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Expulsion of cadmium from a simulated wastewater using CKD as adsorbent: Optimization with isotherm study

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ABSTRACT

Cement kiln dust (CKD) considers as an inexpensive, abundant adsorbent and was used in the present work for removing cadmium ions from a simulated wastewater. CKD was obtained from Al-Duh Cement Factory located in south of Al-Muthanna Governorate/Iraq and identified by X-ray diffractometer (XRD), scanning electron microscopy (SEM) with EDX, and Fourier transform infrared spectrometry (FTIR). Optimization of the main effective parameters like initial cadmium concentration, CKD dosage, shaking speed, pH and contact time on the cadmium removal efficiency was achieved via applying reaction surface strategy (RSM). Results showed that underlying cadmium fixation has the fundamental impact on the cadmium removal efficiency followed by CKD dosage, time, pH, and lastly shaking speed. The preferred operating conditions were found to be an initial Cd concentration of 20 ppm, CKD dosage of 35 g \ L, pH of 8, shaking speed of 300 rpm, and contact time of 90 minutes. Based on these optimum conditions, 99.75% removal efficiency of cadmium was obtained. The adsorption isotherm results showed that the adsorption behavior of Cd ions on CKD agree well with Langmuir model. CKD seems to be a systematic, sustainable and economic material for cadmium removal from wastewater.

Keywords: CKD, Adsorption, RSM, Isotherms models, Cadmium

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1. Introduction

One of the paramount significant topics facing the world is the environmental pollution issue especially by heavy metals pollution, as they are not degradable and not disposed but being processed by mitigated or transformed[1,2]. Because of water is important for countries, especially those suffered from a shortage of water, sewage water must be treated to remove heavy metals using an economical and environmentally acceptable method. Cadmium is one of heavy metals that considered as a carcinogen in addition to poisoning the heart, lungs, and kidneys [3, 4].
There are several approaches for heavy metal treatment such as filtration, reverse osmosis, chemical oxidation and reduction, ion exchange, chemical precipitation, solvent extraction, electrochemical treatment, evaporative recovery, and adsorption [5]. Among these methods, using adsorption method acombined with choicing of an appropriate adsorbent material would be an essential method for heavy metals removal that presented in wastwaters [6]. Activated carbon was used efficiently as an adsorbent in handling of aqouse solutions containing heavy metals [7]. Nevertheless, its production considers as costly approach. Hence, more research interest has been focused on utilizing other low–cost adsorbents. Several adsorbent materials have been examined involving peat, leaf mould, microbial biomass, compost, coal straw, palm press fiber, wool fiber and rice milling byproduct [8,9]. Not all of these are active in elimination of heavy metals. Hence, it is still significant to find appropriate and cheaply adsorbents for the treating heavy metals.
Recently, many research works have been focused on the using of industrial wastes as an adsorbent material so as to reduce processing costs as well as protection of the public health and the environment. Cement kiln dust (CKD) material is one of those materials [10]. CKD is a byproduct from Portland cement production and having

high calcium oxide (CaO) content as well as being very fine particles [11]. Its presence causes pollution to the environment because they affect the respiratory system of a person. It uses in many operations, comprising stabilizing the soil, construction building materials, preparation fertilizers, and as an adsorbent [12-15]. CKD has been used extensively for removal many heavy metals like Cu, Ni, Zn, Cd, Pd [16, 17]. In this field, numerous works have been conducted for removal of cadmium using different sources of CKD [16, 18-25]. However, no previous work has been conducted for the removal of cadmium using CKD generated from Al-Duh Cement Factory located in south of Al-Muthanna governorate in Iraq. Therefore, this work aims to examine the impact of several operating factors such as initial Cd concentration, pH, sorbent dose, shaking speed, and contact time on the adsorption efficiency of cadmium on CKD generated from Al-Duh Cement Factory as a potential and cheep adsorbent material. In this context, response surface methodology (RSM) was utilized as an optimization method. Moreover, the adsorption mechanism was evaluated using Langmuir, and Freundlich isotherm models.

2. Experimental work

2.1. CKD characterization

2.5kg of CKD was gotten from Al-Duh Cement Factory located in south of Al-Muthanna Governorate/Iraq. As a first step, a sample of CKD was dried at 100 ºC for 2hr. before using it at each run. It should be pointed out that no sieving was proceeded to evaluate the average particle size of the dust. The dust was used as received. The structure of CKD was identified by X-beam diffractometer (XRD) utilizing a Shimadzu - XRD-6000, Japan. The working states of XRD were 40 kV and 30 mA utilizing CuKα radiation as the X-beam source, λ=1.54056 Å. The output was accomplished with step season of 0.6 s at a stage size of 0.05° and at examine scope of (3-60°). The investigation of CKD utilitarian gatherings was inspected by using Fourier change infrared spectrometry (FTIR) after just as before the adsorption runs. FTIR spectra of the adsorbent were perceived in the reach 600-4000cm-1using infrared spectrophotometer; model (Bruker – Tensor 27, Germany).
The surface morphology of CKD was inspected utilizing checking electron microscopy (SEM) (Tescan Viga III FESEM, Czech Republic). Boundaries utilized were AV= 20 KV, predisposition = 0, spot = 3.0 and HV = 20 kV, inclination = 1400 V. The absolute surface space of CKD was estimated by BET surface area analyzer model No. Qsurf 9600, thermos Finnegan Co. USA using test method: ISO 9277-2010 with a detection limit from 0.1-200m^2/g.

2.2. Preparation of the simulated cadmium wastewater

Cadmium sulfate ($CdSO_4.8H_2O$) with a purity of 99% supplied from Aldrich was used as a source of Cd ion for preparing the simulated wastewater where a stock solution of 1g/l was prepared initially then the required concentration was obtained by diluting a certain volume of this stock solution with distilled water to get 400 ml of the simulated solution at the required concentration in ppm. Adjusting of pH was achieved using 0.1M HCl and/or 0.1M NaOH. The required amount of CKD was then added at the designated concentration. The solution was then shacked at the specified speed for an interval of time. After completing the adsorption process, a sample of 5 mL was taken and filtered then analyzed for determining the remaining concentration of cadmium by atomic absorption technique (Varian SpectrAA 200 spectrometer). All runs were conducted in a duplicated fashion at room temperature then the average values were adopted in the analyzing of data (the maximum error was lower than 3%).
The removal efficiency (RE %) of cadmium using cement kiln dust as adsorbent was determined by the following formula (Eq. 1) [26] :

$$RE\% = \frac{C_o - C_t}{C_o} \times 100 \quad (1)$$

where C_0 is the initial Cd concentration and C_t is the final Cd concentration at the end of experiment with total time (Δt).

2.3. Design of tests

Deciding the numerical relationship between's the reaction any interaction and their elements can be gotten through applying a bunch of measurable and numerical strategies received by RSM [27]. In this examination, Box–Behnken experimental design with a 3-level 5-factor design was applied to recognize factors that impact on the cadmium removal from a reproduced wastewater. Introductory Cd fixation (ppm);X1, CKD dosage(g/l) ;X2,Shaking speed (rpm) ;X3 ,pH ;X4, and Contact time(min);X5 were considred as interaction factors, while Cd expulsion proficiency was taken as a reaction. The coding of interaction factors were - 1 (low level), 0 (center or main issue) and 1 (undeniable level) [28]. Table 1 shows the interaction factors with their individual levels. Box–Behnken relies just upon a piece of the investigations that required for 3-level factorial to get the suitable quadratic model with the necessary factual properties. Condition (2) can be utilized to indicate the quantity of trails (N) that needed to perform Box–Behnken configuration as follows [29]:

$$N = 2k\,(k-1) + cp \quad (2)$$

Where k alludes to number of factors and cp alludes to the quantity of essential issues that were rehashed.

