Appendix

A.1 Removal of Nickel using Composite

Among the toxicological profile of heavy metals, nickel stands at 57^{th} position with cumulative points of 996 as per the CERCLA section 104 [12], [520]. The concentration of nickel in industrial effluent discharging into potable water bodies ranges from 2-900 ppm [20]. The permissible level of discharge of nickel in potable water as demarked by USEPA, WHO and EU is 0.02 ppm [10], [11].

IACR has enlisted nickel compounds in group 1 (adequate proof for human carcinogenicity) and in group 2B (substances that may be carcinogenic to humans) [87]. Nickel has been reported as a human carcinogen in ACGIH intended notice as a Category A1 [85], [521]. However, nickel in acidic soil becomes more mobile and gets rapidly washed out into the groundwater. Other health issues associated with nickel intake in humans include dermatitis, nausea, chronic asthma, coughing, nephrotoxic, hemolysis and anaphylaxis [25]–[27].

Several physicochemical methods of nickel abatement from aqueous phase like floatation, membrane filtration, photocatalysis, electrochemical process, coagulation and flocculation [8] have been comprehensively practiced in past but most of these methods are expensive, time-consuming and lead to the creation of secondary chemical sludge. The disposal of secondary chemical sludge in environment is another critical issue. On the contrary, adsorption of nickel ions in the liquid phase (real/ synthetic simulated wastewater) has gained exceptional attention by scientists in the last few decades. Adsorption is an inexpensive and eco-friendly method of metal ion remediation from wastewater.

Various scientists [522]–[527] have focused on finding an alternative adsorbent namely clay like kaolinite, montmorillonite, illite and red ochre other than activated carbon for removing nickel from wastewater as activated carbon is uneconomical to use. The surface of bentonite clay is densely packed with ions, which interchange with anions and cations during adsorption [528], [529]. Bentonite clay is produced in bulk volume in the form of ash during volcanic eruptions and is mined from the earth crust [526]. The primary deposit of bentonite clay is in Montana, US [530]. The worldwide production of bentonite clay is 14, 600,000 tonnes per year [531]. Red ochre is a natural red iron oxide that contains hematite as dominant iron oxide. It also comprises of several white pigments such as alumino-silicate (kaolinite or illite), quartz and calcium compounds (calcite, gypsum and dolomite). The varying proportion of hematite and goethite determines the color of ochre from red to yellow and some shades in between. Red ochre is shaped from various natural and anthropogenic sources namely from the natural aerobic decomposition of iron-bearing minerals and as precipitate obtained from coal mine water [164], [170]. The worldwide production of red ochre is 192,000 tonnes per year [171].

It is pertinent to mention here that adsorption of nickel (Ni^{2+}) solely on composite made up of bentonite clay and red ochre has not been done till date.

The adsorption dynamics in the liquid phase is divided into bulk diffusion of sorbate, transfer of adsorbate from the solid-liquid interface to the solid surface and reshuffling of adsorbate ions on solid surface [532]–[534]. Accordingly, the adsorption kinetics is diffusion-limited, transport limited and reshuffling limited. The adsorption dynamics can be singly governed by any of these steps or a combination of them. Very scarce information on adsorption dynamics of metal ions in the liquid phase is available in the literature. The understanding of adsorption dynamics is essential to elucidate the rate-limiting mechanism and in designing and scale-up of continuous bioreactors for effluent treatment plants [535]–[539]. The present investigation based on these facts and in search of persuasive adsorbent aimed at the development of composite made up of bentonite clay and red ochre for removal of Ni^{2+} ions from wastewater. Additionally, physico-chemical characterization of composite and the adsorption dynamics of nickel ions have been studied in detail to comprehend the basic mechanism of Ni^{2+} ions adsorption on the composite surface by deriving dimensionless numbers and by mathematical modeling of experimental data in various isotherms, kinetic, mechanistic and thermodynamic correlations. A comparative study of composite developed in present investigation has also been done with other inorganic adsorbents reported in other research works.

A.2 Results and Discussion

A.2.1 Physico-chemical Characterization

A.2.1.1 SEM-EDX

The SEM micrograph of the composite before and after adsorption have been shown in Figures 4.1a and A.1. It became evident from Figure 4.1a that composite domesticates

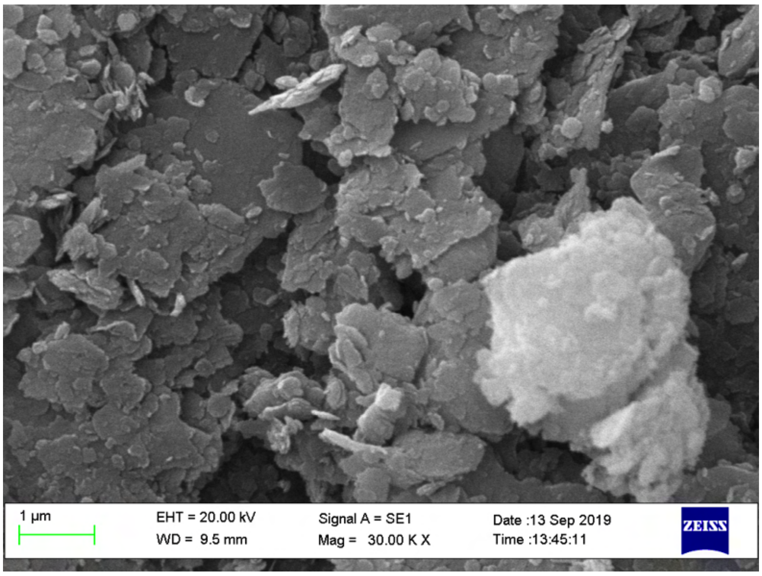


Figure A.1: SEM of composite after adsorption of Ni^{2+} ions

small particles consisting of platelets which conglomerate with the bigger particles. However, it became perceptible from Figure A.1 that fluffy layered sheet structure present in the Figure 4.1a transform into rough, foliated layered structure after the adsorption [247]–[249]. Similar results have been studied by Ogunmodede et al., 2015 [250] in which platelets like arrangement of bentonite clay assembles to form bigger particles. Bilal et al., 2016 [251] observed dispersed structures in bentonite clay due to the presence of alumina with random shapes in SEM micrograph.

The EDX of the composite has been shown in Figure A.2. After adsorption the significant amount of Ni^{2+} ions were observed in Figure A.2 and Table A.1.

