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Research Paper

Activated Kaolinite Sediments as Low-Cost Adsorbents for Cadmium Recovery from Contaminated Wastewater

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Abstract
In this work, activated kaolinite sediments have been investigated for their suitability as economical adsorbents in the removal of cadmium (Cd(II)) ions from polluted wastewater samples. The native kaolinite had a near-neutral pH value (6.9), high cation exchange capacity (97.7 meq/100 g) and low electrical conductivity (0.44 mS/cm), showing a large number of negatively charged surface sites favouring Cd^{2+} adsorption. The activation process created homogeneous sub-micron-sized particles having an average diameter of 458.7 nm, giving rise to an increase in specific surface area and adsorption capability.The batch adsorption tests gave a maximum percentage of Cd(II) adsorption of 98.5% at a pH value of 5, when equilibrium state was achieved in 35–40 minutes with an adsorption capacity of around 70 mg/g under optimal conditions. Adsorption kinetics data followed the pseudo-second-order kinetic model (qe = 81.24 mg/g, R^2 = 0.9517), showing that the adsorption process was mainly governed by surface interactions. The equilibrium data were well fitted by the Langmuir isotherm model (qm = 331.1 mg/g, R^2 = 0.989), implying monolayer adsorption on a limited number of uniforms, high-affinity binding sites.The desorption process through 0.1 M HCl was successful at an efficiency of 96.4%, and the activated kaolinite proved to exhibit remarkable adsorption capacity throughout eight repeated adsorption-desorption processes. From this, we learn that the activated kaolinite is a sustainable, efficient, and economic adsorbent for Cd(II) recovery from wastewater.

Keyword: *Cd(II) adsorption; activated kaolinite; wastewater treatment; ion exchange; regeneration; adsorption isotherm.*

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I. Introduction

Cadmium (Cd) is a soft divalent chalcophile transition metal found naturally in the Earth's crust. But anthropogenic activities like mining, smelting, electroplating, application of phosphate fertilisers, pigment synthesis, battery industry, metallurgy, etc., have led to an increase in the release of cadmium into the aquatic environment. In oxygenated wastewater, cadmium is present mainly in the +2 oxidation state in the form of hydrated Cd^{2+} ions or as soluble complexes with chloride, sulphate, carbonate, and organic ligands. The chemical form of cadmium has a bearing on its mobility, adsorption characteristics, availability and recoverability. Unlike most trace metals, which have some role or the other in living beings, cadmium does not have any biological utility in humans, animals, and higher plants. However, marine diatoms can use cadmium as a replacement for zinc in metalloenzymes in the absence of zinc. **[1]**. Cadmium has been used industrially in nickel-cadmium batteries, electroplating, pigments, coatings, alloys and stabilisers. However, many of these uses are now limited because cadmium is toxic and persists in the environment for a long time **[2].**

Cadmium pollution of water is a serious hazard to public health and the environment. Soluble Cd(II) can transfer from water to soil, groundwater, crops, and food chains. The World Health Organisation (WHO) has recommended 0.003 mg L-1 as the safe amount of Cd in drinking water. This is because Cd accumulates in the kidneys and has a long biological half-life **[3]**. It is linked to renal tubular dysfunction, bone demineralisation, oxidative stress, cardiovascular consequences and carcinogenic risk from chronic exposure. International Agency for Research on Cancer (IARC), classifies cadmium compounds as human carcinogens **[4, 5].** Cadmium (Cd) is toxic to plants and hinders their development, photosynthesis, antioxidant defence and nutrient intake. This reduces production and enables food transfer up the food chain **[6]**. Cd bioaccumulation in aquatic animals could impact oxidative balance, immunity, metabolism and reproduction, endangering the

stability of the ecosystem and food safety **[7]**.Cd(II) needs to be eliminated from contaminated water, and if possible, should be recovered prior to discharge and reuse of the effluents. Various treatment processes have been proposed, such as chemical precipitation, ion exchange, membrane filtration, electrochemical techniques, solvent extraction, biosorption, and adsorption. Chemical precipitation is easy but not effective when cadmium content is low, leading to large amounts of metal-laden sludge. High removal efficiency may be achieved using membrane filtration and ion exchange, although the disadvantages of these techniques include high cost, membrane fouling, and the need for regeneration. In contrast, adsorption is easy, economical, and highly effective for treating dilute Cd(II) wastewater. This method may also use cheap adsorbents and, in addition, allows cadmium recovery due to desorption.**[8, 9]**.

The characteristics of clay minerals include low cost, high chemical stability, and many reactive surface sites; thus, they make excellent adsorbents for heavy metals. The adsorption properties of clay minerals are affected by several factors, including their structure, presence of hydroxyl groups on the surfaces, permanent and pH-dependent surface charge, cation-exchange capacity, particle size, specific surface area, and mineral impurities. Kaolinite, an aluminosilicate clay with 1:1 composition, binds to cadmium ions bycation exchange, electrostatic attraction, and surface complexation on siloxane and aluminol edge sites. Earlier research on cadmium binding on kaolinite indicated that it was pH dependent and followed the Langmuir isotherm.**[10, 11]**. Spectroscopic analysis indicated the presence of structurally different Cd surface complexes on kaolinite**[12]**. Modified kaolinite systems such as tripolyphosphate-impregnated, unrefined, acid-treated and optimised kaolinite adsorbents achieved better adsorption of Cd(II) in controlled aqueous conditions**[13-15]**.Recent reviews point out clay-based materials as low-cost and versatile adsorbents for remediation of heavy-metal pollution **[16].**However, the performance of naturally obtained kaolinite sediments varies greatly depending on the mine location due to factors such as mineral purity and crystallinity, carbonates, exchangeable cations, and Fe/Mn oxides, which can affect Cd(II) removal from contaminated water samples. This research aims to assess the ability of Kalabsha kaolinite sediments to recover cadmium ions from contaminated water. The process of Cd(II) adsorption, along with the physicochemical characteristics that may affect adsorption and Cd(II) recovery, will be studied to investigate whether the sediments can act as efficient, available, and environmentally friendly sorbents. The uniqueness of this study is based on testing locally sourced activated Kalabsha kaolinite sediments and assessing their sorption ability and mechanisms.

