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Original article

Effect of triethanolamine and sodium hydroxide concentration on the activity of Pt/g-C_3N_4 catalyst in the reaction of photocatalytic hydrogen evolution under visible light irradiation

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Abstract In this work, the dependences of the rate of photocatalytic hydrogen evolution under visible light irradiation on the concentration of triethanolamine and sodium hydroxide for 0.1 wt.% Pt/g-C_3N_4 photocatalyst were studied. The kinetic dependences of the reaction rate versus substrate initial concentration described by the Langmuir–Hinshelwood monomolecular model. Optimal initial conditions for highly efficient hydrogen evolution have been studied. It is shown that under such conditions the catalyst is a stable material in long-term experiments. The maximum rate of hydrogen evolution was 7.2 mmol$\cdot$g^{-1}h^{-1}.

Keywords photocatalysis, hydrogen evolution, carbon nitride, triethanolamine, visible light

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1. Introduction

With annual population growth, there is a sharp increase in energy demand. However, the world energy industry is based on fossil raw materials [1]. At the same time, oil and coal combustion products (CO_2, SO_x, and NO_x) cause serious damage to the environment, animals and people [2]. Therefore, the issue of decarbonization of the economy and the search for new environmentally friendly energy sources is becoming acute [3]. Hydrogen is considered as a promising alternative energy source because of its high energy density, zero pollution, etc [4–6] The only method of hydrogen production from renewable energy sources is photocatalytic splitting of water. In this case, the hydrogen produced acts as an energy carrier [1].

Photocatalysts based on graphitic carbon nitrate g-C_3N_4 have attracted attention because of their chemical and thermal stability [7–9]. Also the position of the valence band (VB) and conduction band (CB) corresponds to the thermodynamic conditions of water splitting with the formation of hydrogen and oxygen [10–12]. However, a considerable disadvantage of g-C_3N_4 is, firstly, the low specific surface area, and secondly, the rapid recombination of photogenerated electrons and holes, which significantly reduces the activity of photocatalysts [13, 14]. To increase the efficiency of photocatalytic hydrogen evolution, electron donors, so-called "sacrificial agents", are added to the solution. Electron donors are oxidized by photogenerated holes in the valence band of g-C_3N_4, which avoids recombination of electrons and holes, and hence spatial charge separation occurs. Further, photoexcited electrons from the conduction band of g-C_3N_4 participate in the process of water reduction with the formation of hydrogen. Typically, the sacrificial agents for g-C_3N_4-based catalysts are methanol, ethanol and triethanolamine [15]. However, methanol is decarbonylated at room temperature during the reaction and carbon dioxide is adsorbed on the catalyst surface, so, self-poisoning of the catalyst occurs. In the case of titanium dioxide, surface purification by highly electrophilic forms of oxygen (holes) is possible. Thus, the use of methanol is inefficient for g-C_3N_4-based catalysts due to the lack of surface active oxygen [16]. The use of triethnolamine is of great interest, firstly, because the adsorbed TEOA molecules on the surface of the photocatalyst protect it from photocorrosion and degradation of the p-conjugated graphite-likecarbon nitride structure [17]. Secondly, photocatalytic hydrogen evolution results in the oxidation of TEOA, which is attractive in terms of its carcinogenic nature in the pristine state [16]. Thus, it is possible to combine hydrogen evolution with the destruction of TEOA structure during photocatalytic process.

For efficient photocatalytic process, it is necessary to study the influence of reaction conditions on the activity of the catalyst in the target process. The acidity of the medium is known to have a complex effect on the reaction rate due to changes in the state of the catalyst surface, redox potential H^+/H_2 [18, 19]. The aqueous solution of triethanolamine exhibits weak basic properties. When alkali is added to the reaction suspension, deprotonation of TEOA occurs, which leads to an increase in the pH of the solution. In terms of zeta potential, g-C_3N_4 has zero surface in the pH range of 4.4 – 5.1 [20]. Consequently, the pH of the reaction suspension plays a significant influence on the adsorption-desorption properties on the surface of g-C_3N_4, because at alkaline pH, the surface of carbon nitride is negatively charged, while at acidic pH, on the contrary, it is positively charged.