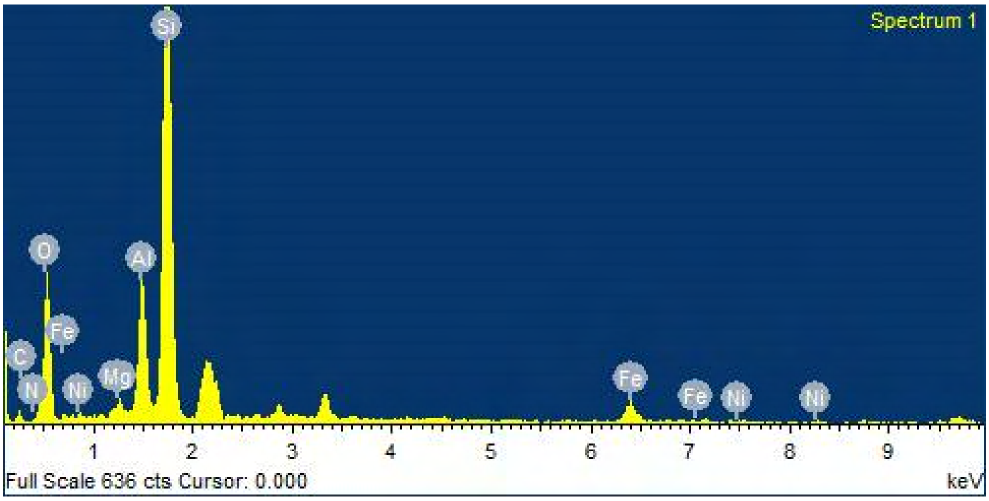


Figure A.2: EDX of composite after adsorption of Ni^{2+} ions

Table A.1: EDX of composite after adsorption

Element	Weight (%)	Atomic (%)
C K	22.13	30.96
N K	9.86	11.83
O K	38.60	40.53
Mg K	0.50	0.35
Al K	5.04	3.14
Si K	20.26	12.12
Fe K	3.18	0.96
Ni K	0.43	0.12
Totals	**100.00**	

A.2.1.2 XRD

The XRD analysis of composite before and after adsorption has been shown in Figure A.3 and Table A.2.

It has been observed from Figure A.3 that intensity of peaks decreases after adsorption process which in turn reflects adsorption of Ni^{2+} ions on composite surface which re-

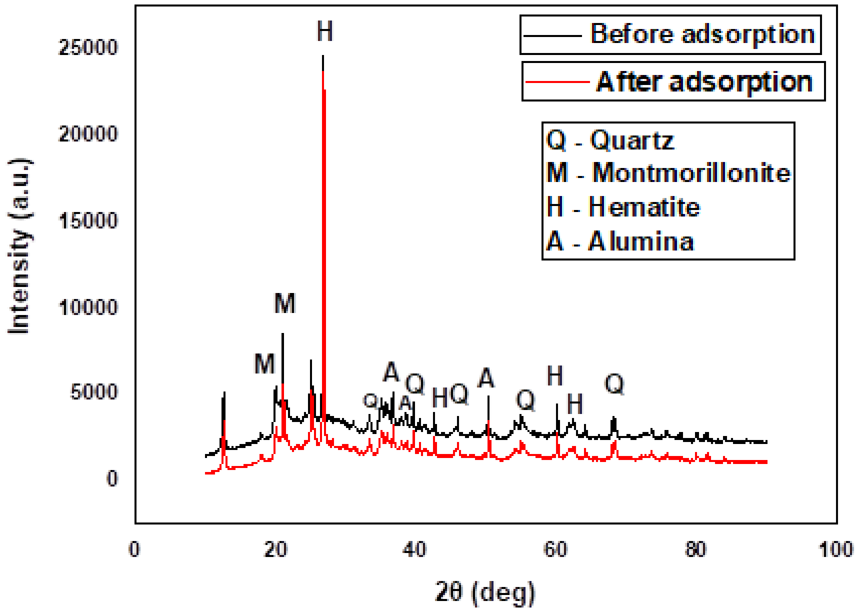


Figure A.3: XRD of composite before and after adsorption of Ni^{2+} ions

Table A.2: Diffractometer major peaks parameters

Peak Center	Area	FWHM
12.48	2290.36	0.28255
19.98	4027.05	0.89497
20.96	4786.98	0.26003
26.7	8347.19	26.63683
26.74	8268.05	0.16658
33.3	2663.88	1.52
36.64	6884.83	2.57765
38.6	2008.9	0.94
39.54	2193.35	0.68468
42.54	2209.02	1.03848
45.88	2610.03	1.7
50.2	2372.7	0.29495
54.92	4027.03	2.18
60.02	3037.15	0.38529
62.36	2145.21	1.12
68.2	2712.58	1.33193

sulted in reduction of crystallinity of the composite. Similar reduction in crystallinity was observed by Rout et al., 2015 [259] during removal of phosphate ions by red soil. In the present work, peak positions before and after adsorption remain same and it was observed that the peaks at 2 θ (19.87°, 21.21° and 68.30°) were dedicated to montmorillonite. Similarly, the existence of quartz in composite was observed at 21.21°, 33.22°, 39.64°, 45.88°, 54.89° and 68.30°. Peaks at 35.89°, 38.60° and 50.39° reflected the presence of alumina in composite. The incidence of hematite was guaranteed at peaks 25.55°, 42.58°, 59.71° and 62.15°.

Quartz, alumina and montmorillonite occur naturally in bentonite clay and hematite is a significant component of red ochre [260]–[262]. Table A.2 gives information about peak center, area and FWHM (Full width at half maxima) of all peaks shown in Figure A.3. Initially, the crystallinity of the composite was found to be 49.90 %.

Bugoi et al., 2008 [261] investigated ceramic pigments using XRD technique and showed the presence of red color due to hematite (Fe_2O_3) which indicated an enormous amount of iron in the pigments. Roman et al., 2015 [263] analyzed red ochre of the burial and found diffractogram peaks of hematite near 42° and 62° which were similar to the present study. Rotondo et al., 2010 [260] did characterization of fifty different kinds of pigments using XRD technique and found peaks of Fe_2O_3 at 42°, 63° and 84° that shows resemblance to the existing result. The presence of quartz was also confirmed by Rotondo et al., 2010 [260] and Bugoi et al., 2008 [261] near 20°, 34° and 48° in the pigments which were similar to the present work. Fil et al., 2014 [262] characterized several properties of montmorillonite and deduced its dioctahedral structure through XRD peak near 68°. Dankova et al., 2010 [264] showed the presence of diffractogram peaks near 20° and 68° that confirmed the presence of montmorillonite in the composite.

A.2.1.3 FTIR

The FTIR of the composite has been shown in Figure A.4. Peaks at 3617.51 cm^{-1} and 3436.11 cm^{-1} were attributed to the asymmetric and symmetric stretching of the hydroxyl group, respectively due to Ca $(OH)_2$ structures in bentonite clay. Peaks at 1648.24 cm^{-1}, 1110.80 cm^{-1} and 1034. 47 cm^{-1} showed bending vibration of the H-O-H group and Si-O bond stretching, respectively. Sharp transmittance between 915.48 cm^{-1} and 789.87