II. Materials and Method

Materials

Allchemicals and reagents utilised in this study are of analytical grade and have not been treated. $CdSO_4.xH_2O$ was purchased from Sigma-Aldrich in Germany. Other chemicals were obtained from Fluka Chemical Corp. and Sigma-Aldrich Inc., Missouri, USA, including H_2SO_4, NaOH, KCl, CH_3COONa, HCl, and CH_3COOH. The kaolinite clay sample was collected from the Kalabsha area in Aswan, Egypt, either from nearby radioactive occurrences or at exploitation localities. The Kalabsha area is located between latitudes 23° 00° and 23° 45° N and longitudes 32° 00° and 32° 30° E. In contrast, 0.2 M KCl–HCl and CH_3COOH–CH_3COONa (acetate) buffer solutions were used to alter pH. A precise amount of $CdSO_4.8H_2O$ was dissolved in distilled water to prepare a 1000 mg/l stock solution, which was then diluted to the proper concentrations.

Activation and Characterization of Kaolinite Clay Sample

The obtained samples were pulverised in a porcelain ball mill, rinsed with distilled water to eliminate contaminants, and then wet-sieved through a 350 mesh sieve (45 μm). The homogenised and <45 μm fraction was obtained. Following washing and drying, the naturally occurring kaolinite clay sediments were activated with either 50% HCl or 20% NaOH at a solid/liquid ratio of 1/10 for two hours before being filtered and dried.

Complete characterisation of used clay sediments including FTIR, physical characterisation, and EDX analysis. The physical characterisation was done to determinecation and anion exchange capacity (CEC, AEC), **[17]**, and pH and electrical conductivity (EC), were determined by a glass electrode and by an electrical conductivity device, respectively. Cadmium analysis was carried out using Inductively Coupled Plasma spectroscopy (Prism ICP) High Dispersion (Teledyne Leeman Labs. USA) during this study. The NicoliteNexeus Model 640 MSA was used. FTIR analysis was performed using an instrument from Thermo Electron Corporation. Dynamic Light Scattering (DLS) was used to determine the particle size distribution. Environmental Scanning Electron Microscope (ESEM) was used to characterise the synthesis material before and after adsorption of REEs, respectively. ESEM model is a Prisma E Thermoficher Scientific America attached with Ultra Dry EDS, using a BSE Detector and 20Kv. Analytical conditions were 25-30Kv acceleration voltage, 1-2 mm beam diameters and 60-120 second counting time. Minimum detectable weight concentration: 0.1-1 wt.%. The precision is better than 1% and the relative accuracy of the concentration measurements is between 2 and 10% for elements with Z>9 (F) and between 10 and 20% for the lighter elements B, C, N, O and F. ESEM-EDAX analyses were also performed to investigate morphological characteristics of theses minerals

as well as to give a semi-quantitative evaluation of their elemental composition. A series of experiments was performed to find the optimum conditions of cadmium adsorption using an activated kaolinite clay sample.Different concentrations of cadmium were used. Different contact times were also used at different ratios of the relevant clay sediments to the cadmium solution.

BatchAdsorption Technique

The experiments utilized a spiked (simulated) wastewater sample containing cadmium, which was added to the spiked sample from a cadmium standard synthetic solution, along with the activated kaolinite clay as adsorbent, which acted as the major adsorption material.In batch studies, feed spiked samples (100 mL of the concerned Cd concentration) were collected in a glass stopper bottle after the acidity had been adjusted to the ideal value. The bottle was then filled with 2 g of NaOH-activatedclay sediment and shaken for varying amounts of time at room temperature. The clay was filtered, and the total cadmium concentrations in the filtrate liquor were determined; the difference was used to calculate the corresponding adsorption concentrations. The removal efficiency (RE) and adsorption capacity (q) of Cd(II) were defined as follows:

$$q_e = (C_0 - C_e)V/M (1)$$

$$\mathrm{R_E}\ (\%) = (\mathrm{C_0} - \mathrm{C_e})\mathrm{C_0} \times 100 (2)$$

Where q_e represents the uptake (mg/g), C_o and $C_e$represent the initial and final metal ion concentrations (mg/L), *V*is the volume (L), and *m*represents the adsorbent weight (g)

III. Results and Discussion

3.1 Characterization of activated kaolinite

The kaolinite sediment was characterized by a nearly neutral pH value (6.9), low electrical conductivity (EC, 0.44 mS/cm), high cation exchange capacity (CEC, 97.7 meq/100 g), and relatively low anion exchange capacity (AEC, 4.79 meq/100 g). Such characteristics point to the predominance of negative charge on the adsorbent surface, which would be very convenient for the uptake of bivalent Cd(II) ions. The near-neutral pH helps keep cadmium in the water-soluble form of Cd^{2+} without interfering with hydrogen ions. High CEC favours efficient ion exchange between Cd^{2+} and exchangeable surface cations, while low EC ensures minimal interference of salts during adsorption. Thus, these properties demonstrate the potential of kaolinite for use in cation exchange and complexation-based adsorption of Cd(II) ions. Because of their abundance in exchangeable sites, hydroxylated surfaces, and reactivity of the aluminosilicate framework, clays have been widely used as adsorbents of heavy metals. **[12, 18-20]**.

The typical FTIR profile of kaolinite before cadmium adsorption is shown in both activated kaolinite samples (Fig. 1). These are structural hydroxyl bands in the high-frequency region and aluminosilicate framework vibrations in the fingerprint region. Main bands in the NaOH-activated kaolinite spectrum (curve a) are at 3679 and 3626 cm^{-1}. These are for the inner surface and inner Al-OH stretching vibrations. Si-O stretching is observed around 1036 cm^{-1} and Al-OH deformation at 934 cm^{-1}, and framework bands are also seen at lower frequencies around 777, 686 and 558 cm^{-1} for Si-O, Si-O-Al and Al-O-Si deformation modes. Similar bands to those at 3670 and 3620cm ^{1} are present in the HCl-activated kaolinite spectrum (curve b), and there are strong bands at 1063, 958, 800, 712, 581 and 488 cm ^{1}. These bands indicate that the reactive hydroxylated aluminosilicate sites were more accessible after the acid and alkaline activation, while the basic structure of kaolinite was preserved. The bands around 3695, 3670, 3650 and 3620 cm^{-1} are usually assigned to the OH stretching vibrations of kaolinite. The bands at 1113, 1033, 1009, 937, 913, 791, 752, 694, 540, 468 and 433 cm^{-1} correspond to Si-O, Al-OH and Si-O-Al framework vibrations **[21, 22]**. In contrast, clear changes in the spectra were seen in the same functional regions after Cd adsorption (Fig.2). This indicates that Cd(II) was interacting with the surface hydroxyl and aluminosilicate groups of activated kaolinite. In the Cd-loaded NaOH-activated sample (curve a, the hydroxyl region shifts to around 3698 and 3640 cm-1, and a broader band appears around 3458 cm^{-1}. This is due to the presence of hydrogen-bonded OH groups and water attached to hydrated Cd(II) at the surface. There is also a new or stronger band at about 1651 cm^{-1} due to H-O-H bending in adsorbed water. The Si-O band shifts from about 1036 cm^{-1} before loading to about 1056 cm^{-1} after loading. The Al-OH region is observed at about 939 cm^{-1} **[12, 23]**. The OH bands of the Cd-loaded HCl-activated sample (curve b) are at approximately 3698, 3640 and 3455 cm^{-1}. The water-bending band is near 1643 cm^{-1}. The Si-O/Al-OH region moves from about 1063 and 958 cm^{-1} before loading to about 1045 and 917 cm^{-1} after loading. The low frequency bands also change from ~581 and 488 cm^{-1} before loading to ~545 and 461 cm^{-1} after loading. These shifts and changes in intensity are the main spectral evidence that Cd removal was not only due to physical retention but also included surface complexation and ion exchange at Al-OH, Si-O and Si-O-Al sites. This interpretation is consistent with the adsorption studies, which show that Cd(II) can be incorporated or

