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Synthesis of Copper/Sulfur Co-Doped TiO₂-Carbon Nanofibers as Catalysts for H₂ Production via NaBH₄ Hydrolysis

Ahmed Abutaleb 

Chemical Engineering Department, Faculty of Engineering, Jazan University, Jazan 45142, Saudi Arabia; azabutaleb@jazanu.edu.sa

Abstract: Copper/sulfur co-doped titanium dioxide-carbon nanofibers (Cu,S-codoped TiO₂ NPs, decorated-CNFs) catalysts were synthesized using the electrospinning process to produce composite nanofibers (NFs). These composite NFs were utilized for the hydrolysis of sodium borohydride (SBH) to generate hydrogen gas (H₂), taking advantage of their catalytic properties. The experimental results demonstrated that using 100 mg of composite NFs yielded the highest catalytic activity for H₂ production, generating 79 mL of H₂ gas within 6 min at 25 °C and 1000 revolutions per minute (rpm) using 1 mmol of SBH. As the catalyst dosage was reduced from 100 mg to 75, 50, and 25 mg, the reaction time increased by 9, 13, and 18 min, respectively. Kinetic studies revealed that the reaction rate followed a first-order reaction, indicating a direct proportionality between the rate of reaction and the catalyst amount. Additionally, it was observed that the concentration of SBH had no influence on the reaction rate, suggesting a zero-order reaction. Increasing the reaction temperature resulted in a reduced reaction time. The activation energy was determined to be 26.16 kJ mol^{−1}. The composite NFs maintained their superior performance over five iterations. These findings suggest that composite nanofibers have the potential to serve as a cost-effective alternative to expensive catalysts in hydrogen production.

Keywords: copper; sulfur; TiO₂; carbon; electrospinning; sodium borohydride; hydrogen



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1. Introduction

Hydrogen (H₂) as an energy carrier has gained recognition as a valuable alternative clean energy source due to its eco-friendly nature and high energy density [1,2]. It is considered a green product and a sustainable energy source. However, the H₂-based economy faces a number of challenges, such as H₂ release and storage control [3]. Metal hydrides play a vital role in the optimal storage of H₂ [4]. Producing H₂ from metal hydrides, such as sodium borohydride (SBH) and lithium borohydride (LiBH₄), is considered a practical, inexpensive, and effective method. SBH stands out as a promising material for storing and producing H₂ through a hydrolysis reaction due to its high H₂ storage capacity (~10.8 wt.%), non-toxicity, and excellent stability in an alkaline solution without the need for a catalyst [5,6]. Proper catalysts can catalyze the dehydrogenation of SBH in an alkaline solution to produce pure H₂ in a controlled manner [7]. It is important to emphasize that the dehydrogenation of SBH is extremely fast in the presence of suitable catalysts without the need for additional heat. Therefore, investigating effective catalysts for the dehydrogenation of SBH is critical for implementing the dehydrogenation process in the sustainable energy industry [7]. While it has been demonstrated that precious metals (e.g., Pd, Ru, Pt, Rh, and their alloys) supported on different matrices exhibit outstanding performance for H₂ generation, their high cost and limited availability restrict their widespread usage [8–11]. To overcome this challenge, researchers are tirelessly working to develop a wide variety of catalysts based on non-precious metals, such as cobalt, nickel, and copper, and their alloys, which can catalyze the dehydrogenation of SBH and produce pure H₂ [12–17]. Copper-based catalysts are widely used because they are readily available

and inexpensive [1]. They have been applied as effective catalysts for the dehydrogenation of SBH to generate H₂ [14]. Carbon-based materials are seen as one of the best alternatives to metal-based catalysts due to their tunable electrical characteristics, availability, and low environmental impact [1]. Doping carbon-based substances with heteroatoms (e.g., S, N, B, and P) allows for the adjustment of their electrical and surface chemistry characteristics, greatly improving the catalytic efficacy of carbon-based materials in numerous reactions. The distinct electronegativities of heteroatoms and carbon atoms contribute to an increase in the conductive properties and electron transmission rate of carbon materials. Cafer Saka [18] demonstrated the effect of S and N on the catalytic activity of carbon in the dehydrogenation of SBH. These atoms enhanced the catalytic activity of carbon, resulting in a hydrogen generation rate (HGR) of 2641 and 4923 mL·min^{−1}·gcat^{−1} for pristine carbon and S-doped carbon, respectively. According to Zhang et al. [7], the addition of S in the form of sodium sulfate and sodium sulfide to a Co-based catalyst for the generation of H₂ from SBH resulted in an HGR of 4425 mL·min^{−1}·gcat^{−1}, which is four times higher than that of cobalt oxide. Dilek Kilinc, [19] demonstrated that a copper (II)-Schiff Base complex-supported TiO₂ matrix exhibited good catalytic activity for the dehydrogenation of SBH compared to unsupported copper (II)-Schiff Base complex, with an HGR of 215 mL·min^{−1}·gcat^{−1} and 14,020 mL·min^{−1}·gcat^{−1}, respectively. In addition to their usage as support matrices, TiO₂ possesses both redox and Lewis acidity characteristics. Various types of nanostructures have been utilized as effective catalysts for the generation of H₂ from SBH. Among them, nanofibers (NFs) have a notable advantage in H₂ production from SBH due to their higher surface-to-volume ratio and highly porous structure [3,20–23]. Electrospinning is widely regarded as a highly convenient and versatile methodology for fabricating NFs. During the electrospinning process, a high electrical potential is applied to overcome the surface tension in the solution, leading to the expulsion of the charged precursor solution from the needle and its subsequent gathering on the collecting mandrel [20]. The electrospinning technique is capable of generating NFs ranging in size from 50 to 900 nm. In this study, Cu,S-codoped TiO₂ NPs decorated on CNFs were synthesized using the electrospinning technique. The utilization of this composite made of Cu,S-codoped TiO₂ NPs-decorated CNFs for the production of H₂ from SBH has not previously been described, to the best of the author's knowledge. The structure and catalytic performance of the new composite are exploited for H₂ production. The prepared composite NFs have shown superior catalytic activity for the dehydrogenation of SBH and subsequent H₂ generation.

