

# Optimization of Synthesis Parameters in Photodegradation of Acid Red 73 Using TiO<sub>2</sub> Nanoparticles Prepared by the Modified Sol-Gel Method

Somayeh Alijani, Mohammad Vaez, and Abdolsamd Zarringhalam Moghaddam

**Abstract**— TiO<sub>2</sub> nanoparticles were synthesized by the P-25 powder modified sol-gel method under different TTIP (Titanium tetraisopropoxide) concentrations, P-25 loading and the gelation pHs. Based on XRD analysis SEM results and BET technique, the crystalline composition, the level of crystallinity, the particle size and the surface area of the as-prepared samples were found to be affected by these parameters. Response surface methodology based on central composite design was used to optimize these synthesis parameters in photodegradation of Acid Red 73. The degradation efficiency was significantly affected by P-25 loading, pH value of gelation and the interaction effect between TTIP concentration and P-25 loading. The optimal values of parameters were found to be a pH of 1.34, a TTIP concentration of 0.25 M and a P-25 loading of 39.76 g/L. Regression analysis with an  $R^2$  value of 0.9549 showed a good agreement between the experimental results and the predicted value.

**Index Terms**—Modified sol-gel method, Response surface methodology, structural properties, titanium dioxide nanopowders.

## I. INTRODUCTION

The most recently completed assessment, published in 2013 by the World Health Organization (WHO) has reported that 768 million people around the world lacked access to “improved water supply” [1]. These figures are expected to increase in the future due to overwhelming discharge of contaminants into the natural water cycle. To slow the growth of the clean water shortage, the development of low-cost and effective technologies for wastewater treatment is needed. In recent decades, a great deal of interest has been devoted to photocatalytic degradation of organic water pollutants utilizing TiO<sub>2</sub> as photocatalyst [2], [3]. It is mainly due to the high potential of TiO<sub>2</sub> in mineralization of a wide range of organic pollutants at ambient temperature and pressure into CO<sub>2</sub> and water. However, so far, little progress has been made in the development of a photocatalytic technology for water treatment in large applications. The presence of a number of system factors that require rapid coverage testing is one of the technical obstacles to scaling up photocatalytic technology processes [4]. The traditional one-factor-at-a-time (OFAT) approach, as the most common method for optimization, is time-consuming and expensive

due to reagent costs [5]. Moreover, this univariate approach does not show the interactions between the independent variable [4]. Therefore, there is currently an effort to replace this inefficient practice with effective chemometric methods [5]. The response surface methodology (RSM), as an efficient chemometric technique, has been successfully applied for various processes to achieve optimization using experimental designs, including TiO<sub>2</sub>/UV oxidation [6], [7]. However, there have been no reports of research on the optimization of synthesis parameters and their interactions using experimental design methodology during the synthesis of TiO<sub>2</sub> via the modified sol-gel method for degradation of Acid Red 73. TiO<sub>2</sub> nanoparticles as photocatalyst have been usually synthesized by the well-known sol-gel method [8]. Some investigators have reported that modification of the sol-gel method by incorporation of P-25 into the sol can improve the structural properties of the TiO<sub>2</sub> nanocatalyst and its photocatalytic efficiency [9], [10]. In the present study, TiO<sub>2</sub> nanoparticles were synthesized by the P-25 powder modified sol-gel method (PPMSG) under different synthesis parameters and the interaction effect of these parameters on the photocatalytic degradation of Acid Red 73 was studied using central composite design (CCD) based on the response surface methodology (RSM) and the optimal values of these parameters were determined.

