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Research Article

Optimizing Microwave Synthesis Parameters of Amorphous MoS_2/CNT Nanocomposites for Enhanced Hydrogen Evolution using the Taguchi Method

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ABSTRACT

Conventional optimization studies that involve changing one parameter while keeping the others constant are frequently regarded as time-consuming and expensive. The Taguchi method, however, is a simpler and equally effective method for optimizing multiple operational variables in the statistical design of experimental methods. In this study, the Taguchi optimization method was used to determine the microwave synthesis conditions of amorphous molybdenum disulfide/carbon nanotubes (MoS_2/CNTs), which included the amount of CNTs, reaction temperature, reaction time, microwave power, and the ratio of Mo source to S source (Mo:S) in the precursor, for the best performance output - Tafel slope. Tafel analysis is an important step in the screening process for all energy conversion electrocatalysis because it provides information on activity (via exchange current density) and reaction mechanism (via Tafel slope). The findings demonstrate that amorphous MoS_2/CNTs possess both catalytic activity and stability within the voltage range of -220 to -230 mV (vs. NHE). The current density at a voltage of -350 mV (vs. NHE) is -8.94 mA/cm^2, and the Tafel slope is measured to be 102 mV/dec.

Key words: Expermental design, Taguchi, MoS2/CNTs, Tafel slope, microwave synthesis, HER

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INTRODUCTION

Optimization studies that follow the conventional approach of altering one parameter while keeping the rest unchanged are often considered to be both time-consuming and costly. The Taguchi method, however, is a simpler and equally effective method for optimizing multiple operational variables in statistical design of experimental methods. Taguchi experimental design reduces costs, improves quality, and provides trustworthy design solutions. With the Taguchi method, multiple factors can be optimized simultaneously, and more quantitative data can be extracted from fewer experimental trials than with other methods. Taguchi's experiment design is an effective tool for modeling and analyzing the impact of control factors on performance output. This method has been applied to nanomaterials for a limited number of syntheses, including carbon nanotubes[1], graphene/cotton nanocomposite[2] or other nanoparticles such as TiO_2[3].

In this study, Taguchi experimental planning is carried out to determine the optimal synthesis conditions of MoS_2/CNTs for enhancing electrochemical performance in the HER catalysis application. The Tafel coefficient is a key parameter used to evaluate the catalytic efficacy of a catalyst for hydrogen evolution reaction (HER)[4]. A smaller Tafel slope value further indicates a higher catalytic activity[5–7]. Tafel slope is chosen as the output data in the Taguchi method, which is used to define the optimal synthesis conditions for amorphous MoS_2/CNTs. A lower Tafel slope and a higher exchange current density are expected for a better electrocatalyst. Figure 1 illustrates the five fundamental steps of the Taguchi method and each step is detailed as follow:

Step 1 - Define the number of factors to be studied and the number of levels for each factor

For optimizing the synthesis of amorphous MoS_2/CNTs via the direct-two-pot-dispersion (D2PD) process, the five factors are as follows:

- Microwave power (W) – P1
- Reaction time (min.) – P2
- Ultrasonication temperature (°C) – P3
- Amount of f-CNTs (mg) – P4
- The ratio of precursor mixture (AHM+TU) to EG solvent $\sum m_{(AMH+TU)}$:V_{EG} ratio (g/mL) – P5

The number of levels considered for each factor is 4. The factors and levels are detailed in Table 1 for the microwave synthesis process.

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Step 2 - Define the response value of experiment

In this work, the response values considered is the Tafel slope value (from Linear Sweep Voltammetry (LSV) results) for amorphous MoS_2/CNTs synthesis.

Step 3 – Select an appropriate orthogonal array (OA)

Create orthogonal arrays for the parameter design based on the number of factors and the number of levels for each factor. Because this study has five factors with four levels each to consider, we determine that the appropriate orthogonal array is L'16[8].

Step 4 – Run experiments

Conduct the experiments following the designed matrix experiment L16, and the experimental conditions are presented in Table 2.

Step 5 – Analyze the data to identify the optimal level

In the synthesis of amorphous MoS_2/CNTs, the values for the Tafel slope should decrease for better catalytic activity. The formula for the "lower-the-better" normalization criteria considered is chosen as follows:

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

EXPERIMENTAL PROCEDURES

Synthesis of nano MoS_2/CNTs

To prepare the precursor solution, 2.47 g of ammonium heptamolybdate (AHM) and 4.56 g of thiourea (TU) were added to ethylene glycol (EG) solvent. The ratio of AHM to TU in the precursor solution was maintained at 1:30. The amount of EG solvent used was adjusted to vary the ratio of the precursor mixture (AHM + TU) to EG solvent. The mixture was magnetically stirred at 50 °C for 1 hour until complete dissolution.

