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Original

Optimization for benzeneacetic acid removal from aqueous solution using CaO_2 nanoparticles based on Taguchi method

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Abstract

Nanoparticles of CaO_2 were synthesized by chemical precipitation method and characterized by X-ray powder diffraction (XRD), scanning electron microscope (HR-FESEM), energy-dispersive X-ray spectroscopy (EDX), transmittance electron microscopy (TEM) and high-resolution TEM (HRTEM). HR-FESEM, TEM, and HRTEM images confirmed the average size of nanoparticles as 10–40 nm. Furthermore, application of synthesized CaO_2 nanoparticles for the removal of benzeneacetic acid was studied by the Taguchi method. The operating parameters are CaO_2 nanoparticles dosage (0.008–0.03 g), initial concentration of benzeneacetic acid (6.8–13.47 g/L), and contact time (5–60 min). The result indicates that the CaO_2 nanoparticles adsorbent dosage was the most effective factors as compared to initial concentration of benzeneacetic acid and contact time. The optimum parameters were CaO_2 nanoparticles adsorbent dosage = 0.03 g, initial concentration of benzeneacetic acid = 6.8 g/L, contact time = 30 min, and the removal efficiency of benzeneacetic acid = 94.49%. ANOVA showed the most significant factors were adsorbent dosage with 93.78% contribution. Regression analysis ($R^2 = 0.91$) showed a good agreement between the experimental and the predicted values.

Keywords: Taguchi method; Calcium peroxide; Nanoparticles; Adsorption; Benzeneacetic acid

1. Introduction

Carboxylic acids such as benzeneacetic acid (phenylacetic acid, α-toluic acid, alpha tolylic acid, 2-phenylacetic acid, β-phenylacetic acid) are a found in the active auxin (Wightman & Lighty, 1982). Benzeneacetic acid is a by-product of the enzymatic hydrolysis of penicillin G by penicillin acylase (PA) (Ramchandran, Krishnamurthy, & Subbaraman, 1996). Benzeneacetic acid is also used to treat type II hyperammonemia to help diminish the amount of ammonia in a patient's blood stream by forming phenylacetyl-CoA, which then reacts with nitrogen-rich glutamine to form phenylacetyl glutamine (Hammad et al., 2003; Xie, Pei, Pei, & Cai, 2014). Benzeneacetic acid is produced by catabolic activities of microorganisms from different synthetic and natural aromatic compounds, such as aromatic amino acid and lignin (Mohamed, Ismail, Heider, & Fuchs, 2002). Benzeneacetic acid can be produced by the fermentation of soya beans using BacillusLicheniformis (Kim, Yang, & Song, 1999; Yong, Choi, Hur, & Hong, 2001). Benzeneacetic acid can be employed as a therapeutic agent for treatment of cancer (Athankar, Wasewar, Varma, Shende, & Uslu, 2015). Benzeneacetic acid has lots of versatile biological, medicinal activities, and industrial application. Therefore, it is necessary to remove benzeneacetic acid acids as well as separation of acid in aqueous solution. Because of low cost, ease, and high efficiency, adsorption method has been intensively investigated to remove benzeneacetic acid from aqueous solution.

Recently, nanoparticles have been believed as an alternative adsorbent because high surface area, cost effective, and easy preparation. Calcium peroxide (CaO_2) nanoparticles have been used as a novel adsorbent to achieve a high adsorption capacity and greater active sites(Madan, Upwanshi, & Wasewar, 2016b). To our best knowledge, however, literature on optimization of benzeneacetic acid adsorption using CaO_2 nanoparticles as an adsorbent by Taguchi is not available.

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Table 1
Summary of Taguchi applications in adsorption processes.

No.	Adsorption process	Adsorbent	Taguchi application	Adsorption capacity/Percent removal	References
1	Batch process	Spent Agaricus bisporus	To optimize the process condition for removal of Lead (II) from aqueous solution.	$\%E = 80.50\%$, $q_{tot} = 60.76$ mmol/g	Huang, Cheng, Chen, Zhu, and Xu (2009)
2	Continuous process	Al- and Ca-impregnated granular clay materials	To optimize the experimental condition for adsorption of phosphorus onto Al/Ca-impregnated granular clay material.	$q_e = 9.12$ mg/g	Yu, Chen, Hu, and Feng (2015)
3	Batch process	Nano-MgO/γ-Al_2O_3	To optimize the parameters for fluoride adsorption using sonochemically synthesized nano composite from aqueous solutions.	$\%E = 97\%$.	Nazari and Halladj (2015)
4	Batch process	Terminalia chebula-activated carbon	To optimize the parameters for removal of phenol.	$q_e = 294.86$ mg/g	Khare and Kumar (2012)
5	Batch process	Bagasse Fly Ash	To optimize the parameters for the removal of Cd, Ni, and Zn metal ions from aqueous solutions.	$q_{tot} = 6.76$ mg/g	Srivastava, Mall, and Mishra (2007)
6	Batch process	Cerium loaded cellulose nanocomposite bead	To optimize the operational variables for fluoride adsorption.	$\%E = 88.66\%$	Santra, Joarder, and Sarkar (2014)
7	Batch process	Oyster shell powders	To optimize the Cd(II) removal from aqueous solutions using oyster shell powders	$\%E = 99.7\%$.	Yen and Lin (2016)

