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Influence of Operational Parameters and Kinetics Analysis on Photocatalytic hydrolysis of Ammonia Borane for H₂ Production Used Heterojunction FeTiO₃/TiO₂-decorated Carbon Nanofibers

Abstract

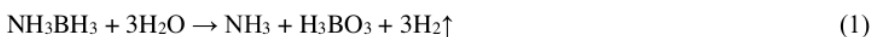
In this work, carbon nanofibers (CNFs) decorated with heterojunction iron titanate/titanate nanoparticles (FeTiO₃/TiO₂ NPs) were effectively prepared using electrospinning for use as a photocatalyst ³⁵ in the production of hydrogen (H₂) from ammonia borane (AB). The as-prepared FeTiO₃/TiO₂@CNFs exhibited strong photocatalytic activity for H₂ release from photohydrolysis of the AB when exposed to visible light. Under visible light illumination, the observed H₂ evolution over TiO₂@CNFs was 0.38 mol in 14 min, whereas over FeTiO₃/TiO₂@CNFs it was 2.97 mol. Using FeTiO₃/TiO₂@CNFs, H₂ generation was shown to be substantially higher under visible light irradiation (2.97 mol/14min) than in the dark (1.87 mol/14min). A mathematical model was ¹⁰ used to predict the photocatalytic activity of FeTiO₃/TiO₂@CNFs. Different operational factors were used to verify the accuracy of the mathematical model; they included the impact of AB ²⁴ concentration, light intensity, reaction temperature, and catalyst dosage. The reaction rate constant and equilibrium concentration were measured experimentally and compared to those predicted by the model. A good correlation was obtained between the calculated and measured photocatalytic activity.

Keywords:

Iron Titanate; ²⁹ Nanofibers; Ammonia Borane Complex; Hydrogen; Electrospinning; Visible Light.

Introduction

Advancement of the hydrogen economy has raised awareness of solid hydrogen storage materials (Simagina et al., 2021). Amongst, Ammonia borane (NH_3BH_3 , AB) has gained interest due to its low molecular weight (30.87 g/mol), high hydrogen content (19.6 wt.%), and durability in solid and aqueous solutions at ambient conditions (Demirci 2017). If an appropriate catalytic material is utilized, H_2 could be easily released from AB. The following equation describes the catalytic hydrolysis:



The first evidence of AB photohydrolysis was published in 2012 by Ayman et al (Yousef et al., 2012). Many investigations have been carried on since then, with the aim of releasing hydrogen from AB using photocatalytic materials of different types and compositions (Yousef et al., 2012, Yousef et al., 2013, Yousef et al., 2015, Song et al., 2021, Yan et al., 2021). The photohydrolysis process may release gravimetric hydrogen capacity in AB, similar to conventional hydrolysis, while light irradiation can limit ammonia formation (Simagina et al., 2021). Photocatalysis, a "green" technology, offers significant promise for producing green energy and environmental remediation by removing hazardous compounds and pathogens (Hoffmann et al., 1995). Recent years have seen much research on photocatalysts incorporating metal oxides, sulfides, halides, etc (Yousef et al., 2012, Yousef et al., 2017). However, it is still difficult to develop photocatalysts that are effective, long-lasting, inexpensive, and kind on the environment. Thus, researchers have placed a premium in recent years on finding ways to modify materials to increase their photocatalytic activity. **Recently, perovskite-type oxides have been presented as a potential photocatalytic material due to their visible light-driven characteristics (Moradi et al., 2021). Amongst them, Iron titanate (FeTiO_3) is attractive because to its non-toxicity, cheapness, and chemical**

and physical stability (Kim et al., 2009, Abdou and Fadl 2019). It is used in a broad variety of electromagnetic, chemical catalysts, photocatalysts optoelectronic and device applications ² due to its low band gap (2.5–2.9 eV) (Kim et al., 2009, Verma et al., 2014). The presence of Fe in the TiO₂ lattice could be improving its band gap and crystallinity (Moradi et al., 2021). Lee and his group developed a heterojunction of FeTiO₃ and TiO₂. The proposed photocatalyst was very effective in photodegrading organic molecules under UV light irradiation ²⁷ (Kim et al., 2009). Quang and et al (Truong et al., 2012), used a hydrothermal process to prepared heterojunction FeTiO₃/TiO₂ photocatalysts. The developed photocatalyst showed excellent ¹³ photocatalytic activity in the conversion of CO₂ to CH₃OH under both visible and UV-light irradiation. Masoud Moradi et al (Moradi et al., 2021), mixed graphene oxide (GO) with FeTiO₃ ²⁵ to reduce the band gap and improve the shift in the visible light range. They found that visible light causes faster photodegradation of phenolic compounds than ultraviolet light. High ¹² photocatalytic activity in the degradation of phenolic compounds ¹² was observed after 240 min of solar light irradiation. Incorporating GO into FeTiO₃ improved visible light absorption with decreasing hole-electro recombination. As is well-known, the shape and structure of nanomaterials, which in turn impact their catalytic activity, are directly influenced by the technique of synthesis used (Yousef et al., 2017). **Previous studies shown that carbon nanofibers (CNFs) may easily capture and transport photo-induce charges during the photocatalytic process owing to their high ¹⁰ electrical conductivity (Liu et al., 2008, Unalan et al., 2008).** The fabrication of heterojunction FeTiO₃/TiO₂@CNFs and their photocatalytic activity toward AB photohydrolysis are ² reported for the first time in the current study. The electrospinning technique was used in the preparation of the photocatalyst. Good

photocatalytic activity towards H₂ production from AB photohydrolysis was shown for the synthesized NFs.

2. Experimental

Experimental part is available in ²³ supporting information.