## II. EXPERIMENTAL

### A. Experimental Design and Optimization by Response Surface Methodology

In this study, the experimental conditions in the synthesis of nanosized TiO<sub>2</sub> powders by P-25 powder-modified sol-gel method were investigated and optimized in the photodegradation of Acid Red 73 using central composite design (CCD) at five levels. The Design Expert 7.0 software was used to analyze the experimental findings obtained under changing synthesis conditions. The concentration of titania precursor (TTIP concentration), P-25 loading and gelation pH were chosen as independent variables and the degradation percentage of Acid Red 73 was considered as the dependent variable (response). The ranges and levels of the studied variables are given in Table I. In according to Table I, TiO<sub>2</sub> nanoparticles were synthesized through 20 experiments with different synthesis conditions.

### B. Synthesis of TiO<sub>2</sub> Nanoparticles

TiO<sub>2</sub> nanocrystals were prepared by the P-25 modified sol-gel method as follow: various volume of titanium isopropoxide (TTIP, Ti(O-i-C<sub>3</sub>H<sub>7</sub>)<sub>4</sub>, purity 98%, Merck) was

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dissolved in a 20 mL of anhydrous ethanol to provide solution with various TTIP concentrations (Table I). The solution was stirred at room temperature for 30 min. Then, a mixture of deionized water and dilute nitric acid was added dropwise to the above solution to adjust the pH solution to a desirable value (Table I). After stirring for 1 h, P-25 powder with a loading according with Table 1 was ultrasonically dispersed in the sol. The obtained white dispersion was then aged for 12 h followed by gelation at room temperature. The resultant gel was dried at 80 °C for 10 min and then calcinated at 600 °C for 1 h with a heating and cooling rate of 1 °C/min.

TABLE I: EXPERIMENTAL RANGE AND LEVELS OF THE INDEPENDENT TEST VARIABLES

Variables	Ranges and levels				
	-2	-1	0	1	2
TTIP concentration (M) ( $x_1$ )	0.1	0.18	0.3	0.42	0.5
P-25 loading (g/L) ( $x_2$ )	10	18.10	30	41.89	50
Gelation pH ( $x_3$ )	1	1.2	1.5	1.8	2

### C. Characterization

The XRD patterns of the prepared titania nanoparticles were obtained using PHILIPS PW1800 analyzer with a Cu K $\alpha$  X-ray source. The main grain size (L) and the rutile content in samples were calculated by the formula as mentioned in our previous work [11]. The morphology of the powders was determined using Field Emission Scanning Electron Microscopy (FESEM, Hitachi S-4160). The Brunauer-Emmett-Teller (BET) method with a Micromeritics 2000 instrument (ASAP 2000, Micromeritics, USA) was used to measure the specific surface area of samples by nitrogen adsorption-desorption at 77 K.

### D. Photocatalytic Activity Measurement

The photocatalytic activities of TiO $_2$  powders (0.1 g/L) prepared by PPMMSG method were evaluated by the photodegradation of Acid Red 73 as the model compound. Experiments were carried out in a batch-mode rectangular reactor made of Pyrex glass with total volume of 1 L. The artificial irradiation was provided by four UV-A lamps (9W, Philips, 1.8 mW/cm $^2$ ), positioned parallel to each other in the box. The scheme of the employed photocatalytic reactor has been presented in our previous work [12]. The dye solution was aerated using two air pumps with a 1 L/min flow rate. The reactor was cooled by a fan placed at the box. Before each experiment, the aqueous solution of acid dye was agitated with gentle air in the presence of TiO $_2$  sample in the darkness for at least 30 min to achieve the complete equilibrium of adsorption process. Photocatalytic degradation processes were performed at 298 K with 250 mL of solution and an initial Acid Red 73 concentration of 20 mg/L. Samples were taken out at regular time intervals, and an Optizen 3220UV Double Beam spectrophotometer was used to analysis the samples. Before the measurement of residual dye concentration, each sample was filtered (pore size < 0.22  $\mu$ m) to remove any possible TiO $_2$  particles present in the solution. The maximum wavelength ( $\lambda_{max}$ ) of Acid Red 73 is 545 nm.