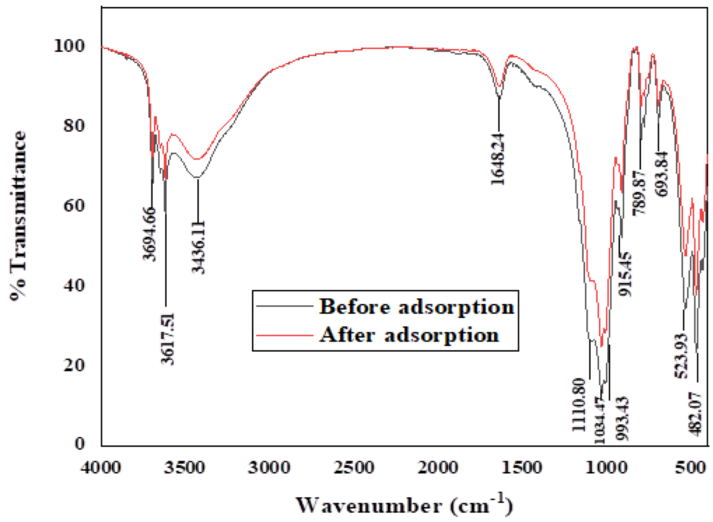


Figure A.4: FTIR of composite before and after adsorption of Ni^{2+} ions

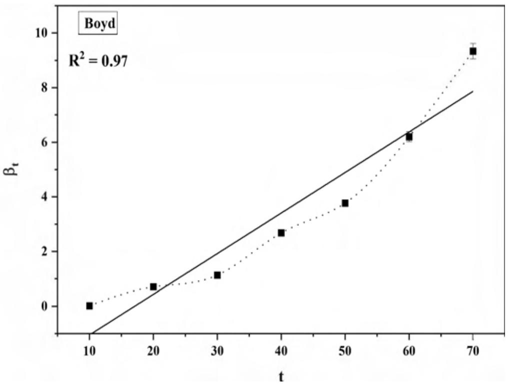
cm^{-1} was due to octahedral sheets present in the composite. Si-O-Al bending was seen near 693.84 cm^{-1} that shows similarity to the results of De Oliveira et al., 2016 [257]. Two prominent bands at 523.93 cm^{-1} and 482.07 cm^{-1} were assigned for hematite of ochre that showed similarity with the study of Mortimore et al., 2004 [258]. A high-intensity band was also observed at 482.07 cm^{-1} that corresponded to Si-O-Si bending vibration. De Oliveira et al., 2016 [257] performed the characterization of bentonite clay and found transmittance peaks at 3698, 3622, 3441, 1638, 1113, 1041, 912, 794, 693, 535, 472 cm^{-1} that were similar to the present study, showing similar functional groups presence on the surface of adsorbents.

The FTIR findings were in agreement with the X-ray diffraction outcomes. No significant shifting of the functional group was observed in FTIRs of composite before and after adsorption. This insignificant shifting revealed the fact that the chemical bonding (chemisorption) between Ni^{2+} ions and functional groups on the surface of the composite was not the key mechanism of adsorption in the present study.

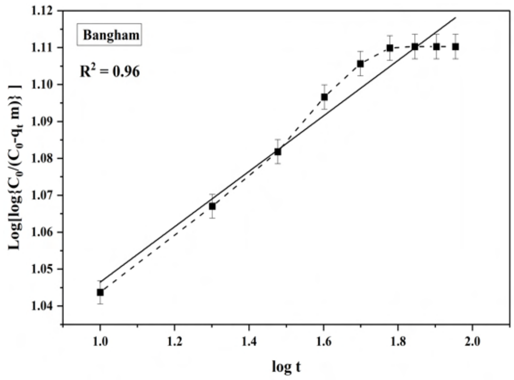
A.2.2 Mechanistic Study

As shown in Figures A.5a and A.5c, the line did not pass through the origin and curves were of multi-linear type which indicated that adsorption was not only controlled by intra-particle model but also by film diffusion [540].

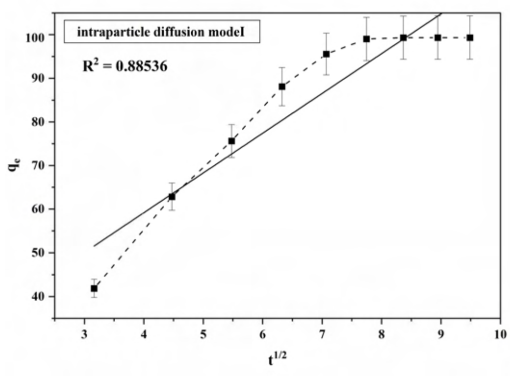
The value of R^2 for Boyd film diffusion model (R^2 = 0.97) was higher than that of intra-particle diffusion model (R^2 = 0.88) implying that adsorption was primarily dominated by film diffusion [541] followed by intraparticle diffusion.



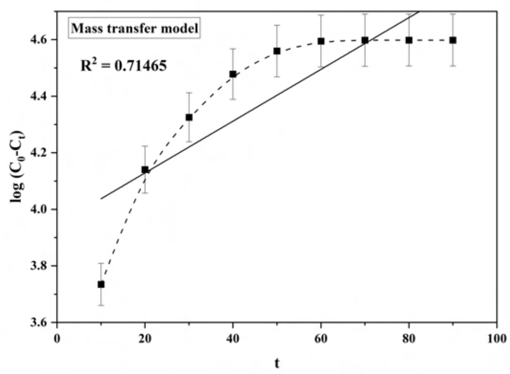
(a)



(b)



(c)



(d)

Figure A.5: Mechanistic models for adsorption of Ni^{2+}ions

Table A.3: Mechanistic model parameters

Model	Parameters	Value	R^2	RMSE	χ^2
Boyd	Slope	0.148	0.97	5.84	0.73
	Intercept	-2.53			
Bangham model	k_0 (mL/g/L)	21.54	0.96	1.83××10^{-4}	0.22 ××10^{-4}
	α	0.0751			
Intraparticle diffusion	k_P (mg/g $min^{1/2}$)	9.12	0.88	386.33	48.29125
	C	22.68			
Mass transfer	D	51.67	0.71	0.200	0.025
	k_0	0.00915			

Additionally, in the present study, it became apparent from Figure A.5d that the plot of log

(C_0 - C_t) vs. t was not linear collectively with low R^2 value which represented the fact that adsorption process was not restricted by mass transfer model. The values of mechanistic model constants are shown in Table A.3.

Similar results have been observed by Dada et al., 2016 [76] during adsorption of copper ions on the bamboo supported manganese nano-composites. The high regression coefficient of Bangham's (Figure **??**) model together with squat values (Table A.3) of SSE and χ^2 also complemented the primacy of film diffusion in the present work [542] over intraparticle diffusion.

A.2.3 Adsorption Dynamics

In the present work, values of the film (0.65×10^{-8} cm^2 sec^{-1}) and pore diffusivity (1.8 $\times$ 10^{-12} cm^2 sec^{-1}) coefficients showed that the adsorption process was dependent upon both film and pore diffusion.

The value of dimensionless numbers φ, λ and N_k were calculated as 2.62, 1.17×10^{-5} and 62.68, respectively. The value of N_k fell between 10^1 and 10^4 which elucidated that adsorption dynamics in the present study is controlled by film diffusion at the onset of adsorption followed by intraparticle at later stage. The value of φ and λ were in range of 10^{-2} to 10^4 and 10^{-12} to 10^8 which showed utmost coverage of composite surface during adsorption with trim downed surface tension [213], [272].

A.2.4 Optimization Study

A.2.4.1 Effect of pH

The effect of pH is shown in Figure A.6.