To examin the impacts of cycle factors on Cd expulsion proficiency, forty-six runs have been achieved. Table 2 shows the BBD proposed to perform these runs.

Table 1. Process factors and their levels in cadmium removal process

Process parameters	**range in Box–Behnken design**		
Coded levels	Low(-1)	Middle(0)	High (+1)
Initial conc. (ppm), X1	20	60	100
CKD dosage(g/l), X2	5	20	35
Shaking speed(rpm), X3	100	200	300
pH , X4	2	5	8
time(min),X5	30	60	90

Table 2. Box- Behnken experimental design

Run	**Blocks**	**Coded value**					**Initial conc. (ppm)**	**CKD dosage (g/l)**	**Shaking rate (rpm)**	**pH**	**Time (min)**
		x_1	x_2	x_3	x_4	x_5					
1	1	-1	1	0	0	0	20	35	200	5	60
2	1	0	1	-1	0	0	60	35	100	5	60
3	1	0	1	0	0	1	60	35	200	5	90
4	1	0	1	0	-1	0	60	35	200	2	60
5	1	0	0	1	0	-1	60	20	300	5	30
6	1	1	0	1	0	0	100	20	300	5	60
7	1	0	0	0	1	-1	60	20	200	8	30
8	1	0	0	1	-1	0	60	20	300	2	60
9	1	0	0	0	0	0	60	20	200	5	60
10	1	0	-1	0	-1	0	60	5	200	2	60
11	1	0	0	0	-1	1	60	20	200	2	90
12	1	-1	0	-1	0	0	20	20	100	5	60
13	1	0	0	1	1	0	60	20	300	8	60
14	1	0	0	1	0	1	60	20	300	5	90
15	1	-1	0	0	-1	0	20	20	200	2	60
16	1	0	0	0	-1	-1	60	20	200	2	30
17	1	0	0	-1	0	-1	60	20	100	5	30
18	1	-1	0	0	1	0	20	20	200	8	60
19	1	1	0	0	0	-1	100	20	200	5	30
20	1	0	1	0	0	-1	60	35	200	5	30
21	1	0	0	0	0	0	60	20	200	5	60
22	1	0	-1	1	0	0	60	5	300	5	60
23	1	-1	0	1	0	0	20	20	300	5	60
24	1	0	0	0	0	0	60	20	200	5	60
25	1	0	0	0	0	0	60	20	200	5	60
26	1	0	0	-1	1	0	60	20	100	8	60
27	1	0	1	0	1	0	60	35	200	8	60
28	1	0	1	1	0	0	60	35	300	5	60
29	1	1	0	0	1	0	100	20	200	8	60
30	1	-1	0	0	0	1	20	20	200	5	90
31	1	-1	0	0	0	-1	20	20	200	5	30

Run	Blocks	Coded value					Initial conc. (ppm)	CKD dosage (g/l)	Shaking rate (rpm)	pH	Time (min)
		x_1	x_2	x_3	x_4	x_5					
32	1	1	0	0	-1	0	100	20	200	2	60
33	1	0	-1	0	0	-1	60	5	200	5	30
34	1	1	0	-1	0	0	100	20	100	5	60
35	1	0	-1	0	0	1	60	5	200	5	90
36	1	-1	-1	0	0	0	20	5	200	5	60
37	1	0	-1	0	1	0	60	5	200	8	60
38	1	0	0	-1	-1	0	60	20	100	2	60
39	1	0	0	0	0	0	60	20	200	5	60
40	1	1	1	0	0	0	100	35	200	5	60
41	1	0	0	-1	0	1	60	20	100	5	90
42	1	1	0	0	0	1	100	20	200	5	90
43	1	0	-1	-1	0	0	60	5	100	5	60
44	1	0	0	0	0	0	60	20	200	5	60
45	1	1	-1	0	0	0	100	5	200	5	60
46	1	0	0	0	1	1	60	20	200	8	90

Based on BBD, a polynomial model with a second order was adopted to fit the interaction terms with the experimental data as shown in equation (3) [30]:

$$Y = a_0 + \sum a_i x_i + \sum a_{ii} x_i^2 + \sum a_{ij} x_i x_j \quad (3)$$

Where Y denotes the dependent variable (RE%), the index numbers for patterns were represented by i and j, a_0 refers to block term, x_1, x_2 … x_k are the cycle factors (free factors) in coded structure. a_i is the first-order(linear) fundamental impact, a_ii second-request principle impact and a_ij is the communication impact. Examination of difference was accomplished then estimateing the regression coefficient (R^2) for confirming the goodness of model fit was achieved.

3. Results and discussions

3.1. CKD properties

Fig. 1 shows The XRD-pattern of fresh CKD where different crystalline peaks could be observed. The main constituting phases in CKD were calcite ($CaCO_3$), quartz (SiO_2), and lime (CaO), beside the existing of portandite ($Ca(OH)_2$). $MgCO_3$ is probably to be exist, but it is not easy to distinguish since its characteristic reflections are very close to those of $CaCO_3$. The presence of lime is a measure of CaO content that readily available for reactions. So, CKD tends to have higher alkalinity behavior because of the mentioned constituents, which play a vital role in the heavy metals precipitation.

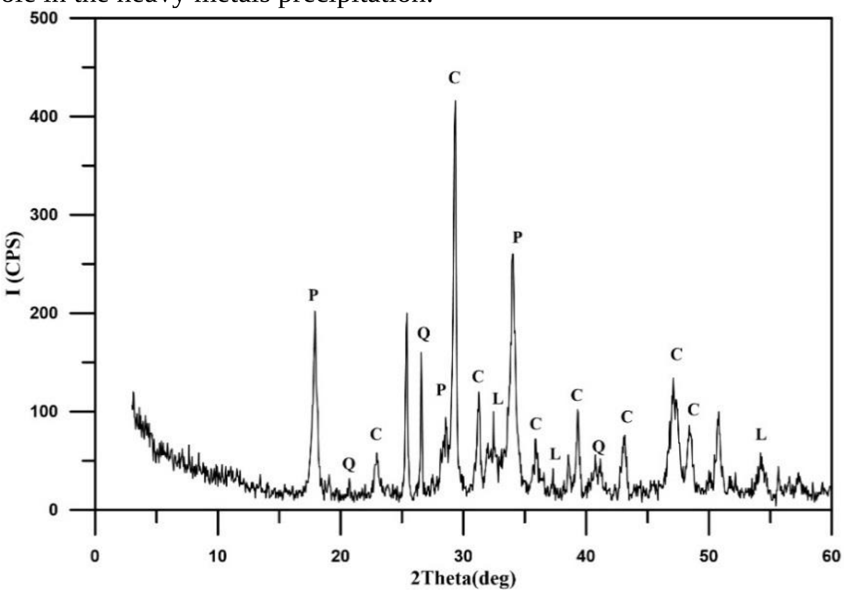
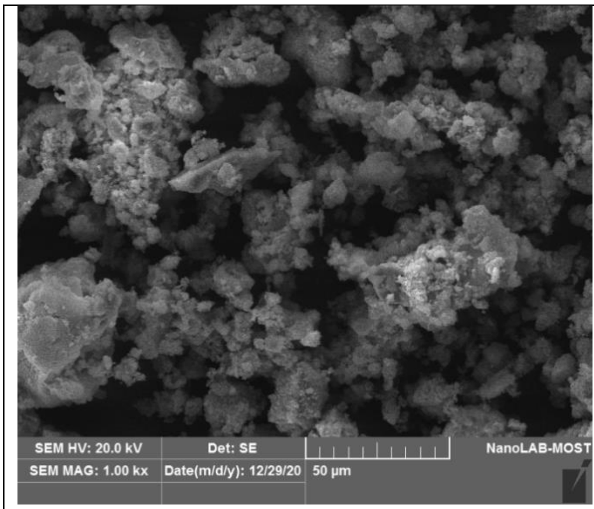
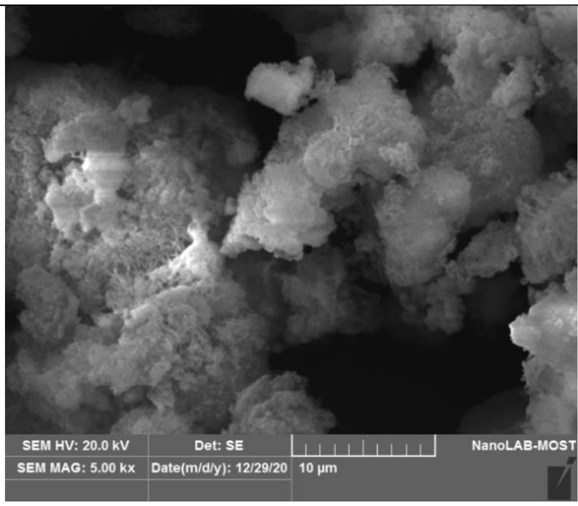