Previously, our group suggested a new method for the synthesis of graphitic carbon nitride. Synthesis of g-C_3N_4 proceeded through the formation of a supramolecular complex melamine-cyanuric acid between melamine and cyanuric acid, which was further calcined at 550 °C for 1 hour [21, 22]. According to X-ray phase analysis data, the coherent scattering region, which characterizes the average crystallite size, is 10.1 nm, which confirms the nanoscale of the obtained material [21,22]. Due to the branched network of pores in the obtained material, easy access of reagents to the co-catalyst is provided. A method of platinum deposition on the surface of the catalyst from labile Pt(IV) nitrate complexes was also developed, which provides a uniform distribution of platinum ionic particles on graphitic carbon nitride [23,24]. The size of platinum nanoparticles is 1 – 2 nm according to the data of transmission electron microscopy [21,22]. The combination of g-C_3N_4 material preparation and Pt particles deposition methods allowed to achieve high activity in the photocatalytic hydrogen production.

Therefore, the aim of this work was studying the influence of triethanolamine and sodium hydroxide concentration on the activity of the previously proposed catalyst 0.1 wt.% Pt/g-C_3N_4 in the reaction of photocatalytic hydrogen evolution under visible light irradiation. Selection of optimal initial conditions for highly efficient process of photocatalytic hydrogen evolution.

2. Experimental

2.1. Photocatalyst synthesis

2.1.1. Synthesis of g-C_3N_4. Graphitic carbon nitrate g-C_3N_4 was prepared according to the procedure described in [21, 22]. Briefly, a suspension consisting of melamine and cyanuric acid was suspended in water, stirred on a magnetic stirrer for 12 hours, and heated at 90 °C. A supramolecular melamine-cyanuric acid (MCC) complex was obtained. After that, the MCC, was washed with water several times, dried in vacuum, and then calcined at 550 °C for 1 hour (heating rate 1 C/min). The prepared sample of graphite-like carbon nitride is hereinafter denoted by the abbreviation CN.

2.1.2. Synthesis of 0.1 wt.% Pt/g-C_3N_4. Platinum deposition on the surface of g-C_3N_4 was carried out according to the procedure described in detail in [24]. A sample of g-C_3N_4 was suspended in acetone, the required amount of previously prepared platinum precursor $(Me_4N)_2[Pt_2(\mu{-}OH)_2(NO_3)_8]$ was added, and stirred for 12 hours. Then the precipitate was washed with acetone several times, dried in an air current. Then the obtained powder was calcined in a hydrogen atmosphere at 500 °C for 1 hour at a heating rate of 10 °C/min. A 0.1 wt.% Pt/g-C_3N_4 catalyst, hereinafter referred to as Pt–CN, was obtained.

2.2. Photocatalytic experiments

The photocatalytic hydrogen evolution experiment was carried out in a horizontally illuminated reactor (Fig. 1). A 50 mL suspension consisting of 25 mg of Pt–CN catalyst, (0 – 70) vol. % triethanolamine (TEOA), and (0 – 5) M NaOH was placed in the reactor. The catalyst was pre-suspended in an ultrasonic bath. The reactor was purged with argon for 15 minutes to remove the oxygen present in the system. An LED with emission maximum at 430 nm, power 50 mW/cm^2, distance from the quartz window to the suspension 6 cm was used as a radiation source. Gas phase analysis for quantitative determination of the evolved hydrogen was carried out using a gas chromatograph CHROMOS GC-1000 equipped with a thermal conductivity detector with a NaX zeolite column.

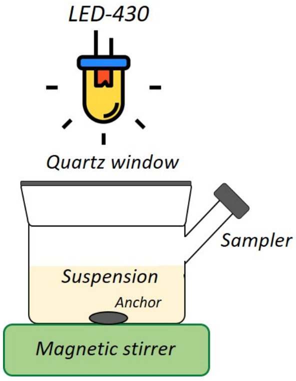

FIG. 1. Schematic representation of the reactor for photocatalytic hydrogen evolution

The apparent quantum efficiency (AQE) was calculated using the following formula:

$$\text{AQE} = \frac{2 \cdot W(\text{H}_2)}{N_f},$$

where $W(\text{H}_2)$ is the rate of photocatalytic hydrogen release (mol/min), N_f is the photon flux.

