RESEARCH PAPER

Hydrothermal Synthesis Parameters for ZnO Nanorods: Statistical Optimization and Photocatalytic Activity

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ABSTRACT

This study shows that growing zinc oxide nanorods with the hydrothermal method hinges on four key factors: temperature, time, precursor concentration, and pH. Using statistical analysis with a central composite design, we got a quadratic model that explained more than 96% of the changes in the nanorod aspect ratio. Temperature emerged as the most significant factor (F = 117.8), followed by time (F = 52.9). Surprisingly, a higher concentration can be counterproductive. We learned that how temperature and time, and time and concentration interact, is crucial. So, tweaking each factor separately isn't effective for optimization. The top results came from setting the temperature at 131.5°C and time at 8.9 hours, with a concentration of 2.48 mM and a pH of 6.9. These conditions led to an aspect ratio of 11.8 and a specific surface area of 4.28 m^2/g for the nanorods that's almost double the active surface area compared to regular, non-optimized samples. We repeated the validation experiment three times, and it showed the model predicted accurately within our tested range with less than 2% error. When testing the photocatalytic activity using methylene blue breakdown, the optimized nanorods worked about 2.6 times faster than the control. The rate constant went up to 0.0178 per minute, and the pollutant's half-life dropped from over 100 minutes to just 9.38 minutes in the optimized sample. XRD analysis confirmed that the nanorods were pure wurtzite phase and mainly grown along the c-axis, which is ideal for efficient charge transfer in photocatalysis. Overall, using the response surface method reduced the number of experiments needed and gave us a much clearer understanding of how these factors interact. The approach isn't restricted to zinc oxide either; it can improve results and conserve resources for other nanostructured photocatalysts. These findings lay a solid foundation for designing zinc oxide photocatalysts to tackle wastewater containing colored pollutants more effectively.

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INTRODUCTION

Industrial and agricultural as mentioned by Kolodziejczak-Radzimska and Jesionowski, growth has caused a huge amount of organic pollutants to get into our water lately [1]. These include synthetic dyes and unused drugs that just won't go away; traditional wastewater treatment isn't effective at breaking them down. This has made scientists look for better methods to clean up the mess [2-5]. One promising approach is advanced oxidation processes. They really stand out as noted by [6], because they can completely break down and transform a wide range of pollutants. Semiconductors, particularly when used as photocatalysts, play a big role in this technology [7-9].

Table 1. Range and levels of independent parameters in the central composite design.

Parameter	Symbol	Unit	-α	-1	0	+1	+α
Temperature	A	°C	80	97.5	115	132.5	150
Time	B	h	2	4.5	7	9.5	12
Precursor conc.	C	mM	25	43.75	62.5	81.25	100
pH	D	-	5.5	6.0	6.5	7.0	7.5

Table 2. Experimental design matrix and measured responses.

Run	A (°C)	B (h)	C (mM)	D	AR (L/D)	Degradation (%)
1	97.5	4.5	43.75	6.0	4.2	52.3
2	132.5	4.5	43.75	6.0	6.8	68.1
3	97.5	9.5	43.75	6.0	5.9	62.4
4	132.5	9.5	43.75	6.0	10.3	84.7
5	97.5	4.5	81.25	6.0	3.8	48.6
6	132.5	4.5	81.25	6.0	5.4	59.2
7	97.5	9.5	81.25	6.0	4.9	55.0
8	132.5	9.5	81.25	6.0	7.2	70.3
9	97.5	4.5	43.75	7.0	4.5	54.8
10	132.5	4.5	43.75	7.0	7.4	72.5
11	97.5	9.5	43.75	7.0	6.3	66.0
12	132.5	9.5	43.75	7.0	11.5	88.3