2. Results and Discussion

2.1. Characterization Results

Copper sulfide demonstrates low solubility in commonly used solvents. Consequently, the addition of sulfide ions (S^{2−}) to a solution containing copper ions (Cu²⁺) leads to the formation of a finely dispersed precipitate known as CuS. Therefore, a green colloidal solution was generated by adding a few drops of ammonium sulfide to the copper ion sol-gel [6]. Electrospinning is a widely used method for producing polymer NFs [24]. Recently, our research group utilized electrospinning to synthesize NFs from a colloidal solution with an optimal nanofibrous shape [25–27]. Figure 1a depicts the SEM image of TIIP-PVP electrospun NF mats after calcination at 800 °C in an Ar atmosphere. As shown in the figure, the generated NFs are smooth, continuous, and free of beads.

Due to the susceptibility of TIIP to rapid hydrolysis upon exposure to atmospheric moisture, the NFs, upon ejection from the electrospinning needle, were able to form continuous networks (gels) of TiO₂ sols [28]. SEM images of the electrospun green colloidal solution following calcination at 800 °C in an Ar environment are presented in Figure 1b. It can be observed that the NFs retained their nanofibrous structure, and continuous NFs were formed despite the unusual sintering conditions. Additionally, densely dispersed white nanoparticles (NPs) were generated on the surface of the NFs. It is well-known that increasing the calcination temperature of electrospun NFs mat while processing it in a vacuum and in the presence of argon gas slows down the rate of polymer decomposition, leading to

the production of carbon as a byproduct of the calcination process [29,30]. The TiO_2 -CNFs exhibited an average diameter of up to 702 nm (Figure 1c), whereas the composite NFs had an average diameter of 33 nm (Figure 1d). This could be attributed to the increased electrical conductivity of the solution due to the addition of Cu precursor and ammonium sulfide, resulting in the stretching of the produced NFs in the electrospinning process.

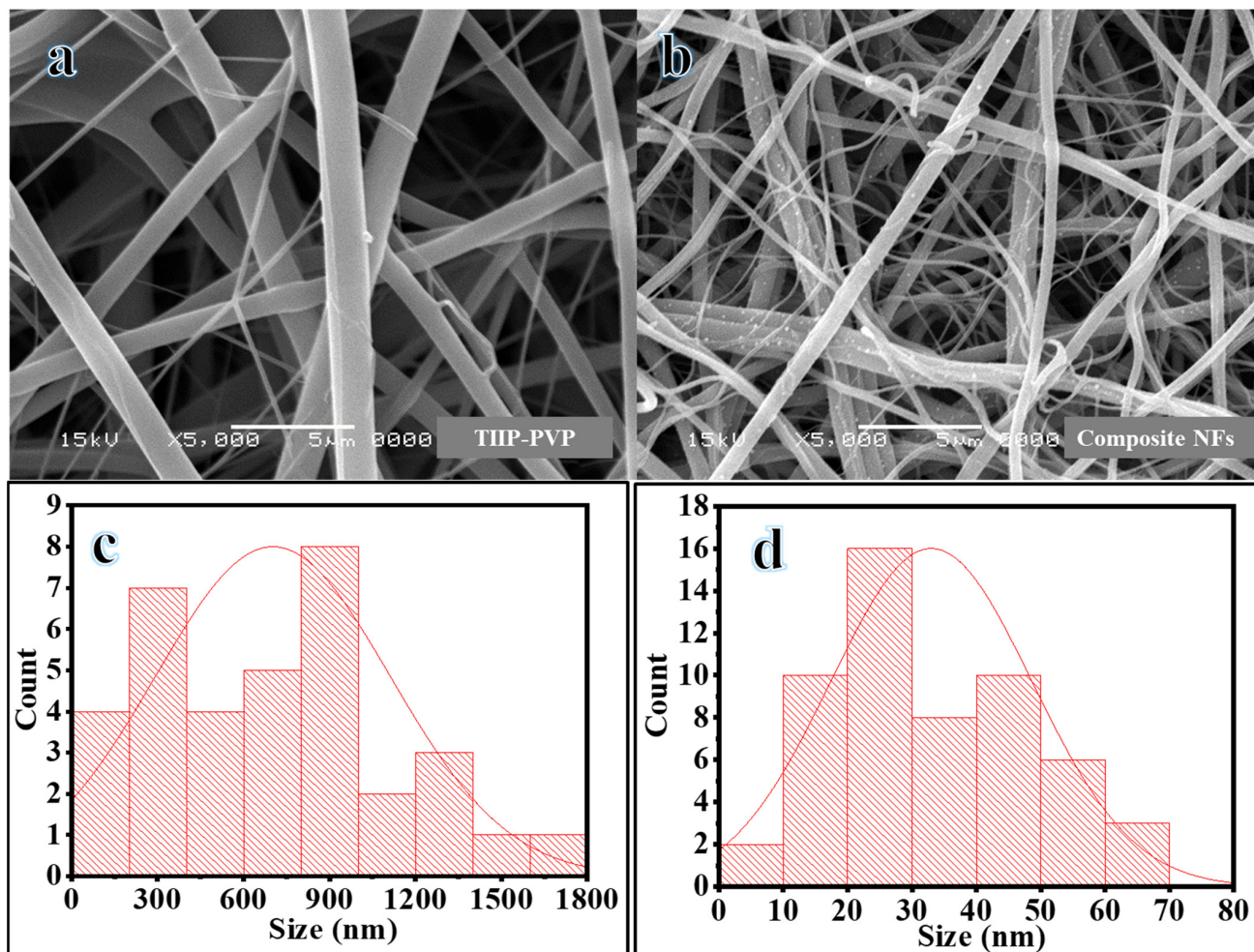


Figure 1. SEM images of obtained powder after calcination of TIIP-PVP (a) and composite NFs (b) at 800 °C in Ar and their respective average size (c,d).