In 1940s, DrGenichi Taguchi developed Taguchi methodology, Taguchi method is statistical tool used to find out the optimum conditions with less number of experiments (Nian, Yang, & Tarng, 1999). Taguchi is a systematic application of design and analysis of experiments and efficient method of optimization which reduces the cost of experiments. Taguchi method is based on number of steps such as identify the quality of characteristics, design parameters selection, number of factor levels determine, choice of suitable orthogonal array, outcome evaluate using signal-to-noise (S/N) ratios, ANOVA, optimum levels of factors selection, optimum process parameters verify during the confirmation experiment, and calculate the confidence interval (Engin, Özdemir, Turan, & Turan, 2008; Sadrzadeh & Mohammadi, 2008).

In this study, CaO_2 nanoparticles were synthesized and the physical, chemical, and structural properties of this nanoparticle were characterized by XRD, HR-FESEM, EDX, TEM, and HR-TEM. The adsorption of benzeneacetic acid on the synthesized CaO_2 nanoparticle adsorbent was carried out by batch adsorption experiments. Optimization of parameters such as adsorbent dosage, initial benzeneacetic acid concentration, and contact time using Taguchi's experimental design methodology were carried out.

For work on various application of Taguchi in the adsorption process are shown in Table 1.

2. Materials and methods

2.1. Materials

The details of chemicals used in present work are given in Table 2. All the chemicals were used without any treatment and purification. The pH of the solution was adjusted with 0.1 M NaOH. All the aqueous solutions were prepared using double distilled water.

Table 2
Details of chemicals used.

Chemicals	Formula	Chemical%	Make
Calcium chloride	$CaCl_2$	99.5	Merck, India.
Hydrogen peroxide	H_2O_2	35	Merck, India.
Polyethylene glycol	PEG 200		Merck, India.
Ammonia	$NH_3.H_2O$	25	Merck, India.
Sodium hydroxide	NaOH		India.
Benzeneacetic acid	$C_8H_8O_2$	98.5	Acros Organics, New Jersey, USA

2.2. Instruments

A digital pH meter (Spectral Lab Instrumental Pvt. Ltd., India) was use to measure pH. X-ray powder diffraction (XRD) analysis was performed by X-ray diffractometer (PAN analytical X'pert PRO) Using Cu X-ray tube (λ = 1.5406 Å). Morphologies of samples were observed with a high resolution field emission scanning electron microscope (HR-FESEM) from Zeiss, model name ULTRA Plus. It comes with a GeminiÒ column that proposes a theoretical resolution of 1.0 nm at 15 kV. Energy dispersive X-ray analysis (EDX) spectrometer was carried out using X Flash 6130 Bruker. Transmission Electron Microscopy (TEM) analysis of particles was performed in PHILIPS-CM 200, operated at 20–200 kV. High Resolution transmission electron microscopy (HR-TEM) of particles was carried out using JEOL JEM-2100.

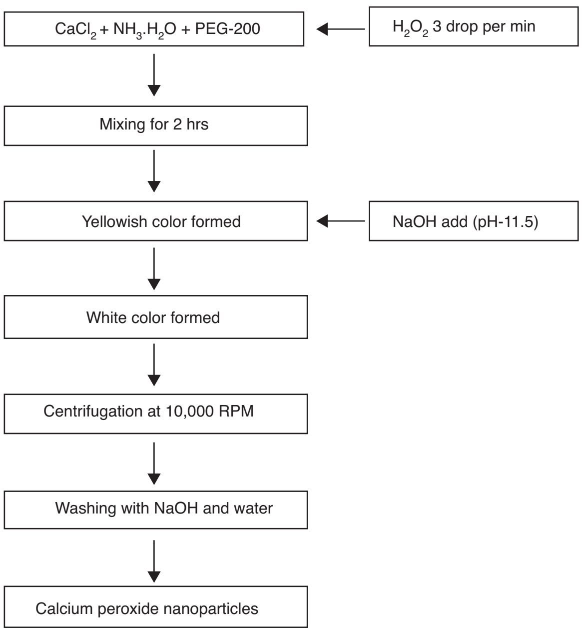

Fig. 1. Experimental procedure for the synthesis of CaO_2 nanoparticles.

