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## Chemistry of Iron Sorption-Desorption in Three Different Soils of Bangladesh

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### **Abstract:**

A laboratory incubation experiment was conducted for ascertaining the chemistry of iron sorption-desorption among the three different soils, namely calcareous 'Ishurdi series' and non calcareous 'Amjhupi' and 'Dhamrai series'. The sorption-desorption behavior was observed under three different iron treatments-0.5 ppm, 1.00 ppm and 2.5 ppm, while the sorption chemistry was studied for 2, 4, 6, 24 and 48 hour of incubation and desorption chemistry was done for 2, 4, 6 and 24 hour of extraction with 0.1N EDTA solution followed by 6 and 24 hour of incubation with different treatments (0.5 ppm, 1.00 ppm and 2.5 ppm) of iron solution. Maximum iron sorption of 35.72 ppm was found for the calcareous Ishurdi soil after 48 hour followed by the non calcareous Amjhupi and Dhamrai soil. There may be higher chemisorption and/or complexation reaction which are favored under conditions provided by the calcareous soils and this might be the reason for greater sorption in Ishurdi soil. The desorption study showed that with increasing extraction period (from 2hour to 24 hour) iron desorption increases. But with increasing sorption incubation period from 6 hour to 24 hour, iron desorption decreases for all the three soils. Concentration of iron in the soil solution and soil properties such as pH, clay content and calcareousness might have positive influence on iron sorption- desorption chemistry. Thus most significant iron desorption was observed for non calcareous soils, Amjhupi ( $r=0.9988$ ) and Dhamrai ( $r=0.9986$ ) soil, followed by calcareous Ishurdi soil ( $r=0.9779$ ).

**Keywords:** Sorption, Desorption, calcareous, Incubation, chemisorption

### **1. Introduction**

Iron comprises about 5% of the earth's crust and is the fourth most abundant element in the lithosphere. Iron is unique geochemically in its ability to form numerous stable compounds both with S and with O plus Si, and in its occurrence. Iron can be either concentrated or depleted during soil development; thus, its concentration in soil varies widely from 0.7 to 55%. Most of this soil Fe is found in primary minerals, clays, oxides and hydroxides (Tisdale *et al.*, 1995). The solubility of the common ions on minerals in soil is very low, only  $10^{-6}$  to  $10^{-24}$  M  $Fe^{3+}$  in solution, depending on pH. Mineral "Soil Fe" represents on amorphous  $Fe(OH)_3$  precipitate, which appears to control solutions  $Fe^{3+}$  concentrated in most soils. As the pH of a soil solution raises adsorption increases and the formation of specific Fe (II) minerals becomes possible. Over the normal pH range in soils, total solution Fe is not sufficient to meet plant requirements for Fe, even in acid soils, where Fe deficiencies occur less frequently than in high pH and calcareous soils (Tisdale *et al.*, 1995). Soil has the ability to immobilize introduced chemicals like heavy metal ions. This is mainly due to sorption properties which are determined by physicochemical properties of the soil such as: amount of clay and organic fraction,  $p^H$ , water content, temperature of the soil and properties of the particular metal ion (Dube *et al.*, 2001 and Stevenson, 1992). The solid state of soils composes an average of 45% of soil bulk. It consists of mineral particles, organic matter and organic-mineral particles. They all play a very important role in giving the soil the ability to adsorb, exchange, oxidize, reduce, catalyze and precipitate chemicals and metal ions in particular (Dube *et al.*, 2001). The inorganic colloidal fraction of soil is the most responsible for sorption by its mineral particles. It is comprised of clay minerals, oxides, sesquioxides and hydrous oxides of minerals. The clay minerals are hydrous iron, aluminium, or magnesium silicates. Clay particles are usually negatively charged. This is a very important factor influencing sorption properties of the soil. There are at least two major possibilities as to how these charges are formed (Loughnan, 1969). Firstly,  $p^H$ -dependent charge and secondly, isomorphism ion replacement in the minerals. The negative charge, which appears as a result of isomorphism ion replacement, is  $p^H$ -dependent and therefore quite persistent. The ability to create negative charges is the highest for 2:1 type clays (Brown, 1998). The understanding of the behaviors of iron adsorption is necessary to provide much needed information on the mobility, persistence and fate iron in the environment. In Bangladesh arsenic-rich iron oxyhydroxides present in aquifer materials appear to be the primary source of arsenic in groundwater. In the subsurface environment, adsorption-desorption of arsenic onto iron oxyhydroxides is an important mechanism controlling its mobility. Presence of ligands, which may compete with arsenic for adsorption sites on iron oxyhydroxides, e.g., phosphate, silicate and sulfate can also influence the mobility of arsenic in the subsurface, if present in large

enough concentrations (Ali and Ahmed, 2003). Therefore this research aims to ascertain the adsorption-desorption of iron in three soils as affected by different soil properties, applied iron treatment and with residence time.