3. Results and discussion

3.1 Morphology study

There are a variety of techniques for making NFs, but electrospinning is by far the most common. The produced NFs have an excellent nanofibrous structure (Yousef et al., ¹⁶ 2015, Al-Enizi et al., 2017, Al-Enizi et al., 2019, Al-Enizi et al., 2020). Electrospun NF mats may have their nanofibrous structure preserved by sintering them at high temperatures in an inert atmosphere (Yousef et al., 2017, Yousef et al., 2017). Electrospun NF mats containing FeAc, TIIP, and PVP are shown in Sem image (**Fig. 1A**) after being vacuum dried at 50 °C overnight. The resulting NFs are shown to be smooth, continuous, and bead-free. ² Sintering at 800 °C for 5 hours in an argon environment maintains the nanofibrous structure, which is an interesting finding (**Fig. 1B**). In addition, tiny, white NPs may be seen growing on the surface of NFs. **Due to polymer partial breakdown, the size of electrospun NF mats was significantly reduced during calcination, from 251.09 nm to 144.81 nm (Fig.1 C and D).** EDX analysis (**Inset Figure 1B**) reveals that iron (Fe), titanium (Ti), oxygen (O), and carbon (C) are the only elements present in the NFs.

3.2 Phase study

Figure 2A ¹⁹ displays the XRD patterns of the powder produced following the calcination process. According to the data, the formation of one peak from tetragonal TiO₂ rutile phase (JCPDS#21-1276) at 2θ value ~36.62° corresponds to (101). The hexagonal ilmenite FeTiO₃ phase (JCPDS#03-0781) has four peaks with 2θ value ~42.67°, 61.58°, 73.56°, and 77.63°, which are consistent with (202), (214), (217), and (223). In addition, ³¹ a low intensity peak at a 2θ value ~26.1° is seen, which is associated with the (002) and suggests the synthesis of carbon-like graphite (Yousef et al., 2017, Xue et al., 2021). **The crystallite diameters of FeTiO₃ and TiO₂ were determined using Scherrer's equation to be 4.56 and 5.19 nm, respectively.** **Fig. 2B** shows a normal TEM image of the introduced NFs. Growth of NPs on the surface of NFs is easily visible in this image. The generated NPs might be FeTiO₃/TiO₂, whereas the NFs could be carbon. The HR-TEM image (**red circle in Fig. 2B**) displays a high crystalline structure with parallel atomic planes (**Fig. 2C**). Furthermore, distinct lattice fringes were identified in the sample, which might be related to the FeTiO₃ and/or TiO₂ phases. The production of polycrystalline material is shown in the SAED pattern of the designated region (**red circle in Fig. 2B**) seen in inset **Fig. 2C**. As demonstrated in **Figure 2D**, the primary NFs are formed of amorphous structure (**blue circle in Fig. 2B**), suggesting the development of amorphous carbon. It may say ² that the NPs are ² crystalline FeTiO₃/TiO₂ NPs, while the NFs are amorphous carbon. ² TEM-EDX was utilized to determine the chemical composition of produced NFs (**Fig. 3**). **Figure 3A** ²⁸ depicts a STEM image of a single chosen NF, as well as a line TEM-EDX analysis and the related EDX analysis, which are displayed in **Figs. 3B, C, D, and E**. The identical distributions of Ti, O, Fe, and C are observed, as illustrated in Figure, providing more evidence for the synthesis of FeTiO₃ and TiO₂. The addition of carbon to the photocatalytic process may result in the following benefits: i) enhance photo-induced e⁻

and h^+ separation, and ii) enhance AB contact with the photocatalyst, via an adsorption process.

3.3. H₂ generation from the photohydrolysis of AB

¹¹ At ambient temperature and under visible light irradiation ($I=25 \text{ Wm}^{-2}$), ¹¹ the photocatalytic activity of TiO₂@CNFs and FeTiO₃/TiO₂@CNFs was evaluated in terms of H₂ release from the hydrolysis of AB (1mmol AB) (Fig.4). The observed H₂ evolution over TiO₂@CNFs and FeTiO₃/TiO₂@CNFs were found to be 0.38 and 2.97 mmol in 14 min, respectively. FeTiO₃/TiO₂@CNFs displayed substantially greater activity than TiO₂@CNFs. This might be because to the quicker electro/hole separation on FeTiO₃/TiO₂@CNFs, which improves H₂ production from AB photohydrolysis compared to TiO₂@CNFs. Also, H₂ generation was shown to be substantially higher ³⁴ in the presence of visible light than in the absence of it (Fig.4). Even when exposed to visible light, no H₂ was detected in the absence of a photocatalyst, showing that AB was extremely stable in water. The effects of starting concentration (C_i), reaction temperature (T), photocatalyst dosage (CNFs), and light intensity (I) on the photocatalytic process have been investigated, and a mathematical model to predict these reactions has been developed (Eq.S2) (Ali et al., 2020):

3.3.1. Effect of initial AB concentration (C_i)

²¹ Under visible light irradiation, the influence of the initial AB concentration (0.1, 0.2, 0.3, and 0.4M) on H₂ generation from AB photohydrolysis was studied (Fig.5A). As indicated in the Figure, the initial H₂ production rate was relatively unaffected by the AB concentration. The photoproduction of H₂ at varying AB concentrations may be attributed to a pseudo-first order reaction, as described by the Langmuir Hinshelwood ⁹ (LH) model (Eq. S3).

The relationship between $\ln C_i/C_f$ and reaction time is seen in Figure 5B. Rate constants (K_1) may be estimated from the slopes of the straight lines shown in Figure

5B. As [AB] increases from 0.1 to 0.4M, K_1 decreases from 0.2318 to 0.0504 min^{-1} (Table S1). By transforming Eq. S7 into a straight-line equation, we can determine the correlation between K_1 and initial [AB] (Fig. 5C).

