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Optimization of Response Surface Methodology for Removal of Cadmium Ions from Wastewater using Low Cost Materials

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ABSTRACT
This study goal to the ability of using low cost materials representing thermestone and aluminum solid wastes in water filtration by using a pilot plant constructed in wastewater treatment plant to remove cadmium ions (Cd(II)). Response Surface Methodology (RSM) used to optimize the optimal parameters that affecting the performance of filter units, these parameters are time, Cd(II) concentration, and filtration rate. These optimized parameters were 9 hr., 5 ppm, 10 l/hr. with removal efficiency of Cd(II) for A-Filter, T-Filter, S-Filter, and A-T-S-Filter was 94%, 95%, 86.8% and 90%, respectively. The result shows that the T-filter has higher cadmium removal efficiency than A-filter, S-filter and S-T-A- filter. While A-filter has a higher removal efficiency of cadmium than the S-filter and S-T-A- filter. While the S-T-A- filter has higher efficiency than S- filter. The result obtained from RSM was good Agreement with the result of experiments. As a result, the optimized process in this paper can be widely utilized with high removal ratio of Cd(II) ions from wastewater samples.

Keywords: Cd(II) removal, RSM, cadmium, thermestone, aluminum, filter media.

INTRODUCTION

The techniques that remove more pollutants from wastewater than the treatment of conventional is called advanced treatment of wastewater (Crini and Lichtfouse, 2019). In the United States, the utilization of advanced wastewater treatment processes has increased significantly (Visa and Chelaru, 2014; Shen et al., 2015). Most of their objectives are to remove heavy metals such as cadmium (Dixit et al., 2015). Reusing wastewater is becoming more important in some location with limit water supply sources. Furthermore, some industrial wastewater must comply with strict rules and regulations regarding the removal of particular toxic compounds and refractory organics, which cannot be accomplished by conventional secondary treatment systems. As a result, advanced wastewater treatment techniques are required (Lin and Lee, 2007; Nasir et al., 2022).

There were reusing industries of small-scale for paper, glass, plastic and scrap metal locally or carried to re-rolling mills of many states. Materials reusing were widely in every considerable cities and in majority of medium-sized cities (Salzmann, 2022). Metals (mostly aluminum), thermostone, cardboard and paper are the substances mostly recycled and reused by large-scale industries. It is worth mentioning that using of the aluminum waste as filter media rather than sand because it has density less than that of water media (Raheem et al., 2022). The aluminum media is available, low cost, and easy to maintain. On the other hand, thermostone is available in large quantities as waste products from construction and demolition operations, may have potential as inexpensive filter materials and also has many indirect importance such as protecting the environment, from the effects of pollution and energy saving and lowering landfill costs. It is important to indicate the using of local solid waste as filters media will certainly be reduced the cost of water and advanced wastewater treatment and sustainability (Voigt, 2022; Abdulhasan et al., 2022).

Previous studies related to this study are as the follow: Alwared and Zeki (2014) proved the ability to use aluminum filings as a monomedia in gravity rapid filter to compare it with the conventional sand filter. The results showed that aluminum filings filter better than the sand filter in turbidity removal and in head loss reduction. Zwayen (2015) examined the ability to use the filings of solid wastes like glass, aluminum, and plastic as a filter media. The results of his study showed that the aluminum media can be considered the best media as compared to the sand media in terms of runtime by 22.7–29.16% when the gradation and its depth of filter were changed. While the plastic media can be considered the best media compared to the sand media in terms of runtime by 19.2–31.5%, when the filtration velocity was increased. Also, the plastic media showed best media and it had long run time in comparison with sand media by 19.2–26.3%, when the concentration was increased. Finally, he proved that the backwashing for these different media needed time and amount of water less than that of the sand media. Martemianov et al. (2017) utilized disposable, cost-effective adsorbents. The adsorbents used were of vermiculite concrete and aerocrete (thermostone) modified with iron oxyhydroxide (FeooH). They showed that adsorbent materials have a greater capacity for adsorbing heavy metal cations than activated carbon and vermiculite-based adsorbents. Dong, C. et al. (2023) shows that the geopolymer adsorbent affect of RSM for treatment of wastewater from cadmium (Cd). The result shows RSM very effective. Lau et al. (2020) proved of using RSM for the effect of contact, flow rate, contaminate concentration. Emami M.R.S. (2021) Proved that optimize the removal % and permeation flux behavior of aqueous Pb^{2+} solutions in nanofiltration processes using RSM. The RSM model determined a regression coefficient $R^2 = 0.99$ for both removal efficiency and permeate flux.