Figure 1. XRD analysis of CKD (C: calcite, Q: quartz, L: lime, and P: portandite)

BET surface area was found to be equal to 3.94 m^2/g. BET result indicates that CKD particles own a high superficial area. This suggests that due to the increasing in the surface area the oxide particles present in CKD can prove great reactivity [31]. Ibreesam and Faisal [18] found that The CKD gotten from Al-Kufa cement factory located at Kufa, Al-Najaf city in Iraq has a specific surface area of 0.5200 m^2/g. Mackie et al [31] studied the performance of Six CKD samples obtained from cement manufacturing plants located at North America and found they have specific surface area between 0.35 to 0.65 m^2/g. In a further study achieved by Mackie and Walsh [32], the same CKD samples were used as a neutralization agent. Coruh and Elevli [33] found that the specific surface area of CKD collected from Bartın Cement factory situated at North West of Turkey was 4.2 $m^2/$ g. larger values of specific surface area of CKD from other sources were also reported in the literature [10]. The specific surface area of the present CKD is approximately closed to that observed by Coruh and Elevli [33]. This designates that the particles of oxide existing in CKD have high reactivity as a result of high specific surface area they have. Increasing the surface area also permits more space to adsorption of metal, because that CKD was acting as sorbent and neutralizing agent [25]. The structural of the sorbents can be obtained by characterization using SEM since this technology has the ability to produce an actual clear image [34]. Fig. 2 (a, b) shows SEM images of fresh CKD at different magnifications: 10000x, and 5000x respectively. From these images, it can be shown that CKD particles have irregular surface, random in shape and size with many pores that may held the adsorbate. They are approximately resemble as a “popcorn structure with clustered fine and densely packed crystals which tend to agglomerate more together [35].



(a)



(b)

Figure 2. SEM images of fresh CKD at different magnifications a) 10000x,b) 5000x

3.2. Statistical analysis

RSM as a statistical method always used to maximize the production of certain product via the optimization of operating parameters. The interaction among process variables can be recognized by RSM whereas these intarctions can not be determined by conventional methods [27].

Forty six runs were conducted with diverse factors of the process via different sets to identify their impact on the removal efficiency and performing the optimization among them. Table 3 displays values of removal efficiency for each experiment. Results showed that the efficiency of Cd removal was arranged between 37.92% and 100% based on the experimental design.

Table 3. Experimental results cadmium removal

Run Order	Blocks	Real Value					RE%	
		Initial conc. (mg/l)	CKD dosage (g/l)	Shaking rate (rpm)	pH	Time (min)	Actual	Prediction
1	1	20	35	200	5	60	97.33	101.695
2	1	60	35	100	5	60	68.55	70.4321
3	1	60	35	200	5	90	83.56	83.3901

Run Order	Blocks	Real Value					RE%	
		Initial conc. (mg/l)	CKD dosage (g/l)	Shaking rate (rpm)	pH	Time (min)	Actual	Prediction
4	1	60	35	200	2	60	69.49	68.1964
5	1	60	20	300	5	30	60.55	68.7258
6	1	100	20	300	5	60	57.36	53.6923
7	1	60	20	200	8	30	65	70.4607
8	1	60	20	300	2	60	55.64	61.735
9	1	60	20	200	5	60	61.87	64.9831
10	1	60	5	200	2	60	50.145	50.4145
11	1	60	20	200	2	90	66.46	70.8899
12	1	20	20	100	5	60	93	91.4308
13	1	60	20	300	8	60	79.82	76.0050
14	1	60	20	300	5	90	91.87	80.1768
15	1	20	20	200	2	60	90.83	89.1951
16	1	60	20	200	2	30	71.36	59.4389
17	1	60	20	100	5	30	68.98	61.6746
18	1	20	20	200	8	60	95.61	100.217
19	1	100	20	200	5	30	42.122	48.148
20	1	60	35	200	5	30	70.33	71.9391
21	1	60	20	200	5	60	61.56	61.735
22	1	60	5	300	5	60	60.19	61.735
23	1	20	20	300	5	60	100	98.4820
24	1	60	20	200	5	60	61.63	59.7015
25	1	60	20	200	5	60	61.64	61.735
26	1	60	20	100	8	60	73.2	77.4834
27	1	60	35	200	8	60	77.51	68.9538
28	1	60	35	300	5	60	73.82	79.2183
29	1	100	20	200	8	60	53.27	55.4272
30	1	20	20	200	5	90	100	104.389
31	1	20	20	200	5	30	98.64	92.9377
32	1	100	20	200	2	60	43.26	44.4053
33	1	60	5	200	5	30	50.5	54.1572
34	1	100	20	100	5	60	48.24	54.1572
35	1	60	5	200	5	90	71.84	46.6410
36	1	20	5	200	5	60	86.85	65.6082
37	1	60	5	200	8	60	63	61.4364
38	1	60	20	100	2	60	58.27	57.9319
39	1	60	20	200	5	60	61.49	61.735
40	1	100	35	200	5	60	68.67	73.1256
41	1	60	20	100	5	90	66.04	56.9055
42	1	100	20	200	5	90	53.1	59.599
43	1	60	5	100	5	60	46.56	39.1236
44	1	60	20	200	5	60	62.22	61.735
45	1	100	5	200	5	60	37.92	52.6502
46	1	60	20	200	8	90	86.22	81.9117

Cd removal efficiency results were analyzed by Minitab-17 program where a quadratic model in terms of real units for process variables was obtained as follows:

$$RE\% = 191.0 - 1.536 X_1 + 0.664 X_2 - 0.2643 X_3 - 6.94 X_4 - 1.633 X_5 + 0.005053 X_1^2 + 0.00262 X_2^2 + 0.000274 X_3^2 + 0.277 X_4^2 + 0.00717 X_5^2 + 0.00845 X_1X_2 + 0.000133 X_1X_3 + 0.0109 X_1X_4 + 0.00200 X_1X_5 - 0.00139 X_2X_3 - 0.0269 X_2X_4 - 0.00451 X_2X_5 + 0.00771 X_3 X_4 + 0.002855 X_3 X_5 + 0.0726 X_4 X_5 \quad (4)$$

Condition (4) shows that the expulsion productivity changes dependent on the elements (squared and straight). Evacuation productivity increments with expanding upsides of positive coefficients, while it diminished by expanding upsides of negative coefficients and results shown that the constructive outcome have a place with CKD dose. The normal qualities for the expulsion proficiency getting from condition (4) are recorded in Table (3).

