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Preparation of TiO_2/Perlite Composites by Using 2^{3-1} Fractional Factorial Design

Dilek DURANOĞLU*

Yıldız Technical University, Chemical Engineering Department, Davutpasa Campus,

Esenler 34210, Istanbul, Turkey

Abstract: Successive impregnation and calcination processes were performed in order to produce TiO_2/perlite composites. 2^{3-1} fractional factorial design was first applied to optimize the production conditions of TiO_2/perlite photocatalysts. Seven TiO_2/perlite composites (including three central point experiments) were produced by manipulating three process parameters (amount of TiO_2 used in impregnation process, particle size of perlite and calcination temperature). Prepared TiO_2/perlite photocatalysts were characterized by X-Ray Diffraction Spectrometry and SEM. XRD patterns indicated that anatase was the main crystalline phase for all produced samples. Degradation capacities of produced TiO_2/perlite composites were investigated in methylene blue degradation process. The linear models of TiO_2 loading (%) and methylene blue degradation (%) of TiO_2/perlite composites were developed by regression analysis of the experimental data. As a result of analysis of variance, it was found that developed models were statistically significant with the p-value of 0.0040 and 0.0003, for TiO_2 loading (%) and methylene blue degradation (%), respectively. According to the coefficient of determination (0.9821 and 0.9970 for the models of TiO_2 loading and methylene blue degradation, respectively) and error analysis, developed models fit well to the experimental data. Effect of process parameters was investigated by using response surface plots. Amount of TiO_2 and particle size were found as the most effective parameters on both TiO_2 loading (%) and degradation efficiency (%). Calcination temperature did not affect TiO_2 loading but methylene blue degradation capacity.

Keywords: 2^{3-1} factorial design; photocatalyst; TiO_2; perlite.

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*Corresponding author. E-mail: dilekdur@gmail.com; dduran@yildiz.edu.tr.

INTRODUCTION

TiO_2 is considered the most suitable photocatalyst due to its higher degradation capacity against to the numerous organic pollutants. However, TiO_2 nanoparticles require very expensive microfiltration processes for separating them from water after their use in water treatment. This problem can be solved by attaching nanoparticles onto a support material [1]. However, photocatalytic efficiency of TiO_2 may decrease by immobilizing it on a support material due to the different reasons like properties of support material and production conditions. Expanded perlite, a kind of glassy volcanic rock, can be used as photocatalyst support due to its high silicon content, porous structure, and be found easily in low-cost in nature. Moreover, comparing to other porous materials like activated carbon, zeolite, and clays, lower density and higher light transparency make perlite a promising photocatalyst support. TiO_2 can be strongly attached to the perlite surface with Ti-O-Si bonds effectively, hence, stable photocatalysts could be developed. Expanded perlite, has been widely used as photocatalyst support in recently published works in order to photocatalytic degradation of different pollutant like ammonia [2,3], sulfamethoxazole [4], ethylbenzene [5], phenol [6,7], and furfural [8]. None of them investigated the effect of preparation conditions onto degradation efficiency by any experimental design approach. In the literature of photocatalysis and photocatalytic degradation, experimental design techniques have been just focused on the optimization of degradation process conditions [9, 10] except a few [11, 12]. However, systematic investigation of optimum preparation conditions is very important to develop effective photocatalysts. Applying experimental design techniques would have great significance in designing and developing high performance photocatalysts for environmental remediation [11]. By means of experimental design methods, higher amount of information can be obtained by performing less amount of experiments. There are many types of experimental design techniques such factorial design (Full and Fractional Factorial Design), response surface methodology (Central Composite Design, Box-Behnken Design, Doehlert Design), Plackett-Burman Design, Mixture Design, Taguchi Design, *etc* [13]. In a Full Factorial Design, coefficient of linear interactions, two-term and three-term interactions can be examined [14]. However, full factorial experiments cannot always be conducted because of economy, time, or other constraints [15]. Especially, in case of material development, by considering the cost of raw materials and characterization, conducting Fractional Factorial Design would be more efficient. Moreover, Fractional Factorial Design gives enough information if there is only main effects, and a small number of low order interactions are important [16].

