

Study on the Effect of Preparation Parameters of K_2CO_3/Al_2O_3 Sorbent on CO_2 Capture Capacity at Flue Gas Operating Conditions

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ABSTRACT

In this paper, study on the effect of preparation conditions of K_2CO_3/Al_2O_3 sorbent was done. Box-Behnken design was applied to study the influence of four parameters involve initial solution concentration, impregnation time and calcination step temperature and time. A quadratic model was used to correlate the sorbent capture capacity. The model was used to calculate the optimum conditions for preparing sorbent. From the analysis of variance (ANOVA), the most influential factor on each experimental design response was identified. The potassium-based sorbents used in this study were prepared by impregnating K_2CO_3 on Al_2O_3 support. The CO_2 capture capacity was measured in the presence of H_2O in a fixed-bed reactor at CO_2 capture temperature of $60^\circ C$ using breakthrough curves. The optimum sorbent prepared by this method showed CO_2 capture capacity of $77.21 \text{ mg } CO_2/g$ sorbent. It was observed that the experimental values obtained were in good agreement with the values predicted by the model, with relatively small errors between the predicted and the actual values. The results obtained in this study can be used as basic data for study on design and operating condition optimization of CO_2 capture process using these sorbents.

Keywords: CO_2 Capture; K_2CO_3/Al_2O_3 Sorbent; Response Surface Method; Box-Behnken Design

1. Introduction

Global warming increasingly thought to be associated with the atmospheric emission of greenhouse gases. Carbon Dioxide is a major greenhouse gas that is released into the atmosphere due to the use of fossil fuels. It can be removed from flue gas by various methods such as membrane separation, amine scrubbing, and using molecular sieves [1-3]. These methods however are costly and energy intensive.

Solid sorbent processes for CO_2 capture are also under study. The use of solid sorbents can be a highly cost-effective and energy efficient way to remove CO_2 [4-7]. Sorbents containing alkali and alkaline-earth metals like potassium carbonate and calcium oxide are investigated for this commercial feasibility [8].

Alkaline-earth method-based sorbents such as MgO and CaO are applicable at much higher adsorption and regeneration temperatures [7]. Alkali metal-based sorbents were employed in CO_2 adsorption at low temperatures ($50^\circ C - 70^\circ C$) with thermal regeneration easily oc-

curing at low temperatures ($<200^\circ C$). CO_2 capture using a dry sodium-based sorbent was also reported [9,10]. However, when CO_2 reacted with Na_2CO_3 , the global carbonation reaction rate was rather slow [11]. The CO_2 adsorption and regeneration of potassium-based sorbents with several supports such as activated carbon [4,5,12], TiO_2 , SiO_2 , MgO , ZrO_2 , CaO and Al_2O_3 were studied [6,9,13, 14]. Al_2O_3 is one of the most proper materials used as sorbent support.

In most of the previous works, the sorbent preparation conditions were fixed and the effect of the different conditions on the capture capacity which is an important specification of the sorbent for commercial applications, has not been studied yet. The objective of this work was to study the influence of preparation condition on sorbent capture capacity and finding optimum condition for preparation of sorbent at larger scales. Therefore, the scope of this research was to carry out a statistical optimization to determine the optimum preparation conditions of K_2CO_3/Al_2O_3 sorbent, in order to achieve high CO_2 capture capacity using the response surface methodology approach

(RSM). Response surface methodology (RSM) is an empirical statistical technique employed for multiple regression analysis by using quantitative data. It solves multivariate data which is obtained from properly designed experiments to solve multivariate equation simultaneously [15]. This method was applied by many of researcher for study on various multi-variable phenomena [16,17].

2. Experimental

2.1. Preparation of Sorbent

The potassium-based sorbents used in this study were prepared by impregnating K_2CO_3 on Al_2O_3 (Aluminum Oxide, Merck) as support. Twenty (20.0) g of Al_2O_3 was added to an aqueous solution of anhydrous potassium carbonate (K_2CO_3 , Merck) with particular concentration in deionized water (Initial solution concentration). Then, it was mixed with a magnetic stirrer at room temperature (Impregnation time). After that, the mixture was dried in a rotary vacuum evaporator at $60^\circ C$. The dried samples were calcined in a furnace under a N_2 flow (100 cc/min) for a particular time at selected temperature (Calcination time and temperature).