The new modifications of the present study are to use other local and cheap materials such as aluminum and thermostone waste as filter media instead of sand and anthracite in granular filtration process for tertiary treatment processes of secondary effluents from a municipal wastewater treatment plant for reclamation and reuse. The RSM demonstrated the optimized removal parameters, as well as time, Cd concentration, and filtration rate. Also, the low cost materials performance in watstewater samples and its reusability were examined.

MATERIALS AND METHODS

Sand and gravel

The sand that are used in current study in Iraqi wastewater treatment plants were brought from the local market, the sand gradations were 0.6–1, where the maximum size of sand was 1 mm. The sand and gradations were shown in Figure 1.

Thermestone materials

The samples of thermestone that utilized were brought and collected as waste from a demolished buildings, construction, and from storage locations and as shown in Figure 2a, then crushed, ground and sieved to obtain two granular degradations (Table 1) as shown in Figure 2b, after that, The samples were cleaned with distilled water before usage to eliminate of any fine powder, and

Figure 1. Sand used in the present study

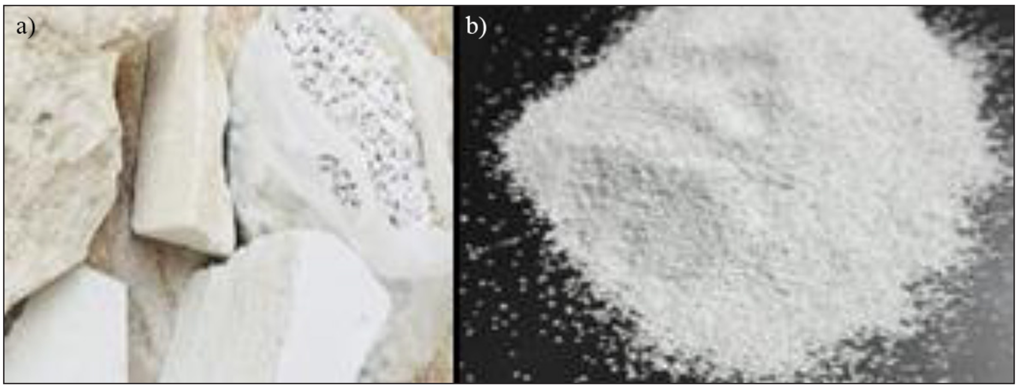

Figure 2. Thermestone

they were then put in an oven at 383 k to dried for 24 hr. (Al-Suhaili et al, 2014).

Aluminum materials

The first source of the aluminum solid waste was the local manufacturing plants for doors, windows and aluminum counters and the second source was the turnery shops for rims which discarded as wastes. The wastes form second source were crushed by the electric grinder machine as shown in shown in Figure 2, then put in HCl acid 10 % to remove the color from wastes' surface which causes additional turbidity. After removing the color, the aluminum filings washed by distilled water until the pH value for washing water became normal. The wastes form the first source were in the form of filings and did not cause an additional color therefore washed by distilled water, mixed with filings from the second source and sieved into gradation of (0.6–1.0) (Table 1) mm like sand media see Figure 3.

Figure 3. Aluminum

Cadmium nitrate

Cadmium Nitrate is salt used for preparing synthetic wastewater containing cadmium. It was bought from the local market with the following main properties see Table 2.

Experimental procedures

The pilot unit consists of four filters of a Polyvinylchloride (PVC) column with an inside diameter of 80 mm and a height of 150 cm, it was partially filled with 50 mm of a media. The filters are designed and built to run in parallel with down-flow direction (Aboubaraka et al., 2022), as shown in Figure 4. The filter columns were used in pilot filtration unit as follows. The mono media filters where sand media (S- filter) with depth of 50 cm in filter No. 1, thermestone media (T-filter)

Table 1. Physical properties of the filter media

Filter media		Permeability cm/sec	Specific gravity	Porosity %
Aluminum	0.6–1.0 mm	0.648	0.974	53
Thermestone	0.6–1.0 mm	0.908	0.974	63

Table 2. Cadmium nitrate main properties

Purity%	Solubility of the salt (mol/L)	Chemical formula	Molecular weight (g/mole)
98	7.21	$Cd(NO_3)_2 \cdot 4H_2O$	236.42

with depth of 50 cm in filter No. 2, and aluminum media (A-filter) with depth of 50 cm in filter No. 3. The multi-media of Aluminum–Sand-Thermestone (A-S-T-filter) with depth of 20 cm of aluminum, 15 cm of sand, and 15 cm of thermestone in filter No. 4. The grain size of 0.6–1 mm for each filter. These filters were subjected to the same operating conditions of filtration rates and influent cadmium concentration. The filters were operated at rates 10, 20 and 30 ml/min and influent cadmium concentration 5, 10, 20, 30.