3.3.2. Effect of reaction temperature (T)

The rate of H_2 production in the presence of $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$ vs irradiation time over a range of reaction temperatures is shown in Figure 6A. Figure shows that when the reaction temperature is raised from 25 to 40 °C, photohydrolysis of AB increases due to enhanced charge carrier mobility and interfacial charge transfer. As the temperature of the reaction rises, photoelectron-hole pair movement increases, accelerating the interaction between adsorbed oxygen and electrons and between holes and OH radicals, speeding up AB photohydrolysis (Ojstršek et al., 2013, Meng et al., 2017, Chen and Hsu 2021). The kinetic analyses of AB photohydrolysis over $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$ at different temperatures are shown in Figure 6B. Increases in the AB photohydrolysis efficiency with increasing temperature are shown by an increase in the reaction rate (K_2) from 0.2318 to 0.4148 min^{-1} as the reaction temperature increases from 25 to 40 °C (Table S1). A straight-line slope from Figure 6B could be used to determine the rate constant (K_2). The plot of K_2 vs $1/T$ reveals a straight-line relationship (Fig. 6C). Using the Arrhenius equation (Eq. S8), we can get the activation energy (E_a) = 3.0162×10^4 of the photocatalytic process (Marandi et al., 2012). Applying the natural log to both sides of Eq. S9 to remove the exponent.

3.3.3. Effect of light intensity (I)

Increases in light intensity increase the rate at which H_2 is produced by AB photohydrolysis (Wei et al., 2021). The influence of light intensity on AB photohydrolysis is shown in Figure 7A. According to the results, the photohydrolysis

rate of AB improved when the light intensity was increased. Enhanced AB photohydrolysis is likely due to an increase ⁷ in the number of photons available per unit of time. The rate constant (K_3) could be determined from the slope of the straight lines produced from the plot of \ln (H₂ generation rate) vs time (**Fig. 7B**) under varying light intensities. With an increase in light intensity, AB photohydrolysis increases, as shown by an increase in K_3 from 0.2318 to 0.3131 min⁻¹ between 25 and 40 Wm⁻² (**Table S1**). Since K_3 is directly proportional to I, we may determine their relationship by plotting $1/K_3$ vs I (**Fig. 7C**) and calculating for I using **Eq. S10**.

3.3.4. Effect of photocatalyst dose (FeTiO₃/TiO₂@CNFs)

H₂ production rate vs irradiation time for different FeTiO₃/TiO₂@CNFs concentrations (0.2, 0.3, 0.4, and 0.5 gm L⁻¹) is shown in **Figure 8A**. Increases in the concentration of FeTiO₃/TiO₂@CNFs result in a higher rate of H₂ production, as indicated in the figure. This could be due to the increased surface area of the FeTiO₃/TiO₂@CNFs facilitates more efficient AB photohydrolysis. The kinetic studies over varying amounts of FeTiO₃/TiO₂@CNFs are shown in **Figure 8B**. When the photocatalyst concentration is increasing from 0.2 to 0.5 gm L⁻¹ (**Table S1**), the reaction rate (K_4) increases from 0.2318 to 0.5533 min⁻¹, showing an enhancement in the AB photohydrolysis. This may be because more adsorption and catalytic sites are available at the surface as a result of the higher concentration of FeTiO₃/TiO₂@CNFs. **Figure 8C** illustrates the relationship ⁷ between the logarithm of the reaction rate (K_4) and the logarithm of the dosage of [FeTiO₃/TiO₂@CNFs]. According to the best-fit line, the generated H₂ ³³ follows pseudo-first order kinetics in terms of catalyst concentration, with a slope of 0.9912. The Langmuir-type relationship (**Eq. S11**) (Ali et al., 2020) was used to determine how the FeTiO₃/TiO₂@CNFs concentration affected K_4 (**Fig. 8D**). This equation can be transforming to straight line equation (**Eq. S12**).

3.3.5. Development of model rate equation

According to **Equations (S7), (S9), (S10), and (S11)**, the rate constant is a function of the FeTiO₃/TiO₂@CNFs dose, AB concentration, light intensity, and reaction temperature. **Table S2** displays the calculated values of KR, Ea, m, and K_{NFs} using multiple regression analysis. **The equation constant k' was calculated using Eq. S2** by substituting the previous obtained values of C_i, T, I, C_{NFs} and values in **Table S2**, K_{app} can be rewritten as follows:

$$K_{app} = K' \left(\frac{-29.52}{1-29.52*25_i} \right) \left(\exp \frac{-3.0162*10^4}{8.314*298} \right) (55*10^{-4} * 25) \left(\frac{0.34*0.2}{1+0.34*0.2} \right)$$

$$0.2318 = K' \left(\frac{-29.52}{1-29.52*25_i} \right) \left(\exp \frac{-3.0162*10^4}{8.314*298} \right) (55*10^{-4} * 25) \left(\frac{0.34*0.2}{1+0.34*0.2} \right)$$

$$K' = 3.39*10^7$$

$$K_{app} = 3.39*10^7 \left(\frac{-29.52}{1-29.52C_i} \right) \left(\exp \frac{-3.0162*10^4}{8.314T} \right) (55*10^{-4} I) \left(\frac{0.34C_{cat}}{1+0.34C_{cat}} \right)$$

Figure 9 depicts a comparison of experimental data and determined K_{app} for AB photohydrolysis under various conditions. The obtained plot demonstrated **that the experimental data is in good agreement with the model** calculated date, confirming the use of the **model to predict the reaction rate constant** under various operational conditions.