2.2. Apparatus and Procedures

A fixed-bed Stainless-Steel reactor (diameter of 15 mm), which was placed in an electric furnace under atmospheric pressure was used for adsorption process. Two (2.0) g of the sorbent was packed into the reactor. In order to prevent condensation of water vapor injected into the reactor and GC column the temperatures of the inlet and outlet lines of the reactor were maintained above $100^\circ C$. The column used in this analysis was a 1/8 in. stainless tube packed with Porapak Q. When the CO_2 concentration of the outlet gas reached the same level as that of the inlet gas in the CO_2 adsorption process, process was stopped. The outlet gas from the reactor was automatically analyzed every 5 min by a thermal conductivity detector (TCD), which was equipped with an auto sampler (Valco online valve). Schematic diagram of the fixed-bed reactor is shown in **Figure 1**.

The feed stream comprises of Nitrogen, carbon dioxide and water. The liquid water flow rate was controlled using a piston pump and the water was vaporized before entering the column. Both CO_2 and N_2 flow rates were controlled by independent mass flow controllers and

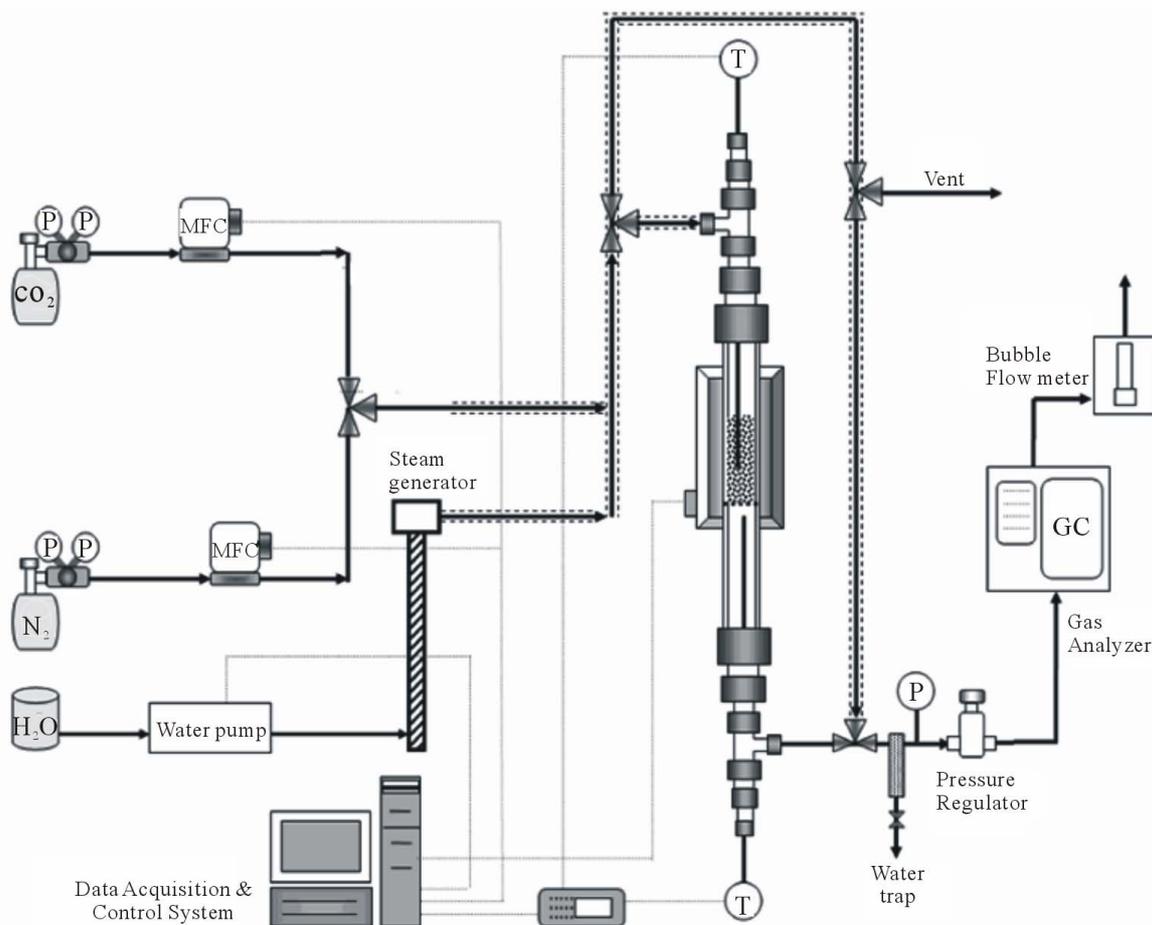


Figure 1. Schematic diagram of the fixed-bed reactor.

these gases were mixed with the vaporized water inside the oven where experiments were done. All the CO₂ sorption tests were performed following the same procedure.

