH 3, 4, 5, 8 & 9 of solution, adjusted by adding requisite amount of dilute alkali or acid solution. It was observed that the pH of solution increased consistently after the removal of  $F^-$  (Table 2 & 3) by HAG. The results also indicate that the adsorption of  $F^-$  and hence % removal of  $F^-$  by HAG & derivatives from water decreases with the increase in initial pH of water, reaches a minima at pH 5, and then increases up to pH 8, after it falls regularly, however in case of HAGG, HASG, HACG the % removal of  $F^-$  decrease with the increase of pH of water continuously. This can be explained by amphoteric nature of HAG & derivatives, the behavior is pH dependent to. In acidic pH, HAG & derivatives are positively charged and behave as anionic exchanger while at higher pH, these behave mostly as cationic exchanger.(R)

X-ray measurements of HAG indicate that the  $Al^{+3}$  is coordinated by octahedral arrangement of oxygen sites and its relationship between the remaining oxygen atoms is essentially same as liquid water.

John I. Pitz Jr., J. Chem. Phys. 48(1968) 909

It has been established that the HAG gels, obtained by precipitation from  $AlCl_3$  and  $NH_4OH$  solutions, are represented by and probably exists as a polymer of 10 fused six

**Effect of Time:** The effect of contact time on the adsorption of F on Starch derivatives were studied at pH 3. The results (Table 5) show that 85 to 90 % adsorption takes place in 30 min and for remaining 10 to 15 % at least 90-120 min. is required.

**Effect of Temperature:** The effect of temperature on the % removal of F by starch derivatives was investigated at 30, 40 and 50°C keeping all other parameters constant, these results are summarized in table 6. The % uptake is highly dependent on  $[F^-]_i$  in solution and increase with the increase in temperature, indicating that the process of adsorption is endothermic in nature.

**Effect of Surfactants:** The effect of nature and concentration of added surfactants on the adsorption of  $F^-$  on the guar derivatives was investigated at room temperature. The results in Table-7 show that the surfactants have a little effect on the adsorption of  $F^-$  on the derivatives. The % removal of  $F^-$  increased with the increase in added cationic surfactants concentration and decreased with the increasing [anionic surfactant], nonionic surfactants however have no effect. It appears that cationic surfactants neutralize some negatively charged sites of the adsorbent resulting in the improved migration of  $F^-$  on the starch derivative. The decreased adsorption by anionic surfactants may be attributed to the competitive adsorption of  $F^-$  and the surfactants.

**Effect of added Co-ions:** The effect of added co-ions e.g.  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$  and  $\text{HCO}_3^-$  on the % removal of  $\text{F}^-$  was investigated with TMAHP- Starch. The results (Table 8) indicate that the addition of co-ions in the range investigated has no appreciable effect, however the increase in  $[\text{HCO}_3^-]$  slightly decrease the adsorption efficiency. This may be due to the competitive rate of adsorption of  $\text{F}^-$  and  $\text{HCO}_3^-$ . The observation is in accordance with the studies on activated alumina and magnesia.

The equilibrium contact time necessary for the batch adsorption study was evaluated from the plots of fraction of residual fluoride ion concentration,  $[\text{F}^-]_r / [\text{F}^-]_i$ , against contact time, t. The plots illustrate the rapid attainment of adsorption equilibrium.

The increase in initial fluoride ion concentration,  $[\text{F}^-]_i$  in the range of 2 to 100 ppm at dose 4 g  $\text{dm}^{-3}$  shows an increase in the amount of adsorbed fluoride.

the effect of agitation time and initial concentration of  $\text{F}^-$  on the adsorption of  $\text{F}^-$  on to HAG, it is clear that the uptake of  $\text{F}^-$  on HAG increase sharply and then become parallel to the time axis.

**Conclusion:** The present study demonstrates that the cationic starch derivatives have potential to remove F from the water. The study has the following salient features-

1. The up take of F is rapid in initial stages and decreases gradually while approaching equilibrium.
2. The major part of total adsorption, nearly 80 % of uptake at equilibrium takes in less than 15 minutes.
3. The up take of F is significantly dependent on the pH of water, increases with the decrease in pH of water.
4. The up take of F by starch derivatives is also influenced by the presence of surfactants. The up take of F increases with the increase in [cationic surfactant], and decreases with the increase in [anionic surfactant], nonionic surfactants have no effect.
5. The material (starch derivatives) after the use can be burnt, thus pose no waste disposal problem.

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**Table 1:** Physical properties of Starch Derivatives

S. No.	Derivatives	Particle size, %	Mesh	Degree of substitution	Water regain	Salt splitting capacity	Weak exchange capacity
1	TMAHP- Starch	60	>100	0.25	2.52	0.66 +0.03	0.25+0.05
		40	60-100				
2.	TEAHP- Starch	60	>100	0.24	2.64	0.58+0.02	0.25+0.03
		40	60-100				
3.	DEAE -Starch	60	40-60	0.21	2.10	0.46+0.03	0.20+0.03
		40	>60				

Stability: 6-8 weeks at pH 3- 10

Swelling in water: slightly.