## III. RESULTS AND DISCUSSION

### A. Model Fitting and Analysis of Variance (ANOVA)

Depending upon the experimental design and the experimental results and the predicted responses, the relationship between the response and the studied variables was derived and expressed by the following empirical second-order polynomial equation in actual form:

$$\begin{aligned} \% \text{ degradation} = & -170.00171 + 4.26293 \times \text{TTIP} + \\ & 4.28598 \times \text{P25} + 150.95228 \times \text{pH} - 0.04118 \times \text{TTIP} \times \\ & \text{P25} + 0.79902 \times \text{TTIP} \times \text{pH} - 0.32880 \times \text{P25} \times \text{pH} - 0.14758 \\ & \times \text{TTIP}^2 - 0.041896 \times \text{P25}^2 - 55.24231 \times \text{pH}^2 \end{aligned}$$

Based on the negative coefficients of quadratic terms  $x_{ii}^2$  in the polynomial expression, an excess of TTIP concentration ( $x_1$ ), the P-25 loading ( $x_2$ ) and pH gelation ( $x_3$ ) in the system produces a negative influence on the photocatalytic performance (decreased Acid Red 73 %degradation). The negative coefficient of  $x_1x_2$  parameter in polynomial expression indicates an antagonistic effect between these variables [5].

According to the ANOVA results (Table II), the model F-value of 45.75 implies the model is significant. There is only a 0.01% chance that the "model F value" could occur due to noise. The p value for the model, which is less than 0.0001, also indicates that the model is significant.

TABLE II: ANOVA RESULTS FOR THE RESPONSE SURFACE QUADRATIC MODEL

Source	Sum of squares	Degree of freedom	Mean squares	F value	p value
Model	2190.23	9	243.36	45.75	<0.0001
Residual	53.19	10	5.32		
Lack of fit	38.47	5	7.69	2.61	0.1578
Pure error	14.73	5	2.95		
Total	2243.43	19			
Std. Dev.	2.31				

The "lack-of-fit F-value" of 2.61 implies that the lack of fit is not significant relative to the pure error. There is a 15.78% chance that the "Lack-of-Fit F-value" could occur due to noise. The non-significant lack-of-fit term indicates the ability of the model to predict the experimental results accurately. The good predictability of the model was also confirmed by the "Pred R-Squared" of 0.8566 which is in reasonable agreement with the "Adj R-Squared" of 0.9549.

The results obtained from the analysis of variance (ANOVA) at 95% confidence level are given in Table III.

In according to the p-value of each factor, the P-25 loading ( $x_2$ ), pH gelation ( $x_3$ ) and the second-order effect of factors ( $x_{ii}^2$ ) are highly significant parameters with  $p < 0.001$ . The interaction effect between the concentration of TTIP ( $x_1$ ) and P-25 loading ( $x_2$ ) is significant at  $p < 0.05$ . Moreover, the first-order effect of TTIP concentration ( $x_1$ ) and the interactions between TTIP concentration and pH gelation and between P-25 loading and pH gelation are insignificant, since these terms have  $p\text{-value} > 0.05$ .

According to the monomial coefficient values of the

regression model,  $p(x_1) = 0.0835$  (TTIP concentration),  $p(x_2) < 0.0001$  (P-25 loading) and  $p(x_3) = 0.0001$  (pH gelation), the order of importance among the factors is P-25 loading ( $x_2$ ) > pH gelation ( $x_3$ ) > TTIP concentration ( $x_1$ ).

TABLE III: COEFFICIENTS OF REGRESSION AND THEIR SIGNIFICANCES

Factor	F-value	95% confidence interval Low	95% confidence interval High	p-value
Intercept	-	36.29	40.48	0.0835
$x_1$	3.69	-2.59	0.19	<
$x_2$	158.72	6.47	9.25	0.0001
$x_3$	36.33	-5.15	-2.37	0.0051
$x_1 x_2$	12.76	-4.73	-1.10	0.1139
$x_1 x_3$	3.00	-0.40	3.23	0.1844
$x_2 x_3$	2.03	-2.98	0.65	<
$x_1^2$	73.76	-6.57	-3.86	0.0001
$x_2^2$	95.11	-7.28	-4.57	<
$x_3^2$	64.59	-6.24	-3.53	0.0001

### B. Response Surface Analysis

The three-dimensional plots display the model-predicted response graphically to provide a clearer view of the response surface and a better understanding of interactions between variables [12].