The photon flux was $3 \cdot 10^{-4}$ mol/min for a 430 nm LED.

3. Results and discussion

3.1. Photocatalytic activity

At the first stage of the work, the effect of TEOA concentration on the activity of Pt–CN in the reaction of photocatalytic hydrogen evolution under visible irradiation (maximum emission of LED 430 nm) was studied. In the absence of electron donor, the rate of hydrogen evolution is low and equals 0.31 mmol$\cdot$g^{-1}h^{-1}. When TEOA is added to the reaction suspension, there is a sharp increase in the reaction rate, a linear increase is observed up to 30 vol.% TEOA. The reaction rate in this case is 3.92 mmol$\cdot$g^{-1}h^{-1}. Further, with increasing TEOA concentration, the reaction rate reaches a plateau (Fig. 2a).

The dependence is described by the Langmuir–Hinshelwood model of monomolecular adsorption (Fig. 2b), implying a one-step oxidation of the adsorbed substrate, with hydrogen release occurring via photoreduction of oxygen-containing molecules proceeding in several stages [25]. Therefore, the obtained data were approximated by the Langmuir-Hinshelwood model according to the following equation:

$$W_0 = k\frac{K \cdot C_0}{1 + K \cdot C_0},$$

where W_0 is the initial rate of photocatalytic hydrogen evolution, k is the apparent rate constant of the reaction, K is the adsorption constant of TEOA, C_0 is the initial concentration of triethanolamine.

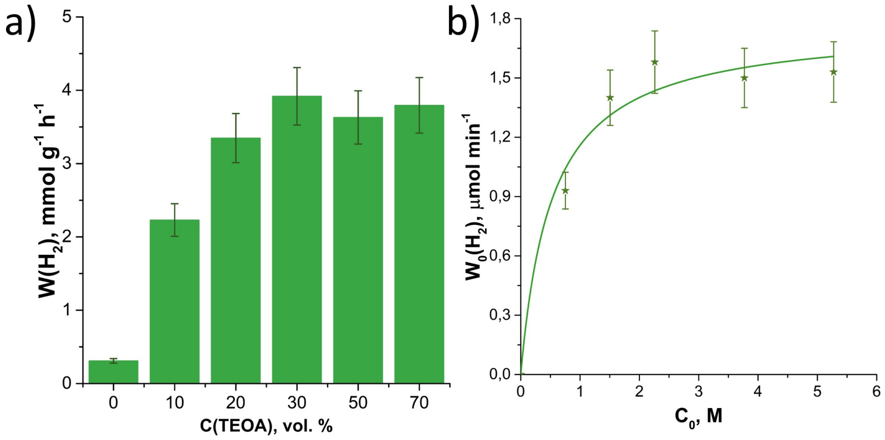

FIG. 2. Dependence of hydrogen evolution rate on TEOA concentration (a) and approximation of experimental data by the Langmuir–Hinshelwood model (b) in the presence of Pt–CN. Experimental conditions: $C(\text{TEOA})$ =0 – 70 vol.%, m_{cat} = 25 mg, V_{susp} = 50 mL, λ = 430 nm

Table 1 shows the obtained values of approximation of experimental data using the above model. The coefficient of determination R^2 is 0.97, which shows the high accuracy of the proposed model.

TABLE 1. Values of approximation parameters of experimental data of TEOA adsorption on catalyst Pt–CN

Parameter	Values
k, μmol$\cdot$min^{-1}	1.8 ± 0.1
K, M^{-1}	1.9 ± 0.7
R^2	0.97

3.2. Influence of pH value

According to the experimental data on determination of the optimum concentration of the sacrificial agent – triethanolamine – the best hydrogen evolution rate was achieved at a volume concentration of 30 vol.% TEOA. Therefore, the influence of pH of the reaction suspension on the activity of the Pt–CN was further studied by adding different amounts of sodium hydroxide to 30 vol.% TEOA. The NaOH concentration value was varied from 0 to 5 mol/L.