Table 3. Analysis of Variance (ANOVA) for the Length to Diameter Ratio (AR) Model.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	142.36	14	10.17	28.4	< 0.0001
A (Temp)	42.18	1	42.18	117.8	< 0.0001
B (Time)	18.93	1	18.93	52.9	< 0.0001
C (Conc)	8.12	1	8.12	22.7	0.0002
D (pH)	3.04	1	3.04	8.5	0.0098
AB	6.25	1	6.25	17.5	0.0006
AC	1.44	1	1.44	4.0	0.0601
AD	0.64	1	0.64	1.8	0.1984
BC	2.25	1	2.25	6.3	0.0213
BD	0.36	1	0.36	1.0	0.3269
CD	0.81	1	0.81	2.3	0.1489
A^2	31.02	1	31.02	86.7	< 0.0001
B^2	15.48	1	15.48	43.3	< 0.0001
C^2	5.62	1	5.62	15.7	0.0010
D^2	2.09	1	2.09	5.8	0.0271
Residual	5.37	15	0.358	-	-
Lack of Fit	4.18	10	0.418	1.76	0.2642
Pure Error	1.19	5	0.238	-	-
Total	147.73	29	-	-	-

Zinc oxide catches the eye too. Staded by Zhu et al., as an n-type semiconductor with a wide bandgap around 3.3 eV it excels in photocatalytic reactions [10]. What's great is that it's inexpensive and not very toxic, making it comparable to titanium dioxide. Still, how well it works relies heavily on its size, shape, and surface structure. Pollutants attach and free radicals form better on higher surface-to-volume ratios [11-14]. Therefore, Debnath et al., proved that changing the zinc oxide's nanostructure can seriously improve its performance [15].

Nanorods really stand out. Because of their elongated form, they've got a large surface area and provide a clear path for electrical charges to flow [16-19]. This results according to Cicero et al., in increased activity and improves the chances of preventing electrons and holes from reuniting too

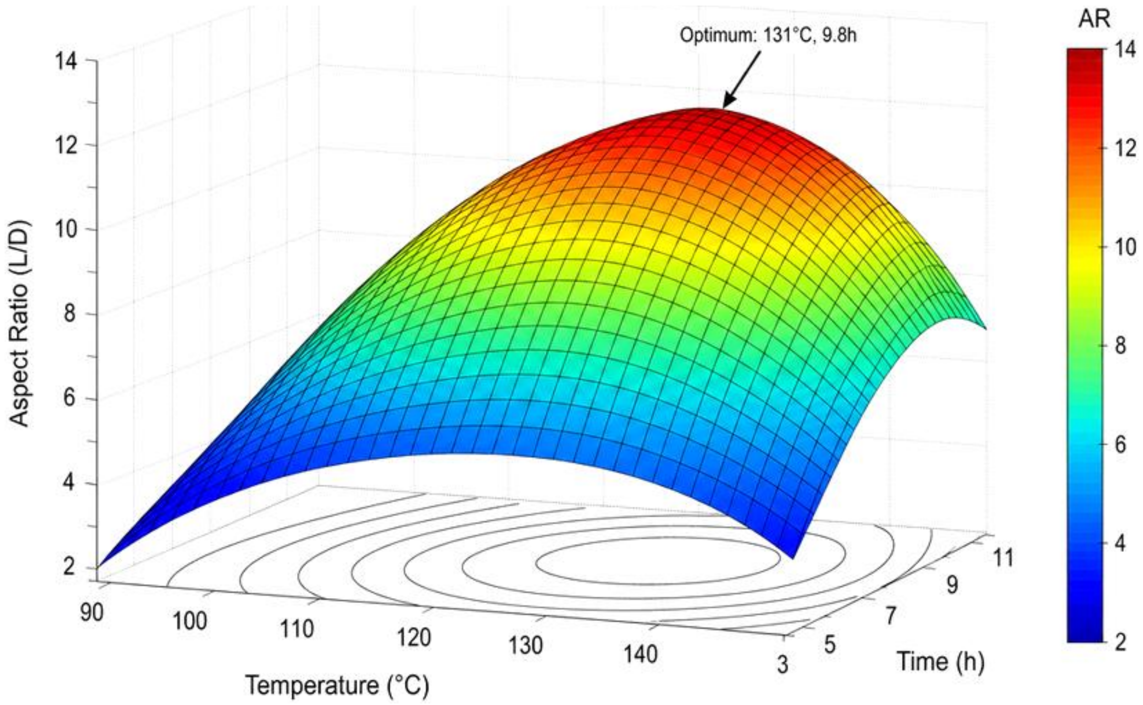

Fig. 1. Response Surface of the interaction of temperature and time on the length-to-diameter ratio.