Table 3
Taguchi controllable factors and their levels.

Factor	Level 1	Level 2	Level 3
(A) Dose (g)	0.008	0.01	0.03
(B) Initial concentration (g/L)	6.8	10.89	13.47
(C) Time (min)	5	30	60

2.3. Synthesis of calcium peroxide(CaO_2) nanoparticles

The CaO_2 nanoparticles were prepared by the existing method from Khodaveisi et al. with slight modification (Khodaveisi et al., 2011). Briefly, 9 g $CaCl_2$ was first dissolved in 90 mL water, followed by the addition of 45 mL NH_3. H_2O (1 M) and 360 mL PEG-200 solution. The mixture was allowed to stir at 400 rpm, and 45 mL H_2O_2 was immediately added to it at the rate of three drops per minute for about 2 h. The stirring was continued, and pH of the mixture was adjusted to 11.5. The change in color of suspension from yellowish to white indicated the formation of CaO_2 nanoparticles. The suspension was centrifuged at 10,000 rpm for 5 min,and CaO_2 nanoparticles were collected. The particles were initially washed thrice using NaOH solution, and then twice with distilled water, and dried at 80 °C for 120 min in vacuum oven. The dried CaO_2 nanoparticles were later used for adsorption experiments. A schematic outline of the synthesis of CaO_2 nanoparticles is shown in Figure 1.

2.4. Batch adsorption studies

Adsorption batch runs were performed for optimization process according to an orthogonal array L_9 (Table 3) and results obtained from each set as are (*%E*) given in Table 4. In each experimental run, 10 mL of aqueous benzeneacetic acid solution of known concentration and the known amount of nanoparticle of CaO_2 were taken in 100 mL Erlenmeyer flask. The flasks were agitated at a constant shaking rate at 22 ± 2 °C in a water bath controlled shaker (REMI, RSB-12, and India). At the end of shaking, the samples were centrifuged and the supernatant used for determination of the benzeneacetic acid concentration.

Removal of benzeneacetic acid is given by Eq. (1) as follows:

$$\%E = \frac{C_0 - C_e}{C_0} \times 100 \tag{1}$$

where C_0 is the initial concentration of benzeneacetic acid (g/L) and C_e is the equilibrium concentration of benzeneacetic acid (g/L).

2.5. Taguchi methodology

The Taguchi method is a simple and vigorous method involves the different experimental conditions through orthogonal arrays to reduce experimental errors and process variation, enhance the efficiency, optimizing the process parameters and reproducibility of experiments. So, the method reduces work time and cost in the processes(Asiltürk & Neseli, 2012). The important parameters affecting adsorption are initial concentration of the adsorbate (g/L), adsorbent dosage (W) and contact time (*t*) and each factor at three levels on the adsorption capacity and removal efficiency was studied. The used level setting values of the main factors (A–C). Taguchi's L_9 orthogonal array matrix was used which incorporates three parameters and three levels.

In Taguchi methodology, the quality characteristics are employed into three different options: "larger is the better", "nominal the-best", and "smaller-the-better. The objective of this study was to remove benzeneacetic acid by CaO_2 nanoparticles, the quality characteristic go for "larger is the better" of benzeneacetic acid removal defined by Eq. (2).

$$S/N_{LB} = -10 \log \frac{\sum_{i=1}^{n} 1/y_i^2}{n} \tag{2}$$

where the subscript LB denoted "larger is the better" and *n* was the number of repetitions under the same experimental conditions and y_i showed the measurement results. Each experiment was repeated twice (1st run and 2nd run) and the S/N ratio was determined using Minitab software (version 14). L and 9 mean Latin square and the number of experiments, and also, 3 and 3 indicate the numbers of factors and their levels, respectively.

3. Results and discussion

3.1. CaO_2 nanoparticles characterization

X-ray diffraction patterns of CaO_2 are shown in Figure 2. The diffraction peaks at $2\theta = 30.47°$, 35.75°, 47.46°, 53.28°, 60.82° and 87.1°can be respectively indexed to (0 0 2), (1 1 0), (1 1 2), (1 0 3), (2 0 2) and (3 1 0) reflections of CaO_2 nanoparticles and match the reference patterns of standard file of CaO_2 (Joint Committee for Powder Diffraction Studies (JCPDS) File No. 03-0865). The morphology of prepared CaO_2 nanoparticles was studied by the HR-FESEM analysis (Fig. 3a and b). It can be clearly indicated that nanoparticles was looked like the

Table 4
L_9 orthogonal array of the operational variables each at three different variables and calculated S/N ratio and removal efficiencies.