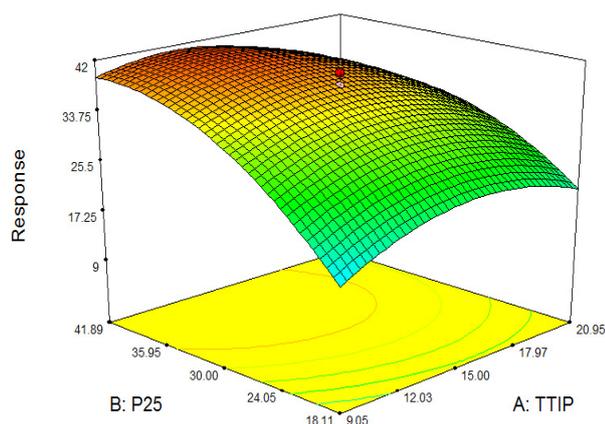


Fig. 1. Effects of TTIP concentration and P-25 loading on Acid Red 73 degradation (Gelation pH: 1.5)

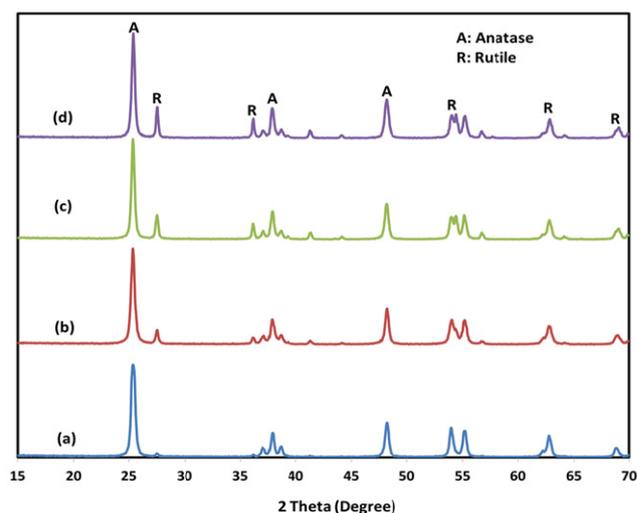
Fig. 1 shows the simultaneous effect of TTIP concentration and P-25 loading on Acid Red 73 degradation. Degradation increases with an increase in TTIP from 0.1 to 0.28 M and decreases gradually when the TTIP concentration increases beyond 0.3 M. This behavior can be attributed to the rutile-anatase distribution in the crystalline framework, as shown in Table IV. Based on the XRD analysis, the rutile content in  $\text{TiO}_2$  catalysts increases with increasing TTIP amount to a middle concentration and decreases as TTIP concentration goes over an optimal value.

 TABLE IV: PHYSICAL PROPERTIES OF  $\text{TiO}_2$  POWDERS OBTAINED FROM MODIFIED SOL-GEL METHOD AT DIFFERENT TTIP CONCENTRATION, GELATION pH: 1.5 AND P-25 LOADING: 30 G/L

TTIP concentration (M)	Crystal size (nm)	Anatase content (%)	$S_{\text{BET}}$ ( $\text{m}^2/\text{g}$ )
0.1	35.75	95	21.36
0.3	42.29	75	22.58
0.5	39.84	89	23.29

Although the anatase phase of  $\text{TiO}_2$  has been reported to be more active as a photocatalyst compared with the rutile, the synergistic effect between rutile and anatase can improve the photocatalytic performance of  $\text{TiO}_2$  [13].