Response surface methodology (RSM)

RSM is a method that uses in design of experiment data during the optimization process (Lee, 2006). A quadratic model using the central composite design (CCD) was used in current study (Box & Draper, 1987). The independent variables namely run time (A), cadmium concentration (B), and flow rate (C) were studied (Cochran & Cox, 1957). Range and levels of Independed Variables are shown in Table 3.

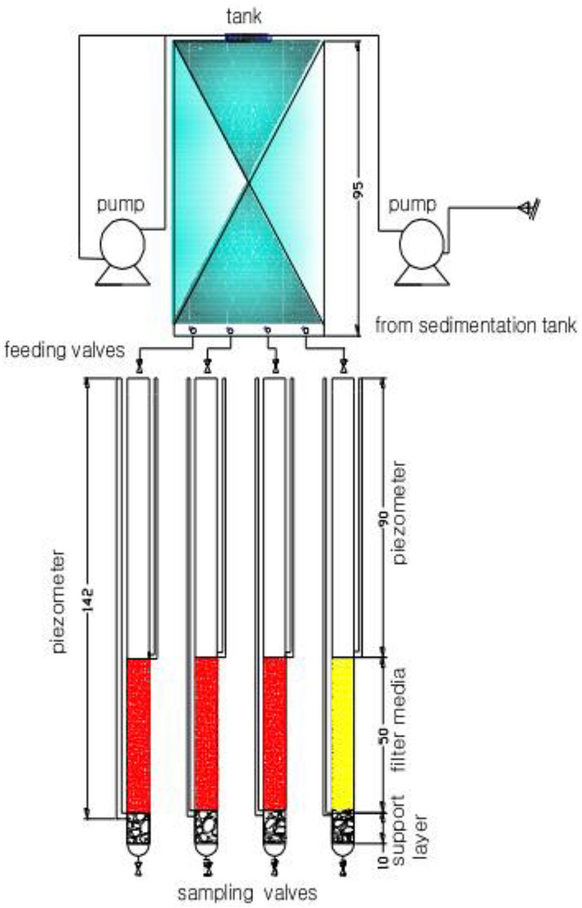

Figure 4. Schematic diagram of the filtration plant

RESULTS AND DISCUSSION

To achieve the main goals of the present study and also, to confirm the ability of the filter used, it was decided to use synthetic wastewater which has been contaminated with cadmium (II) as a type of heavy metals contamination.

The reason for selecting cadmium metal as among the heavy elements in this study is extremely toxicity of this element even in relatively low dosages and it is classified as one of the most toxic heavy metals that is listed through the first category of hazardous waste pollutants for its adverse impact on both human health and natural environment and it is can be discharged in the effluents of many industries such as textile printing industries plastics manufacturing, metallurgical and mining. This justification agrees with that obtained by (Mohammed and Ebrahim, 2012; Zhang et al., 2017).

The different operation conditions of flow rate and initial concentration with a constant certain pH value of 7.6 and preselected running time were used in filtration technique experiments to evaluate the removal performance of filter media as low cost media instead of expensive adsorbents and to eliminate the chemicals that are usually used in adsorption and chemical precipitation processes for removal of heavy metals. The preselected running times were 1–9 hr together with a filtration rate of 10, 20 and 30 ml/min, respectively.

The pH of the solution was adjusted to the average value of the of secondary effluent which is recommended to be of 7.6 (Al-Suhaili et al., 2014). Also, this selection is in agreement with (Najee, 2008) who explained that strongly acidic

Table 3. Range and levels of independed variables

Factor	Name	Units	Low level	High level
A	Run-time	hr.	1	9
B	Cadmium concentration	mg/l	5	20
C	Flow rate	l/hr.	10	30

pH may be caused a poor adsorption as a result of ionization the surface of adsorption sites with a net positive charge (H^+) produced competition between protons and metal ions for sorption sites on the adsorbent surface. Also, at strongly alkaline, a precipitation of metal in solution may occur before the sorption experiments are begun. So, the best adsorption can occur at natural pH, low acidic and alkaline values.