3.3.6. Photocatalytic mechanism

Producing active radicals is the first step in the photocatalytic process. The AB photohydrolysis method for H₂ generation over FeTiO₃/TiO₂@CNFs is shown in Figure 10. Titanium dioxide, which has a band gap of 3.2 eV, is related to a limitation on the number of active radicals that may exist when illuminated by visible light (Kim et al., 2009). FeTiO₃ has a valence band (VB) potential of +2.6 eV relative to a standard hydrogen electrode (SHE) and a conduction band (CB) potential of -0.2 eV (Moradi et al., 2021). The photogenerated positive hole (h⁺)

combines with $\text{H}_2\text{O}/\text{OH}^-$ to form hydroxyl radicals because the VB position of FeTiO_3 is so close to that of TiO_2 (+2.7 eV), allowing the hole to move from the VB of FeTiO_3 to that of TiO_2 (Moradi et al., 2021). With visible light irradiation, electrons in the CB of TiO_2 can be excited to FeTiO_3 CB, as shown in Scheme 1. This suggests that FeTiO_3 can be an effective sensitizer when exposed to visible light. Photogenerated electrons in FeTiO_3 move freely toward the surface of the CNFs, implying that photogenerated electrons and holes are efficiently separated due to low recombination (Zhang et al., 2012). Rather than recombination with holes on the surface of TiO_2 , the photogenerated electron in the CB of FeTiO_3 can react with an oxidant such as an oxygen molecule (O_2) to produce $\cdot\text{O}_2^-$.

Conclusion

Successful preparation of heterojunction $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$ used electrospinning and calcination techniques. Under visible light, $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$ demonstrated fast hydrogen release via photohydrolysis of AB. The measured H_2 evolution over $\text{TiO}_2@\text{CNFs}$ and $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$ were 0.38 mol and .97 mol in 14 min, respectively. This could be due to the faster electro/hole separation on $\text{FeTiO}_3/\text{TiO}_2@\text{CNFs}$, which improves H_2 generation from AB photohydrolysis over $\text{TiO}_2@\text{CNFs}$. It's worth noting that H_2 production increased significantly when exposed to visible light versus when exposed to darkness. The mathematical model predicts the reaction rate constant (k_{app}) by analyzing the effect of various operational conditions. We observed that the experimental data agree well with the model-estimated data.

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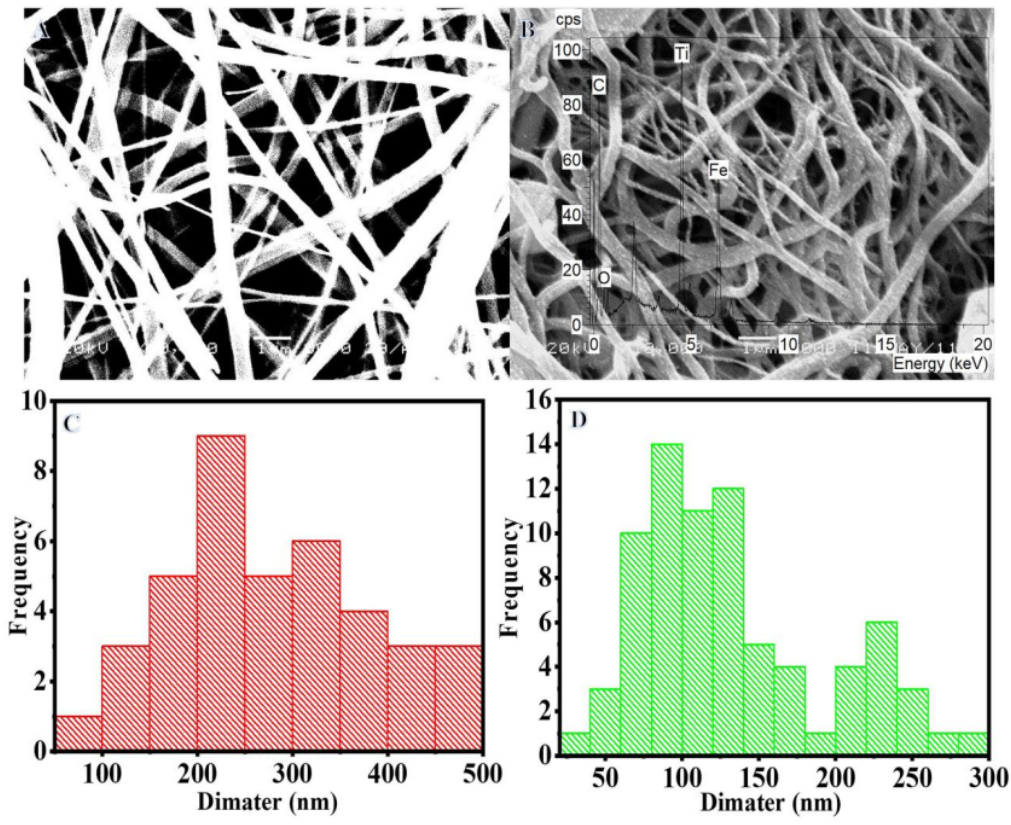


Fig. 1: SEM images of the electrospun FeAc/TIIP/PVP nanofiber mats after drying at 50 °C overnight (A) and the produced powder after calcination in Ar at 800oC (B).