However, the high loading of P-25 slows down the positive effect of TTIP concentration significantly and accelerates its negative effect on the photocatalytic performance. Incorporating P-25 loading into the sol favors the rutile formation in resulting crystal structure, as XRD results shown in Fig. 2.


 Fig. 2. XRD patterns of  $\text{TiO}_2$  powders prepared with TTIP concentration: 0.3 M, pH: 1.5 and the sol containing no P-25 powder (a) 10 g/L P-25 powder (b) 30 g/L P-25 powder (c) and 50 g/L P-25 powder (d)

Although the presence of rutile in the crystalline structure improves the photocatalytic activity, the anatase/rutile proportion can be also an important parameter in evaluation of photocatalytic efficiency. The best result of photoactivity is observed for the sample possesses the crystalline composition of rutile and anatase close to that of commercial P-25  $\text{TiO}_2$  (70/80:30/20) [14]. Generally, both P-25 loading above 35 g/L and TTIP concentration more than 0.3 M decrease the decolorization of catalyst efficiency.

The effects of TTIP concentration and pH gelation on Acid Red 73 degradation efficiency are shown in Fig. 3. The effect of TTIP concentration on the color removal efficiency exhibited the similar trends.

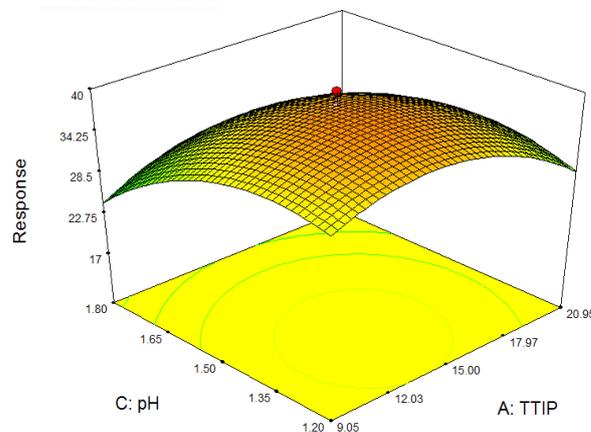


Fig. 3. Effects of TTIP concentration and gelation pH on Acid Red 73 degradation (P-25 loading: 30 g/L)

At higher value of gelation pH, the increased rutile content

and the decreased size of particles can slightly enhance the negative impact of TTIP concentration. The XRD analysis, presented in Fig. 4, show that increasing acidity in medium solution promotes the rutile formation. In addition, it was found that the width of the diffraction peaks of anatase become wider with increasing pH. The size of the crystallites calculated by Scherrer's equation decreases with increasing pH from 56.71 nm at pH=1 to 31.66 nm at pH=2. As suggested by literature, bigger TiO<sub>2</sub> particles are formed at pHs away from the range of isoelectric point (5-6.8) [15].

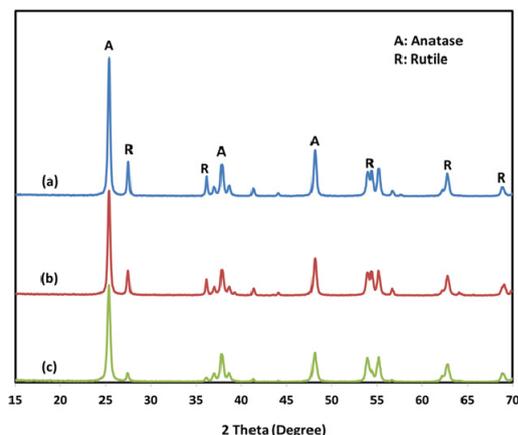


Fig. 4. XRD patterns of TiO<sub>2</sub> powders prepared by modified sol-gel method at TTIP concentration: 0.3 M, P-25 loading: 30 g/L and pH: 1 (a) pH: 1.5 (b) and pH: 2 (c)

Therefore, at higher pH value, the negative impact of TTIP concentration is accelerated, since the powders with a small size of particles are usually associated with large amounts of crystalline defects, which favor the electron-hole recombination leading to a poor photoactivity [16].