Figure 2a illustrates the typical XRD analysis of the composite NFs. Five distinct peaks are clearly visible at 2θ values of 26.98°, 35.72°, 40.88°, 54.08°, and 68.62°, corresponding to the (110), (101), (211), (211), and (002) crystal planes of the rutile phase of TiO_2 (JCPDS#21-1276), respectively. Additionally, there is a prominent diffraction peak at a 2θ value of approximately 42.95°, which corresponds to the (111) crystal planes of metallic copper (JCPDS#04-0836). Furthermore, the presence of a sharp peak at a 2θ value of around 24.89° may indicate the presence of graphite-like carbon resulting from the graphitization of polymer NFs [31]. The absence of a peak in the detection of CuS could be attributed to the thermal degradation of CuS into metallic copper and sulfur at elevated temperatures in a vacuum system containing an argon atmosphere. Moreover, the lack of sulfur peaks may be due to the incorporation of sulfate ions into the TiO_2 and/or carbon structure [32], which could also explain the negative deviation in the peaks of TiO_2 , copper, and carbon. Raman spectroscopy was also employed to investigate the chemical structure of the graphite-like carbon obtained. Figure 2b reveals two peaks centered at approximately 1346.78 and 1593.93 cm^{-1} , corresponding to the D and G bands of graphite, respectively.

This indicates that the polymer has undergone a transformation into graphite with a certain amount of disordered sp² C–C bonding [28].

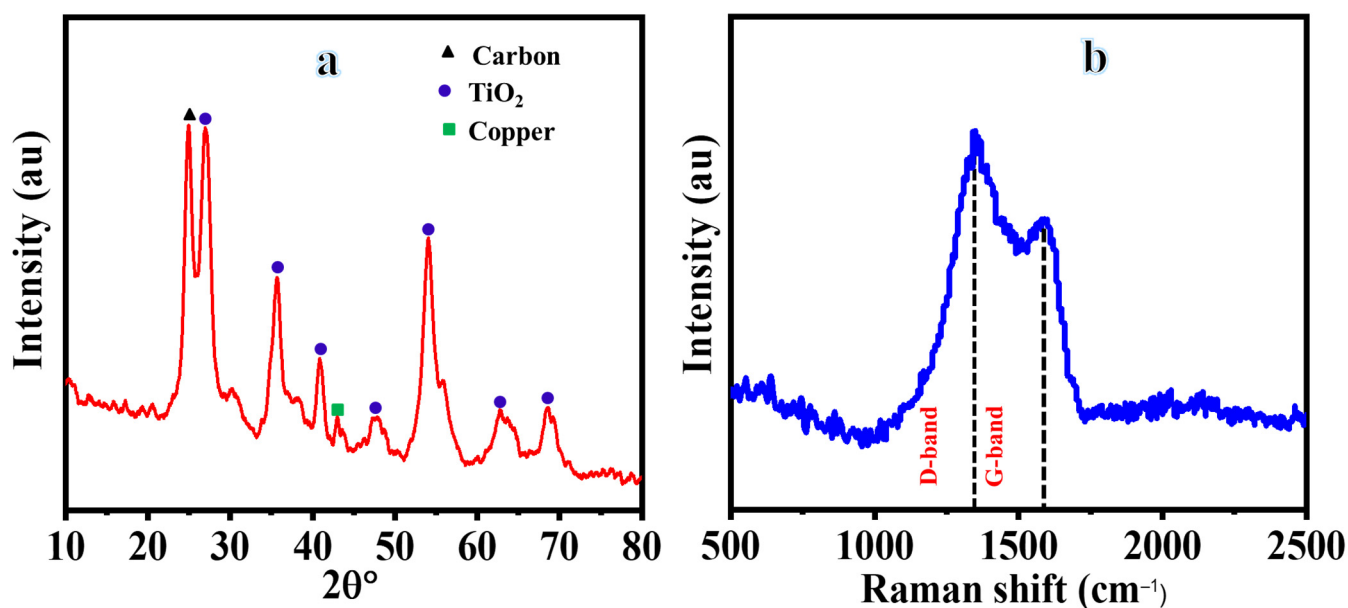


Figure 2. XRD (a) and Raman (b) analyses of composite NFs after sintering at 800 °C in Ar.

Figure 3a shows a typical TEM image of the composite NFs, revealing the nanoparticles (NPs) adhered to the surface of the NFs. Figure 3b displays the EDX spectra of the composite NFs, indicating the presence of titanium, oxygen, copper, and carbon as the only elements in the spectra.

Figure 4a displays the STEM image of the composite NFs, showing the even dispersion of nanoparticles (NPs) inserted within the surface of the selected NF. TEM-EDX analysis was performed along the line indicated in Figure 4a, revealing that the distributions of Ti (Figure 4b), O (Figure 4c), S (Figure 4d), and Cu (Figure 4e) are consistent. Additionally, it has been observed that carbon is the furthest element. The protective carbon coating on the active NPs allows for their long-term preservation during catalytic processes.

2.2. Hydrolysis of SBH

2.2.1. Effect of NaOH Percentages

The hydrolysis reaction of SBH is highly sensitive to the alkalinity of the solution. The rate of hydrolysis in an alkaline solution is slower compared to that in pure water due to the hydrolysis of NaBO₂, which produces NaOH [14,19]. Figure 5 illustrates the volume of H₂ generated by varying concentrations of [NaOH] (0%, 5%, 7%, and 10%) when mixed with 50 mg of catalytic composite NFs and 1 mM of SBH at 25 °C and 1000 rpm. It can be observed that as the [NaOH] increased from 0% to 7%, the completion time of the SBH hydrolysis reaction decreased (17 min, 13 min, and 10 min, respectively). However, when the [NaOH] was further increased to 10%, an increase in the hydrolysis reaction completion time (15 min) was observed. This is likely due to the presence of OH in the hydrolysis of NaBH₄. The HGR (hydrogen generation rate) can be enhanced by increasing the concentration of NaOH, which accelerates the catalyzed hydrolysis of SBH. However, if the NaOH concentration is too high, NaBO₂ may become less soluble, precipitate out of the solution, and attach to the catalyst's surface, obstructing its active sites [14]. This can hamper the contact between BH⁴⁺ ions and the catalyst's surface, resulting in a slower rate of hydrolysis. Based on these findings, the addition of 7% NaOH to the composite NFs significantly enhanced H₂ production, reduced reaction durations, and increased the volume of generated H₂. Therefore, the optimal [NaOH] concentration for SBH hydrolysis catalysis was determined to be 7% for further kinetic investigations.