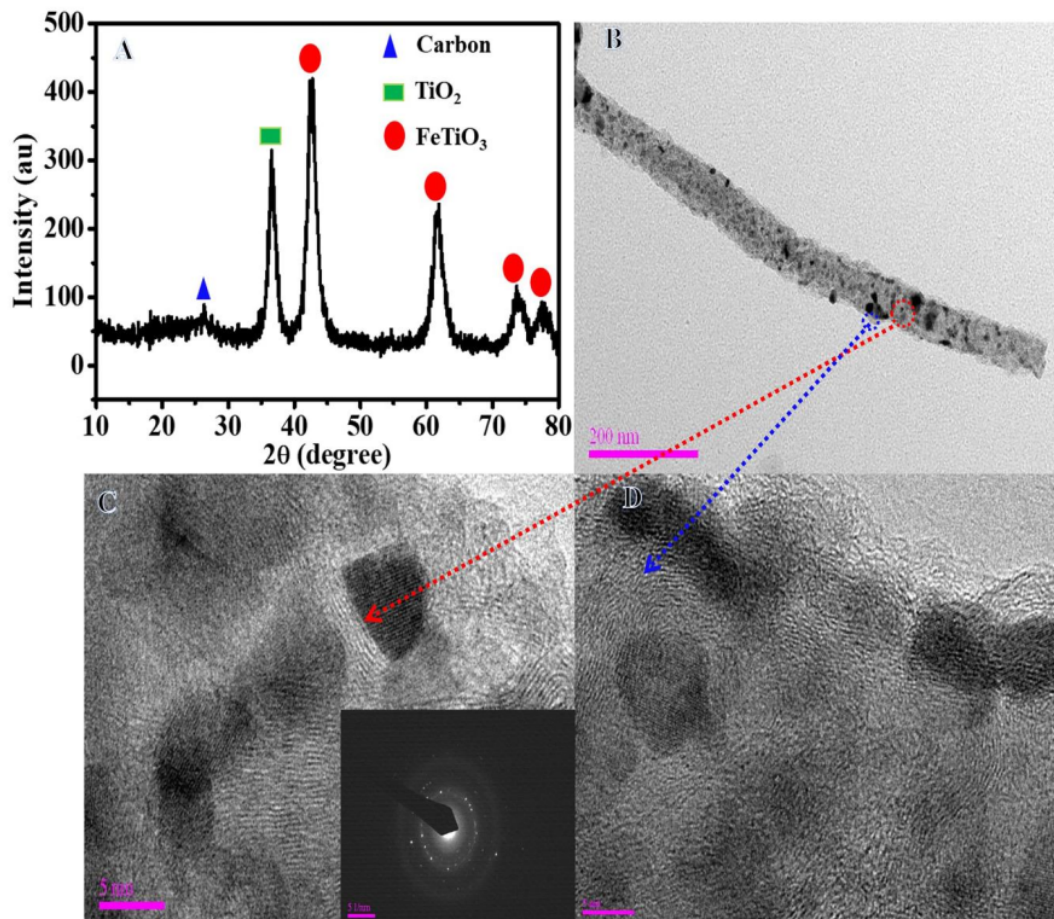


Fig. 2: XRD patterns (A), Normal TEM image of single NF (B) and HR-TEM images (C, D) of produced powder after calcination of electrospun FeAc/TiIP/PVP nanofiber mats at 800 °C in Ar atmosphere. The inset in Panel C represents the SAED of the marked area (red circle).

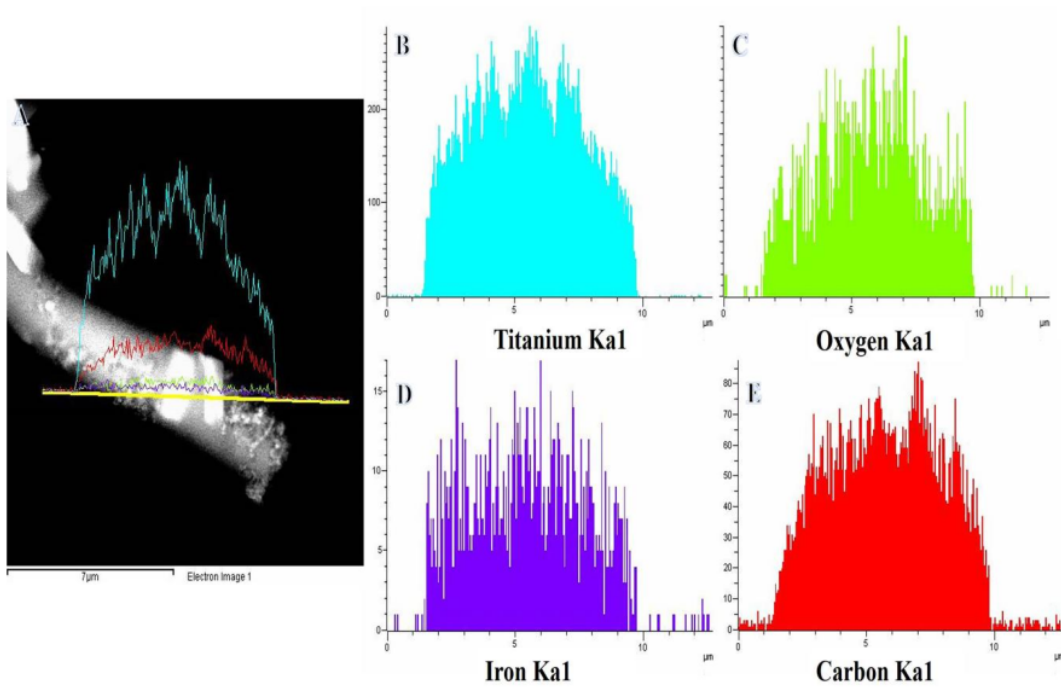


Fig. 3: TEM image of single NF along with the line TEM EDX analysis (A) and the corresponding Ti, O, Fe, and C line analyses TEM EDX (B, C, D).