Next, a suspension of functional carbon nanotubes (f-CNTs) in EG was added to the precursor solution via sonication for 30 mins, ensuring the desired reagent ratio as indicated in column (6) of Table 2. The amount of f-CNTs in EG and the sonication temperature were the variables under investigation.

After sonication, the mixture was subjected to microwave irradiation at various power levels and time settings. The reaction mixture was then cooled down and filtered with ethanol using centrifugation at 5000 rpm for 5 mins. The resulting product was a black paste, which was dried for 12 hours at 90 °C.

Table 2 provides detailed information on the survey conditions conducted using Taguchi design (Figure 1), consisting of 16 experiments corresponding to 16 samples labeled as MSC-D2PD-n (n = 1, 2, ..., 16). The data was analyzed and the Taguchi results were calculated and exported using Minitab software.

The x-ray diffraction (XRD) technique was used to study the structure of amorphous MoS2/CNTs nanocomposites synthesized via the microwave procedure. The Bruker D8 ADVANCE diffractometer is used to do XRD experiments, utilizing Cu Kα radiation (λ=1.5406 Å). The diffraction experiment was conducted over an angular range of 5o to 80o at a scan rate of 1° min-1 and step size of 0.0194 °.

Electrochemical measurements for catalysts

To make the working electrode, 2.5 mg of polyvinylidene fluoride (PVDF) was mixed with 10 mg of the already-prepared materials, specifically the catalysts shown in Table 2. Following this, the mixture undergoes ultrasonic dispersion at 50 ^{o}C for a duration of 60 mins. The dispersion occurred in solutions comprising 1 μL polyvinyl alcohol (PVA) 0.1 wt% and 1 mL EG. Then, a homogeneous suspension with a volume of 50 μL was carefully transferred into the 3 mm glassy carbon electrode (GCE). The working electrode samples used for electrochemical testing are labeled as EC-D2PD-n, where n represents the sample number ranging from 1 to 16.

The following electrolyte solution was prepared for the Tafel plot analysis:

- Dilute H_2SO_4 98% solution with distilled water to a concentration of 0.5 mol/L H_2SO_4.
- Add 0.2 g sodium dodecyl sulfate (SDS) to 100 mL of 0.5 mol/L H_2SO_4 solution and stir thoroughly before inserting the working electrode.
- Throughout the analysis, the solution temperature was maintained at 25 $\pm$ 1°C.

The PARSTAT 2273 (AMETEK) electrochemical instruments was used to perform electrochemical tests in 0.5 mol/L H_2SO_4 (pH = 0.3) solutions at room temperature. The experiments were conducted using a three-electrode arrangement system. A glassy carbon electrode (GCE) loaded catalysts (3 mm in diameter), Pt (99.99%) (10 x 10 mm), and a saturated Cu/Cu^{2+} in $CuSO_4$ were used respectively as the working, counter, and reference electrodes. The value saturated Cu/Cu^{2+} in $CuSO_4$ is 0.34 V (vs. NHE). All final potentials were calibrated to NHE according to the Nernst equation $E_{NHE} = E_{Cu/CuSO4} + 0.34\ V + 0.059 \times pH$. The linear sweep voltammetry (LSV) was measured from 0 to 1 V (vs. NHE) at a sweep rate of 1 $mV{\cdot}s^{-1}$.

RESULTS AND DISCUSSION

XRD patterns of 16 samples MSC-D2PD-n

The X-ray diffraction (XRD) patterns depicted in Figure 2 demonstrate the absence of distinct peaks corresponding to the crystalline phase of the standard pristine MoS_2 (ICDD card # 00-009-0312). This suggests the presence of an amorphous phase of MoS_2/CNTs[6,9,10]. The (103) and (105) peaks undergo a shift from the crystalline to the poorly crystallized states, resulting in a considerable reduction and broadening of their peak intensities. The reversal of the (100)/(103) intensity ratios is particularly remarkable. When comparing the diffraction patterns of MoS_2 that has poor crystallinity to that of MoS_2 with good crystallinity, additional characteristics become apparent, such as the shifting of peaks and the asymmetrical broadening of the peaks. The X-ray diffraction (XRD) analysis, as shown in Figure 2, demonstrated that the background of all 16 samples had an amorphous nature. This suggests the presence of carbon nanotubes (CNTs) in amorphous forms, as opposed to the characteristic diffraction pattern of multi-walled carbon nanotubes (MWNTs) seen in the JCPDS card #96-101-1061. As expected, the CNT-related reflections generally overlap with those of the MoS_2 sheath, and their intensity is greatly reduced.

