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Statistical Optimization of Zinc Oxide Nanorod Synthesis for Photocatalytic Degradation of Methylene Blue

(Pengoptimuman Statistik Sintesis Nanorod Zink Oksida untuk Degradasi Fotopemangkinan Metilena Biru)

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ABSTRACT

In this work, synthesis process parameters of Zinc Oxide nanorods (ZnO NRs) photocatalyst is optimized using Taguchi Method to obtain the highest degradation rate of Methylene Blue dye, MB. The Taguchi L27 (3^8) orthogonal array technique was used to determine the optimum conditions for the synthesis of the nanostructured photocatalyst. Eight important synthesis process parameters were chosen in the analysis while the effects of the parameters were studied using signal-to-noise (S/N) ratio analysis using minitab-16. The ZnO NRs photocatalyst was synthesized via solution process route based on the parameters obtained from the layout of the orthogonal arrays. The optimized synthesized nanorods was then characterized using field emission scanning electron microscope (FESEM), X-ray diffraction (XRD), photoluminescence (PL), ultraviolet-visible near-infrared (UV-VIS-NIR), and Raman spectroscopies while the photodegradation of MB was determined by UV-VIS spectrum analysis under ultraviolet light irradiation. The results show that ZnO NRs with hexagonal wurtzite structure and bandgap energy of 3.25 eV have been obtained. The Taguchi analysis based on simulated experimental runs predicted the highest MB degradation percentage of 17.12% that can be achieved under optimum process conditions. Meanwhile, experimental photocatalytic degradation of MB using ZnO NRs synthesized under the same optimum condition achieved a degradation percentage of 17.27%, which deviates only 0.88% from the predicted value. This analysis could give an approach to optimize the synthesis process to ensure the good performance of nano-photocatalyst for the photodegradation of organic contaminations in industrial wastewater in a short time and cost-effective process.

Keywords: Hydrothermal; MB degradation; synthesis optimization; Taguchi; ZnO NRs photocatalyst

ABSTRAK

Dalam kertas ini, parameter proses sintesis bagi fotomangkin nanorod Zink Oksida (ZnO NRs) dioptimumkan menggunakan Kaedah Taguchi untuk mendapatkan kadar degradasi tertinggi untuk pewarna metilena biru, MB. Teknik tatasusunan ortogon Taguchi L27 (3^8) digunakan untuk menentukan keadaan optimum untuk sintesis fotomangkin berstruktur nano. Lapan parameter proses sintesis penting telah dipilih dalam analisis manakala kesan parameter dikaji menggunakan analisis nisbah isyarat-ke-bunyi (S/N) menggunakan minitab-16. Fotomangkin ZnO NRs telah disintesis melalui laluan proses penyelesaian berdasarkan parameter yang diperoleh daripada susun atur tatasusunan ortogon. Mikroskop elektron pengimbasan pelepasan medan (FESEM), pembelauan sinar-X (XRD), fotoluminesen (PL), inframerah-dekat sinar boleh nampak ultralembayung (UV-VIS-NIR) dan spektroskopi Raman manakala fotodegradasi MB ditentukan oleh analisis spektrum UV-VIS di bawah penyinaran cahaya ultralembayung. Keputusan menunjukkan bahawa ZnO NRs dengan struktur wurtzite heksagon dan tenaga celah jalur sebanyak 3.25 eV telah diperoleh. Analisis Taguchi berdasarkan larian uji kaji simulasi meramalkan peratusan degradasi MB tertinggi sebanyak 17.12% boleh dicapai di bawah keadaan proses optimum. Sementara itu, degradasi fotokatalitik uji kaji MB menggunakan ZnO NRs yang disintesis di bawah keadaan optimum yang sama mencapai peratusan degradasi sebanyak 17.27%, yang menyimpang hanya 0.88% daripada nilai yang diramalkan. Analisis ini boleh memberi pendekatan dalam mengoptimumkan proses sintesis untuk memastikan prestasi nano-fotomangkin yang baik untuk fotodegradasi pencemaran organik dalam air sisa industri dalam masa yang singkat dan kos efektif.

Kata kunci: Degradasi MB; fotomangkin ZnO NRs; hidroterma; pengoptimuman sintesis; Taguchi

Introduction

Azo dyes cover 70% of the dyes used in textile processing, such as Methylene Blue and Methyl Orange, which are complex in structure. Specifically, large quantities of liquid waste containing organic and inorganic compounds are produced by the textile industry (Elliott et al. 2002). After processing 12-20 tons of textiles, approximately 1,000-3,000 m^3 of effluents are released every day (Al-Kdasi et al. 2004). The effluents contain high dye concentrations and trace metals such as Cr, As, Cu, and Zn that can harm human health, including hemorrhage, skin irritation, nausea, and ulceration of the skin (Tufekci et al. 2007).