In the present study, thermostone, and aluminum were acted as an adsorbent matter that can be attributed to physical and chemical adsorption, and this conclusion is in well agreement with the previous studies of (Al-Suhaili et al., 2014) who they recommended the possibility of using a reactive disposal thermostone as low cost alternative adsorbent for heavy metals reduction. Because that thermostone contained some pieces of irregular shape particles and porous network structure consists a lot of small pores generated offers, by its nature, a well evolved surface area of reaction contact, thus these properties lead to facilitate the diffusion of the Cd(II) ions to the surface of the adsorbent. The result shows that the filter of thermestone has a higher removal efficiency of cadmium (Cd) than aluminum and sand filters. While aluminum filters has a higher removal efficiency of Cd than the sand filter.

Optimization representation

The results in the form of cadmium removal from wastewater were appraised based on the CCD were illustrated in Table 4. A regression model for the response of corrosion rate was produced using RSM's historical data design, and

Table 4. Experimental results according to CCD

Time (hr.)	Cadmium conc.	Flow rate	A-Filter %	T-Filter %	S-Filter %	S-T-A-Filter %
1	5	10	85	87	80	83
3	5	10	88	91	86.35	87
5	5	10	86.5	93	86	86
7	5	10	94	95	86.8	90
9	5	10	94.3	94.8	86.8	92
1	5	20	80	85	81	79
3	5	20	85	90	82	84
5	5	20	90	94.2	85	86
7	5	20	90.2	94	85	90
9	5	20	91.8	94.5	88	92
1	5	30	79	80	76	77
3	5	30	84	87	82	83
5	5	30	88	90	85	86
7	5	30	89.7	93	88	88
9	5	30	90	93.6	88	89
1	10	10	76	77	74	75
3	10	10	80	79	78	78
5	10	10	84	83	80	80
7	10	10	85.6	90	82.9	83
9	10	10	87.4	91	81.8	85
1	15	10	73	74	73	70
3	15	10	79	80	77	78
5	15	10	82	82	79	80
7	15	10	83	84	80	80
9	15	10	83	85	80	80
1	20	10	72	73	70	70
3	20	10	73	75	72	68
5	20	10	78	79	77	78
7	20	10	80	82	79	79
9	20	10	81	82	80	79