The CO₂ capture capacity of the sorbent was evaluated by the breakthrough curve for CO₂ adsorption. In this study, the CO₂ capture capacity of sorbent was calculated from its breakthrough curve during CO₂ adsorption in the presence of 9.0 vol.% H₂O and 1 vol.% CO₂. The CO₂ capture capacity describes the amount of CO₂ absorbed until the output concentration of CO₂ reached 1 vol.%, which was the same value as that of the inlet.

2.3. Design of Experiments

The RSM has several classes of designs, with its own properties and characteristics. Central composite design (CCD), Box-Behnken design (BBD) and three-level factorial design are the most popular designs applied by the researchers. In this work, the Box-Behnken design was used to study the effects of the variables towards their responses and subsequently in the optimization studies. This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interaction between the parameters. In order to determine if there exist a relationship between the factors and the response variables investigated, the data collected must be analyzed in a statistically manner using regression. A regression design is normally employed to model a response as a mathematical function (either known or empirical) of a few continuous factors and good model parameter estimates are desired [18].

Replicates of the test at the center are very important as they provide an independent estimation of the experimental error. Each variable is investigated at two levels. Meanwhile, as the number of factors, n , increases, the number of runs for a complete replicate of the design increases rapidly. In this case, main effects and interactions may be estimated by fractional factorial designs running only a minimum number of experiments. Individual second-order effects cannot be estimated separately by $2n$ factorial designs. The responses and the corresponding parameters are modeled and optimized using ANOVA to estimate the statistical parameters by means of response surface methods.

Basically this optimization process involves three major steps, which are, performing the statistically designed experiments, estimating the coefficients in a mathematical model and predicting the response and checking the adequacy of the model.

It is assumed that the independent variables are continuous and controllable by experiments with negligible errors. It is required to find a suitable approximation for

the true functional relationship between independent variables and the response surface [19].

The experimental sequence was randomized in order to minimize the effects of the uncontrolled factors. Regression analysis was performed to determine the surface response as function of second order polynomial equation:

$$Q = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_{ii}^2 + \sum_{i \leq j \leq k}^{k-1} \beta_{ij} x_i x_j \quad (1)$$

where Q is the predicted response (sorbent capture capacity), β_i , β_{ii} , β_{ij} represent linear, quadratic and interaction effects. β_0 is the intercept term and x_i , x_j , ..., x_k are the input variables which affect the Q value [20].

In this work the influence of four sorbent preparation parameters involve initial solution concentration, impregnation time and calcination step temperature and time on sorbent CO₂ capture capacity has been studied. The range of these variables selected base on previous works of other researchers [6,7,13,14] (Table 1).

3. Results and Discussion

3.1. Development of Regression Model Equation

The design of experiment is given in Table 2, together with the experimental results. Eight replicate runs were observed at the centre of the design to allow the estimation pure error.

Using multiple regression analysis, the second order polynomial model (Equation (1)), which characterizes the relationship between sorbent capture capacity and studied variables, was obtained. The coefficients of polynomial are shown in Table 3.

Response surface plots were described by the regression model for BBD which was developed using MINITAB 14 software. The student's t-test was performed to determine the significance of the regression coefficients. The results of statistical analysis including the regression coefficient, t and p values for linear, quadratic and combined effects of the variables are given in Table 3. The statistical significance of the model was also determined by F-test for analysis of variance (ANOVA) and re-

Table 1. Experimental range and levels of independent variables for preparation of sorbents.

Independent variables	Symbols	Unit	Code levels		
			-1	0	1
Initial solution concentration	ISC	wt%	10	25	40
Impregnation time	ITI	hr	1	12	23
Calcination time	CTI	hr	3	4	5
Calcination temperature	CTE	°C	300	400	500

Table 2. Box-Behnken design matrix, the experimental and predicted values of sorption capture capacity.