However, it is also noted that small grain size can confer sufficiently active surface to adsorb organic molecules and promotes photocatalytic efficiency, as a result. The BET analysis of the samples prepared at different pH of gelation show the increase in the surface area from 17.31 m<sup>2</sup>/g to 28.01 m<sup>2</sup>/g as gelation pH increases from 1 to 2. Therefore, as shown in Fig. 3, there is an optimum value for gelation pH

leading to an optimal value for particles size and consequently high level of crystallinity combined with good surface area which contributes to high efficiency of TiO<sub>2</sub> catalysts.

Fig. 5 shows the effects of P-25 loading and gelation pH on decolorization. The influence of loading of P-25 on the degradation of Acid Red 73 is highly significant.

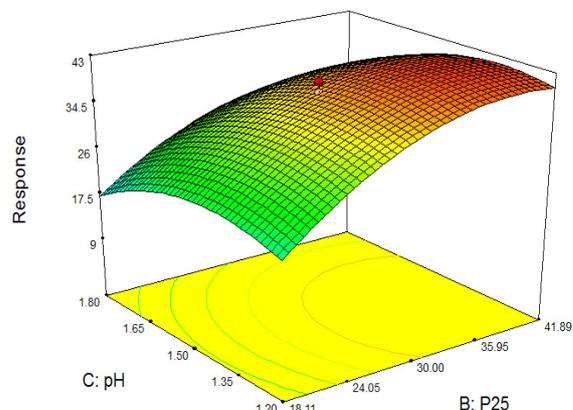


Fig. 5. Effects of P-25 loading and gelation pH on Acid Red 73 degradation (TTIP concentration: 0.3 M)

As the amount of P-25 powder incorporated into sol increases, the degradation percentage of acid dye increases drastically, whether gelation pH has a low or high value. This positive effect can be ascribed to the increased rutile content in the final crystal structure which confers more electron capture traps. However, at higher loading of P-25, a slight decrease in acid dye decolorization is observed. This negative impact can be explained by decreasing the grain size of samples at higher loading of P-25, as FESEM analysis shown.

Fig. 6 shows the morphology of powders prepared at 10, 30 and 50 g/L P-25 in sol.

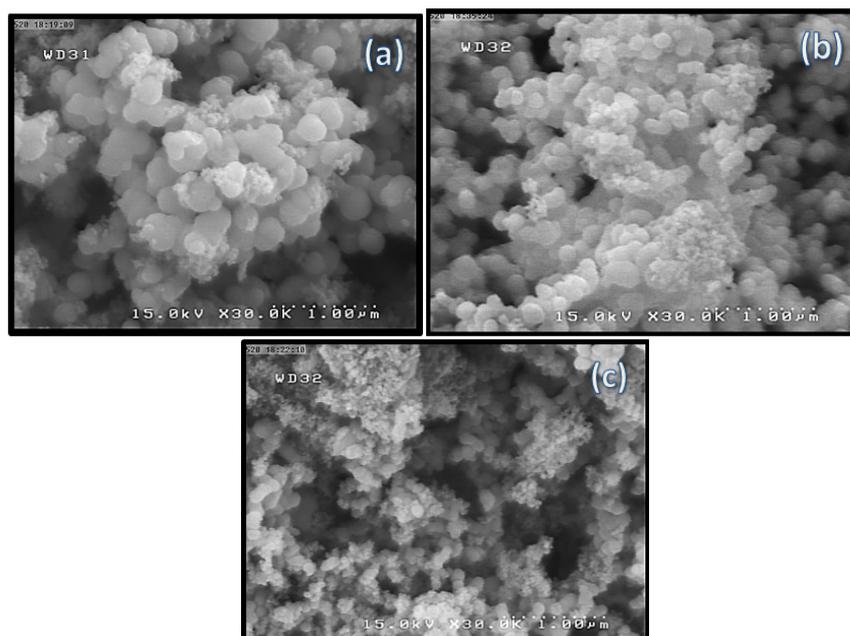


Fig. 6. FESEM images of TiO<sub>2</sub> powders prepared at TTIP concentration: 0.3 M, pH: 1.5 and by the sol containing 10 g/L P-25 powder (a) 30 g/L P-25 powder (b) and 50 g/L P-25 powder