2^{3-1} Fractional Factorial Design matrix is subtracted from the matrix of 2^3 Full Factorial Design [13]. 2^3 Full Factorial Design requires eight experiments and mathematical model of it is given below:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3 \quad \text{(Eq. 1)}$$

However, in 2^{3-1} Fractional Factorial Design, only four experiments are performed. Two-term interactions are reduced according to following relations:

$$X_1 = X_2X_3\ ;\ X_2 = X_1X_3\ ;\ X_3 = X_1X_2\ ;\ I = 1 = X_1X_2X_3 \quad \text{(Eq. 2)}$$

Consequently, mathematical model for 2^{3-1} Fractional Factorial Design is given as follows:

$$Y = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 \quad \text{(Eq. 3)}$$

Where $\beta_0 = b_0 + b_{123}$, $\beta_1 = b_1 + b_{23}$, $\beta_2 = b_2 + b_{13}$, $\beta_3 = b_3 + b_{12}$.

Variation of each parameters involves the effect of two-term interactions of other two parameters. In order to carry out as possible as less amount of experiment, 2^{3-1} Fractional Factorial Design was chosen for investigation of preparation conditions on ultimate TiO_2/perlite composites and methylene blue degradation efficiency. Amount of TiO_2, particle size of perlite and calcination temperature were chosen as independent process variables; both TiO_2 loading percentage and methylene blue degradation percentage were investigated as responses.

MATERIALS and METHODS

Titanium dioxide (Degussa P-25®), as photocatalyst, was supplied from Evonik Company. The ratio of anatase:rutile phases in Degussa P-25 is about 3:1 [17]. Expanded perlite, as photocatalyst support, was obtained from TAŞPER Perlit Company, Turkey. Methylene blue (Sigma-Aldrich, >99%) was used as received and dissolved in distilled water in order to obtain the desired concentration.

Preparation and characterization of perlite supported photocatalyst: After sieving desired particle size, expanded perlite was rinsed with distilled water under 5 mL/min air flow till no powder remains in the filtrate. Typical procedure given in [6], with some changes, was applied in order to prepare TiO_2/perlite composites. According to that, a determined amount of TiO_2 (Degussa P-25), then, 5 mL of diluted nitric acid (at pH 3.3), was added into 50 mL of absolute ethanol in an ultrasonic bath. Then, 5 g of prepared perlite was added to the mixture while stirring in a shaker at 130 rpm, successively, the mixture was kept in ultrasonic bath for 15 minutes to disperse possible agglomerated titanium dioxide powders. After impregnation in an oven at 120°C for 12 hours, the mixture was calcined at high temperature for 1 hour. Once again, TiO_2/perlite composites were washed in distilled water with the assistance of air bubbling for few minutes in order to remove titania powders which had not been precipitated on perlite surface. After filtration and drying, TiO_2/perlite composites were weighed, then used in photocatalytic degradation experiments. TiO_2/perlite composites were investigated by Scanning Electron Microscopy (SEM, CamScan Apollo 300). Samples were attached onto stubs by using double-sided carbon tape, and, in order to obtain high resolution images, they were coated with gold powders using an automated sputter coater. The crystalline structures of TiO_2/perlite composites were determined via X-ray diffractometer. XRD measurement was performed on Philips Panalytical X'Pert Pro X-ray diffractometer using Cu K-alpha radiation. TiO_2/perlite composites were ground to have fine powders before analysis. Crystalline structure of prepared samples has been determined by using software of the instrument.

2^{3-1} Fractional Factorial Design: TiO_2/perlite composites were prepared according to the experimental design matrix given in Table 1. 2^{3-1} fractional factorial experimental design was applied in order to investigate the effects of the selected parameters (amount of TiO_2, particle size of perlite and calcination temperature) on both TiO_2 loading and methylene blue degradation efficiency. Levels of selected parameters and responses for 2^{3-1} fractional factorial design can be seen in Table 2.

Table 1. 2^{3-1} Fractional Factorial Experimental Design Matrix with central points.

Sample No	X_1	X_2	X_3
PT1	+1	+1	+1
PT2	+1	-1	-1
PT3	-1	-1	+1
PT4	-1	+1	-1
PT5	0	0	0
PT6	0	0	0
PT7	0	0	0

Table 2. Factors and levels of 2^{3-1} fractional factorial design.