The adsorption of Ni^{2+} ions were mainly influenced by the surface charge of composite which in turn is controlled by pH of the solution. The effects of pH were investigated within the pH range of 1 to 10 (Figure A.6). There was sharp increase in percentage removal of Ni^{2+} ions from 75 % to 84 % with an increase in pH from 1 to 6. Then, a slight increase in adsorption was observed between pH 6 to 8. Over pH 8, rapid adsorption

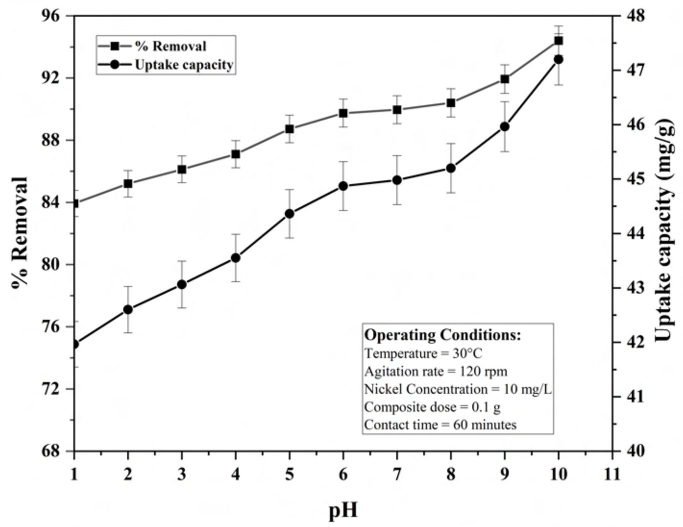


Figure A.6: Effect of pH on the removal of Ni^{2+}ions

of Ni^{2+} ions were observed due to precipitation of Ni^{2+} and $NiOH^{+}$ ions as Ni $(OH)_2$. Similar sorts of results were conveyed by Sandeep and Suresha, 2013 [543] for removal of Ni^{2+}ions from electroplating wastewater. Removal of Ni^{2+} ions were less significant at lower pH due to the prime presence of H^{+} ions in liquid phase resulting in competition between H^{+} and Ni^{2+} ions for the same active site. However, with increase in solution pH there was a decrease in H^{+} ions concentration which resulted in higher adsorption of Ni^{2+} ions on active sites. Similar findings were observed by Bennour, 2013 [544] for adsorption of nickel onto the clay. In the present study the optimum pH was observed as 6.

A.2.4.2 Effect of Composite Dose

The effect of composite dose on the adsorption of nickel has been depicted in Figure A.7.

It became clear from Figure A.7 that with the increase of adsorbent dose from 0.1 to 1.0 g/L, there was an increase in percentage removal of nickel ions due to more availability of active sites. However, with increase in the composite dose from 0.1 to 1 g, uptake capacity declines sharply because Ni^{2+} ions concentration (mass transfer gradient) in solution falls at a higher biomass dose and the system attains equilibrium between solid and aqueous phase at lower uptake capacity (9.9 mg/L in the present work). The maximum uptake capacity observed in the present investigation was 45.61 mg/L at 0.1 g of composite dose.

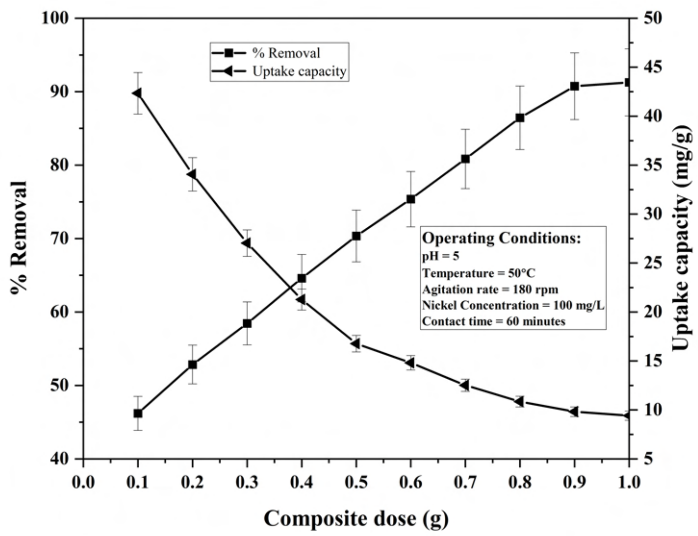


Figure A.7: Effect of composite dose on % removal and adsorption capacity

Similar results have been also reported by Al-Shahrani et al., 2012 [545] and Sandeep and Suresha, 2013 [543]. In the present study the optimum adsorbent dose was observed as 0.1 g.

A.2.4.3 Effect of Initial Concentration of Ni^{2+} Ions

The effect of initial concentration of Ni^{2+} ions has been depicted in Figure A.8.

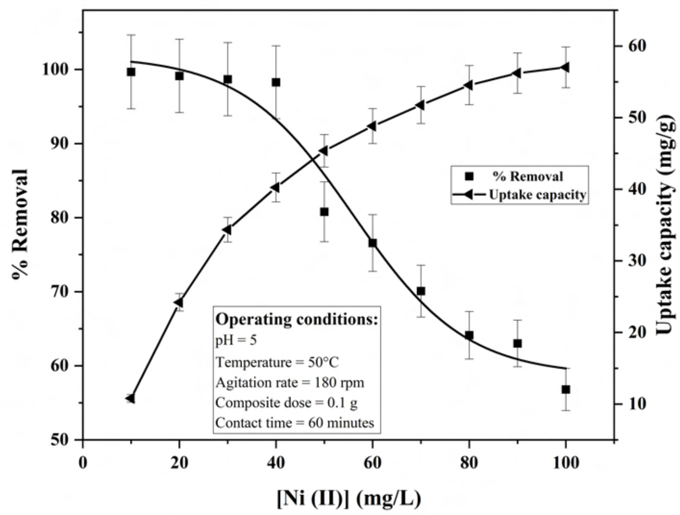


Figure A.8: Effect of the initial concentration of Ni^{2+} on removal and uptake capacity

It was observed from Figure A.8 that the percentage removal of Ni^{2+} ions decline with an increase in concentration of Ni^{2+} ions. The rationale behind this decrease was elevated concentration of Ni^{2+} ions per unit dose of composite. Furthermore, the increase in uptake capacity of composite from 15 to 59 mg/g with an increase in Ni^{2+} concentration from 10 to 100 mg/L was due to enhancement in driving force generated in liquid phase to overcome the mass transfer resistance at solid-liquid interphase. The results of the present study showed similarity with the findings of Ogunmodede et al., 2015 [250] and Zhang and Wang, 2015 [546]. In the present study the optimum concentration of Ni^{2+} ions were observed as 100 mg/L.