immobilized by Si-O and Al-O networks, mesopores, and ion-exchangeable sites in aluminosilicate materials, where chemisorption is generally involved in the adsorption mechanism**[15, 24]**.Post-loading spectral changes are also observed for the NaOH-activated sample. This suggests that alkaline activation enhances the Cd(II) uptake mainly through increased surface basicity, deprotonated oxygen sites and exchangeable Na+ sites that can be substituted by Cd^{2+}.

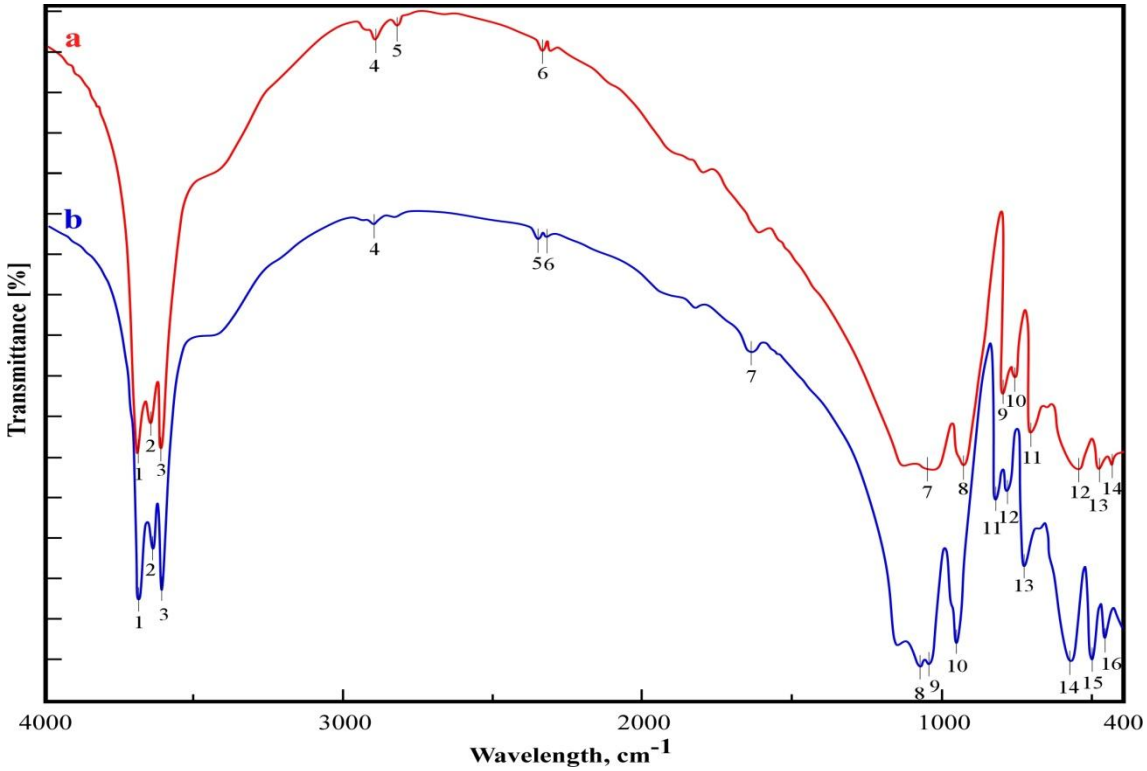

Fig. (1): Infrared spectra of Studied Kaolinite sample either with a) NaOH or b) HCl or before cadmium adsorption

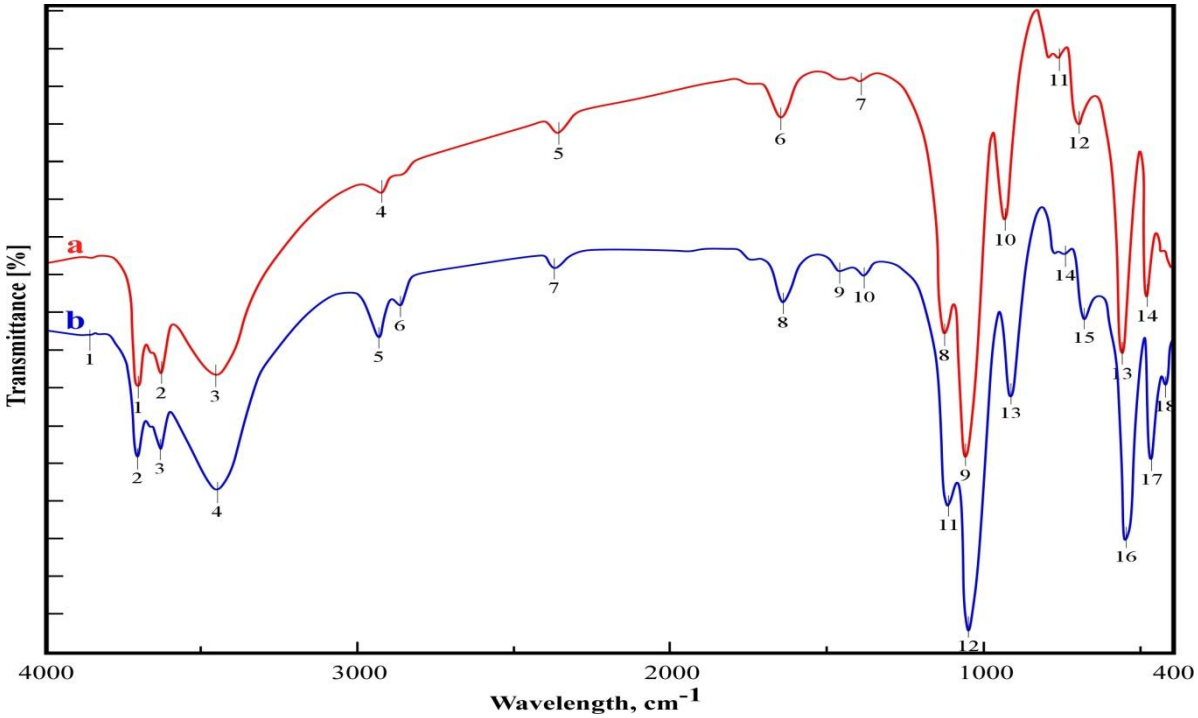

Fig. (2): Infrared spectra of the studied kaolinite clay sample after activation either with a) NaOH or b) HCl and after cadmium adsorption