## 2. Materials and Methods

For the research study three soils were selected. Two of the soils represented the Gangetic alluvium- one is calcareous and the other one is non-calcareous while the third soil represented the Brahmaputra alluvium. The three soils represented the Ishurdi, Amjhupi and Dhamrai series respectively. The descriptions of the three series according to upper and lower categories are represented in the Table 1

Soil Series under Study	Location	General Soil Type	USDA* Soil Taxonomy
Ishurdi Series	Noapara, Jessore Sadar Upazilla	Calcareous Dark grey Floodplain Soil	Aeric Andoaquepts
Amjhupi Series	Shatakali, Shalikhua Upazilla	Non-calcareous Dark Grey Floodplain Soil	Aeric Andoaquepts
Dhamrai Series	Sombhag, Dhamrai Upazilla	Non-calcareous Grey Floodplain Soil	Typic Andoaquepts

Table 1: Location and Correlation of general soil types of the three soil series with the US Soil Taxonomy: (Source: SRDI 1970, FAO, 1988, \* Soil Survey Staff, 2003)

The soil samples were collected, prepared and the common physical, chemical and physicochemical properties of the three soil series were analyzed following the standard methods (Imamul Huq and Alam, 2005).

The three selected soils (Amjhupi series, Ishurdi series and Dhamrai series) were used for this sorption studies. The sorption studies were carried out in batch mode where a series of 50 ml polythene/plastic centrifuge tubes or sorption tubes were used. In each tube 5 g soil sample was weighed. Twenty five ml each of the Iron solution, (made from  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) were added to the tubes. There were three treatment rates for iron solution and the concentrations of Fe used were 0.5, 1.0 and 2.5 ppm along with the control. The sorption time allowed for the soils were 2, 4, 6, 24 and 48 hour. The tubes were shaken on a reciprocating shaker (50 turns/min) for two hours and were allowed to equilibrate at a constant temperature (room temperature) for 2, 4, 6, 24 and 48 hour, respectively. It was established from a preliminary study that two hour shaking followed by 24 hours rest was sufficient for the system to equilibrate. Immediately following the equilibration period, the suspensions were centrifuged at 2000 rpm over 5-50 minutes and filtered (Garcia-Sanchez. A. et al., 1999 and Begum and Imamul Huq, 2007). Iron concentrations in the supernatant were determined with a Varian Spectraa-220 AAS. All assays were carried out in triplicate and only the mean values are presented.

### 2.1. Calculation

Adsorption of iron (Fe) by the soils (X) was calculated by deducting the value in the equilibrium solution (C, supernatant) from the value of the original solution ( $C_0$ ). The zero Fe treatment was used as background Fe for the experiment and these values were subtracted from the others to correct for the Fe that was released from the untreated soil. The following formula was used for the calculation (Joardar et al., 2005, Begum and Imamul Huq, 2007).

$$X = \frac{V_0 C_0 - V_1 C_1}{W}$$

Where,

X = amounts of Fe adsorbed (mg/kg soil),

$V_0$  = initial volume of Fe solution (ml),

$C_0$  = initial concentration of Fe solution (mg/l),

$V_1$  = total volume of equilibrating solution (ml),

$C_1$  = final concentration of Fe in equilibrating solution (mg/l),

W = weight of soil taken (g).

The following form of the Langmuir adsorption equation was tested to calculate the adsorption coefficients:

$$\frac{C}{X} = \frac{1}{kb} + \frac{c}{b}$$

Where,

C = equilibrium concentration of Fe in solution (mg/l),

X = amounts of Fe adsorbed (mg/kg),

b = adsorption maximum (mg/kg),

k = constant related to the bonding energy of to the soil (mg/kg).

A plot of C/X versus C was made for each of the three soils. The values of adsorption maximum and bonding energy were calculated from slope (1/b) and intercept (1/kb) of the plot, respectively.