A.2.4.4 Effect of Temperature

The effect of temperature on adsorption of Ni^{2+} ions is shown in Figure A.9. It became ap-

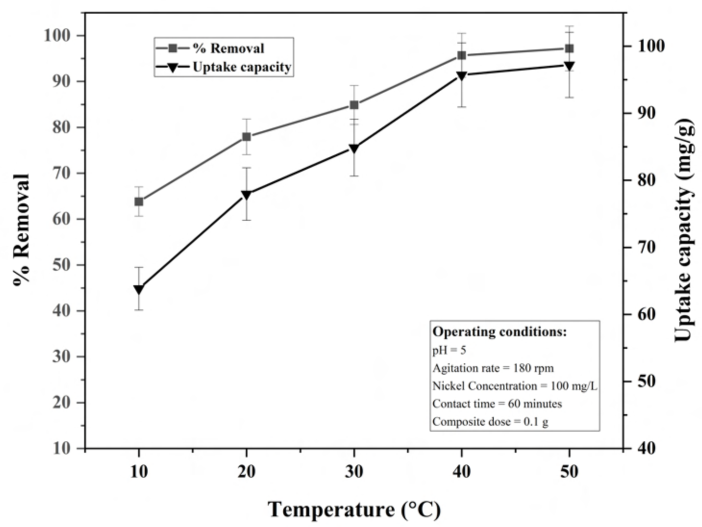


Figure A.9: Effect of temperature on the removal of Ni^{2+} ions

parent from Figure A.9 that with an increase in temperature, there was subsequent increase in uptake capacity and percentage removal. As temperature increases, rate of diffusion of Ni^{2+} ions also get enhanced across the film of liquid around composite. The increase in diffusion rate was due to reduction in thickness of liquid boundary layer surrounding the composite. Similar types of results were observed in the study done by Zhang and Wang, 2015 [546]. In addition to this, elevated temperature stimulated the binding of Ni^{2+} ions with the composite surface, suggesting that an endothermic mechanism regulated the adsorption of Ni^{2+} ions onto the surface of composite. In the present investigation, 50°C was observed as optimum temperature for further experiments. Similar findings were observed by Malkoc and Nuhoglu, 2010 [547] during Ni^{2+} adsorption. The authors discussed the decrement in thickness of boundary layer around the adsorbent with increase in temperature which ultimately resulted in higher adsorption at elevated temperature.

A.2.4.5 Effect of Agitation Rate

The effect of agitation rate on removal of Ni^{2+} ions has been shown in Figure A.10.

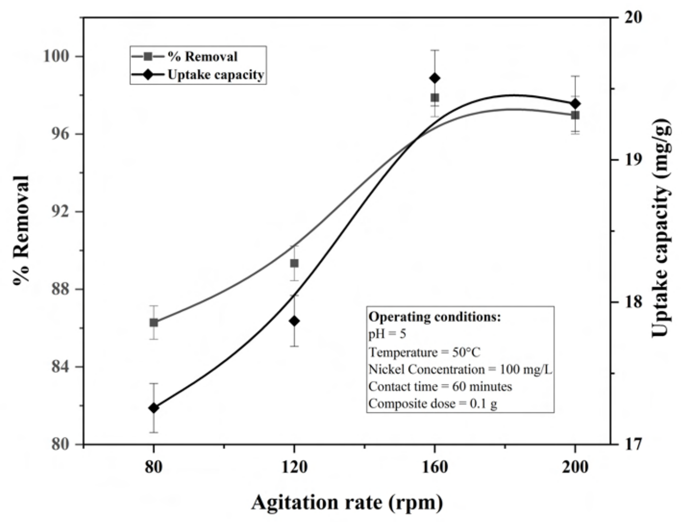


Figure A.10: Effect of agitation rate on the removal of Ni^{2+}ions

It became comprehensible from Figure A.10 that adsorption of Ni^{2+} ions increased with the increase in agitation rate. This happened due to the fact that the increased agitation rate reduced the external mass transfer resistance, allowing the metal ions to dwell on the clay surface. Therefore, the driving force for internal diffusion has increased and lead to higher adsorption. Potgieter et al., 2006 [548] reported similar views, claiming that the rise in agitation rate increases the constant rate of adsorption between metal ions and adsorbent in the adsorption system. Ghodbane et al., 2008 [549] also inferred that diffusion of Ni^{2+} ions from the bulk phase to liquid film surrounding the composite increases with increase in agitation rate. Authors pointed out the fact that higher agitation rate substantially reduces the thickness of the film which in turn augments the rate of adsorption. Similarly, McKay and Gordon, 1982 [550] noticed an increase in uptake capacity with the rise in agitation speed and mentioned the fact that at high agitation speed, external mass transfer coefficient enhances considerably resulting in higher rate of adsorption. Results of McKay and Gordon, 1982 [550] and Ghodbane et al., 2008 [549] were in support of the present investigation. Thus, optimum agitation rate for the adsorption of Ni^{2+} ions on surface of composite was 180 taken as rpm.

A.2.4.6 Effect of Contact Time

The influence of contact time on adsorption of Ni^{2+}ions has been shown in Figure A.11.

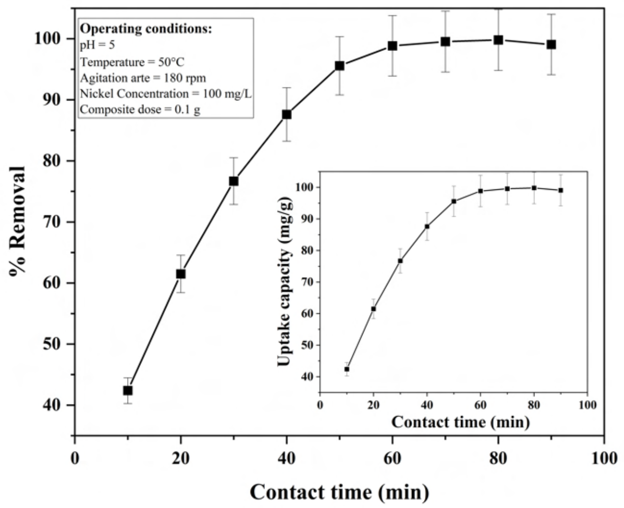


Figure A.11: Effect of contact time on the removal of Ni^{2+}ions

It became obvious from Figure A.11 that with an increase in contact time from 10 to 60 minutes, removal of Ni^{2+} ions increase from 41.88 % to 99 %. However, after 60 minutes, no further removal was observed. This showed that 60 minutes were sufficient to achieve the adsorption equilibrium. Thus, adsorption of Ni^{2+} ions did not increase even when the contact time was extended up to 2 hours. This response was due to tremendous availability of active sites on the surface of composite during the first 60 minutes. Thereafter, the entire surface of composite was saturated and hence no further removal was observed. Similar observations were recorded by Ogunmodede et al., 2015 [250] and Zhang and Wang, 2015 [546].

A.2.5 Adsorption Kinetics

Figure A.12 and Table A.4 shows the adsorption kinetics of Ni^{2+} ions on the surface of composite in the aqueous phase.