The EDX characterization of the NaOH-activated kaolinite (Fig. 3) confirmed a preponderance of aluminosilicates with abundant silicon (48.1 wt%) and aluminum (31.9 wt%), showing the retention of the structure of kaolinite after activation. Sodium (5.8 wt%) suggests effective alkaline activation, implying the insertion of Na^+ ions into exchangeable sites on the surface and hence an increased ability to capture Cd(II) through cation exchange. Other minor constituents identified include iron (2.7 wt%), titanium (3.7 wt%) and calcium (1.0 wt%). These impurities are common in natural kaolinite and could contribute to the heterogeneous surface of the clay mineral. The low abundance of oxygen (6.7 wt%) is most likely an artefact of the analytical method used, and not the real composition of the bulk material. In summary, the EDX analysis confirms that NaOH activation gave sodium-rich kaolinite while retaining the aluminosilicate structure, and hence is suitable for Cd(II) removal by ion exchange and surface complexation. Kaolinite is a natural 1:1 aluminosilicate clay mineral, compared to 2:1 clays like montmorillonite.Though it is a clay stone made mostly of ordered clay minerals with varying amounts of non-clay minerals like quartz, carbonates, gypsum, and halite, the Kaolinite deposits are generally hard, massive, and white to earth grey in colour. The mineral Kaolinite is classified as a two-layer structure of hydrated aluminum silicates.

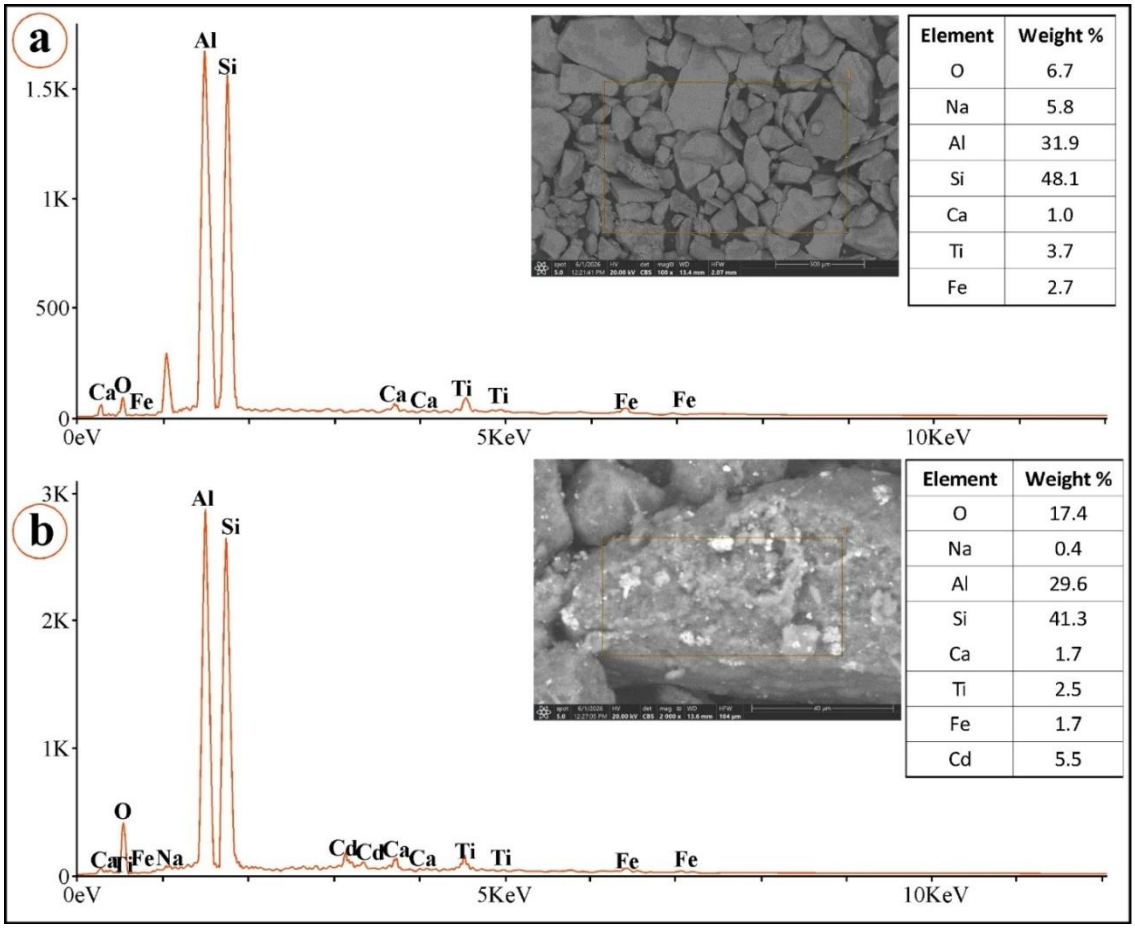

Fig. (3): Semi-quantitative EDX analysis of NaOH-activated kaolinite showing major elements (Si, Al, Na, O) and minor impurities (Fe, Ti, Ca) as weight percentages

The results in Fig. 3 show that the nitrogen adsorption-desorption isotherm of NaOH-activated kaolinite shows a gradual increase in adsorbed volume with increasing relative pressure, which implies limited overall porosity and that adsorption mainly occurred on the available external surfaces and interparticle pores. According to the IUPAC classification, the isotherm corresponds to type IV, indicating a pore structure composed of both micropores and a predominance of mesopores mainly in the range (approximately 2–30 nm), which suggests that NaOH activation revealed or generated diffusion-accessible pores for adsorption processes **[25]**. This interpretation is consistent with recent studies on kaolinite adsorption, where pollutant uptake behaviour was explained by N2 adsorption, pore structure, zeta potential, and granulometry in combination [26]. The negative zeta potential suggests that the surface of the activated kaolinite is negatively charged, possibly due to the deprotonated silanol and aluminol groups, which enables electrostatic attraction and ion exchange with cationic pollutants, such as metal ions or cationic dyes**[26, 27].**However, when the absolute zeta potential is less than about 30 mV, the suspension is only considered moderately stable and more susceptible to aggregation,

consistent with the right-tailed particle size distribution in the grain size plot **[28]**. The particle size distribution is mainly unimodal; that is, the activated clay fraction is quite homogeneous. The tailing towards larger sizes indicates a partial aggregation of the particles. The textural parameters for the modified sample are a pore diameter of 1.57 nm and a specific surface area of 56 m^2/g. Overall, the mesoporous nature, negative surface charge and fine particle distribution of NaOH-activated kaolinite suggest the potential of this material as a low-cost adsorbent, especially for cationic contaminants, where adsorption can be attributed to electrostatic interactions, surface complexation and ion exchange**[26, 27, 29]**.