	Independent variables	**Levels**		
		Low (-1)	Middle (0)	High (+1)
X_1	Amount of TiO_2 (g)	0.25	0.75	1.25
X_2	Mean particle size of perlite (mm)	1.5	2.5	3.5
X_3	Calcination temperature (°C)	300	450	600
	Dependent variables	**Constraints**		
		Low	High	Goal
R_1	TiO_2 loading (%)	0	100	Maximize
R_2	MB Degradation (%)	0	100	Maximize

Statistical analysis: The statistical significance of variables was evaluated using the analysis of variance (ANOVA). Model fitting was evaluated by coefficient of determination (R^2) and error analysis. Design Expert 10.0 software package was used for evaluating model fitting and statistical significance of the developed model.

Photocatalytic degradation study: 500 mL pyrex glass reactor was equipped with 2 x 6 W UV lamps (365 nm) and was used for methylene blue degradation experiments (Figure 2). The photoreactor was covered with aluminum foil to block transition of external light to the reactor. The temperature of the reaction medium was kept constant at 20 ºC by using cooling water recycling. Air flow (12 L/h) was fed into the reactor and flow rate was controlled by a flowmeter. In order to complete adsorption-desorption equilibrium, the reactor was kept in dark in the first 30 min, and then, air flow and UV source was turned on. The sample was collected from the reactor periodically, and methylene blue concentrations were determined at 664 nm by using an Analytic Jena Specord 200 Plus UV Spectrophotometer. Experimental studies were carried with 4 g of PT samples in 500 mL methylene blue solution (initial conc: 20 ppm) and, the mixture was stirred at 500 rpm using a magnetic stirrer throughout the process.

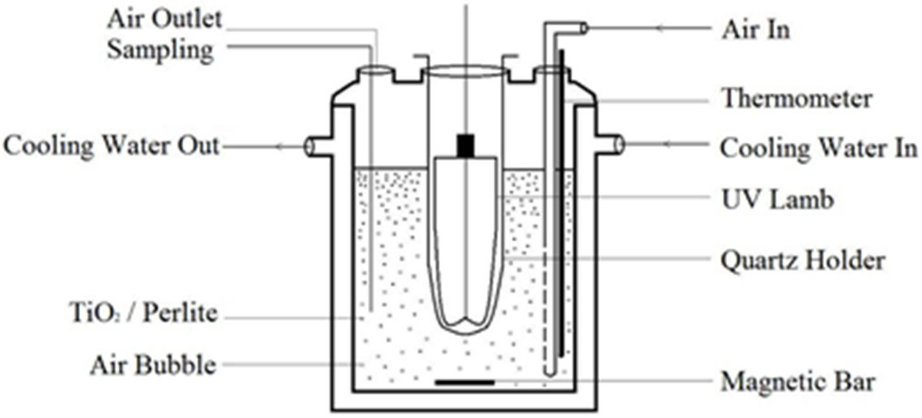
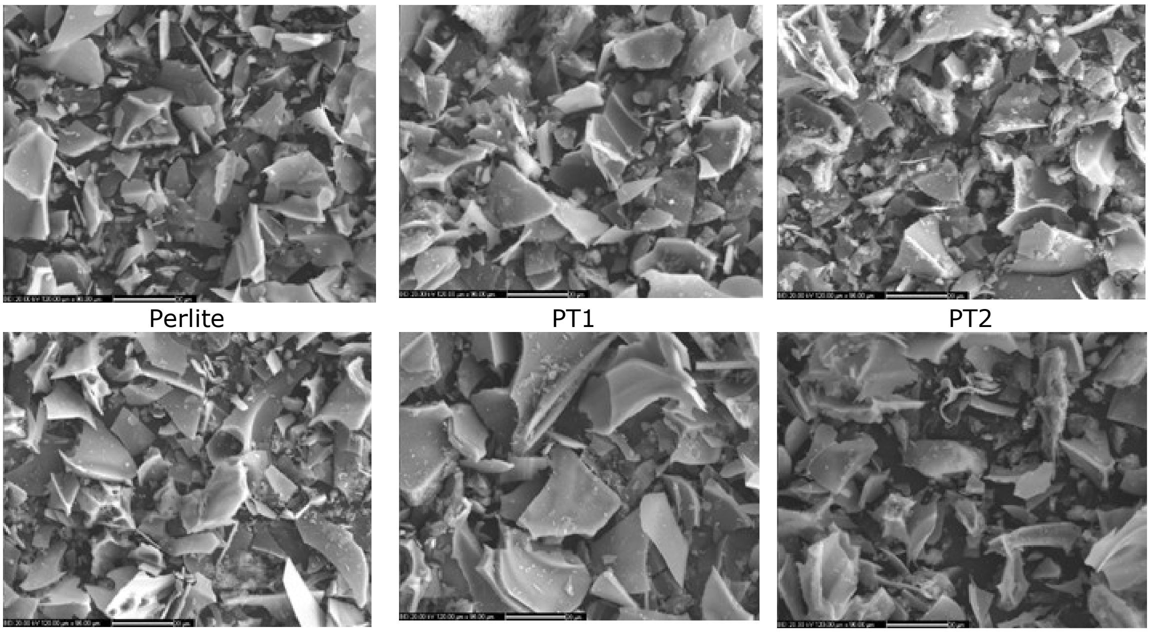