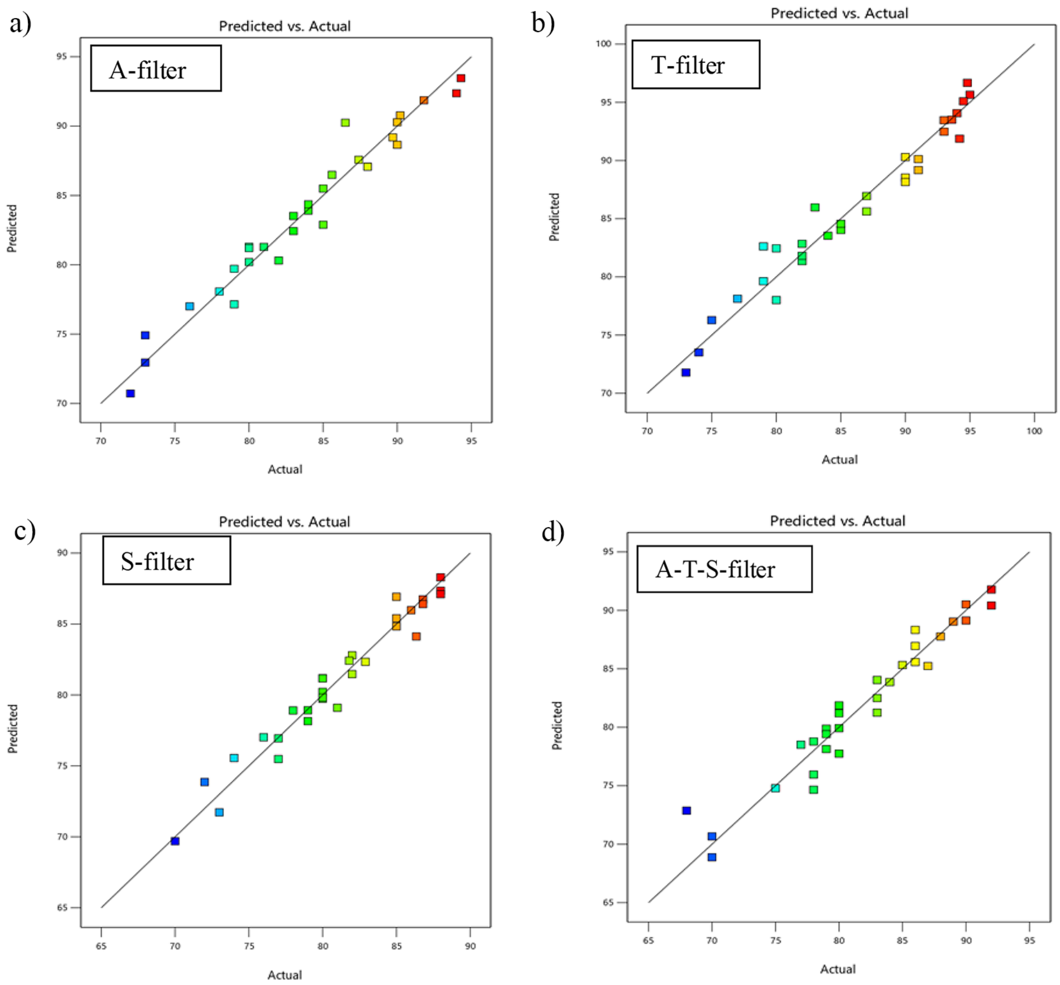

Figure 5. Relation between actual and predicted results of cadmium removal for A-filter, T-filter, S-filter and A-T-S-filter

it recommended fitting the response data with a third-order polynomial model (Salam et al., 2015). Figure 5 showed the actual and expected of cadmium removal efficiency for A-filter, T-filter, S-filter and A-T-S-filter this result agree with (Emami, 2021).

Contour plots and response surface for low cost material (filter media) of cadmium removal

Two-dimensional (2D) contour plot and three-dimensional (3D) response surface of the model-predicted response for filter media to removal of cadmium from wastewater.

Effect of flow rate

It was observed that the ratio of the cadmium removal decreased with the increasing of the filtration rate. From the results of Figure 6, flow rate was 10, 20 and 30 l/hr, while cadmium concentration was 5 ppm, pH was 7.6, it is obvious that the maximum removal efficiencies of S-filter, T-filter, A-filter and S-T-A-filter were 86.8%, 95%, 94%, 90%, respectively at the filtration rate 10 ml/min and thus can be attributed to at high flow rate that caused the reduction in the contact time between the particles of media and the cadmium solution as well as a higher velocity of flow results in less attachment by adhesion between the particles of filter media and the cadmium solution and leads increasing a sloughing precipitate from the media this result agree with (Aziz and Smith, 1996; Mahvi et al., 2004).

Effect of influent concentration

The effect of different influent concentrations (5, 10, 20 and 30) mg/l was also proved in the present study. From Figures 7, it is noticed that the ratio of cadmium removal decreased with the increase in initial concentration. The maximum removal efficiency of cadmium was reduced from