ANOVA was utilized to analyze suspicions about model coefficients dependent on a factual technique in which the absolute variety isolates into singular parts with specific wellsprings of variety [36,37]. ANOVA utilizes F-test and P-test for deciding model ampleness. By the relapse condition, for an enormous worth of F , the vast majority of the variety in the reaction could be explained . For assessing whether F is adequately huge to demonstrate factual importance, P-esteem is constantly received where a P-esteem under 0.05 implies that the model clarifies 95% of the fluctuation [38].
Table 4 presentations ANOVA for the quadratic model. This table presentations factual terms like DF (level of opportunity), SeqSS (amount of the square), Adj SS (changed amount of squares), Adj MS (changed mean of square) , Cr. % (level of commitment for every boundary), F-worth and P-esteem. The worth of F is equivalent to 33.72 at P equivalent to 0.0001 conferms incredible significance for the relapse model. Approval of Model fit quality was accomplished by different connection for the model. For this situation, a various connection coefficient with a worth of 96.43% was gotten which demonstrates that this relapse measurably critical. Upsides of anticipated different connection coefficient (pred.R2 = 85.71%) and changed numerous relationship coefficient (adj.R2 = 93.57%).are viable in the presnt work which implies great model portrayal.
ANOVA results introduced that the percent of commitment of introductory focus is 64.69%, which implies that underlying Cd fixation turned into the fundamental viable boundary on the expulsion effectiveness of cadmium followed by CKD measurements. The straight term has 84.63%, as a principle percent of commitment to the model with followed by the squared with 6.48% and the interaction among the variables with 5.31%. The results confirm that cadmium initial concentration is the most important factor.

Table 4. ANOVA results for cadmium removal

Source	**DF**	**SeqSS**	**Cr%**	**Adj SS**	**Adj MS**	**F-value**	**P-value**
Model	20	11961.2	96.43%	11961.2	598.06	33.72	0.0001
Linear	5	10498.6	84.63%	10498.6	2099.72	118.39	0.0001
X_1	1	8024.5	64.69%	8024.5	8024.5	452.45	0.0001
X_2	1	1264.8	10.20%	1264.8	1264.78	71.31	0.0001
X_3	1	198.9	1.60%	198.9	198.88	11.21	0.0030
X_4	1	485.9	3.92%	485.9	485.93	27.40	0.0001
X_5	1	524.5	4.23%	524.5	524.5	29.57	0.0001
Square	5	803.6	6.48%	803.6	160.73	9.06	0.0001
X_1^2	1	406.8	3.28%	570.4	570.42	32.16	0.0001
X_2^2	1	22.1	0.18%	3.0	3.04	0.17	0.6830
X_3^2	1	6.7	0.05%	65.6	65.61	3.70	0.0660
X_4^2	1	5.1	0.04%	54.2	54.18	3.05	0.0930
X_5^2	1	362.9	2.93%	362.9	362.94	20.46	0.0001
2-Way Interaction	10	659.0	5.31%	659.0	65.90	3.72	0.0040
X_1X_2	1	102.7	0.83%	102.7	102.72	5.79	0.0240
X_1X_3	1	1.1	0.01%	1.1	1.12	0.06	0.8030
X_1X_4	1	6.8	0.06%	6.8	6.84	0.39	0.5400
X_1X_5	1	23.1	0.19%	23.1	23.13	1.30	0.2640
X_2X_3	1	17.5	0.14%	17.5	17.47	0.99	0.3300
X_2X_4	1	5.8	0.05%	5.8	5.84	0.33	0.5710
X_2X_5	1	16.4	0.13%	16.4	16.44	0.93	0.3450
X_3X_4	1	21.4	0.17%	21.4	21.39	1.21	0.2830
X_3X_5	1	293.4	2.37%	293.4	293.44	16.55	0.0001
X_4X_5	1	170.6	1.38%	170.6	170.57	9.62	0.0050

Source	DF	SeqSS	Cr%	Adj SS	Adj MS	F-value	P-value
Error	25	443.4	3.57%	443.4	17.74		
Lack-of-Fit	20	443.0	3.57%	443.0	22.15	304.15	0.0001
Pure Error	5	0.4	0.00%	0.4	0.07		
Total	45	12404.6	100 %				
	S	R^2	R^2(adj)	PRESS	R^2(pred)		
Model Summary	4.21137	96.43%	93.57%	1772.63	85.71%		

Figure 3 shows scattereing of residuals was observed with no any definite pattern confirming the significance of the model because of no outliers are existing in the data of the standard probability plot with a non-linear relationship. Data are not skewed as confirmed by Histogram.

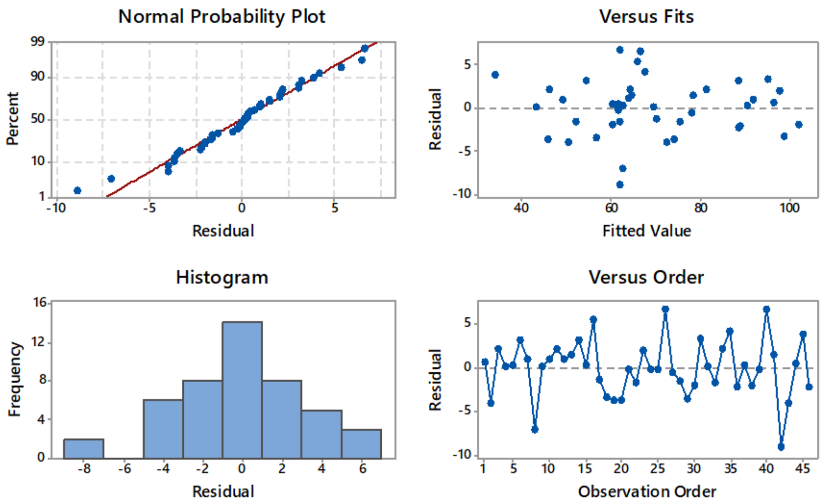


Figure 3. Residual plots

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3.3. *Effect of interaction factors on the cadmium expulsion productivity*

Figures 4-a,b explain the impact of initial concentration (20,60,100)mg/l of Cd on its removal efficiency at different values of shaking speeds (100,200 and 300 rpm) with holding value of CKD dosage at 20 g / l, pH at 5 and contact time at 60 min. Figure 4-a displays the reaction surface plot, while Figure4-b delineates the relating shape plot.

From the plot of reaction surface, it was shown that at a shaking rate of 100 rpm, a decrease in the effectiveness of the evacuation occurs with beginning focus expanding. There was nevertheless a small change in the removal efficiency as the speed of rotation increased from 100 to 300 rpm specifically at a concentration of 20 mg/l, This means that the reactivity of CKD is very high due to generating two mechanisms namely adsorption and precipitation responsible of the removal process; so, the agitation speeds within the adopted range have not a significant effect on the removal process. Similar behavior was observed by Ibreesam and Faisal [18]. The corresponding contouring plot approves that the maximum removal efficiency > 90% occurs in a range in which the shaking speed ranges between 100-300 rpm and Cd concentration between 20-25 mg/l.