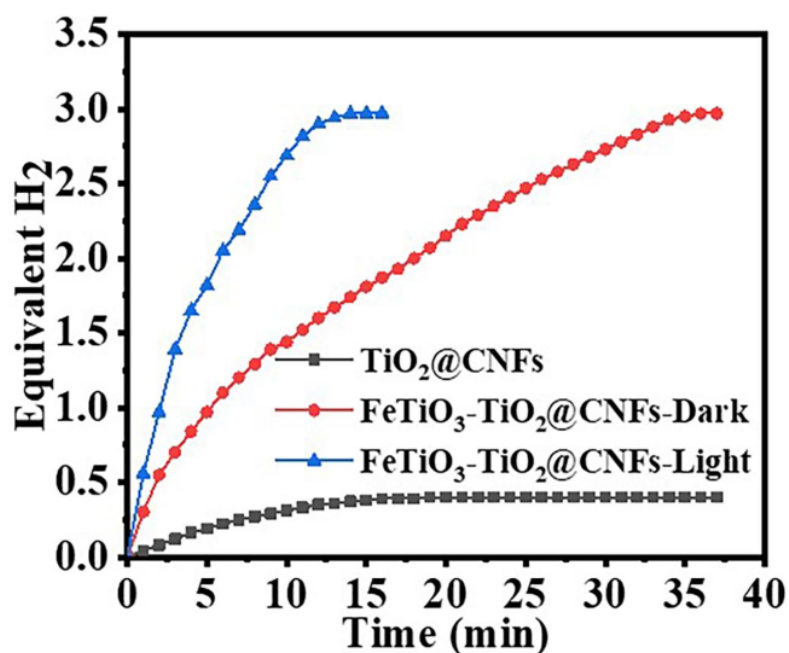


Fig. 4: H₂ generation in the presence of different photocatalysts. (Photocatalyst amount = 0.2 gm L⁻¹, Ci = 0.1M, T = 298 K, and I = 25 W m⁻²).

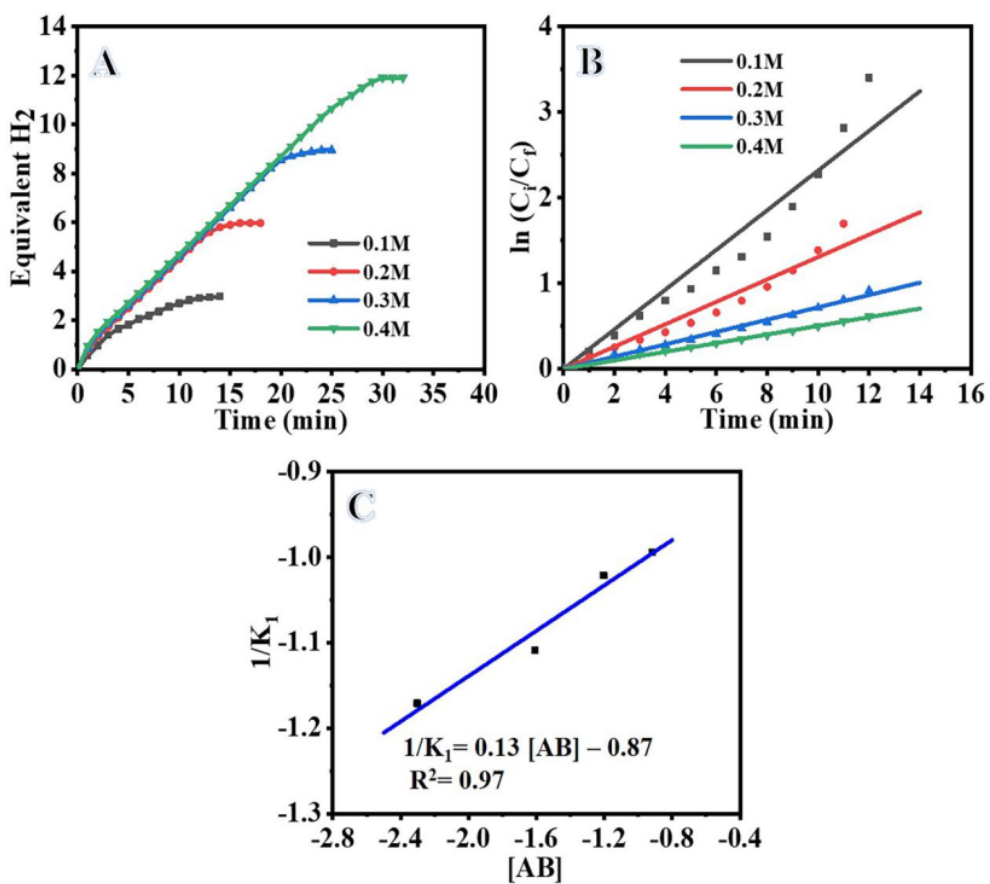


Fig. 5: Influence of [AB] on H₂ generation (A), $\ln C_i/C_f$ vs. time (B), Modified LH plot for AB photohydrolysis (C). (Photocatalyst amount = 0.2 gm L⁻¹, T = 298 K, and I = 25 W m⁻²).