Run	Sorbent preparation variables (Code levels)				Sorbent capture capacity (mg/g)	
	ISC	ITI	CTI	CTE	Experimental	Predicted
1	1	0	-1	0	71.61	75.49
2	0	-1	0	1	69.63	64.98
3	0	0	0	0	74.25	74.19
4	-1	0	0	-1	52.89	55.07
5	0	1	0	1	72.19	68.32
6	1	0	1	0	79.09	76.66
7	1	0	0	-1	74.14	77.37
8	1	-1	0	0	73.19	76.84
9	-1	0	1	0	55.31	54.72
10	0	0	1	1	64.89	65.81
11	-1	1	0	0	54.97	56.99
12	1	0	0	1	71.34	71.67
13	0	1	-1	0	69.87	71.49
14	-1	0	-1	0	49.67	52.82
15	0	0	0	0	71.26	74.19
16	1	1	0	0	75.35	78.83
17	-1	0	0	1	52.69	49.37
18	-1	-1	0	0	53.19	54.07
19	0	1	1	0	75.51	73.06
20	0	-1	0	-1	69.46	71.56
21	0	0	-1	1	66.45	63.96
22	0	1	0	-1	70.04	73.14
23	0	0	1	-1	70.45	71.19
24	0	-1	1	0	72.51	70.57
25	0	0	0	0	76.07	74.19
26	0	0	-1	-1	66.85	69.98
27	0	-1	-1	0	68.71	69.07

Table 3. Estimated regression coefficients of second order polynomial model for optimization of sorbent preparation (R² = 0.971).

Coefficient	Estimated coefficient	t-Value	p-Value
β_0	-57.431	-2.553	0.025
β_1 (ISC)	2.3232	5.599	0.000
β_2 (ITI)	0.1163	-0.290	0.777
β_3 (CTI)	20.014	2.717	0.019
β_4 (CTE)	0.2803	3.757	0.003
β_{11} (ISC ²)	-0.0303	-8.900	0.000
β_{22} (ITI ²)	-0.0057	-0.566	0.582
β_{33} (CTI ²)	-2.4512	-2.432	0.032
β_{44} (CTE ²)	-0.0004	-3.781	0.003
β_{12} (ISC*ITI)	-0.0014	0.090	0.930
β_{13} (ISC*CTI)	-0.0120	0.434	0.672
β_{14} (ISC*CTE)	0.0000	-0.613	0.551
β_{23} (ITI*CTI)	0.0018	0.434	0.672
β_{24} (ITI*CTE)	0.0004	0.467	0.649
β_{34} (CTI*CTE)	0.0016	-1.217	0.247

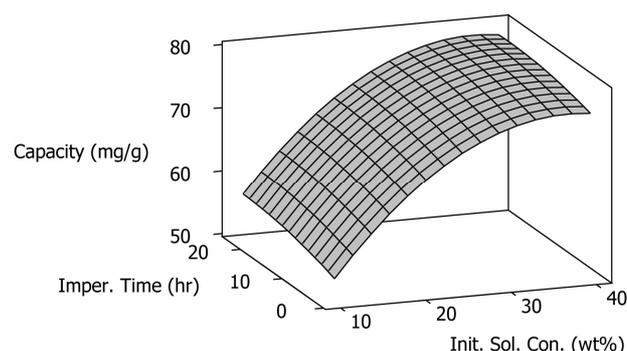
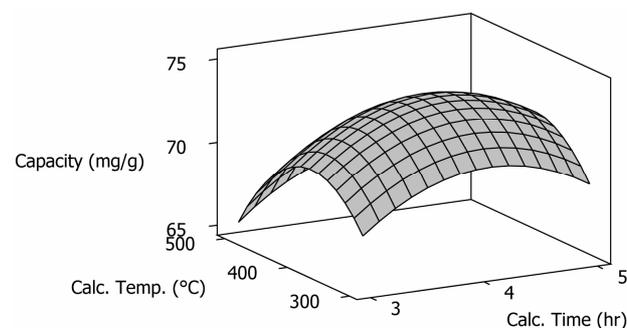
siduals analysis was performed to validate the model at 97% of confidence level. The model fitted well with amylase activity and the optimal values from the model

was justified ($p = 0.000$). The ANOVA given in **Table 4** indicates that the linear, quadratic terms in second order polynomial Model were highly significant ($p < 0.001$) and adequate to represent the relationship between sorbent capture capacity (mg/g) and initial solution concentration, impregnation time and calcination step temperature and time.