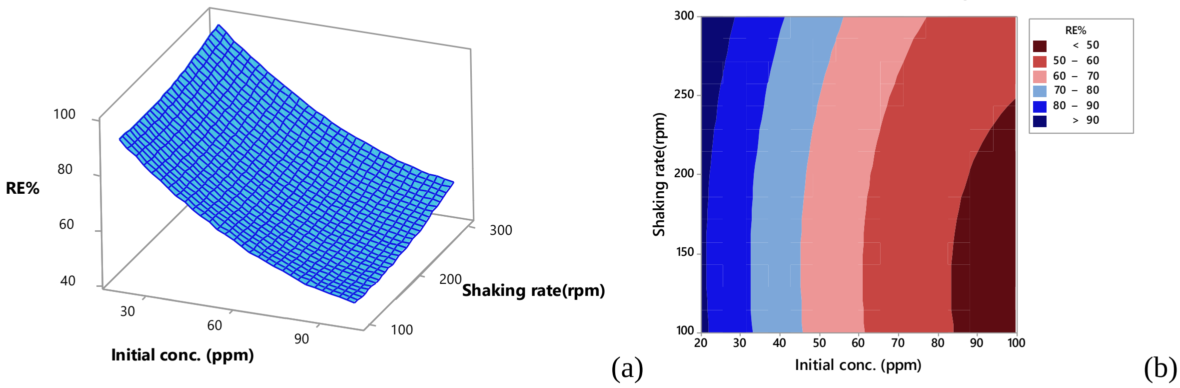
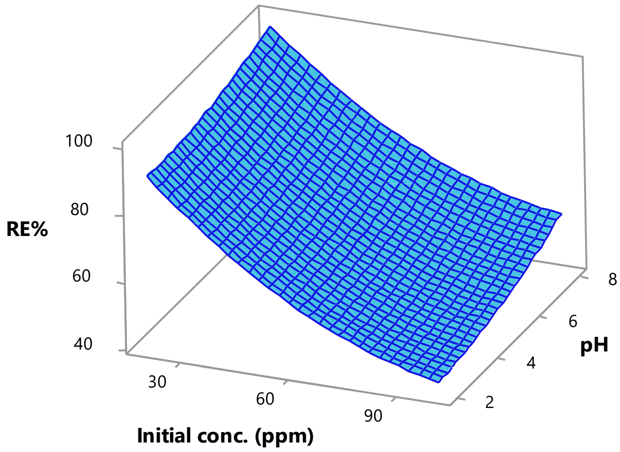
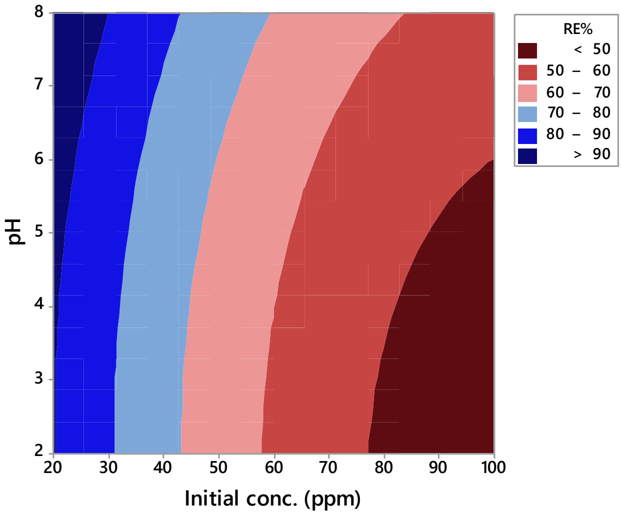


Figure 4. Plot(a) Response surface and plot(b) structure for the impact of early on cadmium spotlight and shaking rate on the adequacy departure of cadmium hold esteems: CKD dosage(20g/l), pH(5),contact time (60min.)

Figures5-a,b illustrate the impact of Cd initial fixation on the expulsion proficiency at various upsides of pH (2,5,8),with holding worth of CKD measurements at 20 g/l, the shaking speed at 200 and the contact time at an hour. Figure (5-a) shows the reaction surface plot, while Figure (5-b) outlines the comparing shape plot. The pace of weighty metals expulsion from wastewater is represented generally by pH as per prevous contemplates [24]. From Figure5-a, it is seen that, at pH 2, the proficiency of evacuation decays with expanding the underlying centralization of Cd. Nonetheless, a little improvement in the expulsion productivity happened with the pH arriving at 8. At a grouping of 20 ppm. The outcomes showed an increment in expulsion proficiency happened by expanding the pH. The comparing shape plot Figures5-b affirms that the greatest worth of CD evacuation proficiency > 90% happens in a little reach where the pH ranges between 5-8 and the metal particle focus is between 20-25 ppm.
As pH expanded from 5.0 to 8, more great electrostatic fascination powers would be upgraded cationic metal particle adsorption because the CKD surface becomes more negatively charged [24]. El Zayat et al [19] observed that, at pH values below 4, CKD has lower sorption capacity for uptaking the metal. At pH values higher than 4, a significant increase in the CKD capacity adsorption was detected especially at pH = 5.0 while their results confirmed that an optimum capacity (>90%) was obtained at pH higher than 6. The present results are compatible with work of El Zayat et al [19]. Similar observation was found by El-refaey [21].



(a)

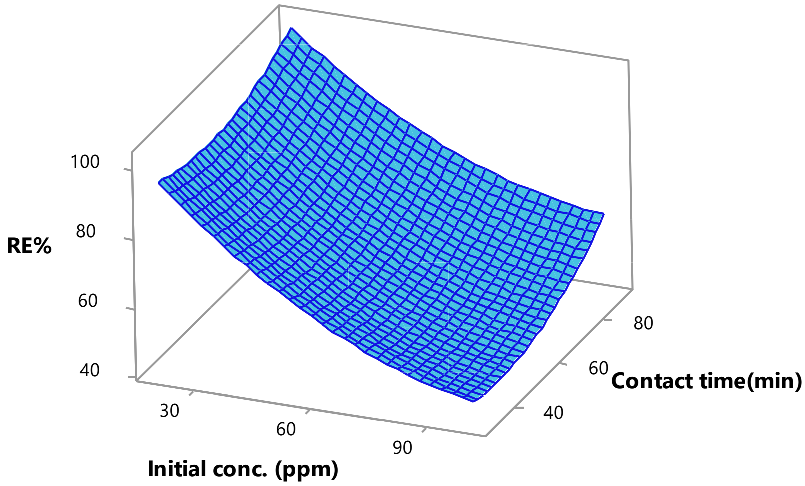


(b)

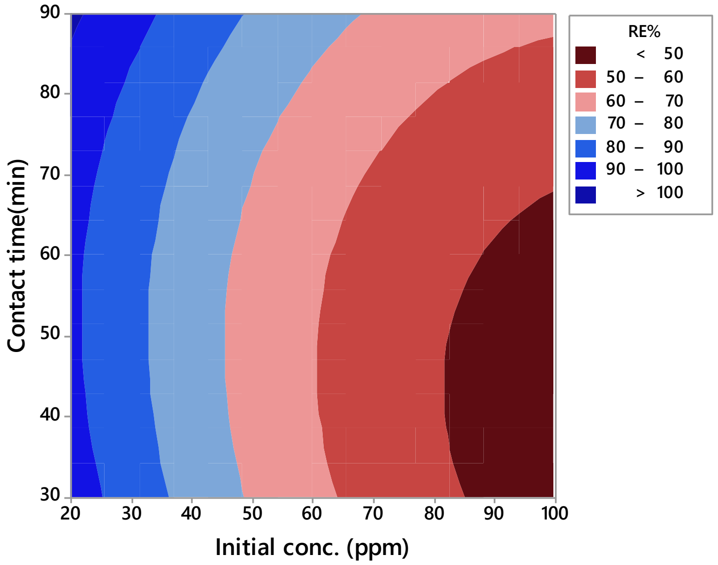
Figure 5 Plot(a) Reaction surface and plot(b) shape for the effect of starting cadmium conc. what's more, pH on the effectiveness removal of cadmium CKD dosage(20g/l), shaking rate(200rpm),contact time(60min.)