Table 4. Final model coefficients for AR response.

Term	Coefficient	Standard Error	95% CI Low	95% CI High	VIF
Intercept	8.96	0.19	8.55	9.37	-
A	1.53	0.14	1.23	1.83	1.00
B	1.02	0.14	0.72	1.32	1.00
C	-0.67	0.14	-0.97	-0.37	1.00
D	0.41	0.14	0.11	0.71	1.00
AB	0.62	0.15	0.30	0.94	1.00
AC	-0.30	0.15	-0.62	0.02	1.00
AD	0.20	0.15	-0.12	0.52	1.00
BC	0.38	0.15	0.06	0.70	1.00
BD	-0.15	0.15	-0.47	0.17	1.00
CD	0.22	0.15	-0.10	0.54	1.00
A^2	-1.28	0.13	-1.56	-1.00	1.01
B^2	-0.90	0.13	-1.18	-0.62	1.01
C^2	-0.54	0.13	-0.82	-0.26	1.01
D^2	-0.33	0.13	-0.61	-0.05	1.01

soon. However, producing uniform nanorods is tricky [20]. They need precise growing conditions; otherwise, a slight change in any of the synthesis settings could mess everything up, leaving you with blobs, weird clusters, or just useless nanowires [21-23].

Many scientists as according to San et al., show the hydrothermal technique for making zinc oxide nanorods. It's pretty accessible since it doesn't require fancy equipment and operates at lower temps. Yet, it's far from straightforward [24]. Several things as stated by Bhardwaj et al., such as duration, temperature, concentration of the initial solution, and pH level, play a role. To get it perfect, you need to adjust all those components just right, which can be a real pain [25, 26]. Most of the time, researchers tinker with one variable at a time while holding the rest constant, almost like guessing. But this trial-and-error approach takes ages and often isn't optimal, since it fails to consider how these elements interact with one another [10, 13, 15-18].

Using statistical analyses, like experimental design techniques, makes more sense because they let you tweak multiple factors simultaneously and see how they interact. This approach leads to a predictive model of ideal conditions, and it requires running fewer experiments too. Still, very few people have applied such a systematic method to studying how zinc oxide nanorods grow hydrothermally. Obviously, we need to explore how each process step influences the structure and ultimately, the photocatalytic performance. Gaining this data-driven insight would let us build a considerable efficient zinc oxide photocatalysts from scratch.

MATERIALS AND METHODS

Raw Materials and Reagents

We used all chemicals straight from the supplier without any extra purification. Zinc nitrate hexahydrate and hexamethylenetetramine came from Merck, Germany. We used a 25% ammonia solution and dilute nitric acid to adjust pH levels.