Exp. no	Level			Benzeneacetic acid removal (%)			S/N ratio (dB)
	A	B	C	R_1	R_2	Mean	
1	1	1	1	40.93	38.93	39.93	26.01
2	1	2	2	38.11	39.36	38.74	25.74
3	1	3	3	29.25	28.24	28.74	23.15
4	2	1	2	60.96	58.95	59.96	29.54
5	2	2	3	51.87	53.12	52.49	28.38
6	2	3	1	53.00	51.99	52.49	28.38
7	3	1	3	94.99	97.00	96.00	33.62
8	3	2	1	81.87	80.62	81.25	32.18
9	3	3	2	80.80	81.81	81.30	32.18

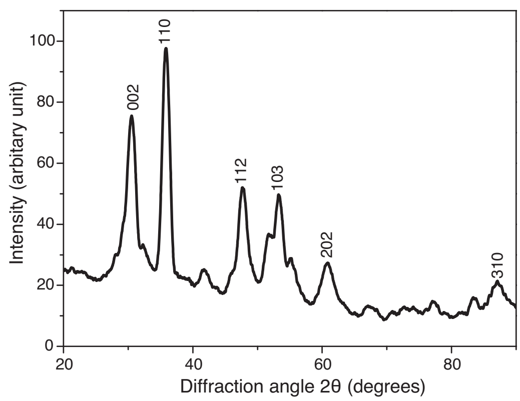

Fig. 2. XRD pattern of synthesized CaO_2 nanoparticles.

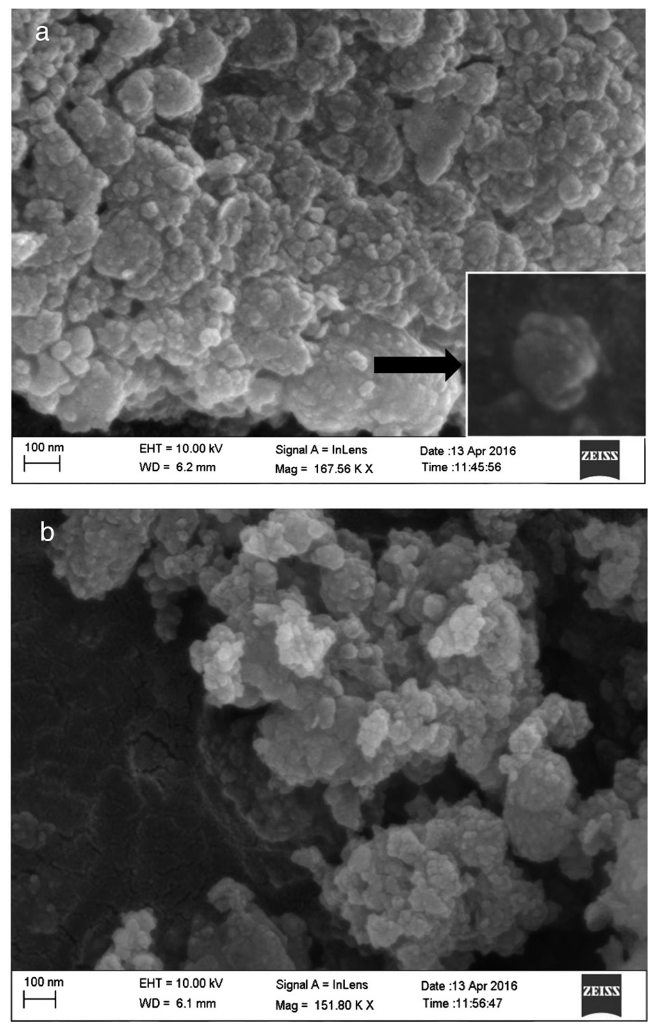

Fig. 3. (a, b) HR-FESEM images of CaO_2 nanoparticles.

aggregated round shape and are mostly spherical in shape. EDS showed the trace spectrum of CaO_2 nanoparticles as shown in Figure 4. The atomic compositions for calcium (Ca) and oxygen (O) were 42.88% and 57.12%, respectively. Figure 5a displays the TEM image of large number of CaO_2 nanoparticles with approximately uniform shape and size. It can be clearly seen is near spherical in shape with average particle size of about 10–40 nm. Figure 5b shows HR-TEM images of synthesized CaO_2 nanoparticles. The HR-TEM images clearly confirms, CaO_2 nanoparticles have a lattice structure with an interplanar spacing about 0.18 and 0.12 nm, which corresponds to (1 1 2) and (3 1 0) plane of CaO_2 respectively. These results are reliable with the XRD pattern.