Figures 6-a,b illustrate the impact of Cd initial concentration on the Cd removal efficiency at different contact time values (30,60,90 minutes) with holding CKD dosage at 20 g / l, shaking speed at 200 and pH at 5 where Figure 6-a addresses the reaction surface plot while Figure 6-b shows the relating shape plot. From the plot, it was shown that at contact season of 30 minutes, the effectiveness of expulsion decreases with expanding of the underlying fixation.

The expulsion effectiveness changes altogether as the contact time got higher than 60 min. for various upsides of beginning Cd focus. The relating form plot supports that the greatest worth > 90% of Cd expulsion effectiveness happens in a little reach wherein the contact time ranges between (60-90 minutes) and the ion metal concentration is between 20-35ppm for Cd.
At the initial stage, the high rate of sorption process is occurred due to the existing of enough numbers of binding sites on the adopted sorbent which then decreased with elapsed time due to the hold of these sites by cadmium ions [18].



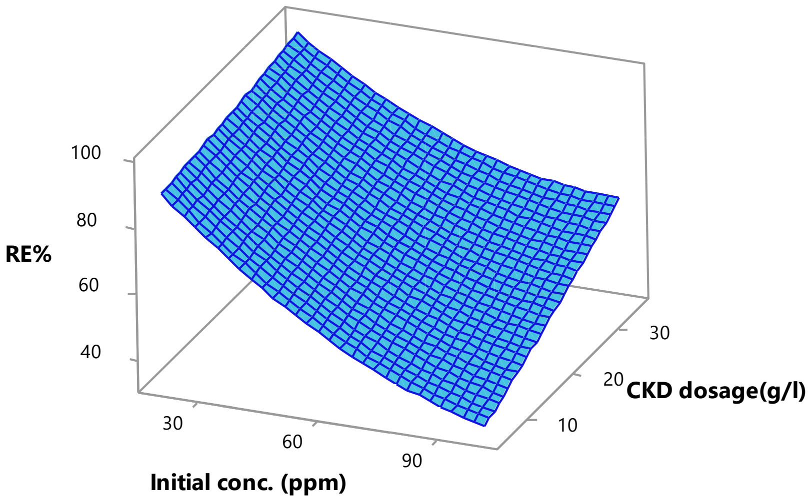
(a)



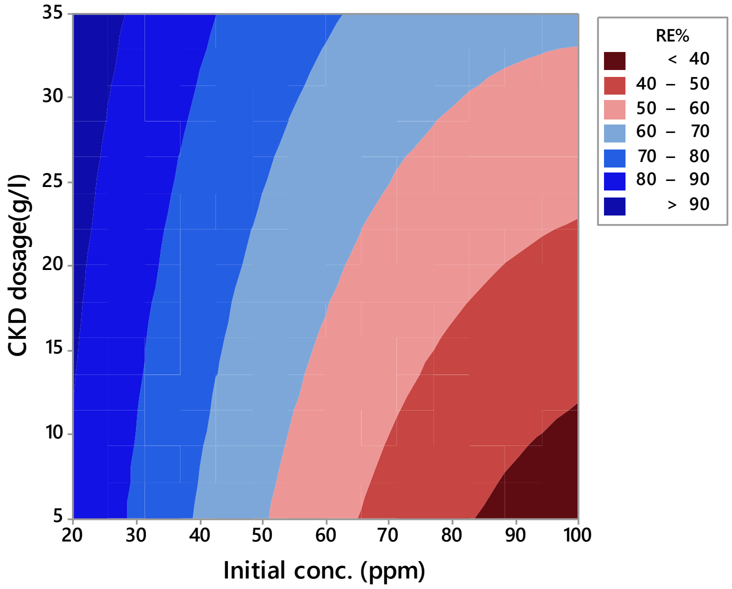
(b)

Figure 6. Plot(a) Reaction surface and plot(b) shape for the effect of starting cadmium conc. also, contact time on the effectiveness evacuation of cadmium. Hold esteems: shaking rate (200rpm), pH (5), CKD dosage (20g/l)

Figures 7-a,b shows the effect of beginning cadmium focus and CKD dosage on the cadmium removal efficiency. The response surface plot Fig. 7-a demonstrates that the CKD dose have a good impact on the removal efficiency, rises rapidly as the dose of CKD increases to 35 g/l specially at higher concentration of Cd. The corresponding contouring plot Fig.7-b approves that the maximum value of the aforementioned expulsion proficiency > 90% located in a small range in which the CKD dose ranges between 13-35 g / l, and the Cd ion concentration between 20-30 ppm. The increase in removal efficiency with increasing CKD dosage may be ascribed to existing greater vacant sites [18]. All Figures (4-6) showed that removal efficiency declined with an expansion in the underlying fixation of cadmium. Similar observations were found in previous works [24].



(a)



(b)

Figure 7. (a) Plot of Reaction surface and(b) plot of shape for the effect of beginning cadmium fixation and CKD measurements on the effectiveness removal of cadmium. Hold values: shaking rate (200rpm), pH (5), contact time (60 min.)

3.3. The optimization and confirmation test

For obtaining the optimum value that increases the desirability function (D_f), a numerical optimization of the program has been applied. The preferred objective was determined by controlling the weight or value that can alter the target characteristics. Five options have been selected for response goal domains: Minimum, Maximum, Focus, inside reach and None [37]. In the present research, the aim is to maximize the removal efficiency so that the "maximum" field with the relating "weight" 1.0 was chosen. 37.92% was taken as a minimum for the evacuation effectiveness while 100% was taken as the most extreme. Under these settings and limits, an optimization procedure was performed and results with the desirability function of (1) are shown in Tables 5. Optimization results shows that using cadmium concentration (20 ppm), shaking speed (300 rpm), CKD dosage (35 g / l), time (90 min) and pH 8 will give a complete removal of Cd.
Two trials with optimized process parameters were performed to confirm optimization results. 20 ppm was taken as the initial concentration of cadmium. The results are shown in Table 6. After 90 minutes of contact time, pH 8, a shaking speed of 300 rpm and CKD dosage of 35 (g / l), Cd removal efficiency(99.75%) was accomplished which is in concurrence with the ideal worth came about because of enhancement using allure work.

Table 5. The ideal upsides of cycle factors for amplifying cadmium efficiency of removal

Response		Goal		Lower	target	upper		weight	importance
RE(%)		maximum		37.92	100	100		1	1
Solution: Parameters					Results				
conc. (ppm)	CKD (g/l)	Shaking speed(rpm)	pH	time (min)	RE (%) Fit	D_f	S E	95% CI	95% PI
20	35	300	8	90	133.917	1.0	7.70	(118.06; 149.78)	(115.84; 151.99)

Table 6. Confirmation the optimum conditions for cadmium removal efficiency

Run	**Initial conc. (ppm)**	**CKD dosage (g/l)**	**Shaking speed (rpm)**	**pH**	**time (min)**	**R E(%)**	
						actual	**Average**
1	20	35	300	8	90	99.80	99.75
2	20	35	300	8	90	99.70	

3.4. Isotherm models study

The adsorption isotherm represents the relationship between the quantity of adsorbate at equilibrium per unit of adsorbent (q_e), with the equilibrium concentration of adsorbate in a solution (C_e). Adsorption modeling will allow us to valuation both the improvement of the adsorption system and the operating conditions required to improve the efficiency of the system [39].

The quantity of contaminants absorbed per unit mass of adsorbent at any time (q_t) was estimated using the following relationship [40]:

$$q_t = (c_i - c_t)\,\frac{V}{m} \tag{5}$$

Where q_t in mg /g, V is volume of solution treated in L, m weight of adsorbent in g.