These contaminations can be reduced through the photocatalytic activity of photocatalyst such as ZnO in the form of nanostructures (Nagaraju et al. 2017; Solis-Pomar et al. 2016). To resolve this, the high photocatalytic effect of ZnO NRs that contributes to the oxidation process of contaminated effluents is important by the generation of hydroxyl free radicals (HO*) and superoxide radical anion (O2-*) to destroy the chemical bond of an organic contaminant (Chung et al. 1992; Kestioğlu et al. 2005). This mechanism can destroy components that are not destroyed under the conventional process and destroy a wide range of organic contaminants in polluted water (Al-Kdasi et al. 2004).

ZnO has been investigated as high efficient photocatalytic material due to its high surface reactivity, higher efficiency to generate hydroxyl ions compared to TiO_2, and strong oxidation power to degrade organic compounds under UV irradiation formed photogenerated (Ong et al. 2018; Qiu et al. 2008). Besides, the synthesis of the ZnO based nanostructure is easy and low cost (Ong et al. 2018). A recent study proved that ZnO NRs acting as an efficient photocatalytic nanomaterial layer to degrade toxic organic compounds (Azzouz et al. 2018).

There are different methods to synthesize ZnO nanorods via chemical routes including reverse micelle, sol-gel, aqueous solution and biomineralization methods (Foo et al. 2014; Garcia & Semancik 2007; Zhang et al. 2007). Among all, the hydrothermal synthesis is commonly used method for growing ZnO nanorods or nanowires, due to its simplicity, low cost, simple instrumentations, and controllable growth temperatures (Kazmi et al. 2021, 2020; Rehman et al. 2016). The photocatalytic activity of the synthesized ZnO NRs affected by some factors that can be optimized to obtain the highest degradation efficiency. The optimization for the synthesis process is required to obtain the ZnO NRs with suitable diameter, roughness, length, and distribution to enhance the photocatalytic activity. However, the traditional single factor optimization method does not consider the interaction between factors.

By performing the design of experiments (DOE), optimal design of the experiment can be determined and selected. DOE consists of several phases, including planning, conducting simulated experiment runs, and analyzing experimental results using statistical methods for optimization (Seyed Mojib et al. 2013). Some classical screening designs include fractional factorial designs, Plackett-Burman, Cotter and mixed-level designs. Full factorial designs are often too expensive to run, since the sample size grows exponentially with the number of factors, which lead to high-cost and time consumption (Jankovic et al. 2021). These limitations can be overcome using experimental design technologies such as Taguchi method to provide optimum levels of affecting factors statistically. Because of its practicality, Taguchi design has become the most often used experimental design in both industry and science. Its strength comes from its effective orthogonal design-matrices with a balanced distribution of factor levels lowering the number of experimental runs necessary. This method is an efficient optimization process, systematic, low cost and time saving (Pouretedal et al. 2017). Previous studies reported the usage of Taguchi analysis in optimizing the photocatalytic degradation of dyes. The optimized parameters included are light intensity, irradiation time, pH and amount of catalyst (Devadi et al. 2014; Pouretedal et al. 2017; Shamsikasmaei et al. 2013). However, there is no report in optimizing synthesis parameters of photocatalyst for photodegradation purposes.

In this experiment, ZnO NRs were synthesized via solution process route (Aini Ayunni et al. 2021; Kazmi et al. 2021, 2020). The photocatalytic degradation of MB was conducted using the ZnO NRs that were synthesized based on the synthesis parameters provided by Taguchi L27 orthogonal array to determine the optimized synthesis parameters of ZnO NRs. The efficiency of the synthesized nanorods is investigated by the degradation rate of MB determined via UV-VIS spectrometer analysis.

Methods

The flow process description to introduce the methods is in Figure 1.

Synthesis of ZnO NRs

The initial synthesis process is the preparation of ZnO nanoparticles (NPs) that will be used as the seed layer. The seed layer solution was prepared by dissolving 0.01 M of ZnA in 20 mL methanol and heated at 60 °C for 30