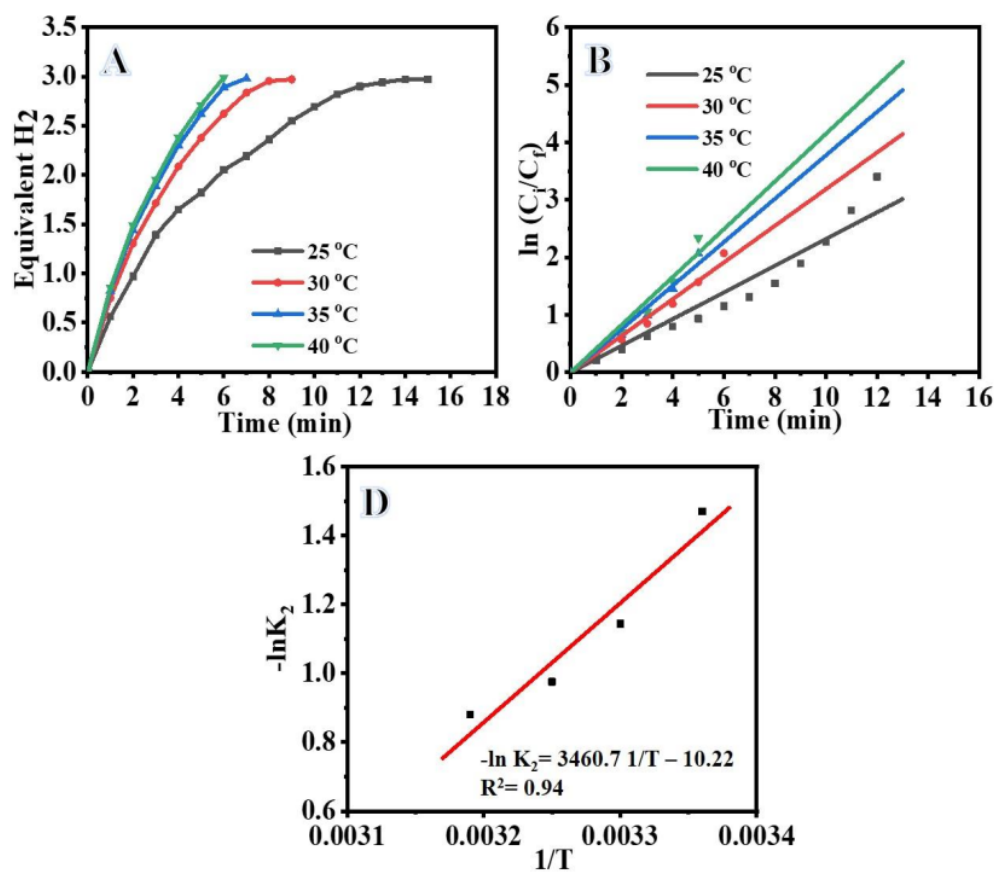


Fig. 6: Influence of Temperature on H₂ generation (A), ln C_i/C_f vs. time (B), and (C) and Arrhenius plot for AB photohydrolysis. (Photocatalyst amount = 0.2 gm L⁻¹, C_i = 0.1M, and I = 25 W m⁻²)

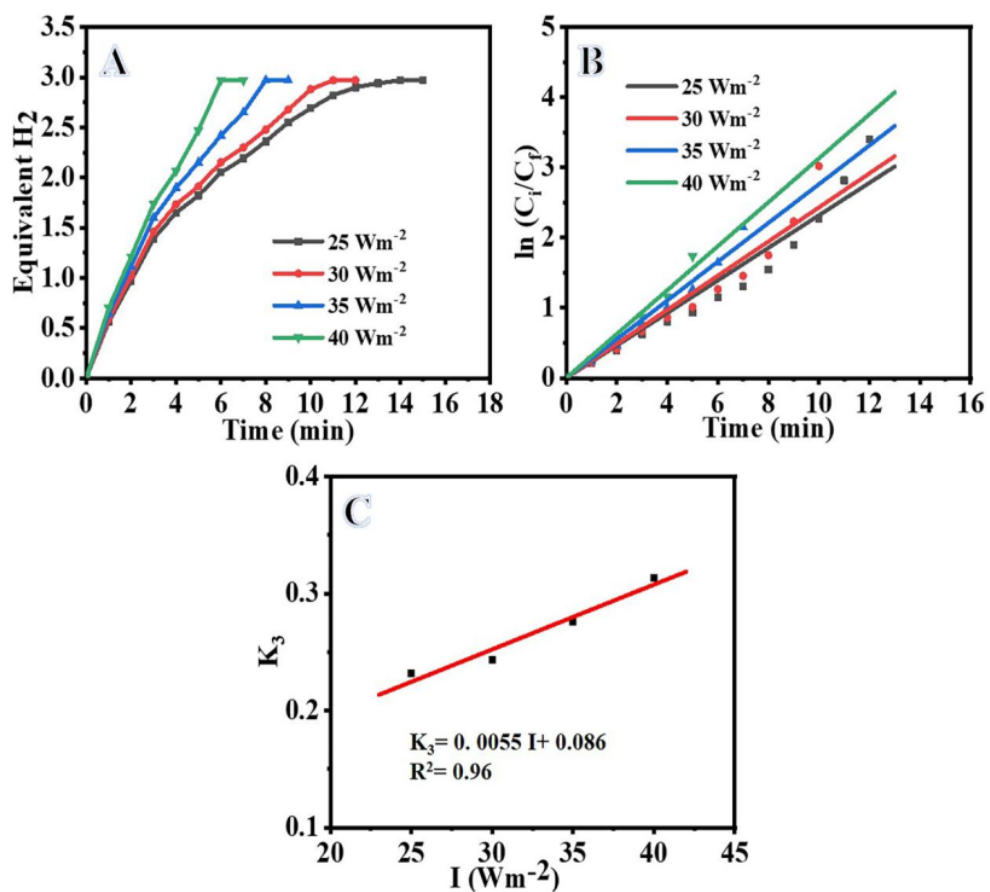


Fig. 7: Influence of light intensity on H₂ generation (A), ln C_i/C_f vs. time (B), and logarithmic value of the H₂ generation vs. logarithmic value of light intensity (C). (Photocatalyst amount = 0.2 gm L⁻¹, C_i = 0.1M and T = 298 K).