SEM images of 16 samples MSC-D2PD-n

The SEM images (Figure 3) of the MSC-D2DP-n samples (n = 1, 2, ..., 16) reveal the formation of interconnected particles that come together to create large fibrous structures. Some regions show the presence of MWNTs with varying structures and diameters ranging from 50 to 100 nm, which are larger than the initial f-CNTs diameter of 20-30 nm. Although the MoS_2 product fully coats the MWNTs structure during the reaction, the characteristic hexagonal morphology of MoS_2 is not visible due to its amorphous form.

Upon exposure to microwave irradiation, the amorphous MoS_2 rapidly forms in all MSC-D2DP-n samples, immediately covering the entire MWNTs surface. The presence of the amorphous structure, which possesses high surface energy, suggests that it is well-suited to act as a catalyst in the hydrogen evolution reaction (HER).

Taguchi optimization calculation

The Taguchi technique utilizes the Tafel slope as the output data to determine the most suitable synthesis conditions for amorphous MoS_2/CNTs. The LSV curve depicted in Figure 4 and the Tafel equation η = b log j + a (2) are employed for the calculation of the Tafel slope presented in Table 3.

The aim of this study is to reduce the Tafel slope of the MoS_2/CNTs materials prepared in this research. Figure 5 visually represents how the parameters directly influence the desired outcome. The graph reveals the optimal synthesis conditions for achieving amorphous MoS_2/CNTs, which are as follows:

- Microwave power: 240 W
- Reaction time: 45 mins
- Ultrasonication temperature: 80° C
- Amount of f-CNTs: 40 mg
- The ratio of precursor mixture (AHM+TU) to EG solvent $\sum m_{(AHM+TU)} : V_{EG}$ = 0.06 g/mL

To evaluate each factor's influence, the average value of the factor's S/N ratio at the same level was determined, and then the factor's primary effect value was calculated (delta). Tables 3 shows the effect of various parameters on Tafel slope b, together with the mean S/N ratio for each

factor level. Factor P2 has the highest significant delta value, as shown in Table 3. The Tafel coefficient is shown to be significantly influenced by the reaction time.

Evaluate the catalytic abilities of nanostructured amorphous MoS_2/CNTs.

The utilization of Tafel equations in electrochemical analysis is a prevalent method for assessing and describing electrocatalysts in the hydrogen evolution reaction. The Tafel slope and exchange current density are the sole parameters consistently calculated and mentioned in the literature about the Tafel equation. The Tafel constant is suggested to be used as the onset potential for the hydrogen evolution reaction (Vonset). When the Tafel slope and exchange current density (j) are equal, the Tafel constant becomes the distinguishing factor between two electrocatalysts.

Tafel plots depict the logarithmic correlation between electrochemical current density (j) and different overpotentials, as described by the Tafel equation (η = a + b log j). The given equation can be employed to calculate two important parameters, namely the Tafel slope (b) and the exchange current density (j_0) when η is equal to zero. The Tafel slope determines the intrinsic catalytic efficiency of the catalyst employed. A highly effective catalytic material typically requires a high j_0 and a low Tafel slope.

Figure 4 illustrates the hydrogen catalytic activity of amorphous MoS_2/CNTs nanocomposites materials generated utilizing the microwave heating method.

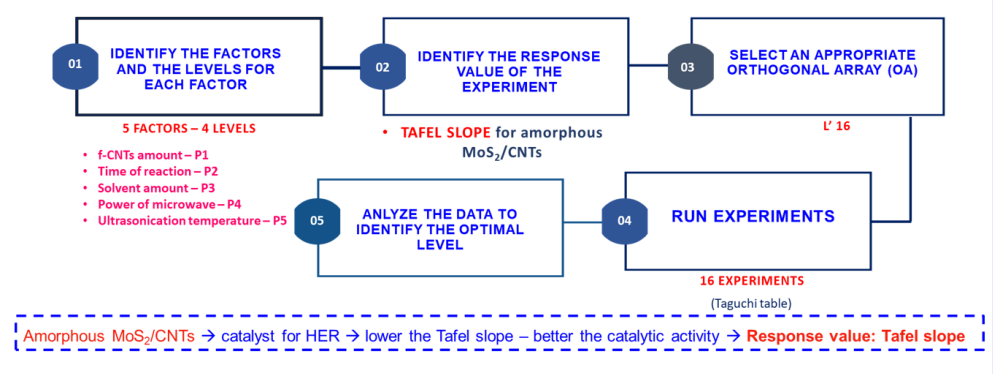

Figure 1: Taguchi design of experiment involves modeling the effect of control factors on performance output.