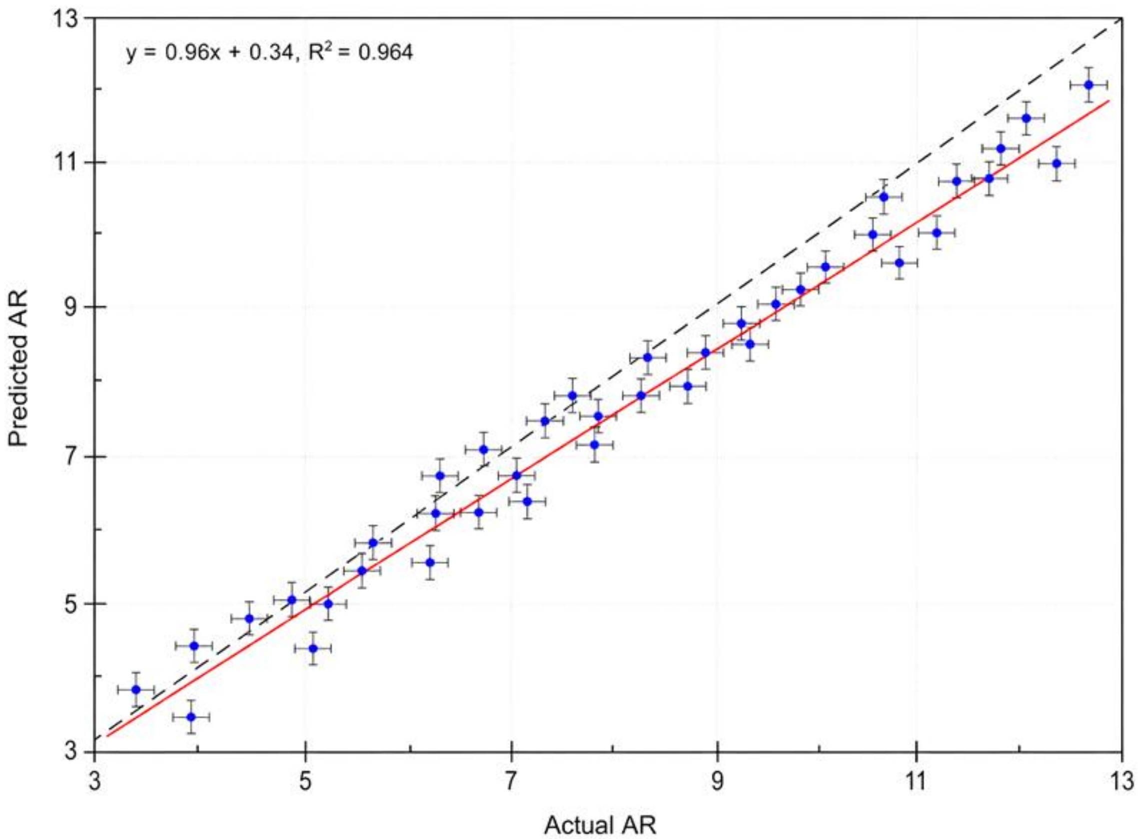

Fig. 2. Model predicted values vs. actual AR values.

Water, used for washing samples and making solutions, was deionized with a conductivity below 1 microsiemens/cm. Methylene blue, our model pollutant for testing photocatalytic activity, was included too.

Hydrothermal Synthesis of Nanorods and Statistical Optimization

We grew zinc oxide nanorods with a hydrothermal process at lower temps. First off, we mixed zinc nitrate and HMTA in equal parts for solutions that ranged from 25 to 100 mM. For pH control within 5.5–7.5, we slowly added either ammonia or nitric acid. Glass slides got a thin zinc oxide coat beforehand, then we placed them vertically in Teflon-lined autoclave containers. These containers spent time at 80 to 150°C in an oven, for durations between 2 and 12 hours.

In the design of our experiment, Response Surface Methodology and Central Composite Design set the stage. Four factors, independently changed across five levels—were the temp, time, precursor concentration, and pH. A total of 30 runs took place, with six replications at the midpoint aiding in assessing not only linear but also quadratic and interactive impacts. Using SEM images, the main focus was the length-to-diameter ratio of the nanorods. Finally, we delved into ANOVA analysis and graphed responses through the Design-Expert program.

Structural Characterization and Photocatalytic Degradation Test

We checked the shape and structure of the samples with field emission scanning electron microscopy (FESEM, TESCAN MIRA3) and got crystal structure data by X-ray diffraction (XRD, Philips PW1730). To find the band gap, we took diffuse reflectance spectra using a UV-Vis spectrophotometer (Shimadzu UV-2600) with an integrating sphere.