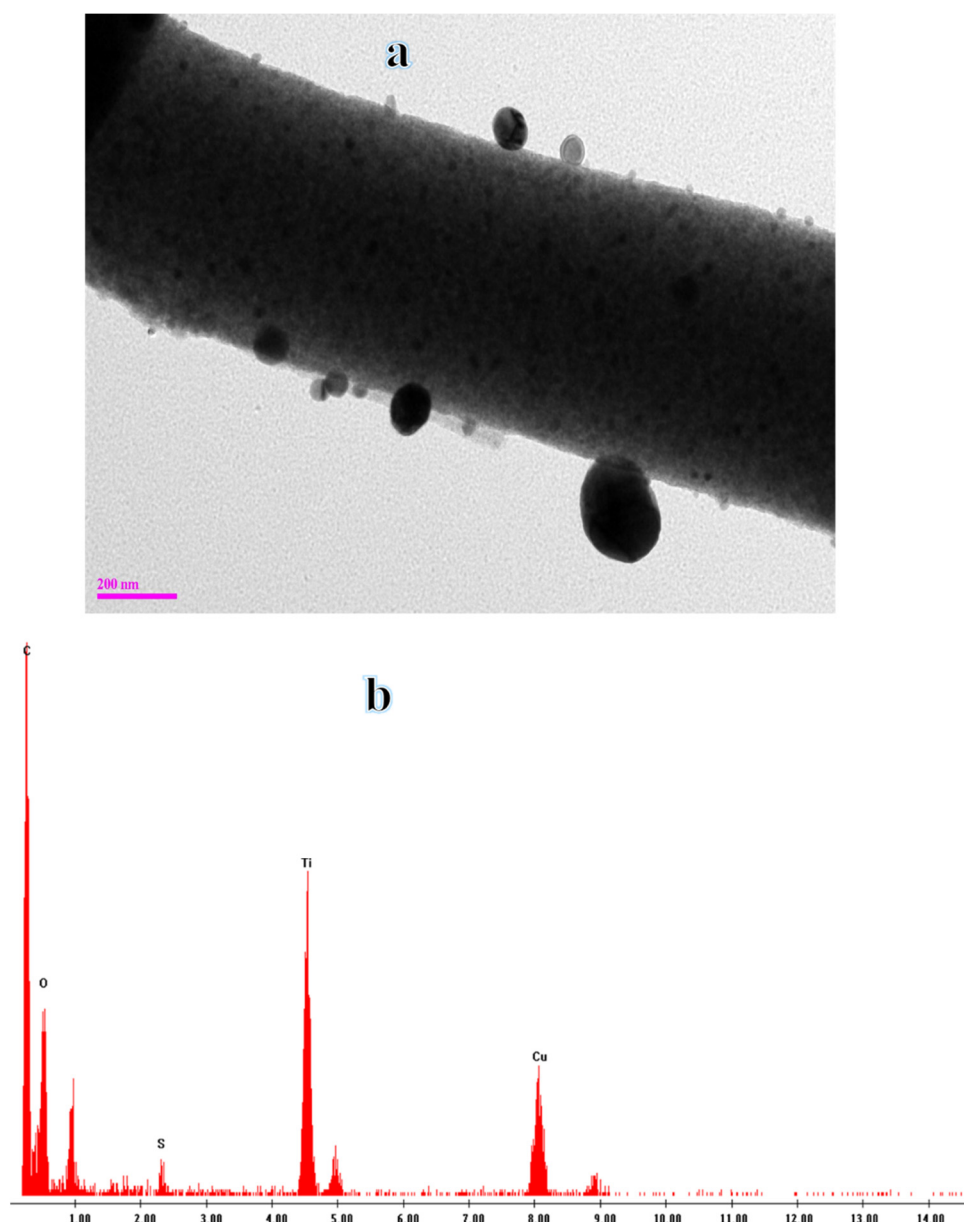


Figure 3. Normal TEM (a) and EDX (b) of composite NFs after sintering at 800 °C in Ar.

2.2.2. Effect of Catalyst Amount

Figure 6a demonstrates a positive correlation between the amount of catalyst (ranging from 25 to 100 mg) and the volume of H_2 generation in the presence of 1 mM SBH, 25 °C, 7% NaOH, and 1000 rpm. In the absence of a catalyst, only 13 mL of hydrogen gas was generated over a duration of 56 min. Although SBH is inherently unstable in atmospheric conditions, it exhibits a slow reaction rate with water in the absence of a catalyst, resulting in limited H_2 production [33]. However, the introduction of a small amount of catalyst (25 mg) significantly increased the volume of H_2 production within 18 min. Hydrolysis reactions were conducted with 50 mg, 75 mg, and 100 mg of catalyst, resulting in the production of 79 mL of H_2 in 13, 9, and 6 min, respectively. The enhanced catalytic activity can be attributed to the increased specific surface area and exposure of active sites facilitated by the incorporation of composite NFs [34]. As the SBH hydrolysis process progressed, a decrease in the rate of H_2 production was observed. This may be due to the formation of H_2 aggregates and the blocking of active sites, hindering the generation of new active sites for subsequent catalytic cycles [34]. The H_2 generation rate (HGR) was determined by

analyzing the linear segment of each plot shown in Figure 6a. The logarithmic scale values of the HGR relative to the initial amount of composite NFs are depicted in Figure 6b. The slope of the straight line is close to first order (slope = 0.89), indicating that the catalytic hydrolysis of SBH follows a nearly first-order dependence on the catalyst amount.