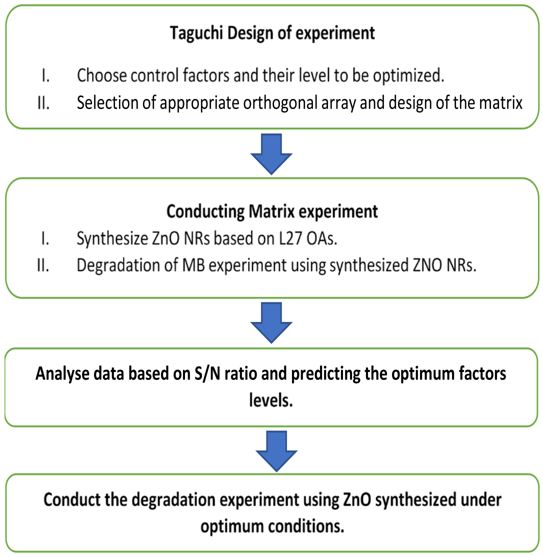

FIGURE 1. Methodology flowchart

min. 0.09 M of NaOH solution was prepared in 20 mL methanol in a separate beaker and stirred for 30 min. The NaOH was then added dropwise into ZnA solution and heated at 60 °C under vigorous stirring for 2 h to prepare stabilized ZnO NPs solution. Next, the prepared ZnO suspension was spin coated on the glass substrates at varying speeds for 30 s to obtain homogenous distribution. Then, the substrates were annealed at varying temperatures. To prepare the growing solution, 0.025 M HMTA and 0.025M $Zn(NO_3)_2$ were added into 100 mL of DI water and stirred for 2 h at room temperature. The ZnO seed-coated substrates were placed upside down in a beaker containing the growing solution. The beaker was then covered with aluminum foil and left to grow the ZnO NRs hydrothermally in the convection oven. Next, the substrates were removed from the solution, rinsed with DI water and annealed to remove any contaminants (Kazmi et al. 2020). The ZnO NRs were synthesized using synthesis process parameters provided by the layout of the orthogonal array in Table 2 and were used as photocatalysts for photocatalytic degradation of MB.

TAGUCHI'S EXPERIMENTAL DESIGN

Taguchi method is a statistical method used for optimization of experimental design. Firstly, the operating parameters were identified and then optimized in a process. The orthogonal arrays were applied to obtain a set of simulated experiments to be conducted. The orthogonal array was in the procedure to study all the parameters with lesser number of experiments. Based on the results of these experiment runs, the data was analyzed using the loss function to measure the proficiency characteristics and the optimum level of each factor will be obtained. The value of this loss function was assigned to signal-to-noise (S/N) ratio. The three sets of proficiency characteristics for analyzing the S/N ration that can be selected are nominal-the-best, larger-the-better and smaller-the-better (Pouretedal et al. 2017; Taguchi et al. 1987).

In this work, eight operating factors, such as spin coat speed, spin count, seed layer annealing temperature, seed layer annealing time, growing temperature, growing time, substrate annealing temperature, and substrate annealing time with three level variations of each were chosen to evaluate the photocatalytic performance of the nanorods on the degradation of the MB. Table 1 shows these parameters and their level. L27(3^8) OAs were used for this study and the layout is given in Table 2. The photocatalytic degradation of MB was conducted using the ZnO NRs photocatalysts that were synthesized based on layout in Table 2.

The designed factors chosen in Taguchi will be completed with the synthesis result for further analysis. In analyzing this Taguchi design, the proficiency characteristic selected for analyzing the S/N ratio is 'the larger is better' of the S/N ratio was used. The S/N ratio was calculated using the equation below.

$$\frac{S}{N} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \qquad (1)$$

where 'n' is the number of repeated experiment and y is the characteristic property.

TABLE 1. Operating factors at different levels

Factors	Level 1	Level 2	Level 3
Spin coat speed	1000 rpm	2000 rpm	3000 rpm
Spin count	1X	2X	3X
Seed layer annealing temperature	150°C	200°C	250°C
Seed layer annealing time	10 min	20 min	30 min
Growing temperature	80°C	95°C	100°C
Growing time	2 hr	4 hr	6 hr
Substrate annealing temperature	150°C	200°C	250°C
Substrate annealing time	30 min	60 min	90 min

TABLE 2. The layout of the L27 OAs

Exp No.	Spin coat speed (rpm)	Spin count (X)	Seed layer annealing temperature (°C)	Seed layer annealing time (min)	Growing temperature (°C)	Growing time (h)	Substrate annealing temperature (°C)	Substrate annealing time (min)
1	1000	1	150	10	80	2	150	30
2	1000	1	150	10	95	4	200	60
3	1000	1	150	10	100	6	250	90
4	1000	2	200	20	80	2	150	60
5	1000	2	200	20	95	4	200	90
6	1000	2	200	20	100	6	250	30
7	1000	3	250	30	80	2	150	90
8	1000	3	250	30	95	4	200	30
9	1000	3	250	30	100	6	250	60
10	2000	1	200	30	80	4	250	30
11	2000	1	200	30	95	6	150	60
12	2000	1	200	30	100	2	200	90
13	2000	2	250	10	80	4	250	60
14	2000	2	250	10	95	6	150	90
15	2000	2	250	10	100	2	200	30
16	2000	3	150	20	80	4	250	90
17	2000	3	150	20	95	6	150	30
18	2000	3	150	20	100	2	200	60
19	3000	1	250	20	80	6	200	30
20	3000	1	250	20	95	2	250	60
21	3000	1	250	20	100	4	150	90
22	3000	2	150	30	80	6	200	60
23	3000	2	150	30	95	2	250	90
24	3000	2	150	30	100	4	150	30
25	3000	3	200	10	80	6	200	90
26	3000	3	200	10	95	2	250	30
27	3000	3	200	10	100	4	150	60