Table 1: Five factors and 4 levels for each factor to be investigated in Taguchi method

Level	P1	P2	P3	P4	P5	Response
	Microwave power (W)	Reaction time (mins)	Ultrasonication temperature (°C)	Amount of f-CNTs (mg)	∑m(AHM+TU (g/mL)	Tafel slope (b)
1	240	15	50	10	0.02	…
2	400	30	60	40	0.04	…
3	560	45	70	70	0.06	…
4	720	60	80	100	0.08	…

Table 2: Experimental plan for the first round of optimizations (following Taguchi method)

Exp Name (1)	Microwave power (W) (2)	Reaction time (mins) (3)	Ultrasonication temperature (°C) (4)	Amount of f-CNTs (mg) (5)	∑m(AHM+TU):V_{EG} (g/mL) (6)
MSC-D2PD-1	240	15	50	10	0.02
MSC-D2PD -2	240	30	60	40	0.04
MSC-D2PD -3	240	45	70	70	0.06
MSC-D2PD -4	240	60	80	100	0.08
MSC-D2PD -5	400	15	60	70	0.08
MSC-D2PD -6	400	30	50	100	0.06
MSC-D2PD -7	400	45	80	10	0.04
MSC-D2PD -8	400	60	70	40	0.02
MSC-D2PD -9	560	15	70	100	0.04
MSC-D2PD -10	560	30	80	70	0.02
MSC-D2PD -11	560	45	50	40	0.08
MSC-D2PD -12	560	60	60	10	0.06
MSC-D2PD -13	720	15	80	40	0.06
MSC-D2PD -14	720	30	70	10	0.08
MSC-D2PD -15	720	45	60	100	0.02
MSC-D2PD -16	720	60	50	70	0.04