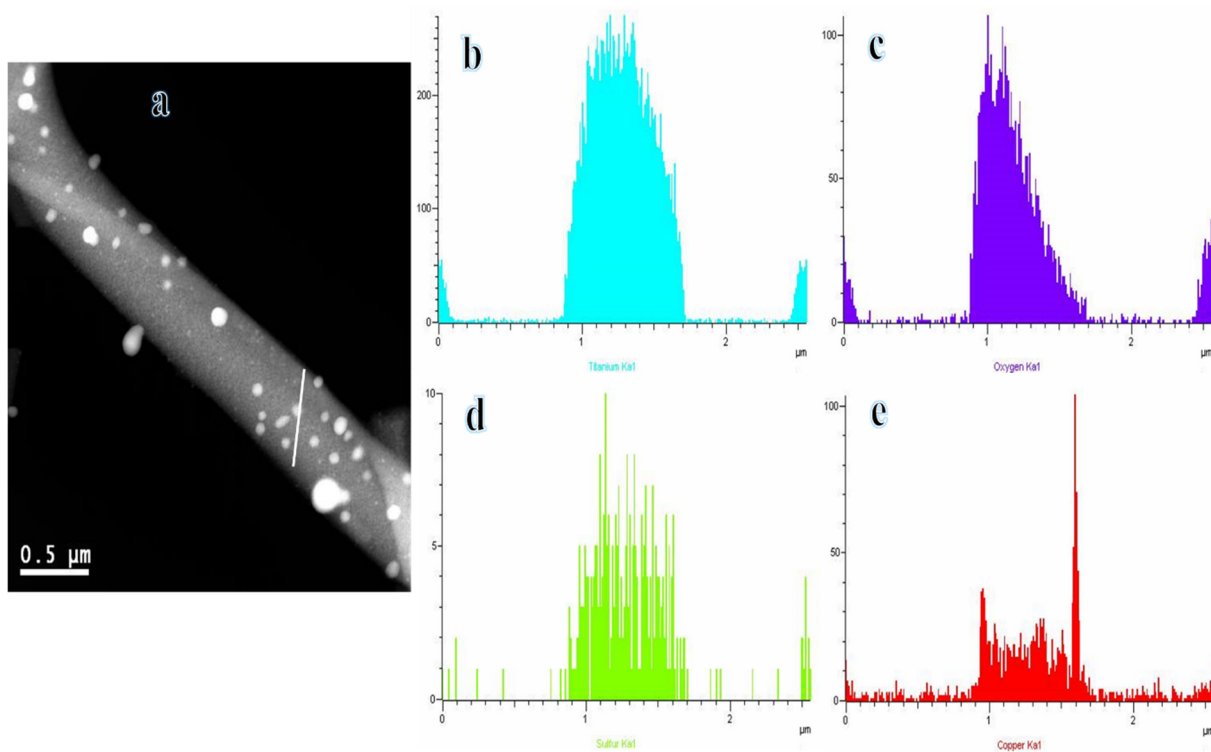


Figure 4. One STEM image (a), and TEM-EDX of Ti (b), O (c), S (d), and Cu (e) of composite NFs after sintering at 800 $^{\circ}\text{C}$ in Ar.

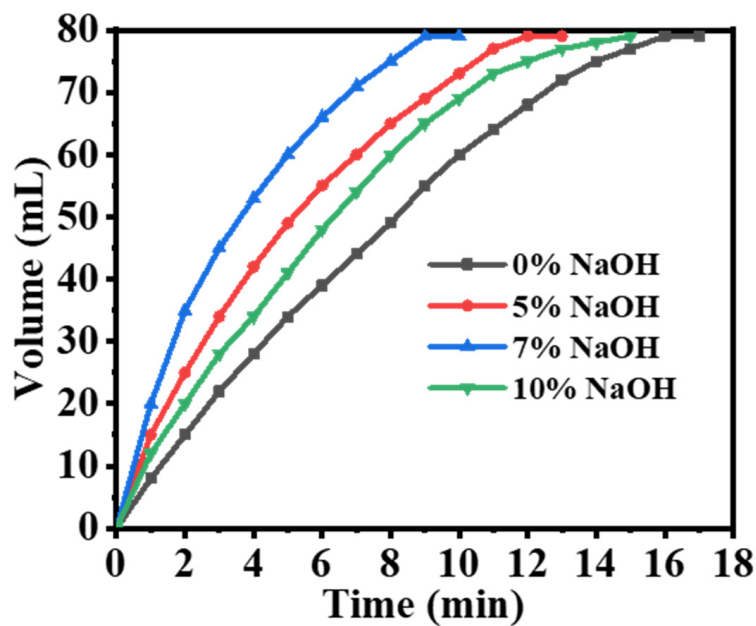


Figure 5. Effect of NaOH % on the hydrolysis of SBH used ([SBH] = 1 mmol, composite NFs = 25 mg, 25 $^{\circ}\text{C}$, and 1000 rpm).