Figure 2. Experimental setup for photocatalytic degradation.

RESULTS and DISCUSSION

Characteristics of prepared TiO_2/perlite composites: SEM images of prepared samples can be seen in Figure 3. Although during the preparation of both PT1 and PT2 the same amount of TiO_2 was used, the amount of loaded TiO_2 onto perlite is much more in case of PT2 sample, which has the smaller particle size. Hence, it can be concluded that not only Ti/Perlite ratio is very important for impregnation process but also particle size is very effective.



Perlite PT1 PT2

PT3 PT4 PT5

Figure 3. SEM images of perlite and TiO_2/perlite composites (x1000).

An X-ray diffractometer was used in order to examine the crystalline structure of TiO_2/perlite composites. As known that crystalline structure is mainly affected by calcination temperature, XRD patterns of TiO_2/perlite composites, which were calcined at different temperatures, and pure TiO_2 were given in Figure 4. Anatase diffraction peaks, which match well with the PDF table (01-084-1285), was observed for all samples, beside, the XRD pattern of PT2 sample, which was calcined at 300 °C, show also rutile diffraction peaks that match well with the PDF table (01-088-1175). As a conclusion, XRD patterns indicate anatase as the main crystalline TiO_2 phase, and rutile crystalline phase in the precursor TiO_2 was converted to the anatase at 450ºC and 600ºC of further calcination.