3.2. OA and the analysis of S/N ratio (Taguchi method)

According to the Taguchi L_9 (3^3) OA, nine experiments were performed, and each experiment was repeated twice which were denoted by R_1 and R_2. The value of the response (removal efficiency) and S/N ratio are illustrated in Table 5. The mean of response and the mean of S/N ratio variable for each factor at a certain level can be determined. The boldfaces refer to the maximum value of the mean of response and the mean of the S/N ratios of a certain factor among four levels as shown in Table 5. The mean response plot for the removal of benzeneacetic acid using CaO_2 nanoparticles is shown in the Figure 6. The plot is used to show the relationship between the variables and output response. Adsorbent dosage 'A' and contact time 'C' increases with increases the removal efficiency of benzeneacetic acid. The effect of initial benzeneacetic acid concentration for removal is shown by factor 'B'. Removal efficiency increases with decreases in benzeneacetic acid concentration. The optimum level of operational variables is determined from the maximum value. The mean of removal efficiency level '3' is 86.18, level '1' is 65.30, and level '2' is 60.00 and its shows this

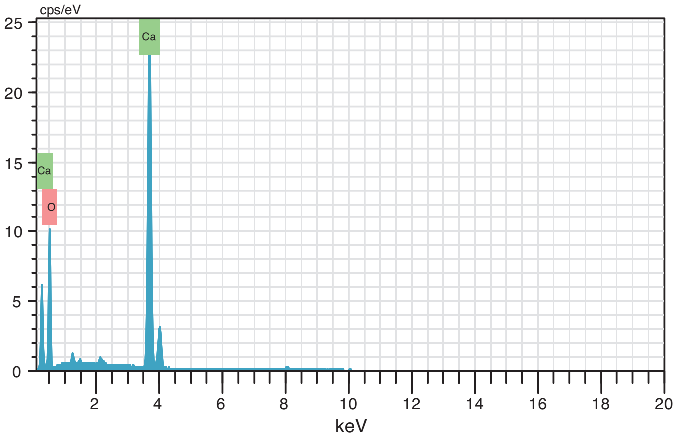

Fig. 4. EDX analysis of CaO_2 nanoparticles showed characteristics peaks.

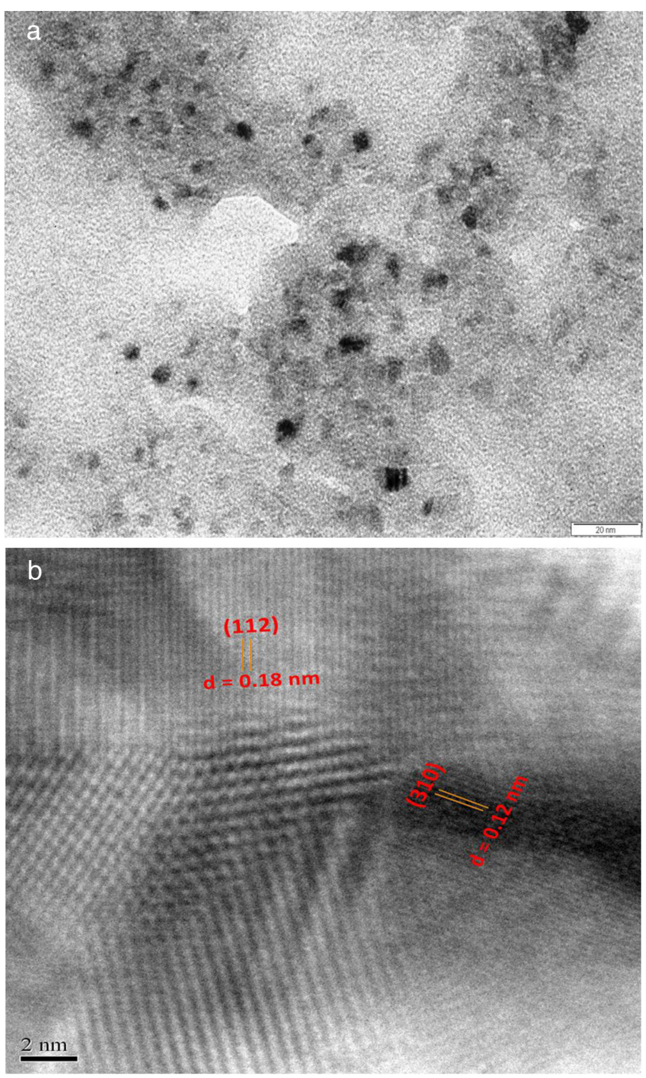

Fig. 5. (a) TEM and (b) HR-TEM images of CaO_2 nanoparticles.

level gives maximum efficiency. The S/N ratio plot as shown in Figure 7. According to these figures, increasing the adsorbent dose with low concentration of benzeneacetic acid concentration and increase the contact time the removal efficiency is increases

Table 5
Calculated mean of response and S/N ratio for data obtained from benzeneacetic acid removal experiments.