MB PHOTODEGRADATION EXPERIMENT

The photodegradation rate was investigated by studying the degradation of 15 mL MB solution with molarity of 0.05mM under UV irradiation. The ZnO NRs coated glass substrate with the dimension of (2 cm × 1.6 cm) was placed in the base of the beaker containing MB solution. The distance of the glass substrate to the UV lamp was kept constant at 10 cm. The UV lamp, 18W (220-240V) has wavelength up to 368 nm was used to irradiate the substrate. The setup was in the dark cupboard to avoid other sources of lights and without any circulation or stirring of the solution. The reactor was exposed to UV irradiation for 1.5 h. This experiment is considered as preliminary test without any flow of solution or stirring. In future work, the ZnO NRs photocatalyst will be implemented in microfluidic system. The analysis of wastewater before and after the photocatalysis process have been performed using observation of methylene blue degradation according to the water and wastewater examination standard method. A double beam UV/VIS spectrophotometer was used to determine the MB concentration. Indication of MB degradation will be presented by peaks between 600 and 700 nm as the absorption of the π-system. According to Beer-Lambert Law, MB's concentration is directly proportional to its absorbance peak. This makes it possible to determine the photodegradation efficiency of MB using the following equation:

$$R = \frac{C_o - C_t}{C_o} \times 100\% \tag{2}$$

where C_o is the initial concentration of MB solution; and C_t is concentration of MB solution during irradiation.

RESULTS AND DISCUSSION

MORPHOLOGICAL AND RAMAN ANALYSIS OF THE SYNTHESIZED NANORODS

One of the synthesized ZnO NRs grown for 2 h at 100 °C under optimized conditions was characterized using FESEM. The FESEM image was used to investigate the cross-sectional of the vertical ZnO NRs on glass substrate with average diameters and heights of 81.81 ± 3 nm and 1400 ± 3 nm, respectively. The ZnO NRs covered the glass substrate with homogenous distribution as shown in Figure 2.

Figure 3 shows the Raman spectra of ZnO NRs. Peaks of ZnO appear at 94, 331, and 430 cm^{-1}, which represents 'E2 Low mode', 'E2 Low-E2 High mode' and 'E2 High vibration mode', respectively. This Raman spectrum analysis showed that ZnO NRs have been successfully grown.

The XRD pattern analysis based on (JCPS # 00-036-145) indicated that ZnO with hexagonal wurtzite structure has been successfully synthesized shown in Figure 4. The average crystallite size of the ZnO NRs was calculated to be 46.21 nm using the Scherrer equation based on diffraction line (101) at diffraction angle of 36.3°. The detailed XRD data analysis can be referred to Table 3.

The optical characteristics of ZnO NRs were investigated using UV-Vis shown in Figure 5. The absorption wavelength spectra from 350 to 800 nm were investigated. The bandgap energy of the optimized ZnO NRs was estimated using Tauc Plot to be 3.25 eV. The ZnO NRs have the predominant absorption in the UV region around 300-390 nm which corresponds to the excitations of electrons from the valence band to the conduction band with a bandgap energy of 3.25 eV.

PL spectra of the ZnO NRs at room temperature is shown in Figure 6 at the wavelength range of 350 nm to 750 nm. The first emission bands centered at approximately 397 nm and multiple peaks in the 425-500 nm region and wide peak centered at 611 nm. The first emission band is due to radiative transition of free electrons from CB to VB of ZnO, known as near band edge emission (NBE) (Xu et al. 2016). Meanwhile, the multiple emission peaks are due to the recombination process of the intrinsic defects in different levels inside the ZnO's band gap, which is known as deep level emission (DLE) (Sun et al. 2011).