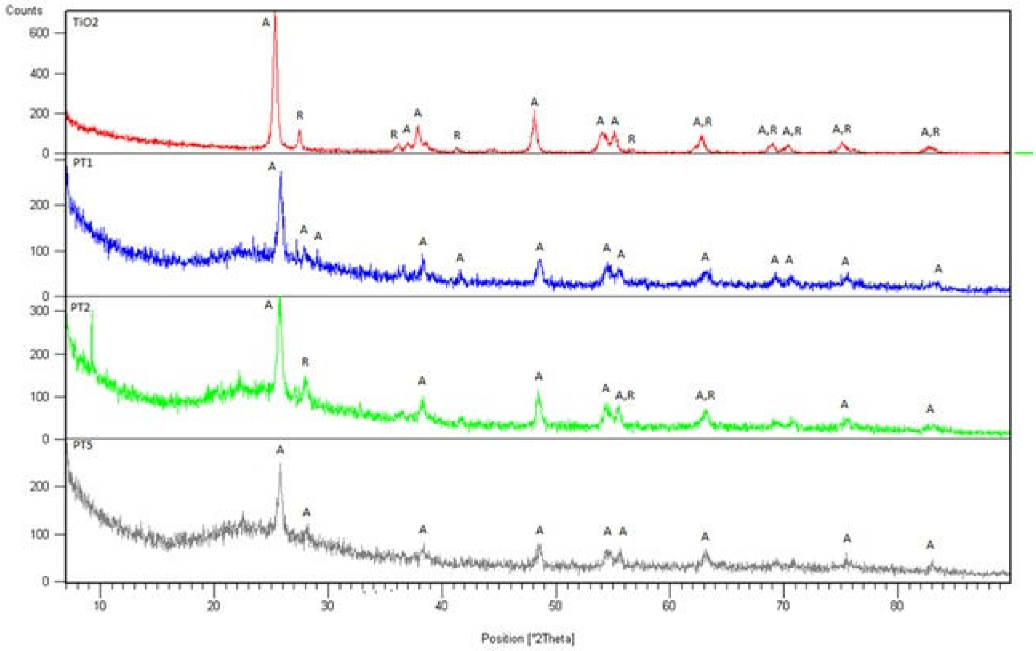


Figure 4. XRD patterns of TiO_2 (Degussa P25) and TiO_2/perlite composites (calcination temperature for PT1, PT2 and PT5 were 600, 300 and 450ºC, respectively).

Photocatalytic degradation of methylene blue: Photocatalytic degradation of methylene blue by TiO_2/perlite composites can be seen in Figure 5. At the first 30 minutes, all samples adsorbed different amounts of methylene blue (%13 - %44) depending on their preparation conditions. Among the PT samples, the best photocatalytic efficiency was obtained with PT2 sample, which has a high amount of TiO_2 and low particle size of perlite. PT3 and PT4, which has low amount of TiO_2 during preparation, showed the worst degradation capacity, even though they had relatively high adsorption capacity. It can be concluded that amount of TiO_2 is the most effective parameter in order to increase photocatalytic degradation efficiency of composites.

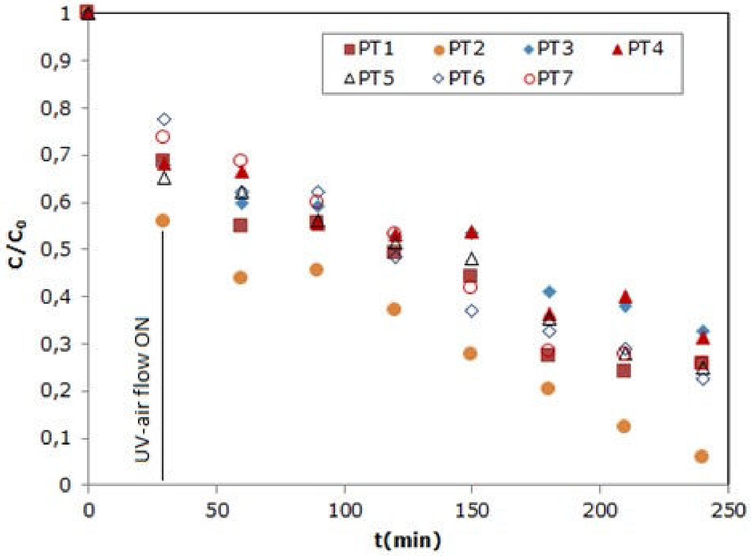


Figure 6. Photocatalytic degradation of methylene blue.

Experimental design approach, model fitting, and statistical analysis: 2^{3-1} factorial design matrix with response values obtained from the experimental studies was given in Table 3. TiO_2 loading (R1) and methylene blue degradation (R2) values were determined by using Eq.4 and Eq.5, respectively.

$$TiO_2 \text{ loading } (\%) \ = [\frac{\text{Amount of TiO2 loaded on perlite (g)}}{\text{Amount of TiO2 (g)}}] * 100 \tag{4}$$

$$MB\ degradation\ (\%) = \left(1 - \frac{MB\ concentration\ at\ 210min\ (\frac{mg}{L})}{initial\ MB\ concentration\ (\frac{mg}{L})}\right) * 100 \tag{5}$$