Desorption of iron by the soils (Y) were calculated by multiplying the Equilibrium concentration (Eq.Conc) in the solution and the volume of extraction (V) and dividing it by the weight (W) of the soil. The following formula was used for the calculation (Begum and Imamul Huq, 2007).

$$Y = (\text{Eq. Conc} \times V)$$

Where,

Y = Amount of desorbed material in the solution (ppm)

Eq. Conc (ppm) = (Machine reading x Dilution factor)/1000

V = Total volume of extraction for the soils taken (ml).

All data were subjected to statistical analysis by MINITAB programs and Microsoft Excel and were used for data representation and figure representation.

### 3. Results and Discussion

The common physical, chemical and physicochemical properties of the three soil series were analyzed and the results of the analyses are presented in the tables- 2 and 3.

Soil Series	Moisture Content (%)	% Sand	% Silt	% Clay	Textural Class
Amjuphi	5.6	37.4	34.5	28.1	Clay loam
Ishurdi	9.0	35.1	33.0	31.9	Clay loam
Dhamrai	1.6	34.9	61.1	4.7	Silt loam

Table 2: Moisture content and Particle size distribution and textural class of the soils studied

Soil Series	pH	Organic Carbon %	Organic Matter%	Total N %	CEC meq%	Exchangeable Cations Meq %			
						Ca	Mg	Na	K
Amjuphi	6.89	0.47	0.81	1.08	35.4	22.5	7.0	0.55	0.42
Ishurdi	7.73	0.62	1.10	0.57	41.6	31.3	4.5	0.45	0.35
Dhamrai	7.08	0.22	0.38	0.30	15.4	8.3	2.5	0.19	0.21

Table 3: Various chemical and physicochemical properties of the three soils

#### 3.1 Sorption Studies of Iron

The amounts of iron adsorbed (X) and the equilibrium concentration of Fe in the solution (C), at various treatments of Fe (Co) in solution followed over different time periods such as 2, 4, 6, 24 and 48 hour are shown in table- 4. The sorption values are presented as-

I. The sorption of Fe with increasing time for each of the three soils at various Fe treatments (T1=0.5 ppm, T2=1.0 ppm and T3=2.5 ppm) are shown in figure- 1, 2 and 3.

II. The sorption of iron for the three soils as affected by increasing concentration of iron along with increasing residence time are represented in figure-4.

The Langmuir coefficients were calculated, the values of the coefficients 'k' and 'b' are presented in the table 5. The statistical significance of the correlation coefficient 'r' for C/X verses C (from Langmuir adsorption equation) is also presented in table-5.

Soil Series	2H -Sorption		4H-Sorption		6H Sorption		24H Sorption		48H Sorption	
Initial Fe concentration (Co) 0.5 ppm										
	C	X	C	X	C	X	C	X	C	X
Amjhupi	0.1411	1.7945	0.1218	1.891	0.1019	1.9907	0.0872	6.1917	0.0691	6.4635
Ishurdi	0.1011	1.9945	0.0925	2.0375	0.0823	2.0926	0.043	6.844	0.0291	7.0635
Dhamrai	0.3438	0.7811	0.3233	0.8835	0.2868	1.0552	0.2585	3.6595	0.1319	5.5215
Initial Fe concentration (Co) 1.0 ppm										
Amjhupi	0.2211	3.8945	0.2019	3.9905	0.1992	4.0233	0.1607	12.589	0.1012	13.482
Ishurdi	0.1521	4.2395	0.1311	4.3445	0.113	4.4348	0.0848	13.747	0.0641	14.0385
Dhamrai	0.6731	1.6345	0.5432	2.284	0.514	2.43	0.424	8.6149	0.2651	11.0445
Initial Fe concentration (Co) 2.5 ppm										
Amjhupi	0.4119	10.441	0.3981	10.51	0.3594	10.703	0.259	33.615	0.191	34.635
Ishurdi	0.2719	11.141	0.251	11.245	0.2205	11.398	0.1918	34.624	0.1181	35.7285
Dhamrai	1.516	4.9199	1.7857	5.5717	1.642	6.2902	1.459	15.615	1.1388	20.4175

Table 4: Equilibrium concentration (C) and sorption (X) of Fe at various Fe treatments (Co) followed over different contact time