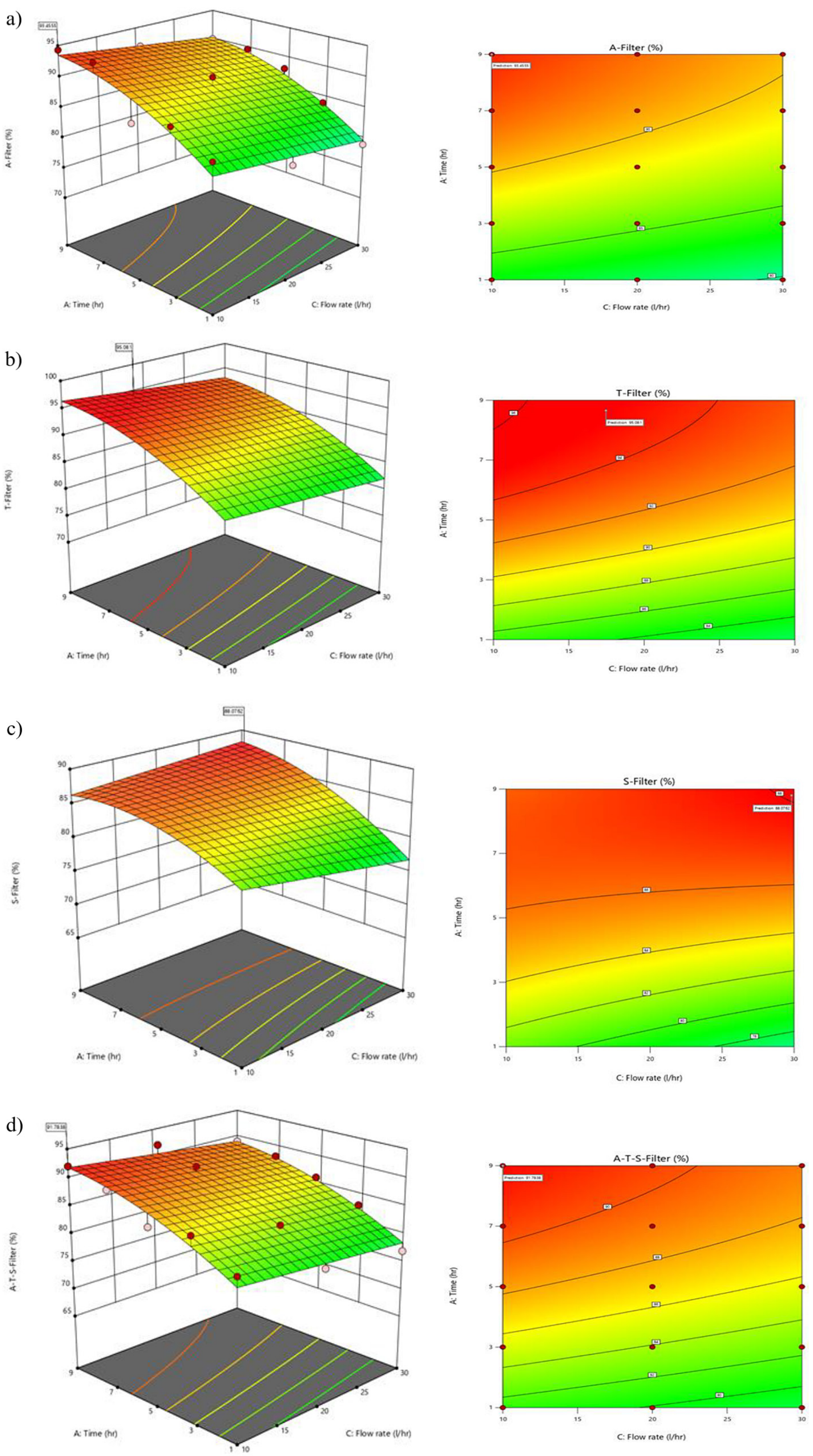

Figure 6. 3D and 2D of Flow rate with removal effcicency of a) A-Filter b) T-Filter c) S-Filter d) A-S-T Filter