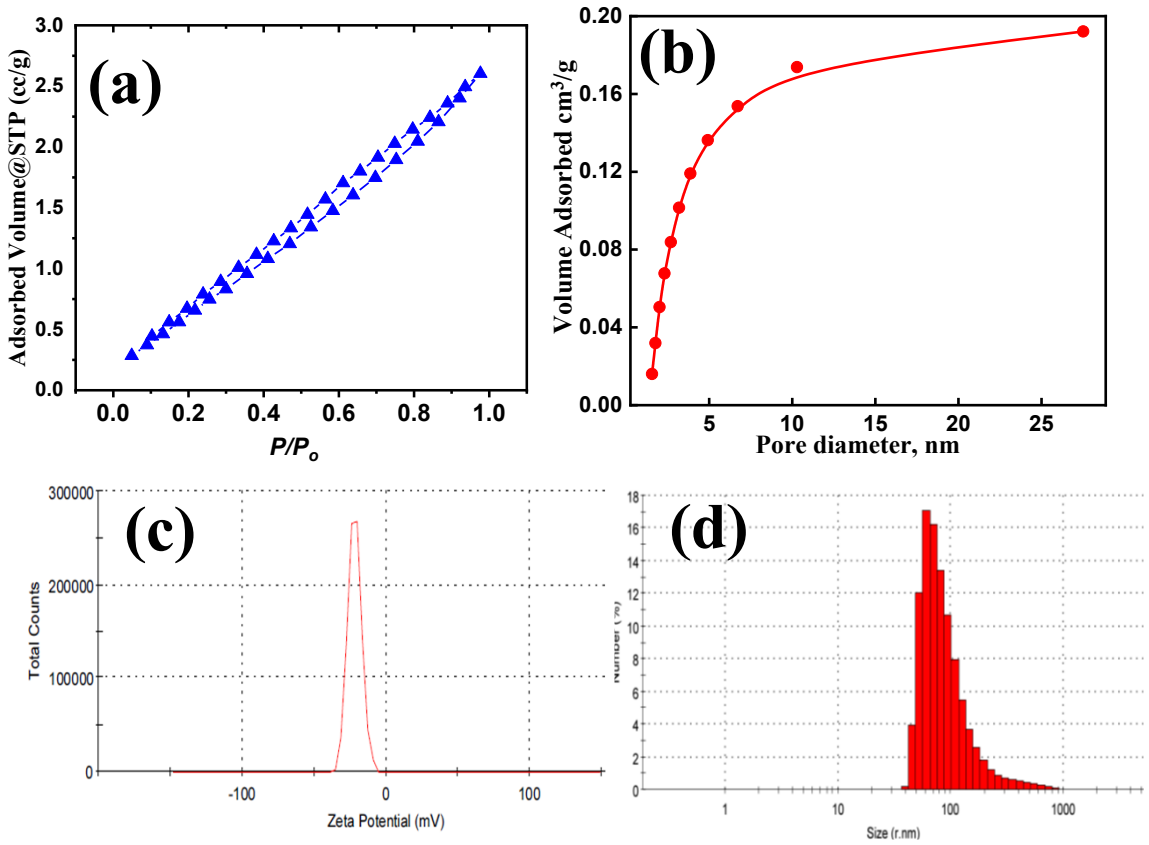

Figure (3): (a)N_2 adsorption-desorption isotherm, (b)Pore diameter, nm, (c) Zeta potential, and (d) Particle size distribution of the NaOH-activated kaolinite clay.

Results of Batch Adsorption Technique

1. Effect of pH upon Cadmium Loading

Adsorption of Cd(II) in relation to the pH (Figure 4) strongly indicates that surface charge and Cd speciation in solution play an important role in the adsorption process of activated kaolinite. At low pH conditions, the efficiency of adsorption and the adsorption capacity of activated kaolinite were determined by the competition of excess hydrogen and cadmium ions for exchangeable sites and protonation of the edge sites of kaolinite. The highest removal rate of Cd(II) (approximately 98.5%) was observed at pH 5 due to partial deprotonation of aluminol and silanol edge sites, which improved electrostatic attraction, ion exchange and surface complexation. The decrease in adsorption at high pH values shows that the removal of Cd(II) was not controlled by precipitation but was influenced by Cd hydrolysis, aqueous hydroxo species and the blocking of active sites of kaolinite. The results obtained are similar to those of other researchers who have noted a strong influence of pH on Cd(II) adsorption onto kaolinite and the change from the outer-sphere adsorption mechanism at low pH to inner-sphere surface complexation at higher pH**[10-12]**.

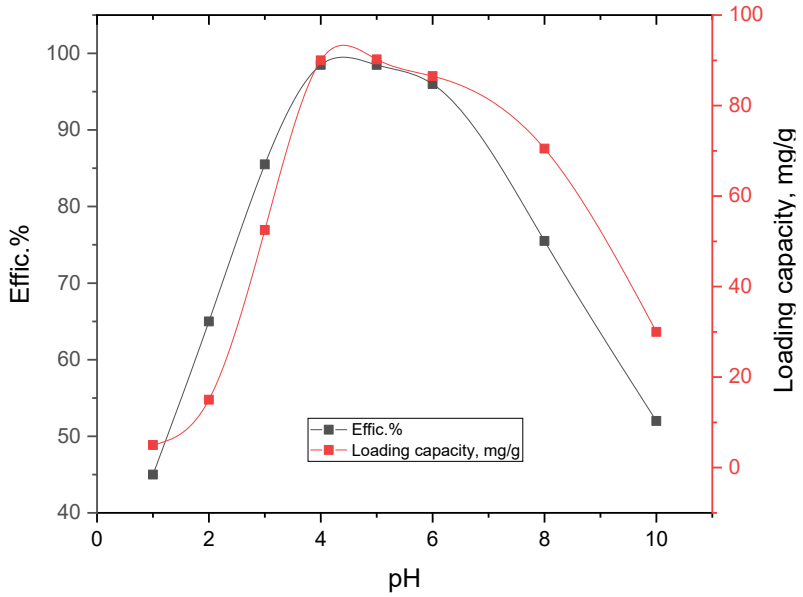

Fig. (4): Effect of pH on Cd(II) loading onto activated kaolinite. Maximum removal (98.5%) occurred at pH 5, where deprotonated aluminol and silanol edge sites enhance electrostatic attraction, ion exchange, and surface complexation.