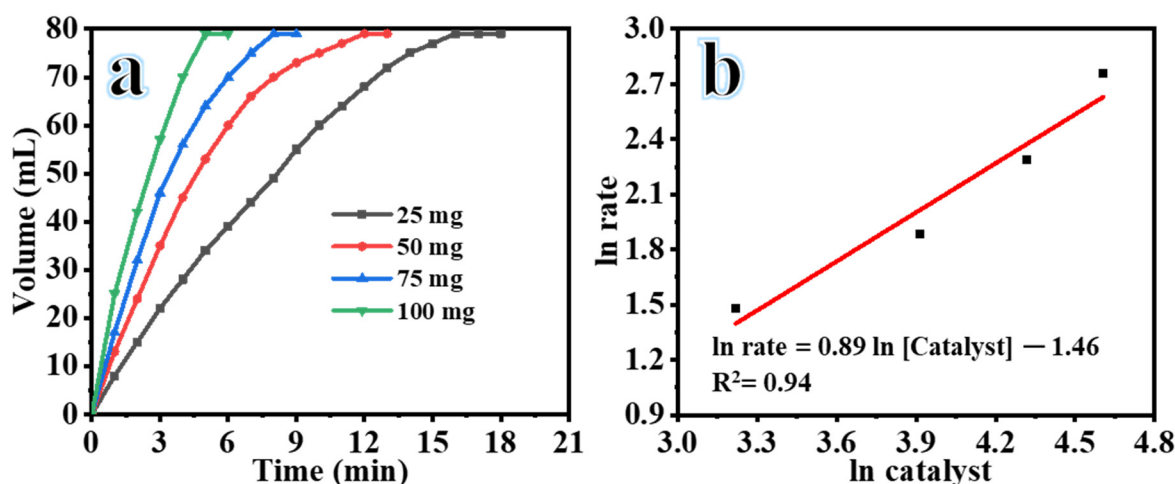


Figure 6. H₂ production volume versus time from hydrolysis of SBH using different composite NF doses at 25 °C, [SBH] = 1 mmol, and 1000 rpm (a), and logarithmic plot of H₂ production rate versus catalyst loading amount (b).

2.2.3. Effect of SBH Concentration

To investigate the influence of the starting concentration of SBH ([SBH]) on the hydrolysis process, experiments were conducted using 25 mg of composite NFs, 25 °C, 7% NaOH, and 1000 rpm. As shown in Figure 7a, the maximum volumes of H₂ produced were 79 mL in 18 min, 144 mL in 32 min, 223 mL in 42 min, and 303 mL in 60 min when utilizing 1, 2, 3, and 4 mM SBH, respectively. This trend indicates that increasing [SBH] from 1 to 4 mM led to a corresponding increase in H₂ production. The correlation between [SBH] and the volume of produced H₂ is evident from the data. Based on these findings, an [SBH] of 1 mM was selected for the remaining experiments due to economic considerations. While higher SBH concentrations could potentially lead to greater H₂ production, it is important to consider the practical aspects. Using a lower SBH concentration allows for greater contact between H₂O and BH^{4−} with the active sites on the catalyst's surface, resulting in H₂ production at higher SBH concentrations. However, it is worth noting that the production of NaBO₂ occurs simultaneously with H₂ production. If the starting [SBH] is increased, NaBO₂ may accumulate on the catalyst and in the solution due to its limited solubility in alkaline solutions. This can increase the viscosity of the solution, reduce mass transfer, and subsequently decrease H₂ production [35,36]. Previous studies have also shown that higher concentrations of SBH can lead to a decrease in catalytic activity due to a limited number of available active sites for the desired reaction [35,37]. Figure 7b depicts the values of the HGR plotted against the starting concentration of SBH on a logarithmic scale. The slope of the linear regression analysis suggests that the catalytic hydrolysis of SBH exhibits a 0.065 order dependence on [SBH], indicating that the reaction follows zero-order kinetics with respect to [SBH].

2.2.4. Effect of Temperature

Figure 8a demonstrates the variation in the volume of H₂ generated during the SBH hydrolysis process at different temperatures: 20, 30, 40, and 50 °C. The experiments were conducted using solutions containing 50 mg of composite NFs, 1 mM SBH, 7% NaOH, and 1000 rpm. As expected, an increase in temperature led to a decrease in the completion time of the SBH hydrolysis process and an increase in the rate of H₂ generation. For example, the time required to reach a cumulative volume of 79 mL of H₂ was 18, 14, 11, and 7 min at temperatures of 298, 303, 318, and 328 K, respectively. Previous studies [38] have shown that increasing the temperature results in a higher availability of highly active reacting molecules leads to an accelerated rate of H₂ generation. This can be attributed to the increased kinetic energy and activity within the reaction system as the temperature is raised. Furthermore,

the kinetic behavior of the SBH hydrolysis process catalyzed by composite NFs was studied at different temperatures (Figure 8b). The linear portions of the H₂ production graphs at different temperatures were used to determine the rate constant (*k*) for the H₂ production from the hydrolysis process. The relationship between *k* and 1/*T* is shown in Figure 8b. The apparent activation energy (*E_a*) was determined using the Arrhenius plot and equation. The slope of the straight line yielded an *E_a* value of 26.16 kJ mol^{−1} for the SBH hydrolysis catalyzed by composite NFs. Comparing the obtained *E_a* value with those of different catalysts for H₂ production from SBH (Table 1), the prepared NFs exhibited a relatively low activation energy. This suggests that the composite NFs are efficient catalysts for the hydrolysis of SBH, resulting in a rapid reaction rate. The entropy (Δ*S*) and enthalpy (Δ*H*) of the hydrolysis reaction process were calculated using the Eyring equation and were found to be 0.044 kJ mol^{−1} K^{−1} and 23.56 kJ mol^{−1}, respectively. These Δ*H* and Δ*S* values can be used to calculate Δ*G* using Equation (1) and rewritten in Equation (2) as follows:

$$\Delta G = \Delta H - T\Delta S \quad (1)$$

$$\Delta G = 28.28 - 0.065T \quad (2)$$

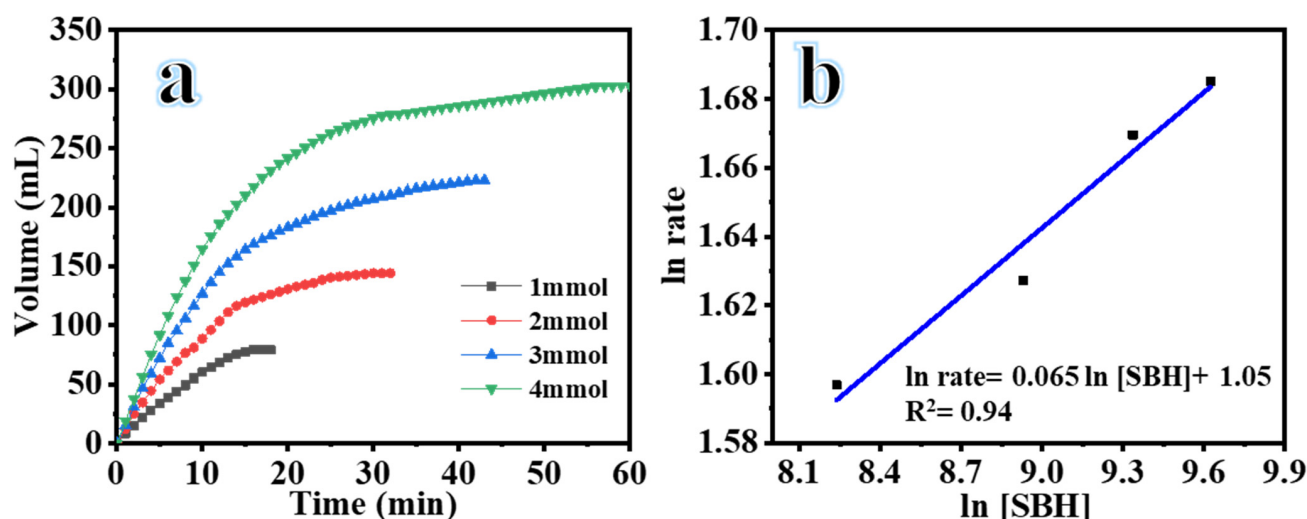


Figure 7. H₂ production volume versus time from hydrolysis of SBH using different [SBH] at 25 °C, composite NFs amount = 25 mg, and 1000 rpm (a), and logarithmic plot of H₂ production rate versus [SBH] (b).

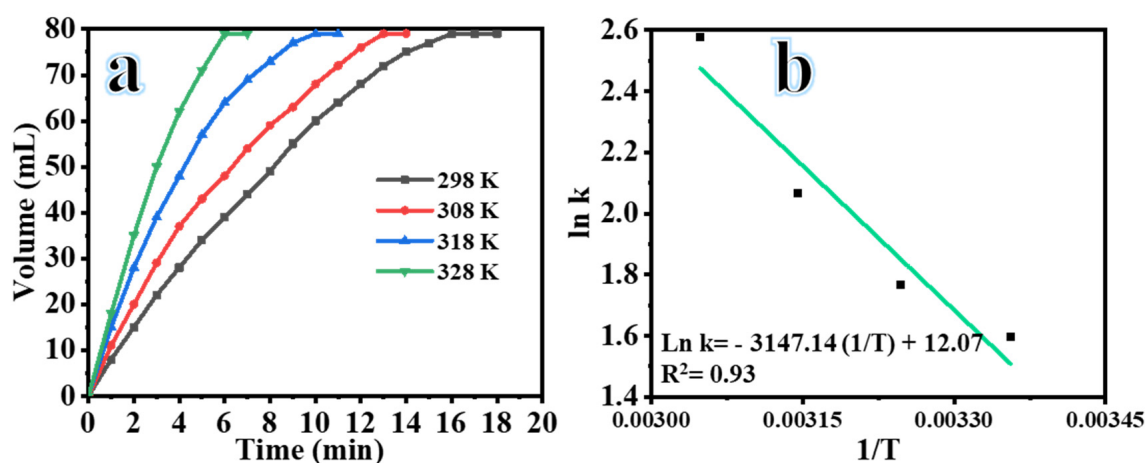


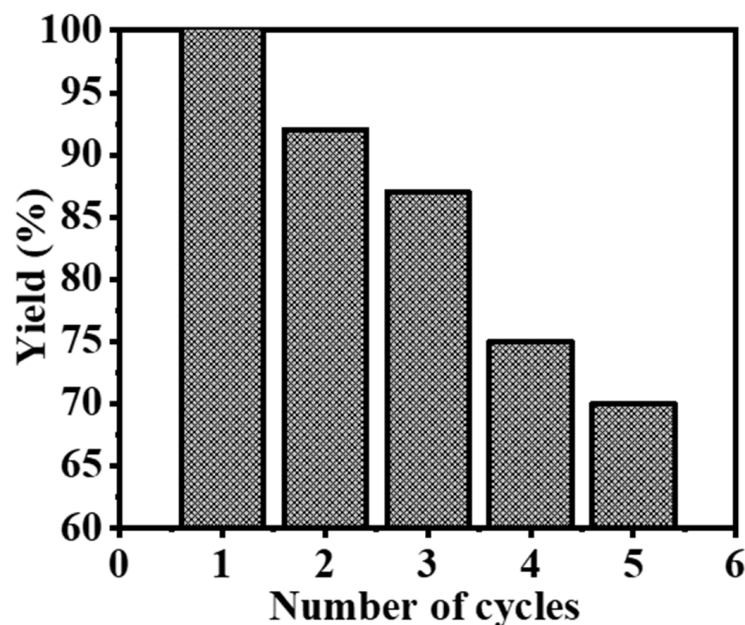
Figure 8. Influence of reaction temperature on the hydrolysis of SBH catalyzed by composite NFs (a) and the Arrhenius plot of ln rate versus 1/*T* (b) used ([SBH] = 1 mmol, composite NFs = 25 mg and 1000 rpm).

Table 1. Activation energies of different catalysts for H₂ generation from SBH.

Catalyst	Ea (KJ/mol)	Ref.
Co-Cu-Ni	40.6	[39]
Co-Cu-B/Ni foam	52	[40]
WSC-Cu	32.7	[41]
P(EP-g-AA)-Cu	42.6	[37]
Co-Ni/AC	68.9	[42]
Co-Ni/MWAC	40.7	[43]
Sm- Ni-Co-P/g-Al ₂ O ₃	52.1	[40]
Ni-Co-B	62	[44]
Cu-Fe-B	56	[45]
Cu based catalyst	61.16	[1]
CuO/Co ₃ O ₄	56.38	[46]
CuNWs	42.48	[14]
Cu,S-codoped TiO ₂ NPs, decorated-CNFs	26.16	This study

2.2.5. Reusability Test

To assess the reusability of composite NFs as catalysts for the hydrolysis of SBH, identical experimental conditions were maintained (25 mg of composite NFs, 1 mM SBH, 7% NaOH, 25 °C, and 1000 rpm) in a recycling study (Figure 9). It was observed that the catalytic activity of the composite NFs declined after the third cycle. In the first cycle, the composite NFs were able to produce the full stoichiometric amount of H₂. However, in the third cycle, only 87% of the total yield of produced H₂ was collected. This decline in catalytic activity can be attributed to the accumulation of boron compounds such as NaBO₂. These compounds can hinder the active sites of the catalyst and reduce its catalytic activity [45].