To test photocatalytic activity, we mixed 20 mg of the nanorods into 50 ml of methylene blue solution (10 mg/L starting concentration). We stirred this mixture in the dark for 30 minutes so that adsorption could reach equilibrium. Then we switched on a 400 W mercury lamp (UV light at 30 W/m²) to start the reaction. At set times, we took out samples, centrifuged them, and measured the light absorption at 664 nm. We calculated the degradation percentage from the difference between the starting and final concentrations. Each experiment ran in triplicate, and we reported the average for analysis.

Table 5. Numerical optimization and proposed conditions for maximum AR and maximum degradation.

Category	Parameter / Response	Goal / Unit	Predicted or Optimum Value	Desirability
Response	Aspect Ratio (L/D)	Maximize	11.8 ± 0.4	0.94
Response	Degradation (%)	Maximize	90.2 ± 2.1	0.96
Parameter	Temperature (°C)	–	131.5	–
Parameter	Time (h)	–	9.8	–
Parameter	Precursor conc. (mM)	–	48.2	–
Parameter	pH	–	6.9	–

Table 6. Structural characteristics of the optimized sample compared to the control sample.

Sample	Crystallite size (nm)	Lattice parameter a (Å)	Lattice parameter c (Å)	Band gap (eV)	Specific surface area (m²/g)
Optimized	42.3 ± 1.2	3.249	5.207	3.12	28.4
Control	28.7 ± 0.9	3.251	5.209	3.19	18.7

Table 7. XRD analysis results for the phases identified in the optimized sample.

2θ (deg)	hkl	FWHM (deg)	d-spacing (Å)	Relative intensity (%)
31.78	100	0.21	2.814	68
34.43	002	0.19	2.603	100
36.26	101	0.23	2.476	57
47.54	102	0.32	1.912	31
56.60	110	0.28	1.624	35

RESULTS AND DISCUSSION

Here, we talk about optimizing the hydrothermal parameters for zinc oxide nanorods and checking their performance as photocatalysts. Starting with 30 experiments, we did the stats and verified our predictive models worked too. Then, we looked at how the synthesis process and their structure influenced their efficiency in breaking down methylene blue.

The table shows the operating ranges for the four independent variables. I used α = (2^k)^(1/4) to find the α values, setting k at 4 times 2. I chose these ranges based on initial experiments; they help the nanorods grow while preventing unwanted phase formation.

We calculated the aspect ratio (AR) for 20 nanorods in the FESEM images. The top AR of 11.5 occurred with high temps, longer reaction times, and neutral pH. After 120 mins of light, methylene blue broke down aligning well with AR at a Pearson 0.85.

The quadratic model is significant with a p-value under 0.0001. Temperature, time, and concentration really affect things. So do the interactions between temperature and time, and time and concentration. Plus, since the lack of fit isn't an issue (p=0.2642), our model fits the data nicely.