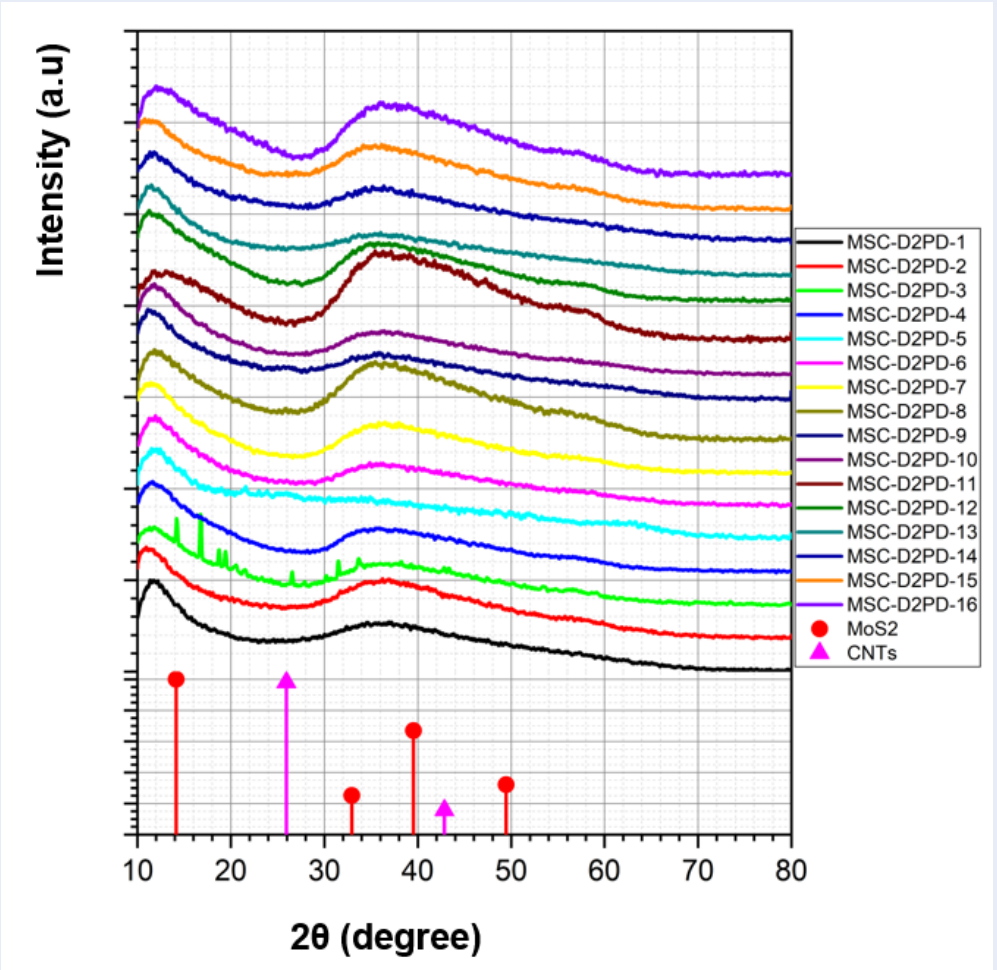

Figure 2: XRD patterns of 16 samples investigated from Taguchi method (Table 2)

Table 3: Response Table for Means exported from Minitab

Level	P1	P2	P3	P4	P5
1	109.5	142	120.8	121.8	120.0
2	122.3	116.3	117.8	114.0	126.5
3	127.3	110.3	133.3	121.3	112.8
4	123.0	113.5	110.3	125.0	122.8
Delta	17.8	31.8	23.0	11.0	13.8
Rank	3	1	2	5	4

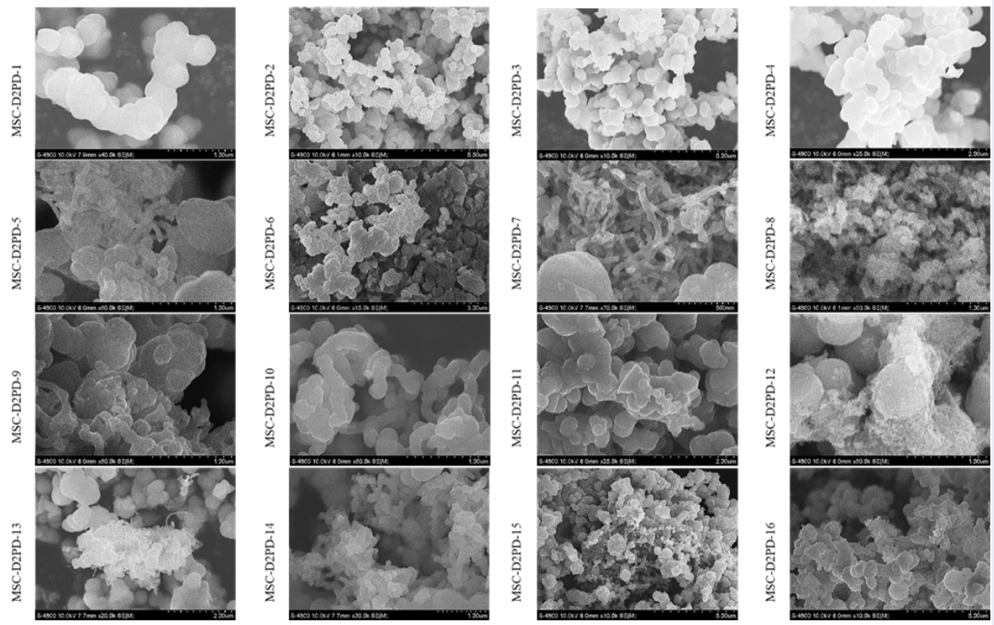

Figure 3: SEM images of 16 samples investigated from Taguchi method (Table 2)

The hydrogen evolution reaction is a sequential procedure occurring on an electrode's surface, resulting in the production of hydrogen gas. The hydrogen evolution reaction from water should theoretically take place at V_{onset} = 0 V (vs. NHE) [11,12]. However, electrode materials are always coated with a layer of hydrogen atoms or hydrogen ions. The existence of this adsorption layer directly affects the resistance of the electrode surface. When the atoms or ions on this adsorbent layer combine to generate H_2, the new reaction becomes more intense, the adsorbent layer thickness increases to its maximum, and an equilibrium is reached. The potential for beginning a reaction is proportional to the resistance of the thickness of the equilibrium adsorbent layer. Thus, the closer the V_{onset} is to 0 V (vs.NHE), the simpler the reaction is and the less energy is required to activate it, implying a higher catalyst activity. The LSV study demonstrated that all of the amorphous MoS_2/CNT samples exhibited the ability to catalyze the H_2 production process, with V_{onset} values ranging from -0.22 to -0.24 V, as indicated in Table 4.