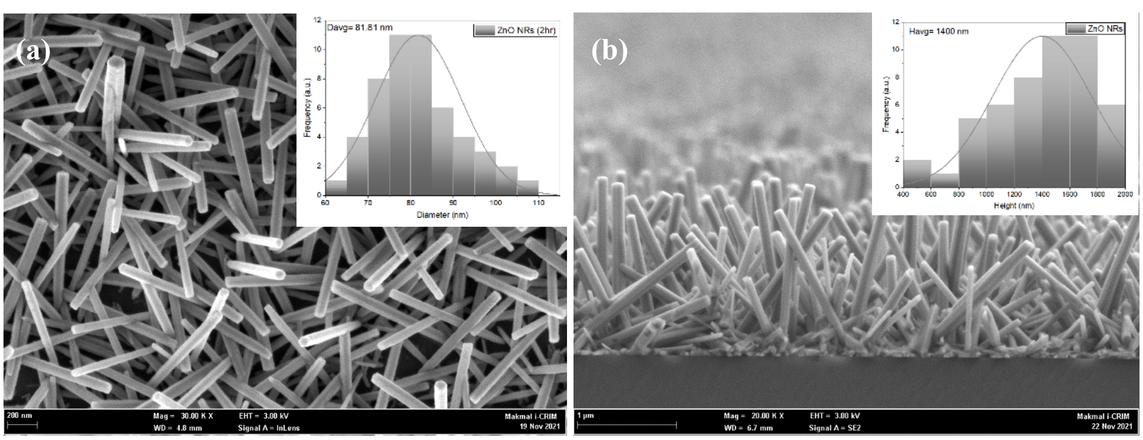

FIGURE 2. SEM image of optimized ZnO NRs (a) top view and (b) cross-section view

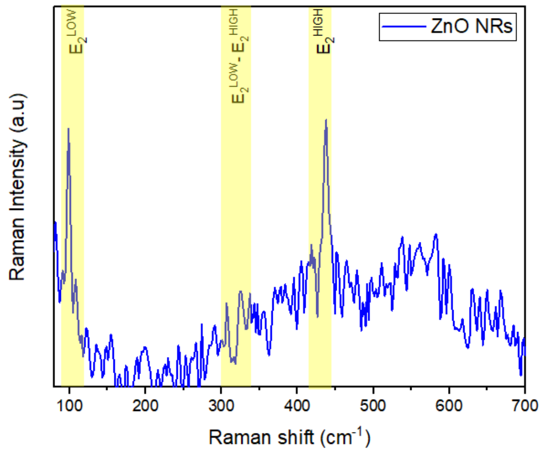

FIGURE 3. Raman Spectra of optimized ZnO NRs

TABLE 3. XRD analysis data

	2θ (101)	FWHM	d-spacing [Å]	D(nm)	Lattice a=b(Å)	Parameter c(Å)
ZnO NRs	36.22	0.189	2.476	46.21	3.2498	5.2066

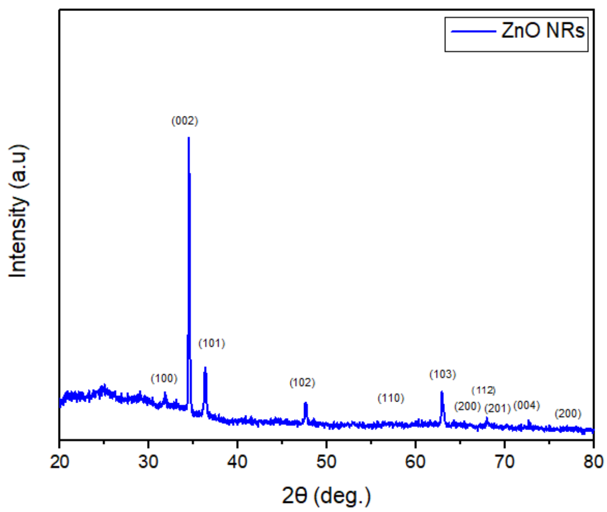

FIGURE 4. XRD patterns of optimized ZnO NRs

S/N ANALYSIS

The nanorods structure was synthesized using the chosen parameter based on the layout of the L27 AOs, photodegradation experiments were carried out accordingly and the results obtained are shown in Table 4. From the Taguchi design analysis set, the quality characteristics are determined by the term 'larger is better' that means that the results of the S/N ratio analysis with the highest photodegradation rate are targeted. The main effects plot of means and S/N ratio was obtained shown in Figure 7.

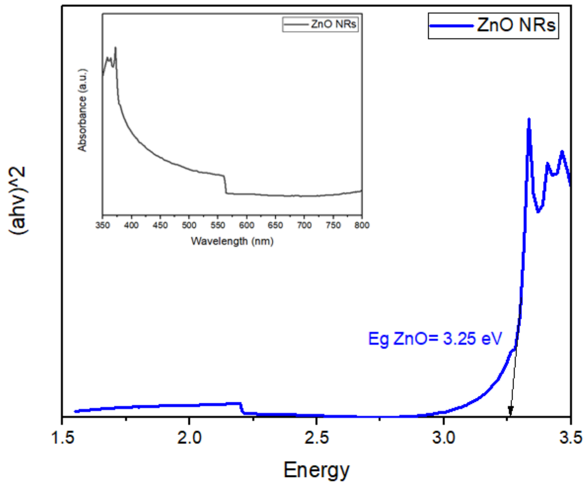

FIGURE 5. Absorption spectra for Tauc Plot Analysis and absorbance spectra (inset picture)