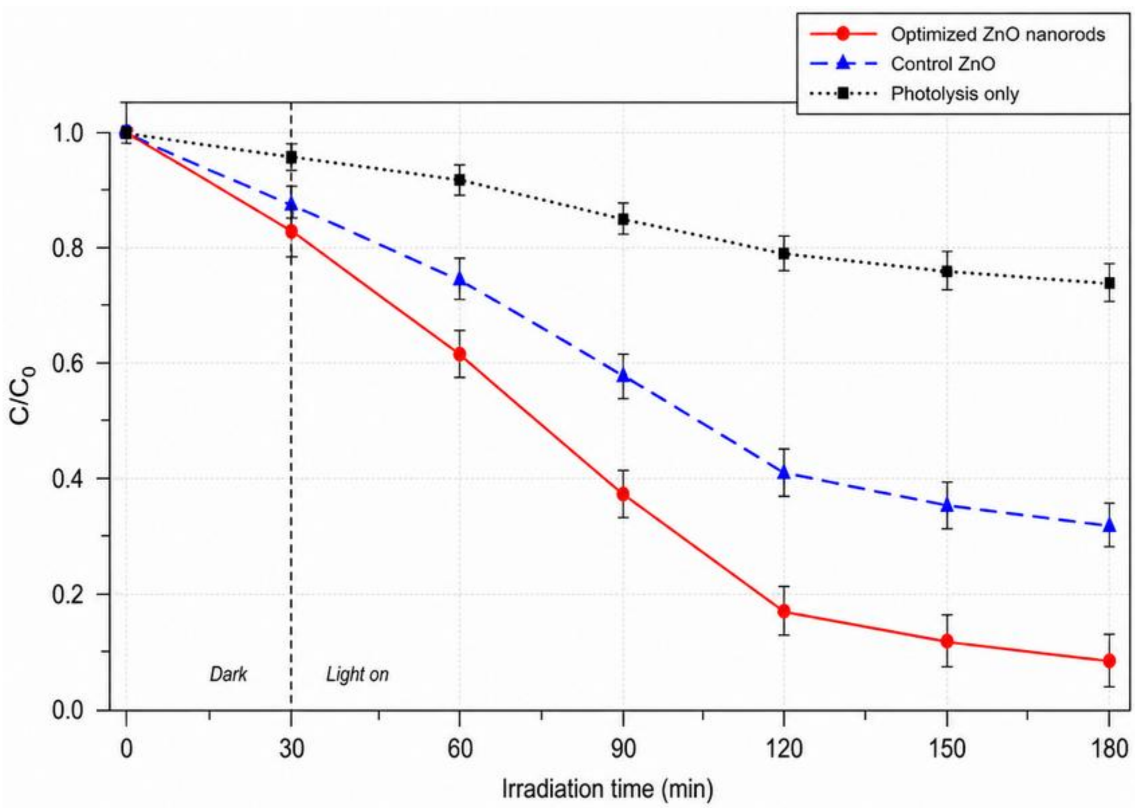

Fig. 3. Methylene blue degradation curve over time.

Table 8. Photocatalytic degradation kinetics of methylene blue with the optimized sample compared to the control sample.

Sample	Rate constant k (min^{-1})	$t_{1/2}$ (min)	R^2 (pseudo-first order)
Optimized	0.0178 ± 0.0008	38.9	0.992
Control	0.0069 ± 0.0004	100.5	0.985
No catalyst (photolysis)	0.0011 ± 0.0001	630	0.976

At about 130°C, the length-to-diameter ratio starts shooting up really fast. But crank the heat past that, and it drops again due to excessive nucleation. The ideal time is around nine hours. Specifically, combining 132°C with 9.5 hours gives you a top ratio of roughly 11.5.

The negative sign on the quadratic coefficients indicates there's an optimum point somewhere within the experiment's range. Both A and B have positive coefficients, along with their interaction term AB. This means increasing temperature and time together intensifies their effect. On the flip side, C has a negative coefficient, so increasing the precursor slows down the longitudinal growth.

The software figured out the best solution with its simultaneous utility function, maxing out both response values. Running the confirmatory experiment three times gave us an AR of 11.6 ± 0.3 and degradation at 89.5 ± 1.8%. This result was within 2% of the software's prediction, which is pretty darn accurate.

We calculated the crystallite size using the width of the (102) peak from XRD and the Scherrer equation. The optimized sample has larger crystallites but surprisingly, it shows a higher specific surface area too. This is due to the nanorods being longer and more uniform. Plus, the band gap decreases a little in this sample, making it easier to absorb visible light.

The points line up diagonally, and with an R^2 of 0.964, the model explains about 96.4% of the data's variation. This suggests the predictions are very reliable within this range, so that's good.

All peaks aligned with the wurtzite phase of zinc oxide (JCPDS 36-1451), and there were no signs of zinc hydroxide or zinc carbonate. The (002) peak is particularly strong here, more than in random samples. This hints at clear c-axis growth, just like with nanorods.