- C = Iron left in equilibrium solution
- Co = Initial iron concentration
- X = Iron sorbed by soils

Time(H)	2 H			4 H			6 H			24 H			48 H		
Soil Series	B	k	r	B	K	r	B	k	r	b	k	R	b	k	r
Amjhupi	3.03	4.747	0.9726	0.44	165.6	0.7812	0.04	63.2	0.9573	0.01	83.3	0.9611	0.34	55.5	0.9813
Ishurdi	0.29	12.9	0.9811	0.83	54.34	0.9568	0.54	85.8	0.9852	0.4	95.3	0.989	0.99	72.3	0.9933
Dhamrai	0.2	66.56	0.9594	0.77	16.47	0.2035	7.97	10.6	0.9112	1.13	10.9	0.9662	1.03	8.44	0.967

Table 5: Values of the Langmuir coefficients 'b' and 'k' and correlation coefficient 'r' for the soils studied

### 3.1.1. Effect of Time

Iron sorption increased sharply with increasing residence time in all the three soils (Figure- 1, 2 & 3). There was a slow increase in iron sorption from 2 to 6 hour followed by a sharp increase from 6 to 24 hour of residence time while after 24 hour, sorption in all the soils were maximum than the initial sorption. However, Ishurdi soil showed a relatively higher sorption than either Amjhupi or Dhamrai soil. The maximum sorption of 35.72 ppm was observed for Ishurdi soil which is calcareous in nature followed by non-calcareous Amjhupi soil(34.64 ppm) and Dhamrai soil (20.42 ppm) at 48 hour of sorption period. The lowest value of sorption was observed for the Dhamrai soil (0.78ppm) at 2 hour sorption, which was non calcareous in nature. It is also interesting to note that, at any sorption period (either 2, 4, 6, 24 or 48 hour) Ishurdi soil showed maximum sorption followed by Amjhupi and Dhamrai soil. The result is supported by the previous study of G Karthikeyan et. al., 2005; who found that adsorption of ferric iron with time was smooth, continuous and sharp by chitin. In another study, the mobility through a calcareous soil of the studied iron chelates differs greatly depending on the type of iron chelate and also on the procedure used to evaluate the retention and the soil/ solution ratio used and sorption increased with the interaction time (Hernandez & Lucena, 2011). The regression analysis showed that the most significant sorption with time has occurred after 48 hour of sorption for the Ishurdi soil( $r=0.9933$ ) which is calcareous in nature, followed by the Amjhupi ( $r=0.9813$ ) and Dhamrai soils ( $r=0.967$ ) which are non calcareous in nature.

### 3.1.2 Effect of Soil Properties

The adsorption study by different types of soil indicated that sorption capacity is dependent on its texture or clay content. The analysis showed that (Table-2) the Ishurdi and Amjhupi soil series have clay loam texture with 31.9% and 28.1% clay content, while the Dhamrai soil have Silt Loam textural class with only 4.7% clay content. The soil with higher clay content exhibits maximum surface area for sorption and thus the Ishurdi series helps in maximum sorption followed by Amjhupi and Dhamrai series. Calcareousness of the soil might have influencing effect in iron sorption. The experiment shows that calcareous soils have maximum sorption capacity irrespective of the treatment of iron and sorption period allowed. Thus the sorption followed the trend-

- **Ishurdi>Amjhupi>Dhamrai**

Previous study by Kang et. al., 2010 showed that surface protonation of variable charges sites increased with decreasing pH yielding positively charged sites on crystal edges and enhancing the attractive force between minerals and iron cyanide complexes. Another study showed that the ability of a soil to retain cation generally improves when soil pH is increased through liming (Thompson & Goyne, 2012). In present study Calcareous Ishurdi soil have highest pH of 7.73 and also have free calcium carbonate which might increase iron sorption.

### 3.1.3. Concentration of Treatment Solution

Adsorption of iron increased in all the three soils with the increase in concentration of the treatment solution. In all the case the highest treatment i.e. 2.5ppm iron treatment caused maximum sorption and 35.73ppm iron was adsorbed after maximum contact period (48 hour) allowed for Ishurdi soil followed by 34.64ppm sorption in Amjhupi soil and 20.42ppm for Dhamrai soil. On the contrary the lowest amount of iron was adsorbed for soils which were in contact with the lowest concentration of iron (0.5ppm) for lowest contract period.