TiO_2 loading values were found between 14% and 55%, while methylene blue degradation varied between 60.1% and 87.7%, at the studied conditions (Table 3). Increasing amount of TiO_2 loaded on perlite surface did not increase degradation capacity at the same level. This can be explained with the agglomeration of TiO_2 particles on perlite surface in case of higher loading (Figure 3). In addition, it should be kept in mind that the other parameters like particle size of perlite particles and calcination temperature also affect degradation capacity. In order to create a relationship between synthetic parameters and responses (TiO_2 loading, MB degradation), experimental data were transferred into Design Expert (10.0) and linear regression model for both TiO_2 loading (R1) and MB degradation (R2) were developed as in Equations 6 and 7, respectively. Significant factors contributing to the regression model can be identified by analyzing the coefficients. According to the coefficients, it is clear that X_1 (amount of TiO_2) and X_2 (particle size of perlite), respectively, are the main factors for both responses. Coefficient of particle size are almost the same magnitude for both responses, but amount of TiO_2 is more effective for TiO_2 loading than degradation capacity, as expected. Calcination temperature (X_3) did not affect TiO_2 loading, whereas there is a significant relation between calcination temperature and methylene blue degradation, due to the different crystalline structure of the samples calcined at different temperature (Figure 4). By looking at the coefficient of X_3 in Eq.7, it can be concluded that increasing calcination temperature resulted in a decrease of MB degradation. Ramli and coworkers [18] had also observed the same trend on their work related to photodegradation of diisopropanolamine by Cu/TiO_2. They indicated that calcination temperature was the least significant variable among the others (calcination time and amount of Cu).

$$R1 = 36 + 17.50X_1 - 3.00X_2 + 1.33 \times 10^{-5}X_3 \qquad \text{(Eq. 6)}$$

$$R2 = 71.53 + 10.40X_1 - 3.40X_2 - 2.5X_3 \qquad \text{(Eq. 7)}$$

Predicted data in Table 3 were obtained by using developed models (Equations 6 and 7) for both responses. Fitting of model to the experimental data was examined with error analysis. Error values of models were measured by using Equation 8. As can be seen from Table 3, error values are between 2.70-10.71% and 0.15-1.42% for TiO_2 loading and MB degradation, respectively. Consequently, it can be concluded that the developed models fit experimental data, very well.

$$\text{Error \%} = \left|\frac{\text{observed data} - \text{predicted data}}{\text{observed data}}\right| * 100 \qquad \text{(Eq. 8)}$$

Significance of the developed models was examined with ANOVA (Table 4). According to ANOVA results, the model F-values were determined as 42.42 and 337.20 for R1 and R2, respectively, indicating that both models were significant. There is only a 0.44% and 0.03% chance error in both models, respectively. p-values less than 0.500, indicate that parameters are significant in 95% confidence level. The correctness and goodness of the models were checked by the coefficient of determination values. The R^2 values were found as 0.9821 and 0.9970 for both models, respectively.

Table 3. Design matrix, independent, dependent variables, error analysis.