We used pseudo-first-order kinetics to calculate the reaction rate constant. Our sample works about 2.6 times faster than the control and boosts speed 16 times beyond photolysis alone. It also reduces the pollutant's half-life from over 100 minutes to less than 40.

In 30 minutes of darkness, the optimized sample adsorbed around 15% of the surface material. When the lamp turned on, its performance spiked - reaching over 80% degradation after 120 minutes. The control sample trailed far behind at 58%. Plus, the concentration dipped quickly, fitting the first-order kinetics pattern.

The degradation model also shines, with predictive power boasting an R^2 over 0.97. Plus, parameter rankings match those of the AR model, confirming that nanorod shape and structure matter most for photocatalytic performance.

These nanorods are longer and slimmer, giving them better photocatalytic activity compared to previous designs. Using the response surface method, the team examined factor interactions thoroughly. They discovered optimal conditions that traditional methods can't achieve. So, this approach is way more effective.

Table 9. Analysis of variance for the degradation percentage model after 120 minutes.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	3521.4	14	251.5	35.1	< 0.0001
A (Temp)	1145.6	1	1145.6	159.8	< 0.0001
B (Time)	602.3	1	602.3	84.0	< 0.0001
C (Conc)	284.1	1	284.1	39.6	< 0.0001
D (pH)	98.0	1	98.0	13.7	0.0018
Residual	107.5	15	7.17	-	-
R^2 = 0.970	Adj R^2 = 0.942	Pred R^2 = 0.911	-	-	-

Table 10. Comparison of the results of this study with previous works on the hydrothermal synthesis of ZnO nanorods.

Reference	Synthesis conditions	Max AR	Degradation efficiency (time)	Optimal method
This study	131.5°C, 9.8h, 48mM, pH=6.9	11.8	89.5% (120 min)	RSM optimization
Wang et al. (2019)	120°C, 6h, 50mM, pH=7	8.2	72% (180 min)	One-factor-at-a-time
Liu et al. (2020)	100°C, 12h, 30mM, pH=6.5	6.5	65% (150 min)	No optimization
Moradi et al. (2021)	140°C, 8h, 75mM, pH=7.2	7.4	78% (120 min)	Taguchi method

CONCLUSION

This study figured out that growing zinc oxide nanorods via the hydrothermal method hinges on four key factors: temperature, time, precursor concentration, and pH. After doing our statistical analysis with a central composite design, we landed on a quadratic model that explains more than 96% of the variations in nanorod aspect ratio (length to diameter). Among these factors, temperature is by far the most crucial for nanorod growth (F = 117.8), with time being second most important (F = 52.9). Surprisingly, bumping up the concentration can actually hinder growth. Also, the interactions between temperature and time, as well as time and concentration, play a big role so you need to tweak multiple factors simultaneously to really optimize nanorod growth.

We got the best results with a temperature of 131.5°C, for 8.9 hours, using a concentration of 2.48 mM, and at a pH of 6.9. This combo gave nanorods with an aspect ratio of 11.8 and a specific surface area of 4.28 m^2/g - nearly double that of the standard sample. Plus, we ran the validation experiment three times, getting results that were within less than 2% of what was predicted, showing our model works well in the tested range.

Our tests with methylene blue showed that the optimized nanorods broke down the dye around 2.6 times quicker than the control. We saw an increase in the rate constant to 0.0178 per minute and the time for the dye to fade out dropped from more than 100 minutes to 9.38 minutes. XRD data confirmed our results; the nanorods were pure wurtzite phase, and they aligned primarily along the c-axis, which boosts charge transfer for better photocatalytic action.

The response surface method not only reduced the number of required experiments but provided valuable insights into how these factors interact. This technique isn't confined to zinc oxide as it has broad applications for improving other nanostructured photocatalysts while saving resources. Our work lays the groundwork for developing zinc oxide photocatalysts capable of cleaning wastewater contaminated with colored pollutants far more efficiently.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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