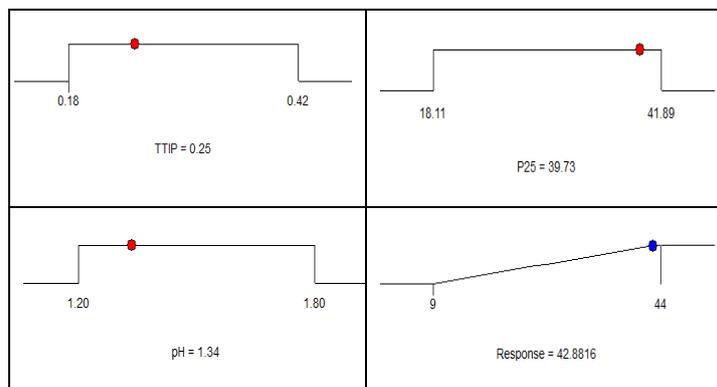


Fig. 7. Desirability ramp for numerical optimization

As it is obvious by comparing Figs. 6 (a), (b) and (c), the decrease in average of grain size is observed with increasing the loading of P-25 in the modified sol, as it is clear from Figs. 6 (a), (b) and (c). The average grain size of powders prepared at various loading of P-25 was measured by Measurement Software. It was found that the average size of grain for the samples obtained at 10, 30 and 50 g/L of P-25 in the modified sol was approximately 185, 141 and 123 nm, respectively. As suggested by literature, the P-25 particles may act as sites for nucleation of TiO<sub>2</sub> nanoparticles derived from alkoxide sol [10]. As P-25 loading increases, the number of crystallites formed from the alkoxide sol is not enough to form a large size grain and the grain size decreases as a result.

#### C. Optimization of the Synthesis Conditions

The optimal conditions for the degradation of Acid Red 73 were determined on the basis of the desirability function. The numerical optimization program was applied to find a set of conditions that maximizes this function. For such studies, the maximum degradation which is the main objective of optimization was considered by adjusting the 'importance' to 5 like the previous work [12] and the 'importance' of 3 was provided for the three independent variables. Based on these settings, the optimal synthesis conditions for maximum Acid Red 73 degradation efficiency (42.88%) were found as shown in Fig.7.

#### D. Model Validation and Confirmation

Verification experiments were carried out at the optimal synthesis conditions obtained from the experimental design proposed by CCD to confirm the adequacy of the model for predicting of the maximum percentage degradation of Acid Red 73. An average maximum degradation of 43.03% was derived from three replicative experiments performed by titania synthesized under optimal conditions. The good agreement between the predicted and the observed results verifies the validity of the model for simulating the photodegradation of Acid Red 73.

## IV. CONCLUSION

TiO<sub>2</sub> nanoparticles were prepared by the modified sol-gel method under different synthesis parameters including TTIP concentration, P-25 loading and gelation pH. Structural properties of nanoparticles were characterized by XRD, FESEM and BET analysis. The experimental design methodology was used to optimize the selected parameters

for synthesis of TiO<sub>2</sub> nanoparticles using the modified sol-gel method for the degradation of Acid Red 73. Under the optimized conditions of a 0.25 M TTIP concentration, 39.76 g/L P-25 loading and a pH value of 1.34, the degradation efficiency of Acid Red 73 approached 42.88% after 1 h.

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