3.2.2. Effect of Time upon Cadmium Loading

Results obtained (Fig. 5), show the contact time curve, where the loading of Cd(II) increased rapidly from about 26 mg/g at 5 min to about 70 mg/g at 35 to 40 min and then reached an apparent plateau. Such a pattern is characteristic of clay adsorbents. The first stage is ascribed to the rapid filling of plentiful external exchange sites and easily reachable aluminol and silanol groups, and the slower later stage to gradual saturation, diffusion into less accessible pores and redistribution onto stronger binding sites. The practical implication is that most of the available Cd(II) can be removed by activated kaolinite in a short contact period. Additional agitation away from the equilibrium region will probably not result in a significant further recovery. Similar time-dependent behaviour was observed for Cd(II) adsorption onto both raw and modified kaolinite systems, with rapid uptake followed by slow approach to equilibrium **[14, 15]**.

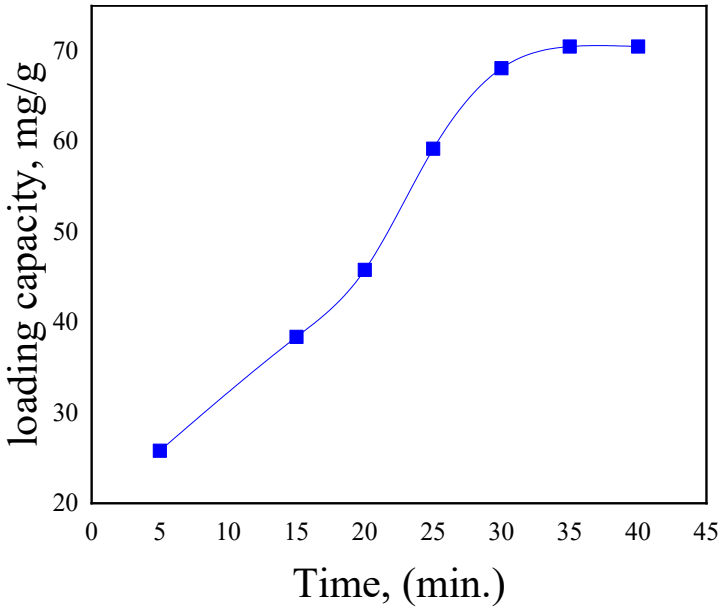

Fig. (5): Effect of contact time on Cd(II) loading onto activated kaolinite. Cadmium uptake increased rapidly from 26 mg/g at 5 min to 70 mg/g at 35–40 min, after which a plateau was reached, indicating equilibrium.

3.2.3. Kinetic study of Cadmium Loading upon Activated Kaolinite

Kinetic parameters (Fig. 6) reveal that the pseudo-first-order equation provides the following adsorption capacity, rate constant and R^2 values q_1 = 73.20 mg/g, k_1 = 0.0498 min^{-1}, R^2 = 0.8981. Considering the high q_1 value, one can conclude that the pseudo-first-order equation appropriately describes the rapid initial adsorption due to the occupation of available adsorption sites. Thus, the half-adsorption time estimated at about 13.9 min also indicates fast initial adsorption. Still, a relatively low R^2 value compared to the pseudo-second-order equation (Table 1) means that the pseudo-first-order equation poorly describes the adsorption. This is expected since Cd(II) adsorption on activated kaolinite involves more complicated processes, such as ion exchange at permanent-charge sites and surface complexation at silanol and aluminol edge groups, rather than diffusion. A better fit was achieved using the pseudo-second-order kinetic model, which yielded q_2 = 81.24 mg/g, k_2 = 0.0008 g mg^{-1} min^{-1}, and R^2 = 0.9517. This model appears to better describe the adsorption kinetics because of the high values of both the calculated initial adsorption rate (h = 5.28 mg g^{-1} min^{-1}) and half-adsorption time ($t_{1/2}$ = 15.4 min). The fast initial adsorption rate coupled with a gradual approach to equilibrium as the active surface sites become filled progressively and diffusion to inactive regions becomes rate-determining shows that the adsorption process is very fast in the beginning and gradually approaches an equilibrium state as the available active adsorption sites get filled progressively and diffusion into inactive regions becomes rate-limiting. The better fit obtained from the pseudo-second-order kinetic model indicates that adsorption kinetics of Cd(II) on activated kaolinite are mainly controlled by the availability of the active surface sites and surface-controlled interactions. **[15, 30]**. The kinetic studies revealed that the removal of Cd(II) by activated kaolinite was better described by the pseudo-second-order model than by the pseudo-first-order model. This supports a mechanism dominated by surface-controlled adsorption rather than simple mass transfer. However, the pseudo-second-order fit does not necessarily suggest that the process is purely chemisorptive because cadmium uptake on kaolinite may involve a combination of mechanisms such as cation exchange, electrostatic attraction by outer-sphere complexes, surface complexation by inner-sphere complexes, and perhaps intraparticle diffusion. This interpretation is in agreement with the adsorption behaviour of Cd(II) on kaolinite and related clay minerals reported in previous studies.