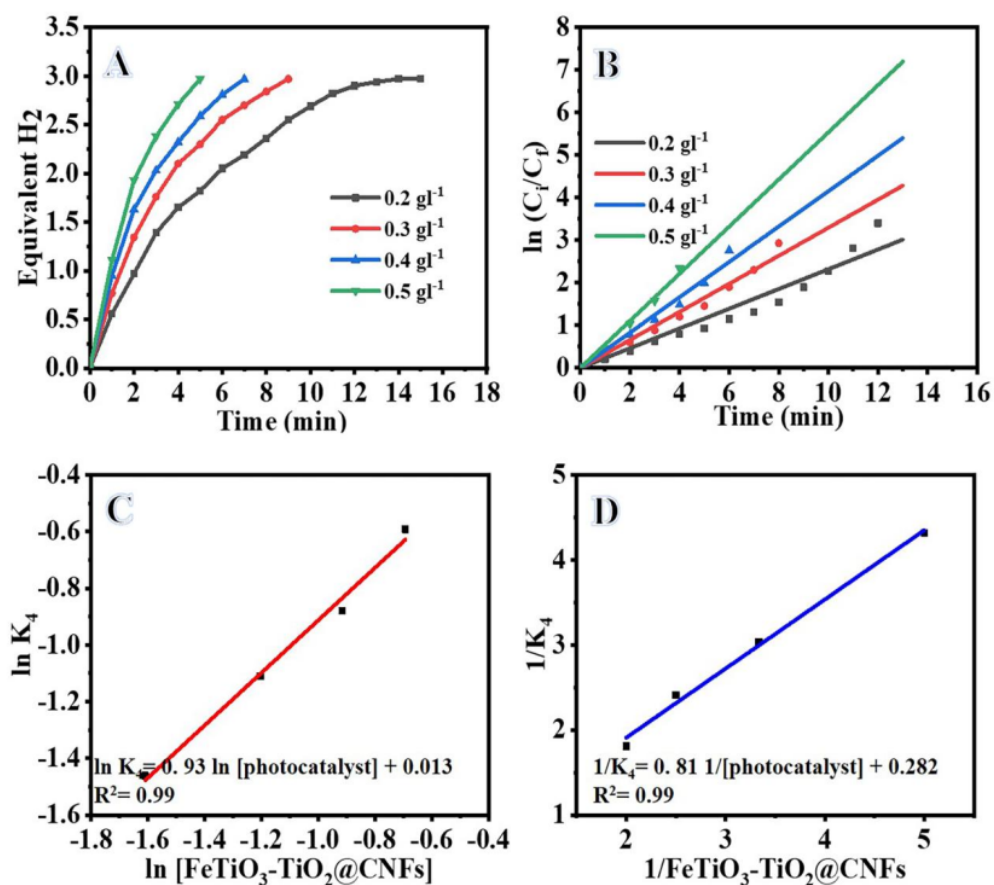


Fig. 8: Influence of catalyst amount on H₂ generation (A), ln Ci/Cf vs. time (B), the logarithmic value of the H₂ generation rate vs. logarithmic value of photocatalyst amount (C), and Langmuir-type plot for AB photohydrolysis (D). (C_i=0.1M, T = 298 K, and I = 25 W m⁻²)

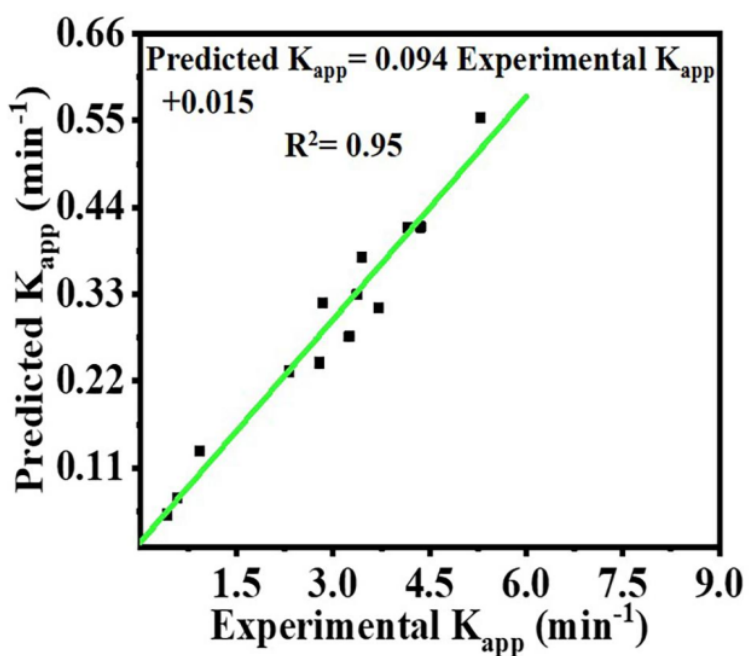


Fig. 9: Comparison of experimental k_{app} and predicted k_{app} values.

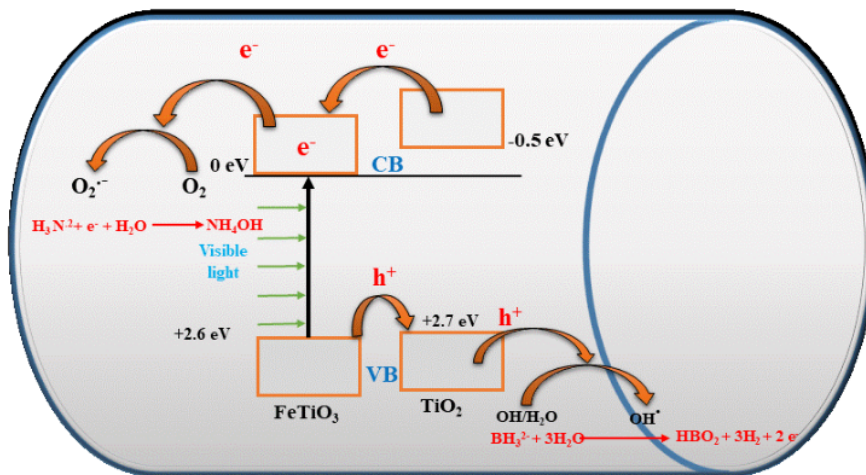


Fig. 10: Schematic diagram for the creation and influence of electrons and holes in the hydrolysis of the ammonia borane complex.

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