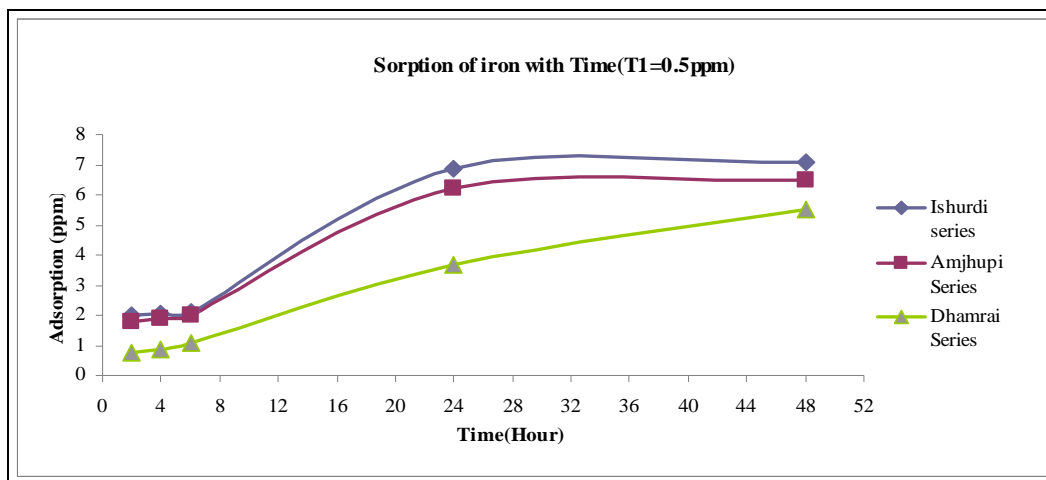


Figure 1: Sorption of iron with time for the three soils at 0.5 ppm treatment

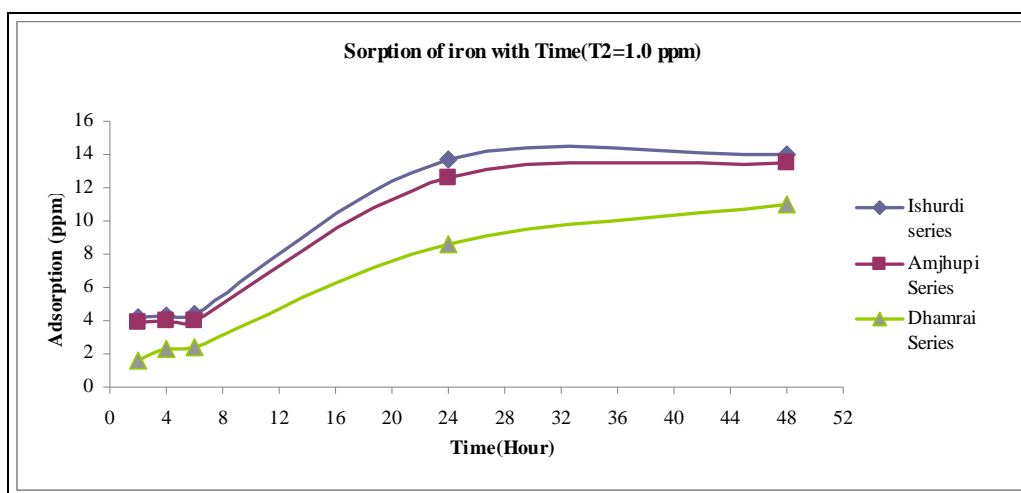


Figure 2: Sorption of iron with time for the three soils at 1.0 ppm treatment

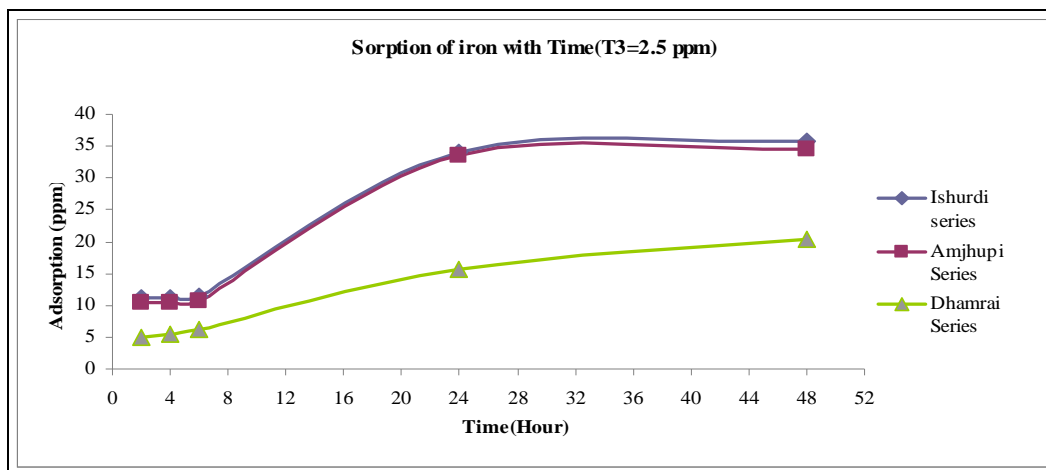


Figure 3: Sorption of iron with time for the three soils at 2.5 ppm treatment

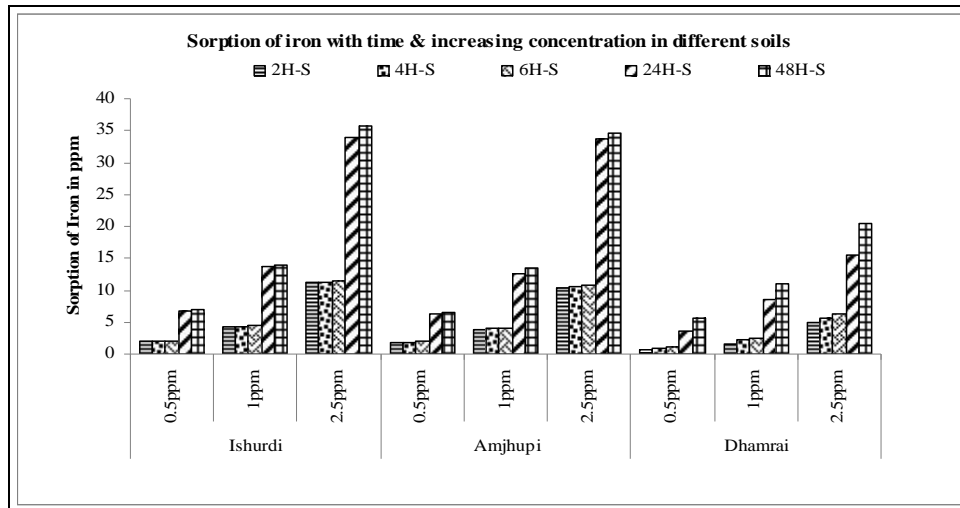


Figure 4: Sorption of iron with increasing time and concentration in different soils

3.2. Desorption Studies of Iron

Desorption of iron from the three soils were done individually for 6 and 24 hour contact time of sorption. Preliminary study showed that 24 hour contract time is the best for maximum iron adsorption. Desorption included 2, 4, 6 and 24 hour of extraction to desorbs iron from the soils. The values of desorption, represented by ‘Y’ and the correlation studies of desorption with increasing time were calculated.

3.2.1. Desorption of Iron after 6 hour sorption

The values of desorption (Y) and the correlation coefficient (r) between the contact time of desorption and desorbed iron for the soils were calculated and are represented in table 6. The desorption of Fe for the three soils as affected by increasing concentration of Fe along with increasing contact time for desorption are represented in figure 5

Soil Series	Time (H)	0.5 ppm		1.0 ppm		2.5 ppm	
		Y	r	Y	r	Y	R
Amjhupi	2	0.167	0.9913	0.22665	0.99659	0.33665	0.99617
	4	0.3544		0.43395		0.54665	
	6	0.88415		0.90515		1.02805	
	24	2.5725		2.918		3.02435	
Ishurdi	2	0.4637	0.95713	0.5475	0.98184	0.60115	0.97884
	4	0.89545		1.0515		1.2515	
	6	2.61565		2.47965		2.38785	
	24	5.2495		5.9545		5.28335	
Dhamrai	2	0.1306	0.99604	0.17275	0.98039	0.23775	0.99966
	4	0.2465		0.32885		0.352	
	6	0.51285		0.6745		0.54315	
	24	1.60735		1.528		2.062	

Table 6: The values of desorption (Y) and the correlation coefficient ‘r’ between desorption of Fe with time for 6 hour sorption