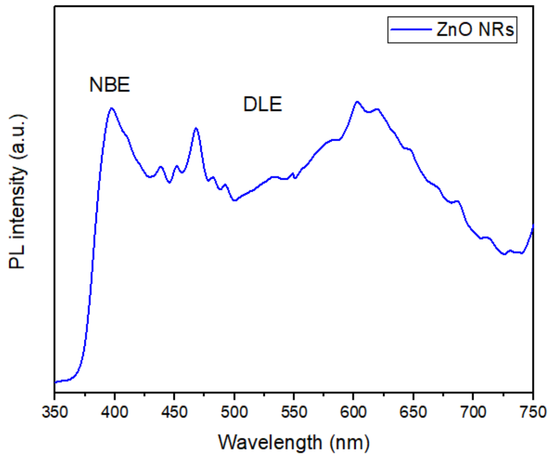

FIGURE 6. PL spectra of optimized ZnO NRs

TABLE 4. The L27 OAs photodegradation rate results

Run	rate of degradation (%)	Run	rate of degradation (%)
1	12.68	15	10.12
2	10.89	16	12.43
3	13.36	17	10.67
4	14.22	18	11.41
5	8.82	19	13.91
6	8.11	20	11.07
7	6.23	21	11.07
8	9.53	22	12.52
9	11.63	23	10.36
10	5.71	24	16.50
11	12.89	25	10.80
12	13.57	26	20.79
13	11.26	27	12.99
14	9.53		

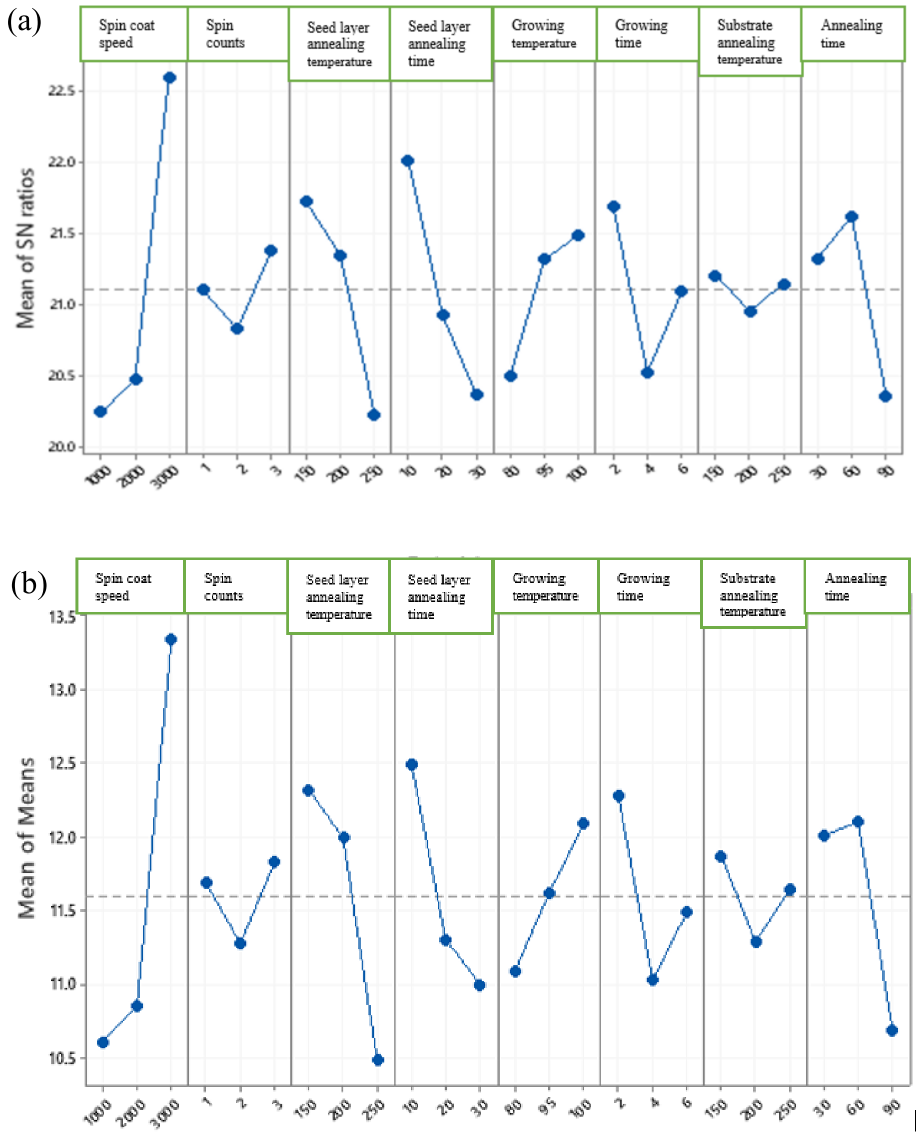

FIGURE 7. Main effects plot for (a) means, and (b) S/N ratio of photodegradation rate for optimization of ZnO NRs synthesis