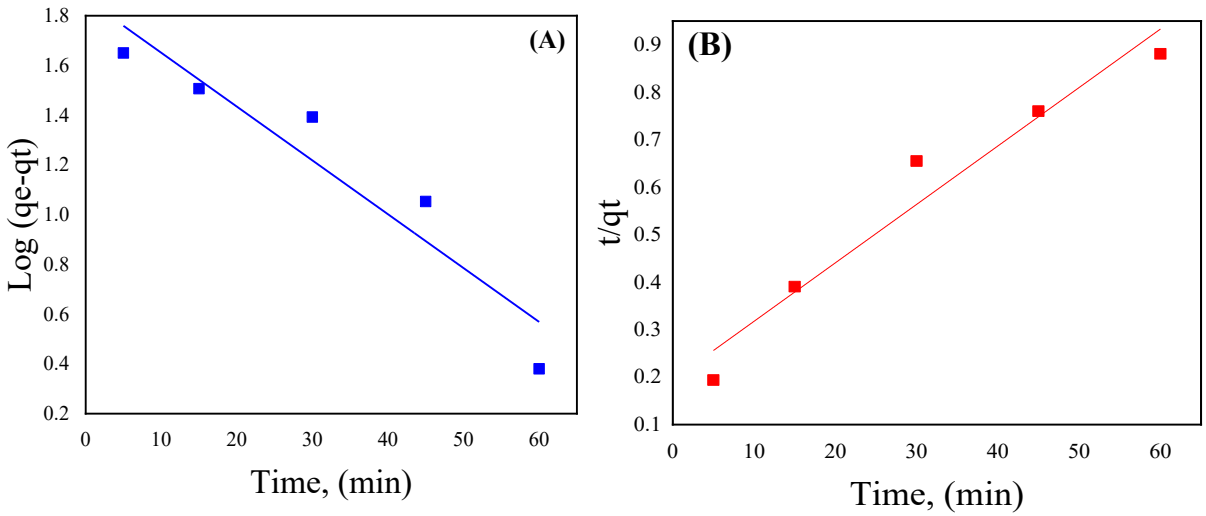

Fig. (6): Kinetic parameters of Cd adsorption upon the concerned activated kaolinite clay according to the Lagergren and Ho& McKay models

Table (1): Kinetic parameters of Cd adsorption upon the concerned activated kaolinite clay according to the Lagergren and Ho& McKay models

The studied kinetic models					
Pseudo-first order			**Pseudo-second order**		
q_1, (mg/g)	k_1 (min^{-1})	R^2	q_2, (mg/g)	k_2 (g/mg.min)	R^2
73.20	0.0498	0.8981	81.24	0.0008	0.9517

3.2.4. Effect of Initial Concentration of Cadmium and Isotherm Study

The obtained results (Fig. 7a–b) show the effect of initial Cd(II) concentration on Cd adsorption by activated kaolinite. The loading capacity increased gradually with increasing initial concentration, reaching about 325 mg/g in the experimental range. The trend is due to a higher concentration gradient that increases

mass transfer and provides more Cd^{2+} ions to the available exchange and complexation sites. However, at higher initial concentrations, the removal efficiency decreased because of saturation of finite active sites with respect to the dissolved Cd(II) load. To understand the adsorption mechanism and surface heterogeneity, isotherm modelling was carried out. Table (2) summarises the fitted parameters of five isotherm models at 298K. The Langmuir isotherm obtained the best correlation coefficient (R^2 = 0.98906), indicating that the adsorption occurs as a monolayer on a finite number of energetically homogeneous high-affinity sites. The maximum monolayer capacity (q_L) was found to be 331.13 mg/g, close to the experimental loading (~325 mg/g), confirming that the uptake at high concentrations of Cd(II) is controlled by surface saturation. The R^2 value of the Freundlich model was smaller (0.80568), which shows that the heterogeneous multilayer adsorption is not predominant. But the n value of 2.9971 (between 1 and 10) still shows favourable adsorption. The Dubinin–Radushkevich (D-R) isotherm yielded a mean free energy E = 12.66 kJ/mol, which is within the normal range for ion exchange processes (8–16 kJ/mol), suggesting the importance of cation exchange in combination with surface complexation. The Temkin model has R^2 of 0.8997, indicating a moderate linear decrease in heat of adsorption with surface coverage. In summary, the combined isotherm evidence is consistent with previous studies on kaolinite and modified clay systems **[10, 14, 16]**confirming that Cd(II) uptake onto activated kaolinite is primarily a Langmuir type, site-limited process driven by ion exchange and surface complexation.

Table (2): Parameters of Cd adsorption isotherms (Langmuir, Freundlich, Dubinin-R, Radushkevich, and Temkin-I models) at 298 K

Isotherm model	**Parameters**	**Value**
Langmuir isotherm	qL (mg/g)	331.1258
	kL	0.08289
	R^2	0.98906
Freundlich isotherm	kF (mg/g)	53.086
	n	2.9971
	R^2	0.80568
D-R isotherm	qDR (mg/g)	146.1145
	kads (mol^2/kJ^2)	3.12E-09
	E (kJ/mol)	12.6645
	R^2	0.89468
Temkin-I isotherm	B (kJ/mol)	64.82315
	AT (L/g)	0.590321
	bT (J/mol)	38.22048
	R^2	0.8997