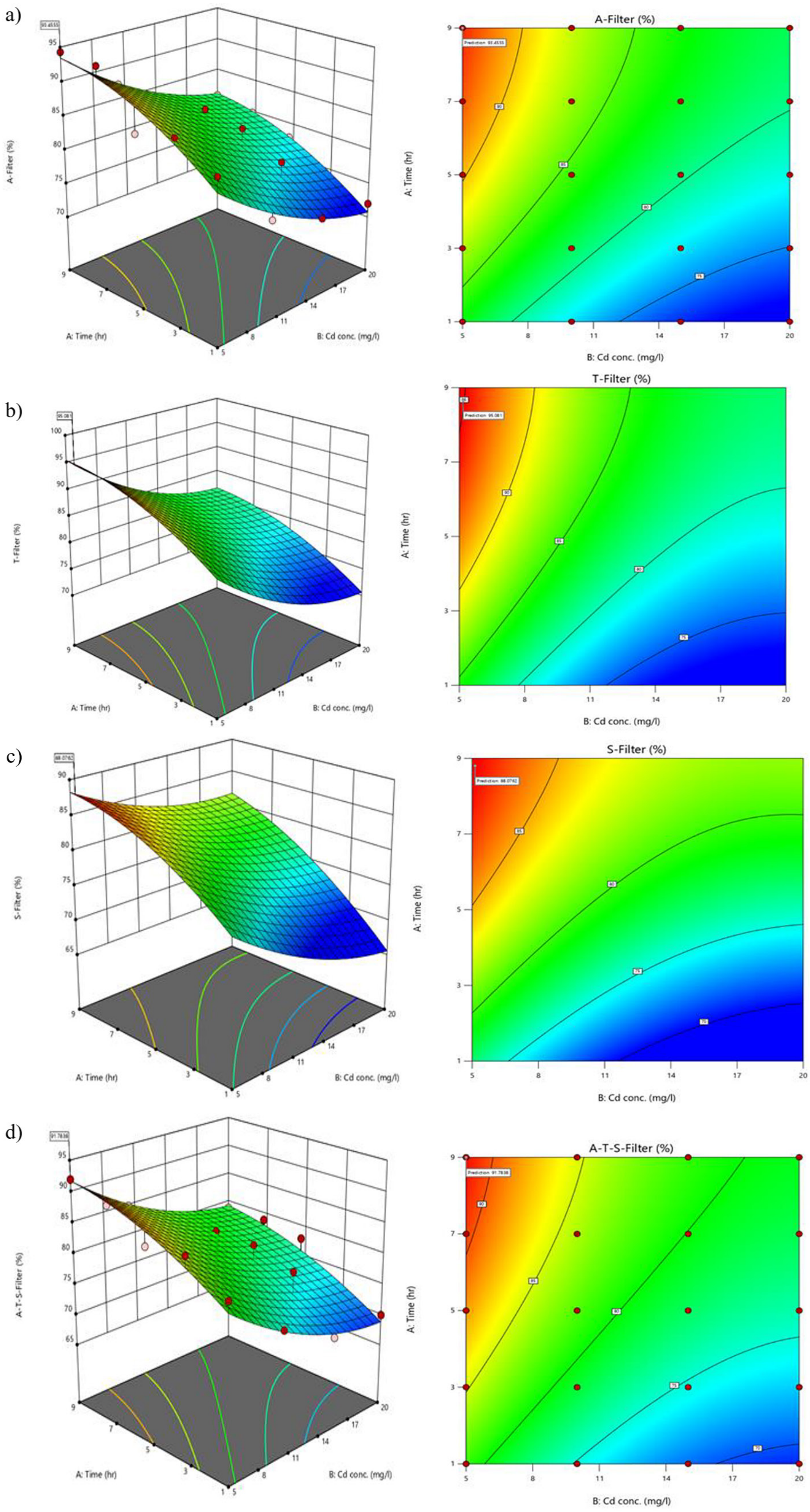

Figure 7. 3D and 2D of Cdmium concentration with removal effcicency of a) A-Filter b) T-Filter c) S-Filter d) A-S-T Filter

86.8–79%, 95–82%, 94–80%, 90–79% for S-filter, T-filter, A-filter and S-T-A-filter, respectively, these results are obtained with a influent concentrationes was increased from 30-5 mg/l.This result agree with (Lee et al 2006).

ANOVA (variances analysis)

The ANOVA analysis's is demonstrated in Table 5. It is showed that the Predicted R^2 of (A-filter = 0.9340, T-filter = 0.9319, S-filter = 0.9208 and A-T-S-filter = 0.9003) is in reasonable agreement

Table 5. ANOVA results for the response quadratic models

Filter media	Std. Dev.	Mean	CV %	R^2	R^2Adj.	R^2Pre	Adeq precision
A-filter	1.36	83.75	1.62	0.9583	0.9496	0.9340	37.4385
T-filter	1.60	85.94	1.86	0.9552	0.9458	0.9319	34.8442
S-filter	1.19	80.99	1.46	0.9568	0.9431	0.9208	30.3768
A-T-S-filter	1.75	81.83	2.13	0.9364	0.9231	0.9003	29.3296

Table 6. ANOVA for reduced quadratic model for A-filter

Parameter	Sum of squares	df	Mean square	F-value	p-value	Characteristics
Model	1017.11	5	203.42	110.35	< 0.0001	Significant
A-Time	418.70	1	418.70	227.14	< 0.0001	
B-Cd conc.	444.74	1	444.74	241.26	< 0.0001	
C-Flow rate	25.89	1	25.89	14.05	0.0010	
A^2	22.53	1	22.53	12.22	0.0019	
B^2	17.39	1	17.39	9.43	0.0052	
Residual	44.24	24	1.84			
Cor Total	1061.36	29				