The optimum level of each factor contributes to the highest photocatalytic activity of ZnO NRs, thus, the highest photodegradation rate will be obtained. It is clearly seen that the optimum spin coat speed is 3000 rpm. This is because the faster the rotational speed will provide a more homogenous distribution of the ZnO seed layer on the glass substrate. Thus, forming a homogenous distribution of ZnO NRs on glass substrate because the formation of the ZnO NRs is dependent on the initial size of the seed layer (Rehman et al. 2016). Next, the spinning count is also important as it affects the formation of the nanorods. Based on the previous study (Kim et al. 2009), the ordering in nanorods decreases as the spinning count increases. The highest spin count, which is 3 times is suitable as there is more ZnO NRs will be formed, and more reactions happen between ZnO and MB, thus increasing the absorption of MB molecules for higher degradation.

The purpose of annealing of seed layer before ZnO NRs are grown is to remove organic residuals. Based on reported study, the orientation of the synthesized nanorods was severely disrupted when the annealing temperature was raised above 200 °C (Kim et al. 2009). Thus, the lowest temperature which is 150 °C with shorter annealing time, 10 min is the most optimum in annealing the seed layer.

For the hydrothermal growth, a shorter growth time will give a shorter height of ZnO NRs. Based on simulation by Xia (2018) on how the height influences the light intensity of the micropillar, results show the higher the micropillar leads to the low intensity of light on the surface and the decrease in photocatalytic activity. Thus, a shorter growth time, which is 2 h is the optimum condition as it leads to a shorter height of nanorods. Next, a higher generation of current density can be obtained with a high hydrothermal growing temperature which for our case is 100 °C. However, at the temperature above 130 °C, vaporization of Zn^{2+} will occur as it is reaching the boiling point of the precursor and resulting in the inhibition growth of the ZnO seed (Mohd Fudzi et al. 2018).

Lastly, for the annealing of the substrate, the crystal size of the nanorods decreased as the annealing temperature increased as a response to the thermal energy generated by the annealing process. It is concluded that the root mean square roughness of ZnO nanorod decreased with the increase of annealing temperature (Kareem 2020). If the surface is rougher, then, it has larger surface area in comparison to smooth surface and less reflection, so more photon is absorbed in this case. In conclusion, a higher degradation rate will be observed if the annealing temperature of the ZnO NRs is lower, which is 150 °C.

Based on this analysis, the ZnO NRs synthesized under the parameters of spin coat speed (3000 rpm), spin count (3X), seed layer annealing at (150 °C for 10 min), growing at (100 °C for 2 h) and substrate annealing at (150 °C for 60 min) obtained the highest S/N ratio indicating the optimum process conditions for the synthesize of ZnO. This prediction allows us to get the highest photodegradation rate of MB solution.

TABLE 5. Response table for the signal to noise ratios

Level	Spin coat speed	Spin count	Seed layer annealing temperature	Seed layer annealing time	Growing temperature	Growing time	Substrate annealing temperature	Substrate annealing time
1	20.25	21.10	21.73	21.70	20.50	21.38	21.21	21.02
2	20.48	20.83	21.05	20.93	21.02	20.53	20.96	21.62
3	22.28	21.08	20.24	20.38	21.49	21.09	20.85	20.37
Delta	2.03	0.27	1.49	1.33	0.99	0.85	0.36	1.26
Rank	1	8	2	3	5	6	7	4

TABLE 6. Analysis of variance for noise ratios

Source	DF	Seq SS	Adj SS	Adj MS	F	P-Value
Spin coat speed	2	41.121	41.121	20.5603	1.50	0.269
Spin count	2	1.509	1.509	0.7545	0.06	0.947
Seed layer annealing temperature	2	17.158	17.158	8.5788	0.63	0.554
Seed layer annealing time	2	11.203	11.203	5.6016	0.41	0.675
Growing temperature	2	4.514	4.514	2.2569	0.16	0.850
Growing time	2	7.188	7.188	3.5941	0.26	0.774
Substrate annealing temperature	2	1.531	1.531	0.7656	0.06	0.946
Substrate annealing time	2	11.205	11.205	5.6027	0.41	0.675
Residual Error	10	136.983	136.983	13.6983		
Total	26	232.413				