Level	Dose	Concentration	Time
Mean of response			
1	35.80	**65.30**	57.89
2	54.98	57.49	**60.00**
3	**86.18**	54.18	59.08
Delta	50.38	11.12	2.11
Rank	1	2	3
S/N ratio			
1	30.99	**35.74**[a]	34.87
2	34.79	34.79	**35.17**[a]
3	**38.68**[a]	33.92	34.41
Delta	7.69	1.82	0.77
Rank	1	2	3

Note: The bold values at each column of this table refer to maximum calculated mean of response (benzeneacetic acid removal efficiency).
Difference: maximum − minimum values for each column.
Rank: The order of importance of factors for the removal of benzeneacetic acid.
Note: The bold values at each column of this table refer to maximum calculated S/N ratio according to the "larger is better" criterion.
[a] Maximum mean S/N ratio indicative optimum level.

Table 6
ANOVA for mean response.

Operational variable	DOF	SS	MS	F-ratio (F)	*P* (%)
Dose	2	3879.54	1939.77	70.49	93.78
Concentration	2	195.55	97.77	3.55	4.72
Time	2	6.71	3.36	0.12	0.16
Error	2	55.04	27.52		1.33
Total	8	4136.83			100.0

and S/N ratio increases. The terms 'signal' to 'noise' ratio signifies the desirable and undesirable value for the output response, respectively. The *S/N* ratio of benzeneacetic acid removal was achieved. The process parameters were optimized based on S/N ratio. Lager the S/N ratio higher the benzeneacetic acid removal. The value of the S/N ratio, illustrate in Table 5. The mean of S/N ratios of level '3' is 38.68, level '1' is 35.74 and level '2' is 35.17 and its shows this level gives higher efficiency. The optimum parameters were adsorbent dose (A) of 0.03 g, initial benzeneacetic acid concentration (B) of 6.8 g/L, and time (C) of 30 min. The optimum combination was found to be A3-B1-C2 with corresponding mean of response value 86.18, 65.30, and 60.00and mean of S/N ratio value 38.68, 35.74, and 35.17 respectively.

3.3. Statistical analysis of variance (ANOVA)

Analysis of variance (ANOVA) was performed to investigate which process parameters significantly affect the process responses and to determine the percent contribution of each operational variable to the response (Engin et al., 2008; Sadrzadeh & Mohammadi, 2008). Table 6 shows the result of ANOVA test for mean response. ANOVA also used for estimate the error variance. The most significant factors that affect benzeneacetic acid removal was found to be adsorbent dose (93.78%) >initial

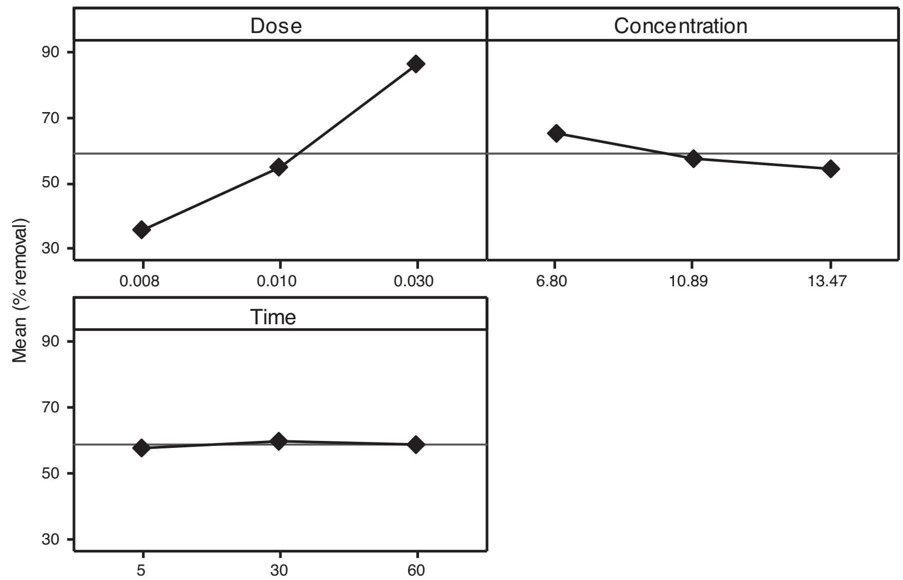

Fig. 6. Mean removal efficiency plotted against different factor levels.