**Table 2:** Freundlich constants for the adsorption of fluoride ion on Starch derivatives\*

[F <sup>-</sup> ] = 0.01 g dm <sup>-3</sup>		Time of Contact = 30 min			
Adsorbent = 0.04 g		Temperature = 30° C			
Volume = 0.1 dm <sup>3</sup>		pH = 8			
Adsorbent	Average K <sub>d</sub>	Freundlich constants			
		K	n	SD	R <sup>2</sup>
TMAHP- Starch	4450 + 250	5.76X10 <sup>-3</sup>	0.59	2.38X <sup>10</sup> <sub>2</sub>	0.99
TEAHP - Starch	2773 + 160	1.71X 10 <sup>-3</sup> <sub>3</sub>	0.74	3.25X <sup>10</sup> <sub>2</sub>	0.99
DEAE - Starch	2290 + 200	1.94X10 <sup>-3</sup>	1.24	5.24X <sup>10</sup> <sub>2</sub>	0.99

**Table 3:** Effect of Adsorbent Dose on K<sub>d</sub> and % Removal of F ion by Starch-Derivatives\*

[F <sup>-</sup> ] = 0.01 g dm <sup>-3</sup>		Time of Contact = 30 min				
pH = 8		Temperature = 30° C				
Volume = 0.1 dm <sup>3</sup>		\ max = 570 nm				
Amount of Adsorbent	TMAHP- Starch		TEAHP- Starch		DEAE – Starch	
	K <sub>d</sub>	%[F <sup>-</sup> ]	K <sub>d</sub>	%[F <sup>-</sup> ]	K <sub>d</sub>	%[F <sup>-</sup> ]
0.02	3198	39	2812	36	1400	22
0.04	3181	56	2819	53	1346	35
0.06	3095	65	2719	62	1309	44
0.08	2426	66	2321	65	1200	49
0.10	2571	72	2333	70	1222	55
0.15	2507	79	2363	78	1254	65



**Table 4:** Effect of pH on  $K_d$  & % removal of Fluoride ion, by Starch-derivatives\*

[F <sup>-</sup> ]		= 0.01 g dm <sup>-3</sup>		Time of Contact = 30 min			
Adsorbent		= 0.04 g		Temperature = 30 °C			
Volume		= 0.1 dm <sup>3</sup>		\ max = 570 nm			
S/No	pH	% fluoride removed					
		TMAHP- Starch		TEAHP- Starch		DEAE – Starch	
1	3	5564	69	4643	65	2045	45
2	4	4078	62	3314	57	1666	40
3	5	3452	58	3055	55	1468	37
4	8	3181	56	2819	53	1346	35
5	9	2217	47	1964	44	858	23

**Table 5:** Effect of Time on  $K_d$  & % removal of Fluoride ion, by Starch-derivatives\*

[F <sup>-</sup> ]		= 0.01 g dm <sup>-3</sup>		Volume = 0.1 dm <sup>3</sup>			
Adsorbent		= 0.04 g		Temperature = 30 °C			
pH		= 8		\ max = 570 nm			
S No.	Time	% fluoride removed					
		TMAHP- Starch		TEAHP- Starch		DEAE – Starch	
		$K_d$	%[F <sup>-</sup> ]	$K_d$	%[F <sup>-</sup> ]	$K_d$	%[F <sup>-</sup> ]
1	15	1532	38	1468	37	1287	34
2	30	3181	56	2819	53	1598	43
3	60	3452	58	3181	56	2500	50
4	90	4078	56	3910	61	3055	55

**Table 6:** Effect of Temp on  $K_d$  & % removal of Fluoride ion, by Starch-derivatives\*

[F <sup>-</sup> ]		= 0.01 g dm <sup>-3</sup>		Volume = 0.1 dm <sup>3</sup>			
Adsorbent		= 0.04 g		Time of contac = 30 min.			
pH		= 8		\ max = 570 nm			
S No.	Temperature	% Fluoride removed					
		TMAHP- Starch		TEAHP- Starch		DEAE – Starch	
		$K_d$	%[F <sup>-</sup> ]	$K_d$	%[F <sup>-</sup> ]	$K_d$	%[F <sup>-</sup> ]
1	30 °C	3181	56	2819	53	1346	35
2	40 °C	3829	60	3313	57	1598	39
3	50 °C	4253	65	3468	62	1768	41

**Table 7:** Effect of nature & Concentration of surfactants on the % removal of Fluoride by TMAHP-Starch

[F <sup>-</sup> ] = 0.01 g dm <sup>-3</sup>		Adsorbent = 0.04 g		
pH = 8		Time of Contact = 30 min		
Volume = 0.1 dm <sup>3</sup>		Temperature = 30 °C		
S. No.	[Surfactant]	% removal of fluoride		
		Arkoline SPW	Arkoline N-45	Arkoline-HCS
1.	0.05	56	56	59
2.	0.10	53	58	62
3.	0.15	51	57	66
4.	0.20	46	56	71
5.	0.25	43	57	76
6.	0.30	38	58	78

an average of three sets.

**Table 8:** Effect of added ions on the % removal of fluoride by TMAHP-Starch

[F <sup>-</sup> ] = 0.01 g dm <sup>-3</sup>		Time of Contact = 30 min		
pH = 8		Volume = 0.1 dm <sup>3</sup>		
Adsorbent = 0.04 g		\ max = 5		
Concentration in g dm <sup>3</sup>	Cl <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	SO <sub>4</sub> <sup>-</sup>	HCO <sub>3</sub> <sup>-</sup>
10	56.0	56.0	56.0	49.0
20	55.5	55.0	55.0	48.0
30	55.5	55.0	55.0	45.0
40	55.0	55.0	55.0	43.0
50	55.0	55.0	54.5	42.0

an average of three sets.

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