**Figure 9.** The reusability of the composite NFs catalyst.

3. Experimental

3.1. Preparation of Cu,S-Co-Doped TiO₂ NPs, Decorated-CNFs

Stock solution of 15% polyvinylpyrrolidone (PVP, average Mw ~1,300,000, Sigma-Aldrich, St. Louis, MO, USA) solution was prepared using a mixture of PVP powder, acetic acid (100%, Showa Chemicals Co., Ltd., Tokyo, Japan), and ethanol (99%, Sigma-Aldrich, St. Louis, MO, USA). The preceding mixture was kept in stirrer overnight at room temperature. A total of 2 g of titanium tetraisopropoxide (TIIP; 97%, Sigma-Aldrich) solution is added

dropwise to the previous solution. The two solutions were kept magnetically agitated at room temperature until they produced a translucent yellow sol-gel. After the sol-gel had been prepared, 1 g of copper (II) acetate monohydrate (CuAc, 98%, Sigma-Aldrich) was introduced and vigorously stirred for 3 h at 60 °C to ensure a uniform mixture. After that, ammonium sulfide ((NH₄)₂S, 40–48 wt.%, Sigma-Aldrich) was added dropwise to the aforementioned CuAc- contained Sol-gel. The formed colloidal green sol-gel that resulted was kept in the magnetic stirrer at room temperature for 5 h. For the purpose of fabricating NFs, the formed colloidal solution was put through an electrospinning apparatus, much as was carried out in our earlier research [18]. A micro-tip plastic syringe of 5 cm was filled with the colloidal solution. The plastic end was positively charged by a metallic wire that was immersed in the tip. The wire was connected to a high-voltage power supply. There was a gap of 18 cm that separated the cylindrical steel rotating drum covered with wax foil negative side from the tip of the plastic needle. Then, 20 kV was applied between the two electrodes. After the consumption the colloidal solution, the system was turned off and the formed mats were detached from the foil. The NF mats were vacuum dried at 50 °C overnight. After drying, they were calcined under vacuum in an Ar environment at 800 °C for 5 h. Pristine TiO₂-CNFs were also prepared using the same procedure excluding the CuAc and ammonium sulfide addition procedures.

3.2. Characterization

Scanning electron microscopy (SEM) (Hitachi S-7400, Tokyo, Japan) was used to examine the surface morphology of the composite NFs. X-ray diffraction (XRD) (Rigaku Co., Tokyo, Japan) using Cu K radiation ($\lambda = 1.54056 \text{ \AA}$) was employed to study the crystalline structure of the composite NFs. The high-resolution pictures were captured using a 200-kilovolt JEOL JEM-2200FS transmission electron microscope (TEM) from JEOL Ltd., Tokyo, Japan, equipped with energy-dispersive X-ray spectroscopy (EDX). The identification of the structure of CNFs was achieved through the utilization of Raman spectroscopy, with a laser excitation wavelength of 532 nm.

3.3. Hydrolysis Reaction

The hydrolysis process for H₂ generation was carried out in a 150 mL two-neck round-bottom flask used as a reaction vessel, and the volume of H₂ was estimated using the traditional water-displacement method. Typically, 25 mg of composite NFs catalyst, 1 mM alkaline SBH, 7% NaOH, 1000 rpm, and 25 °C were mixed in the flask. The yield of H₂ was calculated based on the volume of H₂ obtained from the SBH hydrolysis reaction vessel. Several independent experiments were conducted to determine the optimal values for the parameters involved in the reaction, such as NaOH concentration, catalyst amount, SBH concentration, and temperature. The yield of produced H₂ was determined using Equation (3).

$$\text{H}_2 \text{ yield (\%)} = (\text{Produced H}_2 / \text{theoretical H}_2) \times 100 \quad (3)$$

4. Conclusions

In this study, a composite NFs catalyst was synthesized using electrospinning techniques and employed as a catalyst for the hydrolysis of SBH, resulting in the production of H₂. The catalyst demonstrated significant catalytic efficiency, with a notable production of H₂ achieved using 100 mg of the catalyst. The presence of NaOH in the reaction mixture played a crucial role in the hydrolysis process. Increasing the NaOH concentration from 0% to 7% led to a reduction in the reaction time for SBH hydrolysis. However, further increasing the NaOH concentration to 10% resulted in an increased reaction time. This suggests that the optimal NaOH concentration for efficient SBH hydrolysis lies within the range of 7%. Moreover, increasing the amount of catalyst led to an improvement in the rate of H₂ production, indicating a near first-order dependence of the hydrolysis reaction on the catalyst dosage. On the other hand, the hydrolysis reaction exhibited a zero-order

dependence on the SBH concentration, indicating that changes in SBH concentration did not significantly affect the reaction rate. The composite NFs demonstrated exceptional catalytic activity, as evidenced by the low activation energy (E_a) value of $26.16 \text{ kJ mol}^{-1}$. This low E_a suggests that the composite NFs exhibit efficient catalytic performance for the hydrolysis of SBH. Additionally, the composite NFs exhibited stable and consistent H_2 generation during the reusability and stability tests for up to five cycles. This indicates their potential as a feasible catalyst for practical applications, as they maintained their catalytic activity over multiple cycles without significant degradation. Overall, the study establishes the effectiveness of the composite NFs catalyst for SBH hydrolysis, highlighting its potential as a promising catalyst for hydrogen production.

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Data Availability Statement: Not applicable.

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Conflicts of Interest: The authors declare no conflict of interest.

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