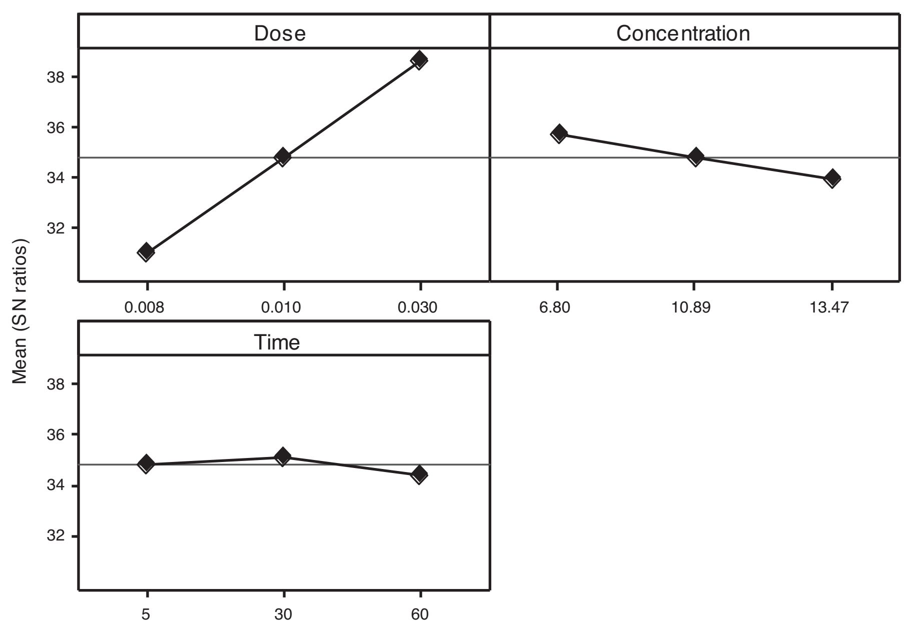

Fig. 7. Mean SN ratios plotted against different factor levels.

concentration (4.72%) >contact time (0.16%). Percent contribution (P-ratio) is defined as a relation between the parameters' sum of square to the total sum of square, which indicates the contributions of these parameters (Nik, Sadrzadeh, & Kaliaguine, 2012):

$$P_A = \frac{SS_A}{SS_T} \times 100 \quad (3)$$

3.4. Development of regression model

Regression analysis was developed to investigate the relationship between the variables (Deniz, 2013). The mathematical model for benzeneacetic acid removal through the statistical analysis is shown as

$$\text{Benzeneacetic acid removal efficiency (\%)} = 44 + 1994A - 1.69B + 0.020C \quad (4)$$

Regression equation provides predicted data from different experimental condition and it's compared with experimental data shown in Figure 8. Table 7 shows the regression analysis. Experimental and predicted values of the removal efficiency of benzeneacetic acid are almost equal. So as to the model tells the interaction of process parameters. ANOVA was derived to observe the null hypothesis for the regression shown in Table 8. A p value less than 0.05 was chosen which show the

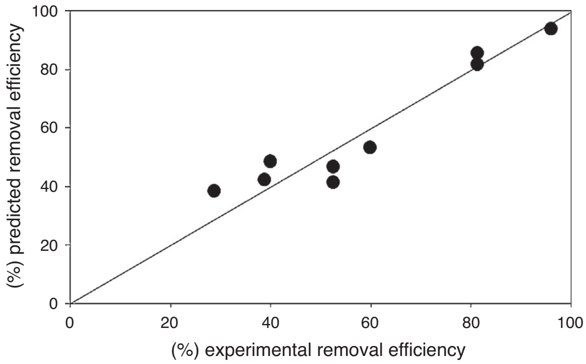

Fig. 8. Comparison of predicted and experimental adsorption data for removal of benzeneacetic acid.

Table 7
Regression analysis results.

Predictor	Coefficient	SE coefficient	T	P
Constant	44.00	13.46	3.27	0.022
Dose	1994.1	304.0	6.56	0.001
Concentration	−1.689	1.099	−1.54	0.185
Time	0.0198	0.1343	0.15	0.888

Table 8
ANOVA table for the regression analysis.