Various models of adsorption isotherm have been presented in the literature [40]. The simplest approach mainly applied to determining isothermal constants is the linear model relationships and then the application of linear regression. Appropriate models are determined using linear regression and model parameter detection [41].

Isotherm adsorption from Langmuir assumes monolayer deposits with respect to adsorption on the surface of homogeneous adsorbents (thrombosis). Such isotherm is provided mathematically in the following way [42]:

$$q_e = (K_{max}\,K_L C_e)/(1 + K_L C_e) \tag{6}$$

Where q_e in mg /g refers to the quantity of pollutant absorbed on the surface of CKD at equilibrium concentration C_e(mg/l), q_{max} refers to the maximum absorption capacity, and K_L refers to the constant of Langmuir in L / mg. Eq 6. can be written in a linear form:

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{q_{max} K_L C_e} \tag{7}$$

Thus, the graph of 1 / q_e versus 1 / C_e creates a straight line with the intersection 1 / (q_{max}) and the slope 1 / K_L q_{max}.

A separation factor or equilibrium constant R_L is used to characteristize Langmuir isotherm, which can be defined as follws:

$$R_L = \frac{1}{1 + K_L\,C_O} \tag{8}$$

The separation factor R_L specifies the isotherm shape and whether the adsorption is favorable or not, as per the criteria given below. Unfavorable; $R_L > 1$, linear; $R_L = 1$, favorable ; $0 < R_L < 1$, and irreversible; $R_L = 0$

Isotherm adsorption from Freundlich binds the adsorption force associated with the absorbent towards the adsorbent. This model takes into account the formation of multi-layer adsorption through active sites that are distributed in a heterogeneous manner. This model is based on a premise that strong anchor sites will be bonded at first and that the adsorption strength will be low as the site's occupancy increases. The mathematical expression related to Freundlich model can be determined by Ghanim et al [42]:

$$q_e = K_F C_e^{\frac{1}{n_f}} \tag{9}$$

Where K_F represents the Freundlich constant which indicates the adsorption power with respect to absorption in (L/g) while the experimental constant (n_f) corresponds to the range associated with the driving force of the adsorption.
The Freundlich model can be presented as follows:

$$\ln(q_e) = \ln(K_F) + \frac{1}{n}\ln(C_e) \tag{10}$$

Thus, the plot of ln (q_e) vs. ln (C_e) creates a straight line with the interception of ln (K_F) and a slope of $1 / n$. To investigate isothermal models, different experiments were performed at different CKD dosage (5-35g) using initial cadmium concentration 50ppm at contact time up to 120 min. Due to the fact that pollutants have negatively charge, they might play the role of donors of electron which are attracted to ions of metals in situ and form gelatinous precipitates [19]. The chosen time in the present section has been higher than 90 minutes for ensuring the equilibrium behaviors of the isotherm. Every isotherm in the present work was taken in the linearized form, for obtaining the isotherm model of the optimal fit with equilibrium data of the experiments. The correlations of the isotherm have been plotted in Figures 8 and 9 for cadmium and with inserting its regression equation and corresponding the linear regression coefficient R^2. Figures 8 have shown the fact that the Langmuir model can draw sufficiently experimental data calculated using a regression coefficient of 0.992, for cadmium, while freundlich model shows R^2 value equal to 0.987. This point out that the equilibrium model of Langmuir was more favorable to elucidate extremely the uptake process by the adsorbent in the present study, representing just one layer adsorption coverage of cadmium and happened in adsorbent surface that was not uniform [20].

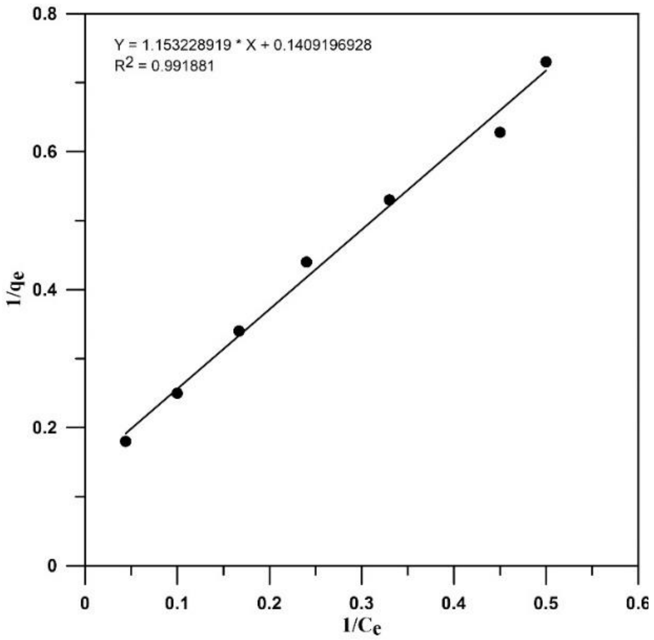


Figure 8. Isotherm for cadmium in langmuir adsorption

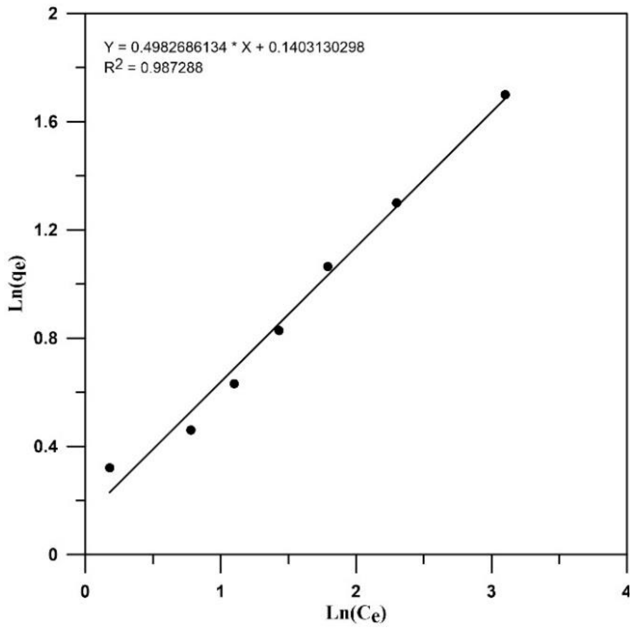


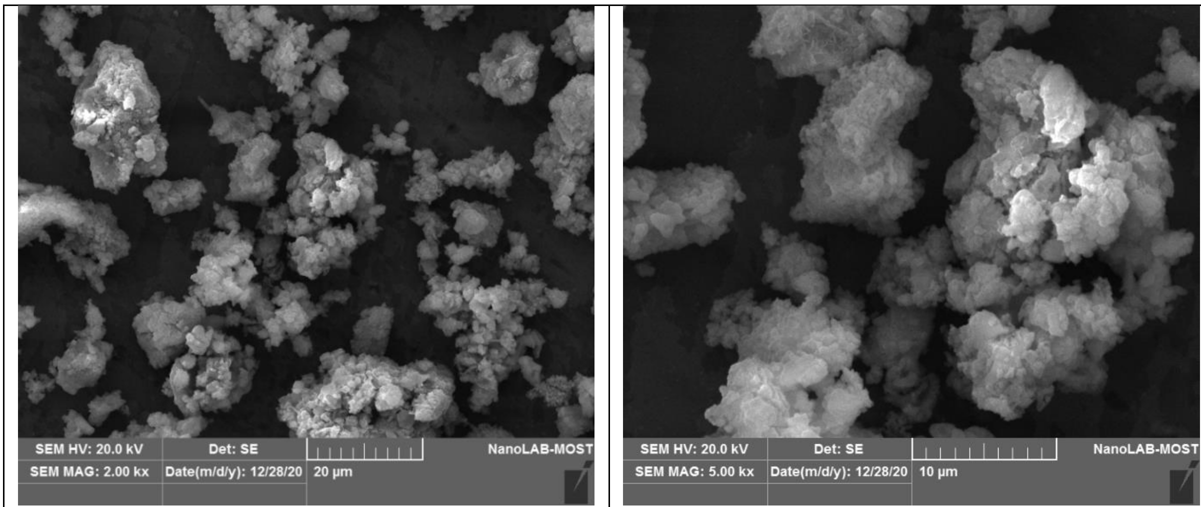
Figure 9. Isotherm for cadmium in freundlich adsorption

Table 7 shows the parameters of the two models. Based on Langmuir constant presented in table 7 the value of R_L is 0.1406, which occurred between 0 and 1 approving that the adsorption of cadmium ions is favorable. From Freundlich model, the value of n lied between 1 to 10 indicating favorable adsorption [24].