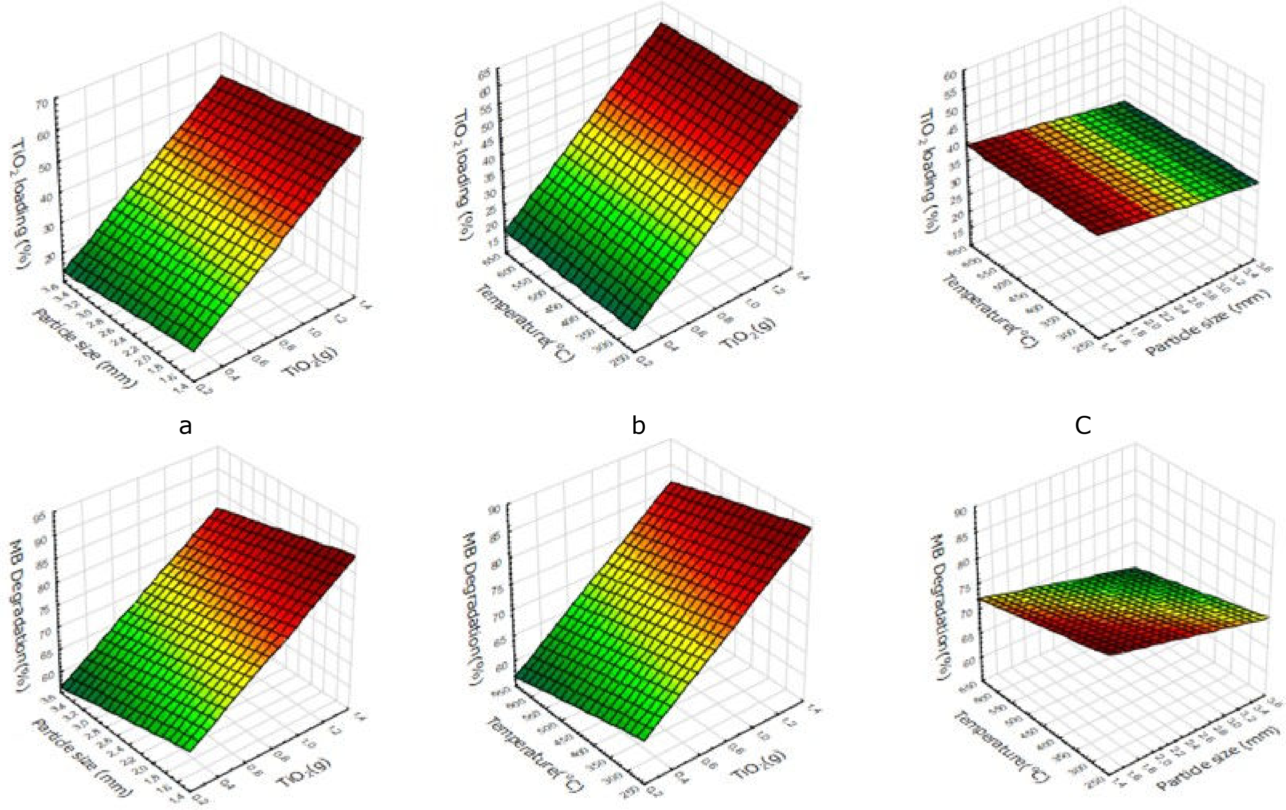
	Independent variables			**Dependent variables**					
	X_1	X_2	X_3	R1 (TiO_2 loading,%)			R2 (MB degradation,%)		
				Observed	Predicted	Error	Observed	Predicted	Error
PT1	+1	+1	+1	49	50.5	3.06	75.9	76.03	0.17
PT2	+1	-1	-1	55	56.5	2.73	87.7	87.83	0.15
PT3	-1	-1	+1	20	21.5	7.50	61.9	62.03	0.21
PT4	-1	+1	-1	14	15.5	10.71	60.1	60.23	1.42
PT5	0	0	0	39	36.0	7.69	71.9	71.53	0.51
PT6	0	0	0	37	36.0	2.70	70.8	71.53	1.03
PT7	0	0	0	38	36.0	5.26	72.4	71.53	1.20

Table 4. Analysis of variance (ANOVA) of developed models for both responses.

	Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**p-value Prob > F**
R1 (TiO_2 loading,%)	Model	1261.00	3	420.33	54.83	0.0040
	X_1	1225.00	1	1225.00	159.78	0.0011
	X_2	36.00	1	36.00	4.70	0.1188
	X_3	0.0000133	1	0.000	0.000	1.0000
	Residual	23.00	3	7.67		
	Lack of Fit	21.00	1	21.00	21.00	0.0445
	Pure Error	2.00	2	1.00		
	Cor Total	1284.00	6			
R2 (MB degradation,%)	Model	503.88	3	167.96	337.20	0.0003
	X_1	432.64	1	432.64	868.59	< 0.0001
	X_2	46.24	1	46.24	92.83	0.0024
	X_3	25.00	1	25.00	50.19	0.0058
	Residual	1.49	3	0.50		
	Lack of Fit	0.15	1	0.15	0.23	0.6787
	Pure Error	1.34	2	0.67		
	Cor Total	505.37	6			

Effects of process parameters: Response surface plots, obtained by using Statistica 8.0, were used to evaluate the effects of process parameters on both TiO_2 loading and MB degradation. In order to evaluate the effect of each parameter on the responses, the obtained regression surfaces were plotted as function of two process variables (Figure 6).