Figures 2 and 3 show the three-dimensional response surface which were constructed to show the interaction effect of the sorbent preparation variables on the sorbent capture capacity.

Table 4. Analysis of variance (ANOVA) of second order polynomial model for optimization of sorbent capture capacity.

Source	Degree of freedom	Sum of square	Mean of square	F-value	P-value
Regression	14	1794.5	128.182	28.54	<0.001
Linear	4	1387.6	43.678	9.73	<0.001
Square	4	395.87	98.967	22.04	<0.001
Interaction	6	11.06	1.843	0.41	0.858
Residual error	12	53.89	4.491		
Lack-of-Fit	10	42.09	4.209	0.71	0.709
Pure error	2	11.80	5.898		
Total	26	1848.4			

**Figure 2. The combined effect of initial solution concentration and impregnation time on sorbent capture capacity (CTE = 400°C, CTI = 4 hr).****Figure 3. The combined effect of calcination temperature and calcination time on sorbent capture capacity (ISC = 25%, ITI = 12 hr).**

As can be seen in **Figure 2**, the sorbent capacity generally increases with increase in initial solution concentration and impregnation time. This trend is the result of increase of potassium carbonate loading on sorbent with the increase of these parameters; but the rate of increasing is slow at higher values since the K_2CO_3 loading is limited. The initial solution concentration is more effective than impregnation time; it shows that the rate of impregnation of K_2CO_3 on alumina support is high.

As can be seen in **Figure 3**, the sorbent capacity has a maximum point with increase in calcination temperature and time and after this point it decreases. This trend is the result of improvement of sorbent structure in calcination step that increases the sorbent capture capacity. At calcination step, the higher temperature and longer time damage some formed structures and decreases the sorbent capacity.

3.2. Process Optimization

In the production of commercial sorbent, relatively high sorbent capture capacity are expected. Therefore, in order to optimize preparation condition, Minitab 14 is used. The sorbent was prepared under the experimental conditions given in **Table 5**, together with the predicted and experimental values for sorbent capture capacity. The optimum sorbent prepared was obtained by using initial solution concentration of 32.3 wt%, impregnation time of 13.4 hr, calcination temperature of $367^\circ C$ and calcination time of 4.1 hr. The optimum sorbent showed capture capacity of 77.21 mg CO_2/g sorbent. It was observed that the experimental values obtained were in good agreement with the values predicted from the model, with relatively small errors between the predicted and the actual values (**Table 5**).

Figure 4 shows the variation of sorbent capture capacity with the parameters at the optimum conditions. For the two parameters involve calcination time and temperature, the optimum point locate at the curve maximum point. The overall trend of four curves represents that the initial solution concentration is the most effective parameters on the final sorbent capture capacity.

Figure 5, shows the breakthrough curves of the sorbent prepared at the optimum conditions during CO_2 adsorption in the presence of 9.0 vol.% H_2O and 1 vol.% CO_2 at $60^\circ C$. It was observed that the breakthrough time was 16 min. Further study is required to enhance the CO_2 adsorption rate.

Table 5. Model validation.

Initial solution concentration (wt%)	Impregnation time (hr)	Calcinations temperature ($^\circ C$)	Calcination time (hr)	Sorbent capture capacity (mg/g)	
				Predicted	Experimental
32.3	13.4	367.0	4.1	78.66	77.21