Table 7. Isotherms models parameters

Isotherm model	**Parameter**	**Value**
Langmuir Adsorption	Q_m(mg/g)	7.096
	K_L(L/mg)	0.12219
	R^2	0.991881
Freundlich Adsorption	K_F(L/g)	1.1506
	n	2.0
	R^2	0.987288

The level of Langmuir model's applicability was proven in numerous studies [43]. Similar observation was found by El Zayat et al [19] and Waly et al [24] for removal of Cd using CKD. Abu El-Eyuoon Abu Zied Amin and Selmy [20] showed that the Langmuir model can draw sufficiently experimental data for Cd removal using Assiut cement kiln dust, Egypt. Ibreesam and Faisal [18] in their study observed that the applicability of two models to characterize the sorption of cadmium by CKD obtained from Al-Kufa cement factory, Iraq.
Fig. 10 (a, b) shows SEM images of CKD after cadmium adsorption at different magnifications: 10000x, and 5000x respectively. From these images, it can be shown that CKD particles after Cd adsorption looked like the fresh CKD in their structure but with more agglomerated structure and less pores structure. The EDX analysis results (Fig.11 and Table 8) show that Ca is the main constituent of CKD in addition to considerable amount of Si, Al and Mg in CKD. Beside, cadmium was observed in EDX with a substantial amount as a minor constituent confirming achieving the adsorption process with high activity of CKD for adsorbing the cadmium.



(a) (b)

Figure 10. SEM images of the used CKD after adsorption at the optimum conditions with different magnifications a) 10000x,b) 5000x

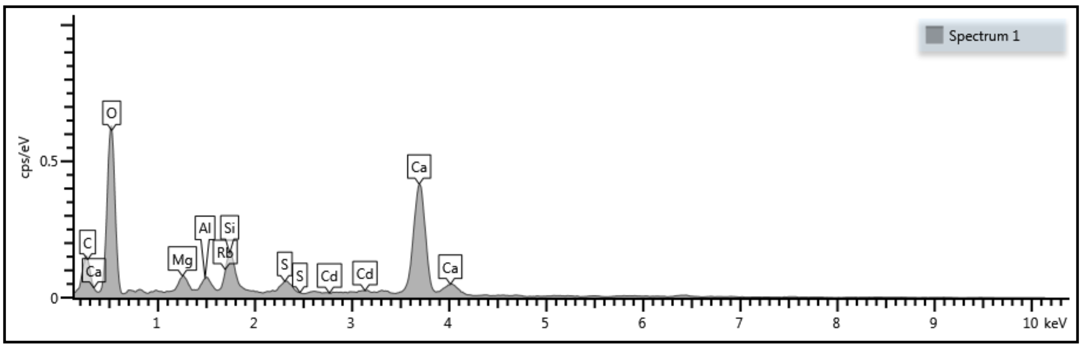


Figure11. EDX image of the used CKD

Table 8. EDX results of the used CKD

Element	Wt%	Wt% Sigma
C	7.20	1.55
O	37.05	1.78
Mg	1.62	0.35
Al	1.41	0.36
Si	4.40	0.60
S	1.69	0.47
Ca	44.13	1.87
Rb	0.23	1.32
Cd	2.26	1.39
Total:	100.00	

FTIR method is an essential technique to identify the structures of the functional groups as well as the existing of definite surface functional groups as a part of sorbents structure. By this technique, it is possible to find the impact of these groups on the metal binding process [21]. Figure 12 shows FTIR spectra of the CKD before and after Cd ions sorption from aqueous solutions. The FTIR spectrum of CKD displays peak positions at 3435.73, 2926.25, 1413.05, 1109.55, and 871.78 cm^{-1}. The band at 3435.73is owing to O–H (hydroxyl) while the bands at 1413.05, and 1109.55 reveal the carbonate and silicate; 871.78 reveal (C-H) bond. Similar data were obtained by El-Refaey [21] and Saraya and Aboul-Fetouh [44]. Slight shifts were detected in FTIR bands of studied sorbent after the sorption reaction with cadmium ions confirming the attachment of cadmium ions on CKD.

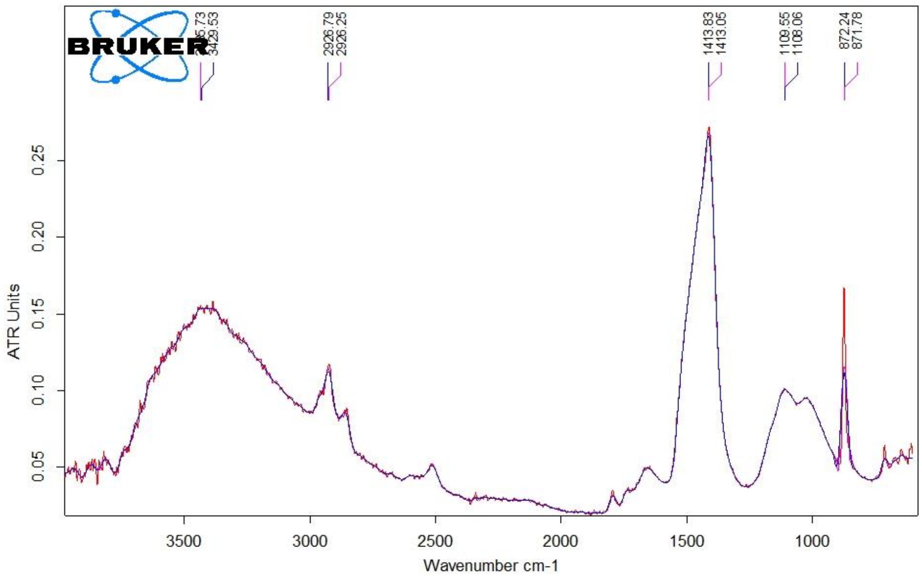


Figure 12. FTIR spectra of the CKD before (red) and after (blue) sorption of Cd from simulated wastewater

4. Conclusions

In current study, RSM method combined with Box-Behnken Design was used to obtain the mathematical model and optimize process factors for cadmium removal using cement kiln dust generated from Al-Duh Cement Factory situated in south of Al-Muthanna Governorate, Iraq. RSM strategy showed that underlying convergence of Cd basically affects the evacuation proficiency followed by CKD dosage. EDX and FTIR confirms the good adsorption of Cd on CKD. It was found that solution pH has an essential effect on the Cd removal efficiency and pH value higher than 5 is recommended. Studies of adsorption isotherm evidently showed that the behavior of Cd ions adsorption on CKD agree well with Langmuir assumptions. The results of current study confirmed that CKD which considered as a cheap product from cement industrial processes was active in the removal of cadmium ions from a simulated wastewater.

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