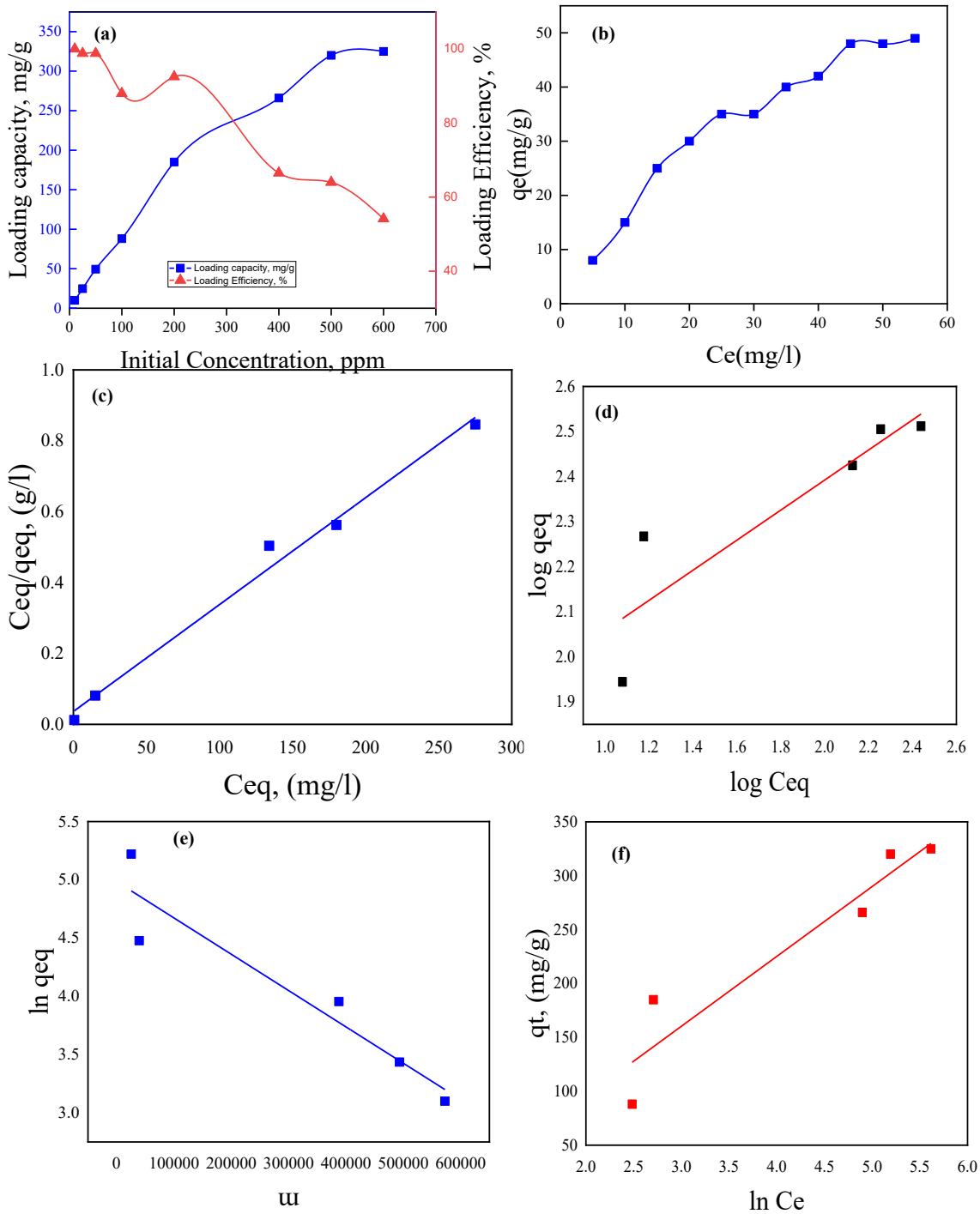

Figure (6): (a) Effect of initial concentration upon Cd adsorption upon studied material, (b) effect of equilibrium concentration, (c) Langmuir isotherm, (d) Freundlich isotherm, (e) D-R isotherm (f), Temkin isotherm, for REEs adsorption upon studied material

3.2.5. Effect of Temperature upon Cadmium Loading upon Activated Kaolinite

The impact of temperature on the adsorption of Cd(II) ions (Figure 7) shows that the adsorption capacity of the system remained relatively unchanged (about 185 mg/g) at 30 and 45 °C, while decreasing to about 170 mg/g at 60 °C. It can be concluded that adsorption of Cd(II) ions on activated kaolinite is favourable at temperatures up to moderately elevated ones, but it is not favourable at high temperatures. The decrease in the adsorption capacity at 60 °C could be associated with changes in the degree of hydration of Cd(II) surface complexes, higher desorption rates, or decreased stability of Cd(II) ions bound through ion exchange and surface complexation. The observed trend indicates that adsorption is temperature-dependent and is consistent

with previous research stating that the adsorption of Cd(II) ions on kaolinite and other activated clay minerals depends on temperature. **[14, 24]**.

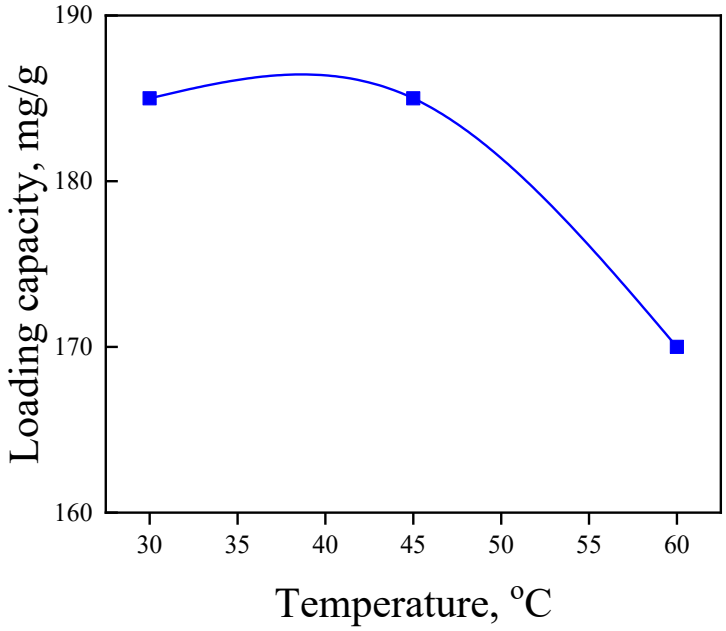

Figure (7): Effect of temperature on cadmium loading on activated kaolinite

Overall, the batch adsorption studies prove that activated kaolinite can be considered an effective adsorbent for Cd(II) removal from contaminated water. High efficiency of the Cd(II) removal at the optimal pH value, quick reaching the equilibrium, good correspondence of the adsorption kinetics to the pseudo-second-order model, and good fitting of the equilibrium adsorption data to the Langmuir isotherm can serve as evidence of the predominant role of the cation exchange and surface complexation of Cd(II) ions on a limited number of hydroxylated aluminosilicate surface sites in the adsorption process. The changes in the FTIR spectrum peaks after Cd(II) adsorption in the O-H, Si-O, and Al-OH regions also support this statement. In practical terms, the optimal conditions of the process correspond to a pH of about 5 with a contact time of 35-40 minutes without heating, with high removal efficiency. It is obvious that this process has certain advantages compared with conventional precipitation methods by generating large amounts of metal sludge. **[8, 9, 16]**.

3.3. Desorption of Loaded Cadmium and Reusability Efficiencies

The desorption studies with 0.1 M HCl as the eluting agent confirmed the elution efficiency of the Cd(II)- loaded activated kaolinite clay. The effective role of proton competition in the acidic medium was revealed in the displacement of adsorbed Cd(II) ions from the active surface sites, leading to a high desorption efficiency of about 96.4%. The high percentage of elution indicates that most of the retained cadmium was reversibly bound, presumably by ion-exchange and electrostatic interactions **[15].** But the desorption efficiency diminished after eight consecutive adsorption-desorption cycles, indicating a gradual exhaustion or partial blockage of the active sites, possible structural fatigue of the clay surface and a stronger retention of a part of Cd(II) by less reversible surface complexation mechanisms.

IV. Conclusion

From the findings in this research, it is apparent that activated kaolinite sediment particles can be used as economical and efficient adsorbent materials for the removal and recovery of Cd(II) ions from polluted water systems. For instance, a maximum Cd(II) removal percentage of 98.5% was recorded at pH 5 when equilibrium state was attained after 35–40 minutes under the batch conditions studied in the laboratory. The process followed a second-order pseudo-kinetic equation and the Langmuir isotherm model, implying that the adsorption of Cd(II) ions occurred mainly through a finite number of active surface sites. The use of Dubinin–Radushkevich adsorption energy and FTIR analysis after the adsorption of Cd(II) indicates that ion exchange and surface complexation are the main mechanisms in the adsorption process. Recovery of Cd(II) using 0.1 M HCl was effective, and the activated kaolinite particles remained capable of adsorbing ions during repetitive cycles.

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