Incorporating conductive supporting materials, such as CNTs, as the growth matrix for MoS_2 nanomaterials in order to create a hybrid structure, brings two distinct advantages in enhancing catalytic activity. (1) These carriers enable the transfer of electrons with greater speed and efficiency, leading to enhanced kinetics of the hydrogen evolution reaction (HER). (2) The specific structure of these carriers allows H^+ ions to penetrate the active regions of MoS_2 nanoparticles through the electrolyte [13]. The bonding of amorphous MoS_2 material onto the surface of CNTs enhanced the conductivity of the material. The disordered surface of MoS_2 enables direct collision of H atoms/H^+ ions on the material's surface, thereby expediting the process. The Pourbaix diagram is a valuable tool for assessing the stability of an electrocatalyst in a water-based environment. It accomplishes this by graphing the chemical potential of different species based on the voltage of the electrode and the pH of the surroundings. When subjected to an acidic environment with a concentration of 0.5M H_2SO_4, it is evident that MoS_2/CNTs is highly stable at the usual operating potentials for hydrogen evolution reaction (-0.22 ÷ -0.23 V (vs. NHE)) [14].

The Tafel slope (mV/dec) is the quotient of the overvoltage divided by the logarithm of the current density during continuous electrolysis. The Tafel slope served as a measure of the reaction kinetics and the rate-determining step (RDS) in the process of hydrogen evolution reaction (HER). The Tafel slopes of 120, 40, and 30 mV/dec correspond to the Volmer, Heyrovsky, and Tafel rate-determining steps, respectively. The HER can arise from either the Volmer-Heyrovsky or Volmer-Tafel processes. [4,15,16] According to the study [17], the active center responsible for H_2 production in MoS_2 nanocrystals is localized at