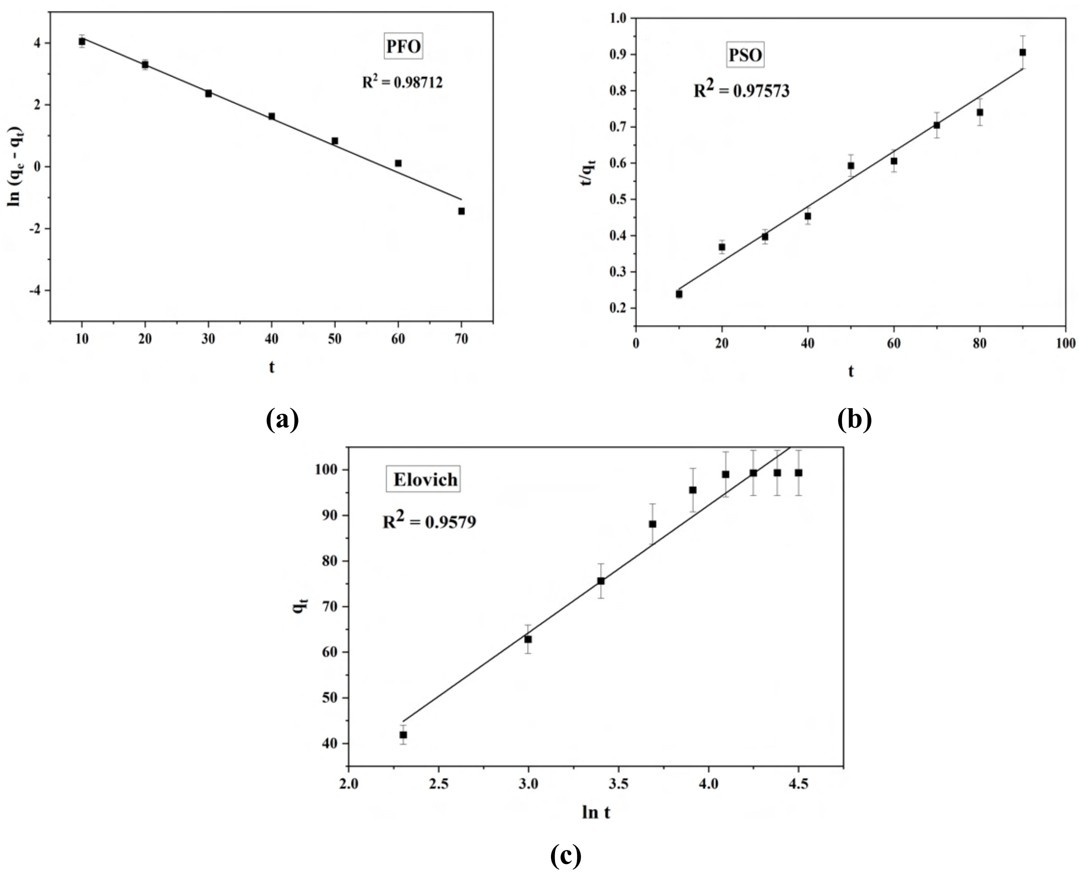


Figure A.12: (a) PFO, (b) PSO model and (c) Elovich kinetic models for removal of Ni^{2+} ions using composite

In the present work, the adsorption kinetics of Ni^{2+} ions have been studied by using three kinetic models. In an attempt to find out the parameters for each kinetic model, experimental results were verified for the best linear fit. The result showed that the adsorption of Ni^{2+} ions on the composite was best defined by the c kinetic model with a high coefficient of regression (R^2 = 0.99) compared to Elovich (R^2 = 0.95) and PSO kinetic model (R^2 = 0.97), thus signifying that the rate of adsorption was limited by physisorption mechanism. Similar results were provided by Ghogomu et al., 2013 [551] during removal of lead ions with kaolinite and Inam et al., 2017 [552] during optimization of process parameters in dye abstraction.

Table A.4: Adsorption kinetic models parameters for Ni^{2+} ions removal

Model	Parameters	Value	R^2	RMSE	χ^2
Pseudo-first order	q_e (mg/g)	153.24	0.99	0.27	0.04
	K_1 (min^{-1})	0.08			
Pseudo-second order	q_e (mg/g)	131.75	0.97	0.008	0.0008
	K_2 (g/mg min)	0.0003			
Elovich	α (mg/g min)	13.88	0.95	141.57	0.0004
	β (g/mg)	0.04			

A.2.6 Adsorption Isotherm

In the present study, Langmuir (Type-I, II, III, IV, V), R-P, F-H, Temkin, Toth, Hill, Sips, K-C, FS-5, Khan, Radke-Prausnitz, D-R, F-G, Elovich, Freundlich and Halsey models have been tested (Figure A.13 and Table A.5).

Table A.5: Adsorption isotherm model parameters for removal of Ni^{2+}ions using composite

Isotherm	Parameter	Value	R^2	SSE	χ^2
Langmuir (Type - II)	q_m(mg/g)	56.31	0.99	0.01	0.00096
	KL (L/mg)	0.75			
Langmuir (Type - I)	q_m (mg/g)	44.72	0.96	$2.4\times\times10^{-4}$	0.00003
	K_L (L/mg)	8.25			
Langmuir (Type - V)	K_L (L/mg)	7.73	0.95	32.43	4.05
	q_{max} (mg/g)	45.74			
R-P	K_R (L/g)	470.70	0.94	120.72	17.24
	a_R (L/mg)	0.90			
	g	12.44			
F-H	n_{FH}	-0.47	0.93	0.5451	0.07
	K_{FH} (L/mol)	0.01			
Temkin	A_T (L/g)	262.43	0.93	153.23	19.15
	b (kJ/mol)	+421.69			

Toth	q_{mT} (mg/g)	60.60	0.92	160.46	22.92
	a_T	0.23			
	z	0.39			
Hill	q_H (mg/g)	55.85	0.92	171.65	24.52
	K_D	0.56			
	n_H	0.56			
Sips	K_s (L/g)	55.88	0.92	171.65	24.52
	α_s	1.77			
	β_s	0.55			
K-C	A	99.67	0.92	171.65	24.52
	B	1.78			
	n_K	0.56			
F-S (V)	K_1 (mg/g)	59.43	0.90	216.22	43.24
	K_2 (mg/g)	114.75			
	q_m (mg/g)	59.43			
	α_{FS}	0.221			
	β_{FS}	0.058			
Khan	q_{max} (mg/g)	1.84	0.90	216.24	30.89
	a_K	0.84			
	b_K	$2.87\times\times10^{7}$			
Radke-Prausnitz	q_{mrp} (mg/g)	1.83	0.90	216.24	30.89
	K_{RP}	$2.87\times\times10^{7}$			
	m_{RP}	0.83			
D-R	β	$2.3\times\times10^{-8}$	0.89	0.29	0.04
	E (J/mol)	4662.52			
	q_m (mg/g)	46.06			
F-G	w (kJ/mol)	-31923	0.89	20.88	2.61
	K_{FG} (L/mg)	$0.3\times\times10^{-8}$			
Elovich	K_E (L/mg)	2.48	0.88	4.481	0.56
	q_m(mg/g)	8.04			
Freundlich	n	5.01	0.86	0.07	0.01
	K_F (L/mg)	4.25			
Halsey	n_H	5.01	0.86	0.37	0.04
	K_H (L/mg)	$1.8\times\times10^{7}$			
Langmuir (Type - III)	K_L (L/mg)	6.66	0.80	441.04	55.13
	q_{max} (mg/g)	48.28			
Langmuir (Type - IV)	K_L (L/mg)	5.34	0.80	15668.78	1958.60
	q_{max} (mg/g)	50.36			

The evaluated isotherm constants, regression coefficients, Chi-square and sum of square of errors for each isotherm have been shown in Table A.5. Based on regression coefficients, the equilibrium adsorption data fitted better into Langmuir, R-P, F-H, Temkin, Toth, Hill, Sips and K-C models. The critical feature of Langmuir isotherm was expressed in terms of a separation factor, R_L which is a dimensionless constant defining the favorability of adsorption. In the present study, the value of R_L was less than 1 depicting favorable adsorption [277]. R-P isotherm is a combination of Langmuir and Freundlich isotherm. It reduces to Langmuir if the value of $a_R C_e^g = 1$.