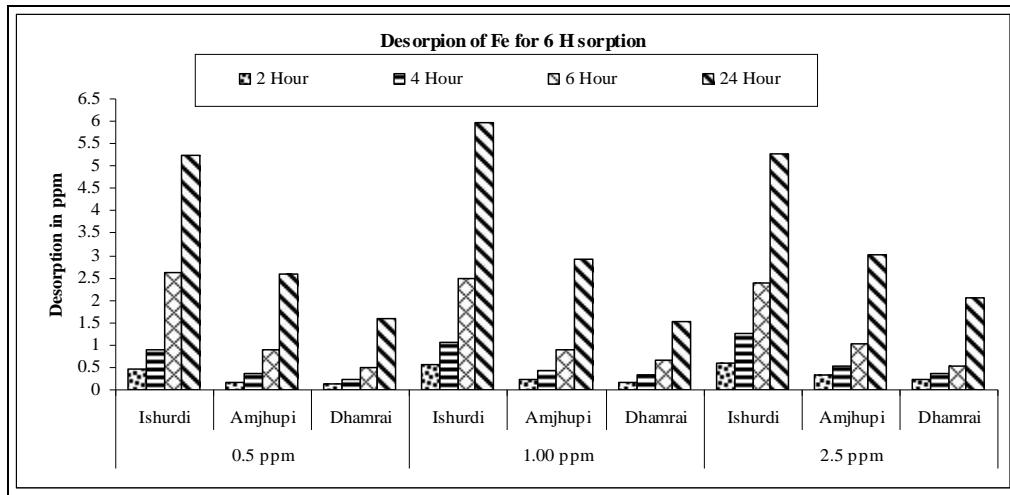


Figure 5: Desorption of Fe by the soils for each Fe treatment after 6 H sorption

The graphical representation and the table-6 showed that, desorption of iron varied with changes in contact time, solution concentration or the nature of the soils. After 2 hour the highest desorption was found for Ishurdi soil which were 0.4637 ppm, 0.5475 ppm and 0.6012 ppm at 0.5 ppm, 1.0 ppm and 2.5 ppm iron treatment. It was observed that, with increasing desorption period, iron desorption increases for all the soils. At 24 hour of extraction period desorption was highest for all the soils. The highest value of desorption was 5.2495 ppm, 5.9545 ppm and 5.2833 ppm at 0.5 ppm, 1.0 ppm and 2.5 ppm iron treatment for 24 hour desorption. The desorption pattern followed the order-

- **Ishurdi > Amjhupi > Dhamrai**

When the solution concentration of iron was increased then desorption varied from soil to soil. Among the three treatments, 1.00ppm showed maximum significant desorption (0.5475 ppm, 1.0515 ppm, 2.4796 ppm and 5.9545 ppm after 2, 4, 6 and 24 hour) but when the concentration of iron in the solution increased then desorption were relatively lower ( 0.6012 ppm, 1.2515 ppm, 2.3879 ppm and 5.2834 ppm after 2, 4, 6 and 24 hour). This indicates that when concentration of the treatment solution increased there may be further adsorption which decreases the concentration of iron in solution.

Moreover the study has showed that with change in soil properties desorption pattern is also changing. Soil properties such as pH, clay content, organic matter content, cation exchange capacity, calcareousness of the three soils (table 2 and 3) are different, which have a great influence on this desorption. The correlation analysis showed that the most significant desorption with time was observed for Amjhupi soil followed by Dhamrai and Ishurdi soil series.

3.2.2. Desorption of iron after 24 hour of sorption

The values of desorption (Y) for 24 hour adsorption and the correlation coefficient ‘r’ for the soils are represented in table 7. The graphical representation of desorption of Fe for each of the three soils as affected by increasing concentration of Fe along with increasing contact time for desorption are represented in figure 6.