The ranking data and contribution factor have been determined in Tables 5 and 6. Based on the S/N ratio, the order of significance of factors is as follows: spin coat speed > seed layer annealing temperature > seed layer annealing time > substrate annealing time > growing temperature > growing time > substrate annealing temperature > spin count. Multiple linear regression (MLR) was applied to validate the relationship between a dependent variable and independent variables. The regression equation below was obtained in Minitab-16 to predict the degradation rate. Based on this regression, the degradation rate under optimum conditions is predicted to be 17.12%.

$$Y=12.15 + 0.001364\ X_1 + 0.074\ X_2 - 0.0183X_3 - 0.0747X_4 + 0.0467X_5 - 0.195X_6 - 0.0023X_7 - 0.0219X_8 \quad (3)$$

where Y is the dependent variable of rate of degradation and X is independent variables; parameters of spin coat speed (X_1), spin count (X_2), seed layer annealing temperature (X_3), seed layer annealing time (X_4), growing temperature (X_5), growing time (X_6), substrate annealing temperature (X_7), and substrate annealing time (X_8). The values of coefficients were determined by the method of least squares. The rate of degradation can be predicted for operational factors in selected ranges. A linear model with the regression coefficient (R^2) of 31.73% was obtained by the multiple linear regression analysis shown in Table 7. The changes in the predictor's value are due to changes in the response variable.

Finally, to validate the S/N analysis, ZnO NRs was synthesized under optimum condition and a degradation experiment was conducted. Figure 8 shows the result of the MB photodegradation done under the optimum condition and a degradation rate of 17.27% was achieved. The degradation rate is calculated based on equation (1). The experimental MB degradation rate obtained is higher than the value predicted by the Taguchi analysis, which is 17.12%. This is due to other factors, such as environment effect, sample conditions, and solution age that are not included in the analysis but significantly influences the growth of the ZnO nanorods. Based on our comparison with non-optimized parameters we found that this optimum value resulted in significant improvement in the degradation of MB.

TABLE 7. Model summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.96898	31.73%	1.39%	0.00%

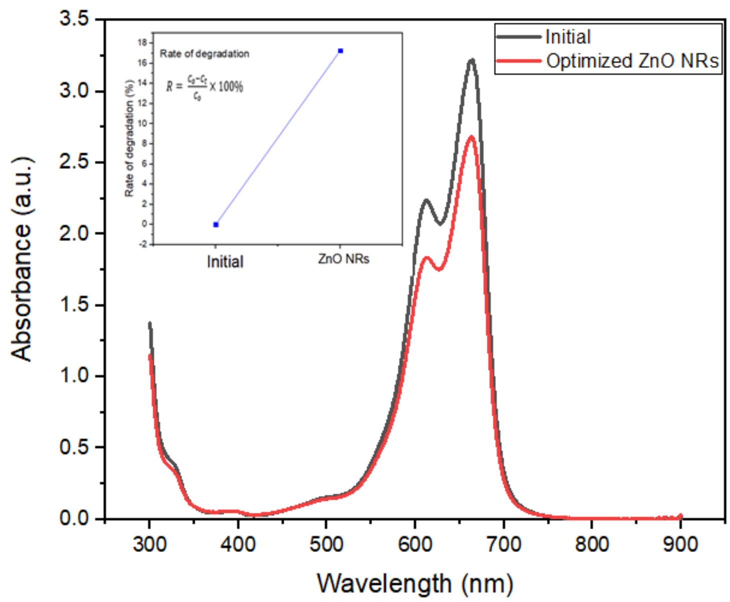

FIGURE 8. MB photodegradation under optimum conditions

Conclusions

In this study, the Taguchi experimental design has been done to determine the optimum parameters for the synthesis process of ZnO nanorods (NRs) photocatalyst to obtain the highest degradation rate of MB. The NRs on glass substrate were synthesized hydrothermally. The optimum parameters obtained are spin coat speed 3000 rpm, spin count 3X, seed layer annealing at 150 °C for 10 min, growing at 100 °C for 2 hours and substrate annealing at 150 °C for 60 min. Based on S/N ratio analysis using Minitab system software, a predicted degradation rate of 17.12% was achieved under optimum conditions. Based on the experimental photodegradation of MB under optimum conditions, 17.27% of MB was degraded which deviates 0.88% from the predicted rate. The analysis will be possibly more accurate if more levels and factors are included. It can be concluded that the Taguchi method is a useful tool that has succeeded to determine the optimum conditions for synthesizing the ZnO NRs photocatalyst. Through this method, a large number of experiments can be reduced that finally greatly reducing the cost and time for optimization purposes.

Acknowledgements

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