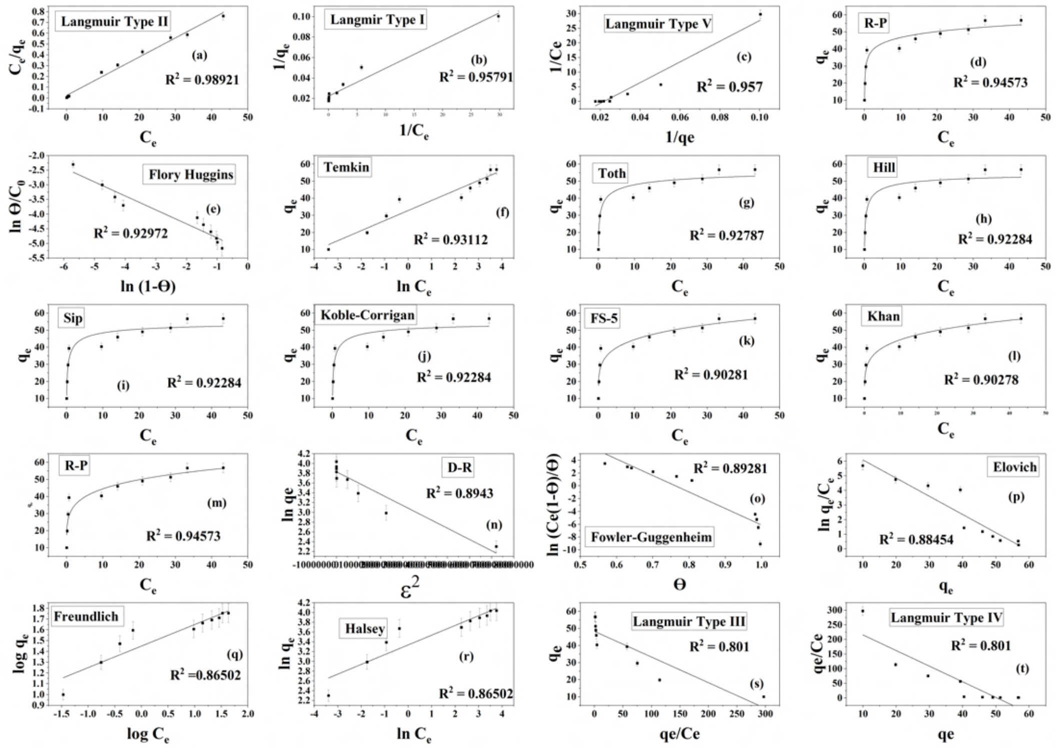


Figure A.13: Adsorption isotherm models for removal of Ni^{2+}ions using composite

However, in the present work, it has been found more than 1 showing the supremacy of Freundlich isotherm [278]. However, the values of SSE and χ^2 were higher for R-P isotherm showing its unsuitability in the present work. The value of ΔG from F-H isotherm disclosed the spontaneous nature of interaction of Ni^{2+} ions onto composite [553]. The value of Temkin model constant 'b' was observed positive in the present study which reflected endothermic in nature of adsorption. This result is in accordance with thermodynamic data obtained in present investigation. In the present work, the parameter 'z' of Toth isotherm was < 1, which again demonstrated the ascendancy of Langmuir isotherm [223]. Similarly, the positive value of 'n_H' in Hill isotherm shows negative cooperativity in the binding of ions which further complemented the preeminence of Langmuir isotherm [223]. Heterogeneity factor 'B_s' and 'n_k' of Sips and K-C isotherm were < 1 which further indicated the suitability of Langmuir isotherm in the present work. Similarly, in Khan isotherm the value of constant 'a_k' was ≈ 1 and in D-R the value of 'ε' was observed as 4.6 kJ/mol establishing the role of physical adsorption on homogeneous surface of composite which is also in accordance with the result of kinetic study in the present work [278]. The negative value of 'w' estimated from F-G isotherm and the values of Elovich and Halsey isotherm constants proved the sustainability of Langmuir isotherm in the present investigation. The value of 'n' (Freundlich isotherm) was > 1 indicating favorable adsorption process [279]. Thus, in the present work, Langmuir model fits best in the experimental data due to the homogenous distribution of active sites on the composite [224].

In Figure A.13, isotherms have been arranged in decreasing order (left to right) of goodness of fit. Here, best isotherm model that fit experimental data was Langmuir isotherm type-II that depicted monolayer coverage on the surface of composite with R^2 - 0.99.

A.2.7 Thermodynamic Study

In the present work, thermodynamic parameters were calculated by plotting the curve between ΔG and T (Figure A.14) and the results have been tabulated in Table A.6.The value of ΔG was negative as depicted in Table A.6 and it decreased with increase in temperature from 283 to 313 K which pointed towards the feasibility and spontaneity in adsorption of Ni^{2+} ions onto composite at a higher temperature [274].

Furthermore, the value of ΔG was observed < + 20 kJ/mol which ruled in the possibilities of electrostatic interaction between active sites of composite and Ni^{2+} ions [283]. The positive value of ΔH (+ 10.557 kJ/mol) in the present study showed that the adsorption of Ni^{2+}ions was endothermic [284] in nature. This could be explained by the fact that the hydration sphere of Ni^{2+}ions got destroyed before adsorption took place on the surface of