Table 7. ANOVA for reduced quadratic model for T-filter

Parameter	Sum of squares	df	Mean square	F-value	p-value	Characteristics
Model	1304.35	5	260.87	102.27	< 0.0001	Significant
A-Time	458.16	1	458.16	179.61	< 0.0001	
B-Cd conc.	575.13	1	575.13	225.47	< 0.0001	
C-Flow rate	25.80	1	25.80	10.11	0.0040	
A^2	28.12	1	28.12	11.02	0.0029	
B^2	43.46	1	43.46	17.04	0.0004	
Residual	61.22	24	2.55			
Cor Total	1365.57	29				

Table 8. ANOVA for reduced quadratic model for S-filter

Parameter	Sum of squares	df	Mean square	F-value	p-value	Characteristics
Model	685.23	7	97.89	69.66	< 0.0001	Significant
A-Time	180.20	1	180.20	128.24	< 0.0001	
B-Cd conc.	245.65	1	245.65	174.82	< 0.0001	
C-Flow rate	3.36	1	3.36	2.39	0.1364	
AB	9.03	1	9.03	6.43	0.0189	
AC	13.58	1	13.58	9.66	0.0051	
A^2	25.03	1	25.03	17.81	0.0004	
B^2	16.84	1	16.84	11.99	0.0022	
Residual	30.91	22	1.41			
Cor Total	716.15	29				

Table 9. ANOVA for reduced quadratic model for A-T-S-filter

Parameter	Sum of squares	df	Mean square	F-value	p-value	Characteristics
Source	Sum of squares	df	Mean square	F-value	p-value	
Model	1077.00	5	215.40	70.66	< 0.0001	Significant
A-Time	416.07	1	416.07	136.48	< 0.0001	
B-Cd conc.	459.68	1	459.68	150.79	< 0.0001	
C-Flow rate	19.34	1	19.34	6.34	0.0188	
A^2	17.19	1	17.19	5.64	0.0259	
B^2	28.53	1	28.53	9.36	0.0054	
Residual	73.16	24	3.05			
Cor Total	1150.17	29				

with the Adjusted R^2 of (A-filter = 0. 9496, T-filter = 0.9458, S-filter = 0.9431 and A-T-S-filter = 0. 9231); i.e. the difference is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 37.438 of A-filter, 34.844 of T-filter, 30.377 of S-filter and 29.330 of A-T-S-filter indicates an adequate signal. This model can be used to navigate the design space. The coefficients of correlation were discovered to be relatively close. Table 6-9 demonstrated the quadratic model statistical significance. The relevance of the model term was assessed using the P-value and F-value utilized for the answer. In this investigation, a higher F-value and a lower probability number denoted a stronger relevance of the related model. Given that the probability value (0.0001) was found to be quite low, the term's significance for the model was demonstrated.

RSM optimization

The main goals of RSM is the ability to establish the optimal parameters for pollutant removal (Yang et al., 2020) which offers a practical solution to the diffusion limitations associated with traditional agglomerated zeolites. Moreover, the self-supported sorbents can be easily recycled after treatment. The derived γ-FeOOH based monolith exhibits higher BET surface area while slightly decreased thermal stability compared with those unmodified sample (labeled as HZ. In the optimization of time (A), Cd(II) concentration (B), flow rate (C) were selected as the responses including (% Cd removal) for A-Filter, T-Filter, S-Filter and A-S-T-Filter were maximized. The time (9 hr), the Cd(II) concentration (5 ppm), flow rate (10 l/hr) are optimum condition including the Cd (II) removal of A-Filter, T-Filter, S-Filter and A-S-T-Filter were 94%, 95%, 86.8% and 90%, respectively.

CONCLUSIONS

This paper Investigate the ability of filings low cost material representing thermestone and Aluminum solid wastes as a mano and multi filter media to remove Cd(II) ions from wastewater. RSM was utilized to optimize predict parameters that done by change three different parameters which were the time, filtration rate and influent cadmium concentration. These optimized parameters were 9 hr., 5 ppm, 10 l/hr with removal efficiency of Cd(II) for A-Filter, T-Filter, S-Filter, and A-T-S-Filter was 94%, 95%, 86.8% and 90%, respectively. The result shows that the filter of thermestone has a higher removal efficiency of cadmium (Cu) than aluminum and sand filters. While aluminum filters has a higher removal efficiency of Cu than the sand filter. As a general result, the offered process utilizing low cost material as filter media has a good potential to eliminate heavy metal from wastewater samples effectively.

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