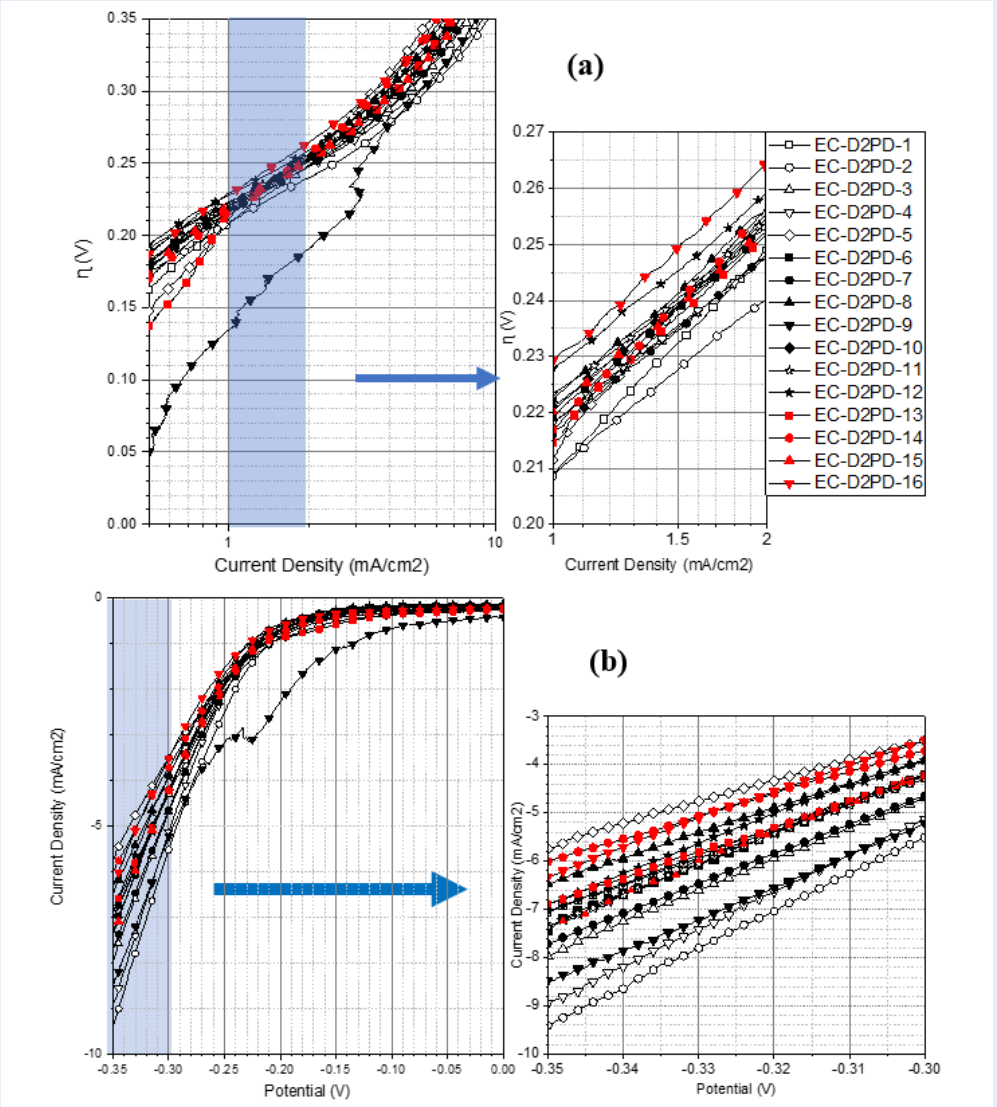

Figure 4: (a) Tafel plots of EC-D2DP-n (n = 1, 2, 3… 16); and (b) LSV curves of EC-D2PD-n (n=1, 2, 3,…16) electrodes in 0.5 M H_2SO_4

the edges and unsaturated Mo or S atoms. The integration of the amorphous structure of MoS_2 and CNTs was employed to enhance the density of catalytic sites, resulting in enhanced electrode performance. The Tafel plots demonstrate slopes of around 120 mV/dec, suggesting that the MSC-D2PD-n samples undertake hydrogen evolution processes through both the Volmer-Heyrovsky and Volmer-Tafel mechanisms. The MSC-D2PD-2 has the most significant catalytic activity, achieving a current density of -9.41 $mA.cm^{-2}$ at -0.35 V. The Tafel slopes of the MSC-D2PD-4 and MSC-D2PD-2 samples are the lowest, with measurements of 99 and 102 mV/dec, respectively. Yuxue Dai and colleagues successfully created MoS_2 on carbon nanotubes using ethylene glycol. The Tafel slope of EG-MoS_2/CNTs was measured to be 87 mV/dec, indicating a significant improvement in electrochemical HER performance compared to MoS_2 and MoS_2/CNTs in an acidic solution. These findings align with our own results [18]. The incorporation of functionalized carbon nanotubes (f-CNTs) enhances the electrical conductivity of the catalyst and inhibits the aggregation of MoS_2. The microwave heating process used to manufacture MoS_2 results in the formation of amorphous microstructures, which in turn leads to a higher number of exposed Mo edges

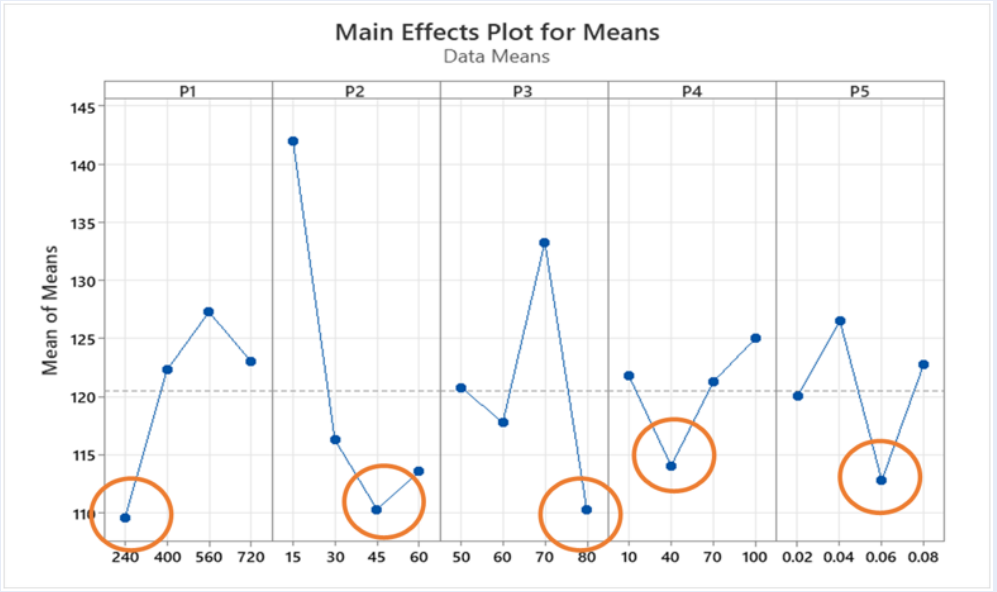

Figure 5: S/N (Ra) analysis exported from Minitab

Table 4: Electrocatalytic performance of D2PD-n (n=1, 2, 3, ...16)