Soil Series	Time(H)	0.5 ppm		1.0 ppm		2.5 ppm	
		Y	r	Y	r	Y	r
Amjhupi	2	0.0955	0.99844	0.1150	0.9988	0.1383	0.99816
	4	0.1511		0.2017		0.2462	
	6	0.3642		0.4837		0.5235	
	24	1.9424		2.1184		1.8797	
Ishurdi	2	0.3375	0.9778	0.3817	0.95561	0.4361	0.97798
	4	0.6796		0.8923		0.7660	
	6	1.2400		1.6910		1.6042	
	24	2.6690		3.1687		3.5194	
Dhamrai	2	0.0611	0.99725	0.0710	0.99869	0.1127	0.98572
	4	0.0969		0.1361		0.2517	
	6	0.1975		0.2625		0.4927	
	24	1.3234		1.5079		1.2102	

Table 7: The values of desorption (Y) and the correlation coefficient ‘r’ between desorption of Fe with time after 24 hour sorption

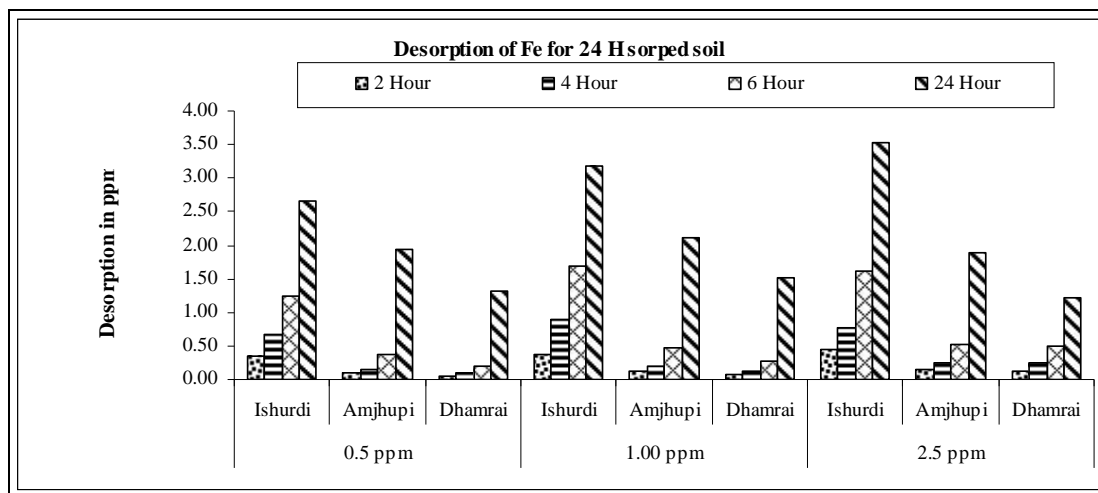


Figure 6: Desorption of Fe for Amjhupi; Ishurdi and Dhamrai at various treatments after 24 H sorption

The graphical representation and the table-7 showed that, desorption of iron varied with changes in contact time, solution concentration or the nature of the soils. It was observed that, with increasing adsorption period iron desorption decreases for all the soils. The reason for this is, when the sorption period was increased from 6 hour to 24 hour a significant amount of iron remained sorbed on the soil solids preferably by ion exchange or by complexation. The research revealed that, when the soils were subjected for 24 hour sorption followed by 2 hour extraction, maximum desorption were 0.3575 ppm, 0.3817 ppm, 0.4361 ppm at 0.5 ppm, 1.0 ppm and 2.5 ppm treatment which decreased from desorption of soils subjected to 6 hour sorption followed by 2 hour desorption (0.4637 ppm, 0.5475 ppm and 0.6012 ppm). At 24 hour sorption followed by 24 hour desorption concentration of iron were also decreased than the soils which were subjected to 6 hour sorption followed by 24 hour desorption. The highest value of desorption for 24 hour sorbed soils were 2.6690 ppm, 3.1687 ppm and 3.5194 ppm at 24 hour desorption for Ishurdi soil. Excessively higher desorption was observed for Ishurdi soil series at 24 hour of desorption irrespective of the solution concentration. Here desorption followed the similar trend as described for 6 hour sorption period.

The desorption pattern after 6 hour and 24 hour sorption of the soils with iron have been shown to be similar when the concentration of iron in the solution was increased. However the relatively higher desorption was observed at 1.00 ppm treatment than the other treatments. The study showed that at although the solution concentration of iron was increased but relatively lesser amount of desorption was viewed, that is with increasing treatment there may be further sorption which may be due to chemisorptions and other soil properties. With changing properties of the soils desorption also changes and the most significant iron desorption was observed for non calcareous soils, Amjhupi ( $r=0.9988$ ) and Dhamrai ( $r=0.9986$ ) soil, followed by calcareous Ishurdi soil ( $r=0.9779$ ).

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