Source	df	SS	MS	F	P
Regression	3	3726.6	1242.2	15.14	0.006
Residual Error	5	410.2	82.0		
Total	8	4136.8			

estimated model is considerable. The predicted and the experimental response (benzeneacetic acid removal) show a very good agreement with each other, and the coefficient determination (R^2) of 0.91 implicates a good fitted of the model (Fig. 8).

3.5. Confirmation experiment

In Taguchi method, confirmation testis a necessary for the optimization study (Santra et al., 2014). The confirmation test is performed with optimum levels and factors. From optimum conditions, to validate the predicted optimal conditions, new experiments were carried out and 94.49% benzeneacetic acid removal efficiency was achieved. Table 9 shows the predicted percent removal and the experimental percent removal (response) at the optimal conditions.

Table 9
Results of the confirmation experiments.

	Optimal condition	
	Prediction	Experiment
Level	A3B1C2	A3B1C2
%(Removal efficiency)	92.92	94.49

3.6. Adsorption Isotherm

Adsorption isotherm models such as Langmuir isotherm (Langmuir, 1918), Freundlich isotherm (Freundlich, 1906), and Temkin isotherm (Tempkin & Pyzhev, 1940) were tested to explain the equilibrium adsorption of benzeneacetic acid from aqueous solution (Table 10).

The initial benzeneacetic acid concentration (C_0) was varied from 4.8 to 13.47 g/L, at a constant contact time of 60 min, with 1 g/L CaO_2 nanoparticles at 25 °C. The q_e values were calculated using

$$q_e = \frac{C_0 - C_e}{W} \times V. \quad (5)$$

where q_e is the equilibrium adsorption capacity of benzeneacetic acid on the adsorbent (mg g^{-1}), C_0 is the initial benzeneacetic acid concentration (g/L), C_e is the equilibrium concentration of benzeneacetic acid in solution (g/L), V is the volume of the solution (L), W is the mass of CaO_2 nanoparticles (in g).

Langmuir isotherm assumes that monolayer adsorption takes place over the homogeneous CaO_2nanoparticles adsorbent surface. The Freundlich isotherm considers the heterogeneous surface of an adsorbent and is used to describe the adsorption data. Temkin isotherm based on heat of adsorption of all the molecules in the layer would decrease linearly with coverage.

The best fit of data was evaluated from the correlation coefficient (R^2), sum of the squares of errors (SSE), and Marquardt's percent standard deviation (MPSD) values. The value of R^2 is higher and the value of SSE and MPSD shows the lower for the most favored situation. From the Table 10, it clearly shows that the Langmuir isotherm has high R^2 value and low SSE

Table 10
Adsorption Isotherms.

Models	Linear form	Parameters		R^2	SSE	MPSD
Langmuir	$C_e/q_e = 1/K_L q_m + C_e/q_m$ q_m – adsorption capacity of benzeneacetic acid (g g^{-1}) K_L – Langmuir adsorption constant (L g^{-1})	K_L (L g^{-1}) 4.624×0^{-6}	q_m (g g^{-1}) 4.504	0.99	1961.63	0.015
Freundlich	$\ln q_e = \ln K_F + (1/n) \ln C_e$ K_F – Freundlich isotherm constants n – adsorption intensity	K_F 721.981	n 4.901	0.71	5098.25	0.026
Temkin	$q_e = B_1 \ln K_T + B_1 \ln C_e$ K_F – Tempkin isotherm constants	K_T (L g^{-1}) 0.413	B_1 857.8	0.71	7509.65	0.032

and MPSD as compared to Freundlich and Temkin isotherm. Thus, this study confirms that adsorption of benzeneacetic acid takes place on the CaO_2 nanoparticles, and follows the Langmuir model(Madan, Ravikumar, & Wasewar, 2016a).

4. Conclusion

In present study, nanoparticles of CaO_2 were synthesized by chemical precipitation method. This was confirmed by a characterization XRD, HR-FESEM, EDX, TEM and HRTEM. Taguchi method has been applied to optimize the process parameters for adsorption of benzeneacetic acid on CaO_2 nanoparticles. A set of orthogonal array L_9 (3^3) follows the "large is better" category and it shows only 9 experiments are sufficient for design of experiments. ANOVA indicated that the most significant factors were adsorbent dosage with 93.78% contribution. The removal efficiency of benzeneacetic acid ($\%E = 94.49$) was obtained by using adsorbent dosage = 0.03 g, initial concentration of benzeneacetic acid = 6.8 g/L, and contact time = 30 min. The experimental optimum conditions confirmed by the confirmation test which shows the value of $\%E$ is in permissible limit.

Conflict of interest

The authors have no conflicts of interest to declare.

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