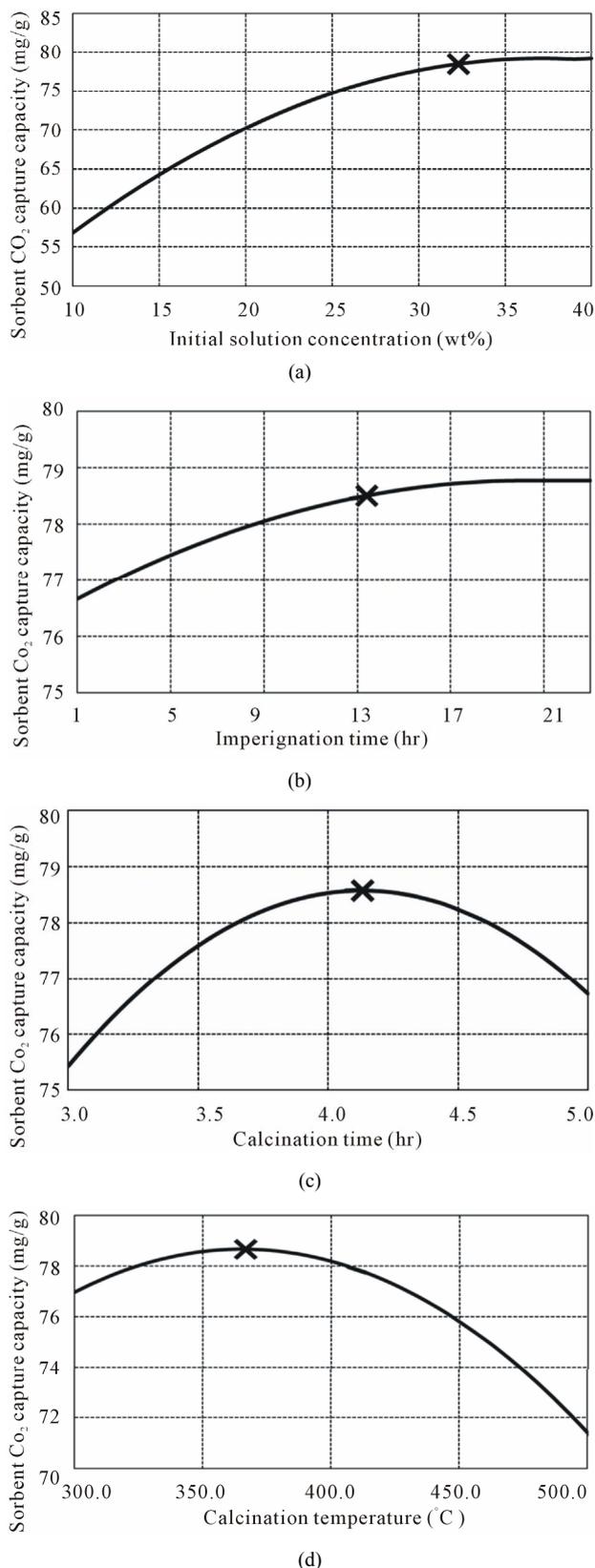


Figure 4. The effect of four parameters on sorbent capture capacity at optimum conditions (x symbol shows optimum point for each curve).

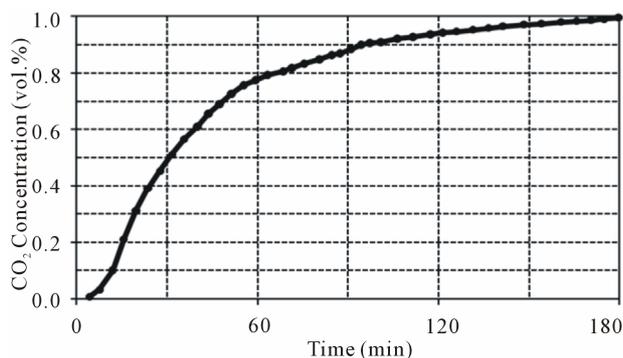


Figure 5. The breakthrough curves of the sorbent prepared at optimum conditions during CO₂ adsorption.

4. Conclusions

The sorbent capacity for carbon dioxide capture from a gas stream is important for the industrial application of solid sorbents. The present investigation was carried out to study combined effects of the initial solution concentration, impregnation time, calcination temperature, and calcination time on the sorbent capture capacity using a Box-Behnken design under the Response surface methodology (RSM). The obtained results demonstrate that sorbent capacity increased with increasing the initial solution concentration and impregnation time. Sorbent capacity has a maximum point for variation of calcination temperature and time and further increases of these variables lead to decrease of sorbent capacity. However, ANOVA analysis as well as 3D surface plots revealed that initial solution concentration has the greatest effect on sorbent capacity. On the basis of the results it can be concluded that RSM presents an excellent tool which enables the evaluation of interactions and competitive effects in multivariable systems and reduces the number of needed experiments in contrast to the classical method of changing one variable at a time.

The optimized values obtained for initial solution concentration, impregnation time and calcination step temperature and time were 32.3 wt%, 13.4 hr, 367°C and 4.1 hr, respectively, with the predicted maximized response of sorbent capture capacity (78.66 mg CO₂/g sorbent). Study on other sorbent characterization such as mechanical strength require for application of this procedure for preparation of industrial sorbents.

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