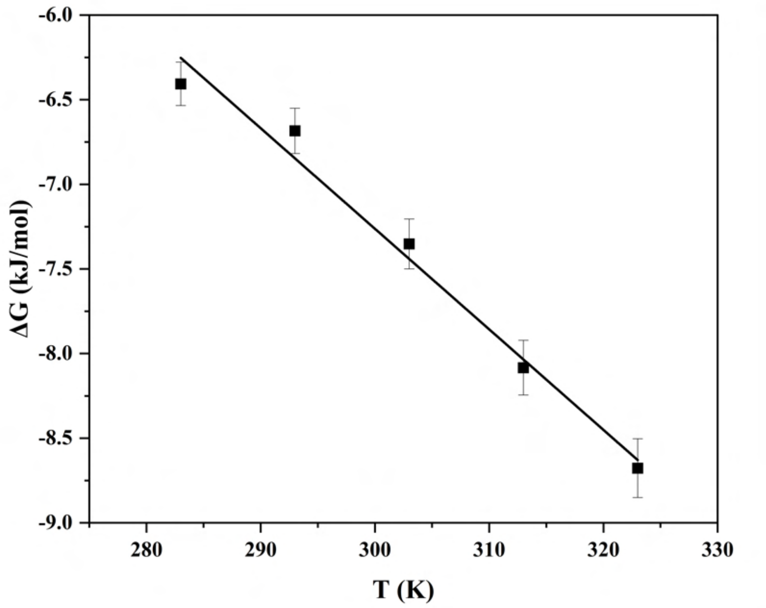


Figure A.14: Plot of ΔG vs. T for removal of Ni^{2+}ions using composite

Table A.6: Thermodynamic parameters for Ni^{2+} ions adsorption

Temperature (K)	ΔG (J/mol)	ΔS (J/mol K)	ΔH (kJ/mol)
283	-6406.98	+ 59.4	+ 10.55
293	-6684.35		
303	-7352.7		
313	-8083.82		
323	-8677.25		

composite. The dehydration process needed energy, and hence it was favored at higher temperature ranges [284], [285]. The positive value of ΔS (+ 59.40 J/mol K) signified increased randomness at the solid-aqueous interface [274].

A.3 Comparative Study

Table A.7 of supporting information shows the comparison of Langmuir adsorption capacities of inorganic adsorbents with the composite developed in the present investigation under varying environmental conditions.

It became evident from Table A.7 that Langmuir adsorption capacity of composite developed in the present work was fairly higher and practical against other inorganic adsorbents cited in the other research findings. It is our belief that this composite can be used as an efficient adsorbent for removal of Ni^{2+} ions from liquid phase in future.

Table A.7: Comparison of composite adsorption capacity with other adsorbents

Adsorbents	Adsorption capacity (mg/g)	Adsorption Thermodynamic	pH	Temp (K)	Ref
Red mud	13.69	Endothermic	5	303	[554]
Clarified sludge	14.3	Endothermic	5	303	[554]
Vermiculite	25.4	Endothermic	6	313	[555]
Sodium mixed bentonite	13.96	Endothermic	6	328	[224]
Local Bentonite	1.91	Spontaneous and exothermic	5.3	293	[556]
Ball clay	0.007 mmol/g	Endothermic	6	303	[557]
Na-Bentonite	13.96	-	9	298	[224]
Chitosan immobilized bentonite	15.82	Exothermic	4	298	[558]
Kaolinite	10.4	Exothermic	5.7	303	[559]
Acid-activated kaolinite	11.9	Exothermic	5.7	303	[559]
Montmorillonite	28.4	Exothermic	5.7	303	[559]
Acid-activated montmorillonite	29.5	Exothermic	5.7	303	[559]
ZrO-kaolinite	8.8	Exothermic	5.7	303	[560]
ZrO-montmorillonite	22	Exothermic	5.7	303	[560]

TBA-kaolinite	8.4	Exothermic	5.7	303	[561]
TBA-montmorillonite	19.7	Exothermic	5.7	303	[561]
Montmorillonite K10	2.1	-	6	298	[562]
3-Mercaptopropyl-trimethoxysilane modified montmorillonite K10	4.73	-	6	298	[562]
composite	**56.31**	**Endothermic**	**6**	**323**	**Present study [246]**

A.4 Conclusion

In the present study, composite was prepared using bentonite clay and red ochre and has been used as adsorbent for removal of Ni^{2+} ions from liquid phase. The optimum parameters for maximum removal of Ni^{2+} ions were observed as pH 6, adsorbent dose 0.1 g, Ni^{2+} ions concentration 100 mg/L, temperature 50°C, agitation rate 180 rpm and contact time 60 minutes. Adsorption of Ni^{2+} ions followed Langmuir isotherm which showed that surface of composite was homogeneous. The homogeneity was also confirmed by model constants of Toth, Hill, Sips and K-C. Adsorption process was favorable in the present work which was further proved by separation factor 'R_L' of Langmuir isotherm and model exponent 'n' of Freundlich isotherm. It was also observed that adsorption of Ni^{2+} ions followed PFO kinetic model elucidated physical adsorption of Ni^{2+} ions on the composite surface. Results of mechanistic modeling showed that not only film diffusion but also intraparticle diffusion governed the rate of adsorption of Ni^{2+} ions on the surface of composite. The thermodynamic analysis demonstrated that the adsorption process was inherently endothermic. Increased randomness was also observed for Ni^{2+} ions on the composite surface. The values of diffusivity coefficients showed the role film and intraparticle diffusion in the present work. The adsorption dynamics was assessed by dimensionless numbers φ, λ and N_k and the results showed that the adsorption is film-cum intraparticle diffusion-limited together with maximum surface area coverage including sluggish surface tension. This research showed that composite could be better alternative for the adsorption of Ni^{2+} ions effectively. This adsorbent is inexpensive and has demonstrated great potential over other inorganic adsorbents reported by various researchers for environmental restoration.

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[1] **J. Singh** and V. Mishra, "Modeling of adsorption flux in nickel-contaminated synthetic simulated wastewater in the batch reactor," *Journal of Environmental Science and Health, Part A*, vol. 55, pp. 1059–1069, 9 2020, ISSN: 1093-4529. DOI: 10.1080/10934529.2020.1767983. [Online]. Available: https://doi.org/10.1080/10934529.2020.1767983

[2] **J. Singh** and V. Mishra, "Synthesis and characterization of activated carbon derived from tectona grandis sawdust via green route," *Environmental Progress |& Sustainable Energy*, vol. 40, e13525, 2 2021, ISSN: 1944-7442. DOI: https://doi.org/10.1002/ep.13525. [Online]. Available: https://aiche.onlinelibrary.wiley.com/doi/abs/10.1002/ep.13525

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[8] **J. Singh**, P. Sharma and V. Mishra, Genetic algorithm-based optimization for simultaneous removal of Cu2+, Ni2+ and Zn2+ ions from aqueous phase using mould (under review)

[9] **J. Singh**, S. Kumar, S. Swaroop and V. Mishra, Quality assessment of the Ganga River during pandemic COVID-19 (under review)

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[1] **J. Singh**, P. Yadav, A. K. Pal, *et al.*, "Water pollutants: Origin and status," in, Springer, 2020, pp. 5–20

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Patent

[1] **Title:** A novel material for treating wastewater and a method of preparation thereof (Filed on 22 May 2019)

Media Coverage of Research Work

Media Coverage

[1] IIT-BHU ने प्राकृतिक विधि से कारखानों के प्रदूषित पानी को बना दिया पीने योग्य

[2] राख से साफ होता था बर्तन, अब साफ होगा पानी...

[3] Teak-Neem-ash purifies contaminated water

[4] गंदे पानी को सागौन और नीम की राख बना देगी शुद्ध, शोध में मिली सफलता

[5] BHU IIT के शोधकर्ताओं को मिली सफलता, दूषित पानी को पीने लायक बनाएगी सागौन और नीम की राख

[6] IIT-BHU scientists use teak, neem ash to extract toxins from water

[7] पानी को साफ़ करेगी मिटटी के चोटी चोटी गोलियां

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