Linear effects of particle size and amount of TiO_2, and also their interactions can be seen in Figure 6a and 6d. Increasing amount of TiO_2 caused a significant increase on both TiO_2 loading and MB degradation. Particle size of perlite was also effective on both responses; smaller particle size resulted in higher loading and degradation capacity. Due to the higher surface area of small size particles, more amount of TiO_2 was impregnated on the perlite surface, hence, higher degradation capacity was obtained. Linear effects of calcination temperature and amount of TiO_2, and also their interactions can be seen in Figure 6b and 6e. Calcination temperature (X_3) did not affect TiO_2 loading, whereas methylene blue degradation was negatively affected by increasing temperature. This result can be explained with the crystalline structure of TiO_2 on perlite surface. Lower temperature resulted in both anatase and rutile phase (Figure 4) crystalline structure that it is similar to the commercial one (Degussa P-25). As can be seen from Figure 6f, smaller particle size and lower temperature gives higher degradation capacity.



a b C

d e F

Figure 6. Response surface plots.

CONCLUSION

Experimental design method was effectively used in order to prepare higher performance TiO_2/perlite photocatalysts for methylene blue degradation. Effects of TiO_2 amount, particle size of perlite, and calcination temperature on both TiO_2 loading (%) and methylene blue degradation (%) were investigated by applying 2^{3-1} Fractional Factorial Design. The linear models of TiO_2 loading (%) and methylene blue degradation (%) of TiO_2/perlite composites were developed by regression analysis of the experimental data. Amount of TiO_2 and particle size were found as the most effective parameters on both TiO_2 loading (%) and degradation efficiency (%). Optimum solution for the highest methylene blue degradation

(%86.94) was obtained via Design Expert Software with 1.143 g of TiO_2, 1.5 mm of perlite particle size and 300 °C of calcination temperature. As a conclusion, experimental design methods can be effectively used in order to prepare photocatalysts with higher capacity for degradation of target pollutant.

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Türkçe Öz ve Anahtar Kelimeler

2^{3-1} Kısmi Faktöriyel Deney Tasarımı Kullanarak TiO_2 / Perlit Kompozitlerinin Hazırlanması

Dilek DURANOĞLU

Öz: TiO_2 / Perlit kompozitleri emdirme ve ardından uygulanan kalsinasyon prosesleri ile elde edilmiştir. TiO_2 / Perlit fotokatalizörlerinin üretim koşullarını en uygun hale getirmek için 2^{3-1} kısmi faktöriyel deney tasarımı ilk kez uygulanmıştır. Yedi adet TiO_2 / Perlit kompoziti (üç adet merkez nokta deneyleri dahil olmak üzere) üç farklı proses parametresi (emdirme aşamasında kullanılan TiO_2'in miktarı, perlitin parçacık boyutu ve kalsinasyon sıcaklığı) değiştirilerek üretilmiştir. Hazırlanan TiO_2 / Perlit fotokatalizörleri X-Işını Difraksiyon Spektrometrisi ve SEM ile karakterize edilmiştir. XRD desenlerine göre bütün üretilen örneklerdeki ana kristal yapı anataz fazıdır. Üretilen TiO_2 / Perlit kompozitlerinin bozunma kapasiteleri metilen mavisi bozunma prosesinde incelenmiştir. TiO_2 / Perlit kompozitlerinin TiO_2 yüklenmesi (%) ve metilen mavisi bozunmasına (%), ait model denklemleri deneysel verilerin regresyon analizi ile geliştirilmiştir. Varyans analizi sonucunda, geliştirilen modellerin sırasıyla TiO_2 yüklenmesi (%) ve metilen mavisi bozunması (%) için 0,0040 ve 0,0003 p değerleriyle istatistik olarak anlamlı olduğu bulunmuştur. Belirleme katsayıları (sırasıyla TiO_2 yüklenmesi ve metilen mavisinin bozunması için 0,9821 ve 0,9970) ve hata analizlerine göre, geliştirilmiş modellerin deneysel verilere iyi uyum sağladığı bulunmuştur. Proses parametrelerinin etkisi cevap yüzey eğrileri ile incelenmiştir. TiO_2 yüklenmesi (%) ve bozunma kapasitesi (%) üzerindeki en etkili parametreler TiO_2 miktarı ve parçacık boyutu olmuştur. Kalsinasyon sıcaklığı TiO_2 yüklemesine etki etmemiştir, ancak metilen mavisinin bozunma kapasitesi üzerine etkili olmuştur.

Anahtar kelimeler: 2^{3-1} kısmi faktöriyel deney tasarımı, fotokatalizör, TiO_2, perlit.

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