Samples	V_{onset} vs. NHE (V)	*j* at -0,35 V (mA cm^{-2})	Tafel slope (mV/dec)
EC-D2PD-1	-0,22	-7,05	132
EC-D2PD-2	-0,23	-9,41	102
EC-D2PD-3	-0,22	-7,96	105
EC-D2PD-4	-0,23	-8,94	99
EC-D2PD-5	-0,22	-5,74	144
EC-D2PD-6	-0,23	-7,45	115
EC-D2PD-7	-0,22	-7,71	109
EC-D2PD-8	-0,23	-6,47	121
EC-D2PD-9	-0,22	-8,48	172
EC-D2PD-10	-0,23	-7,05	113
EC-D2PD-11	-0,23	-7,35	113
EC-D2PD-12	-0,24	-6,89	111
EC-D2PD-13	-0,22	-6,89	120
EC-D2PD-14	-0,22	-6,00	135
EC-D2PD-15	-0,23	-7,26	114
EC-D2PD-16	-0,24	-6,33	123

or active sites[6].

CONCLUSION

The synthesis parameters for producing amorphous MoS_2/CNTs using the microwave technique were optimized using the Taguchi approach. The experimental parameters yielding the most effective results were determined as follows: a microwave power of 240 W, a reaction time of 45 mins, an ultrasonication temperature of 80 °C, an amount of f-CNTs of 40 mg, and a ratio of $\sum m_{(AHM+TU):} V_{EG}$ of 0.06 g/mL.

The empirical results indicated that the Tafel slope was most strongly influenced by the reaction time. The nanomaterial formed, consisting of amorphous MoS_2/CNTs, demonstrated exceptional catalytic efficacy and durability throughout the potential range of -220 to -230 mV (vs. NHE). The measurement showed a current density of -8.94 mA/cm^2 at a potential of -350 mV (vs. NHE) and a Tafel slope of 102 mV/dec.

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CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHORS' CONTRIBUTION

Vinh-Dat Vuong: Methodology and Investigation; Minh Nguyet Nguyen: Data curation, Conceptualization, Writing – original draft; Nguyen Huu Huy Phuc, Thang Van Le: Reviewing and Supervision.

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Research Article

Ứng dụng phương pháp Taguchi tối ưu hóa các thông số của quá trình tổng hợp vật liệu vô định hình MoS_2/CNT bằng phương pháp vi sóng nhằm mục tiêu cải thiện hoạt tính xúc tác của chúng trong HER

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TÓM TẮT

Thông thường, thực nghiệm được thực hiện trong các lĩnh vực khoa học, kỹ thuật, công nghệ,... tốn nhiều công sức, thời gian và chi phí. Vì vậy, việc làm sao giảm được chi phí thực nghiệm, cụ thể là giảm thiểu số thí nghiệm mà vẫn cho kết quả nghiên cứu khảo sát chính xác, hợp lý, có tính khoa học chính là mục đích của quy hoạch thực nghiệm nói chung và quy hoạch Taguchi nói riêng. Phương pháp Taguchi là một phương pháp đơn giản và hiệu quả, cho phép giảm đáng kể các thí nghiệm cần thiết với mô hình toán học thống kê thực nghiệm theo các tiêu chuẩn thống kê, từ đó cho phép xem xét ảnh hưởng của các yếu tố với độ tin cậy cần thiết. Trong nghiên cứu này, phương pháp quy hoạch Taguchi được sử dụng để xác định các điều kiện tổng hợp vật liệu nano molybdenum disulfide/cacbon nanotubes (MoS_2/CNTs) cấu trúc vô định hình bằng phương pháp vi sóng, bao gồm các thông số: lượng CNTs, nhiệt độ phản ứng, thời gian phản ứng, công suất vi sóng và tỷ lệ tác chất Mo:S, nhằm mục tiêu cải thiện hoạt tính xúc tác của vật liệu đầu ra. Biến đáp ứng (Response – thông số đầu ra) được chọn là hệ số Tafel (Tafel slope) – đại lượng đặc trưng cho hoạt tính xúc tác của vật liệu MoS_2/CNTs vô định hình tổng hợp được. Hệ số Tafel là một đại lượng quan trọng để đánh giá hoạt tính xúc tác và cơ chế phản ứng xúc tác. Kết quả cho thấy vật liệu MoS_2/CNTs cấu trúc vô định hình thể hiện khả năng xúc tác tốt và ổn định trong khoảng điện thế -220 mV đến -230 mV (so với NHE), mật độ dòng điện -8,94 mA/cm^2 (V = –350 mV so với NHE), độ dốc Tafel là 102 mV/dec.

Từ khoá: Quy hoạch thực nghiệm, Taguchi, MoS2/CNTs, hệ số Tafel, tổng hợp vi sóng, HER

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