The dependence of hydrogen evolution rate on NaOH concentration is presented in Fig. 3. The pH value of the reaction suspension strongly affects the hydrogen evolution. The dissociation constant pKb of TEOA is 6.2, hence, in acidic media, the amino groups of TEOA are protonated, which can deactivate the α-CH bond [26]. When the pH increases, two forms coexist in solution: molecular and deprotonated. The latter is more efficient as a reducing agent [27], reacting efficiently with hydroxyl radicals.

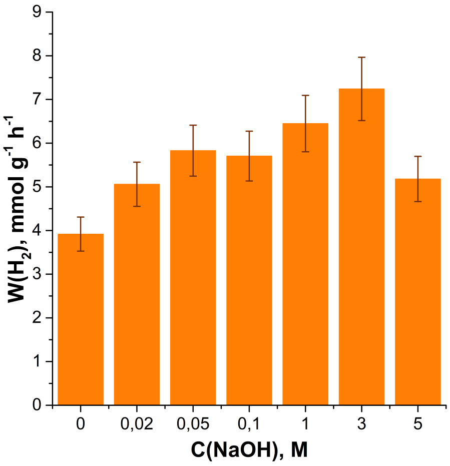

FIG. 3. Dependence of hydrogen evolution rate on NaOH concentration at fixed TEOA concentration in the presence of Pt–CN. Experimental conditions: $C(\text{TEOA}) = 30$ vol.%, $C(\text{NaOH}) = 0.02$ –5 M, $m_{cat} = 25$ mg, $V_{susp} = 50$ mL, $\lambda = 430$ nm

Based on the experimental data (Fig. 3), the addition of NaOH leads to an increase in the reaction rate, which takes place due to the formation of hydroxyl radicals [28]. The photocatalytic reaction involves the oxidation of TEOA, which is characterized by a low oxidative potential of $+0.64$ V vs. SCH. Therefore, from the thermodynamic point of view, the one-electron transfer of the lone nitrogen pair is more favorable [29]. In [30], the authors confirm that the oxidation of TEOA proceeds not by the hydroxyl group but by the lone nitrogen pair. Next, the formation of iminium cation occurs. The iminium cation undergoes hydrolysis to form diethanolamine and glycolaldehyde [29].

In the NaOH concentration range of 0 – 3 M, a linear increase in the reaction rate is observed, reaching a maximum at conditions of 30 vol.% TEOA + 3M NaOH. The hydrogen evolution rate under these conditions is 7.24 mmol$\cdot$g^{-1}h^{-1}, with an apparent quantum efficiency of 1.9 %. However, further, there is a sharp decrease in the activity of Pt–CN. Probably at high pH the amount of H$^+$ cations become very small for the subsequent reduction to form hydrogen H_2, which is one of the reasons for the decrease in the reaction rate. The process of photocatalytic hydrogen evolution becomes more difficult. Thus, the optimal conditions for hydrogen evolution are a solution of 30 vol.% TEOA and 3M NaOH.

3.3. Photocatalytic stability

One of the important characteristics of the photocatalyst is not only the activity of the material in the target process, but also its stability in long-term experiments and the possibility of reuse, which is attractive from the practical point of view. Therefore, cyclic experiments were carried out to study the stability of Pt–CN. According to the results on determining the optimal composition of the electron donor solution, the following systems were used: a) 30 vol.% TEOA; b) 30 vol.% TEOA + 3M NaOH. The number of runs was 4, after each run the reactor was purged with argon to remove hydrogen and oxygen.

As shown in Fig. 4(a,b), in the absence of NaOH, the activity of the Pt–CN photocatalyst does not decrease significantly after four runs, indicating that the catalyst exhibits high stability. However, when 3M NaOH is added, the hydrogen evolution rate decreases almost twofold after the first run. In the case of runs in 3M NaOH medium, a decrease in the hydrogen evolution rate by almost 2.5 times is observed already after the second run. After the fourth run, the hydrogen evolution rate was 1.39 mmol$\cdot$g^{-1}h^{-1}. Thus, a fourfold decrease in activity was observed. Probably, carbonization of the Pt–CN catalyst surface occurs in alkaline medium [21].

Thus, the optimum conditions for hydrogen evolution on Pt–CN is 30 vol.% TEOA, since the addition of NaOH results in catalyst deactivation.

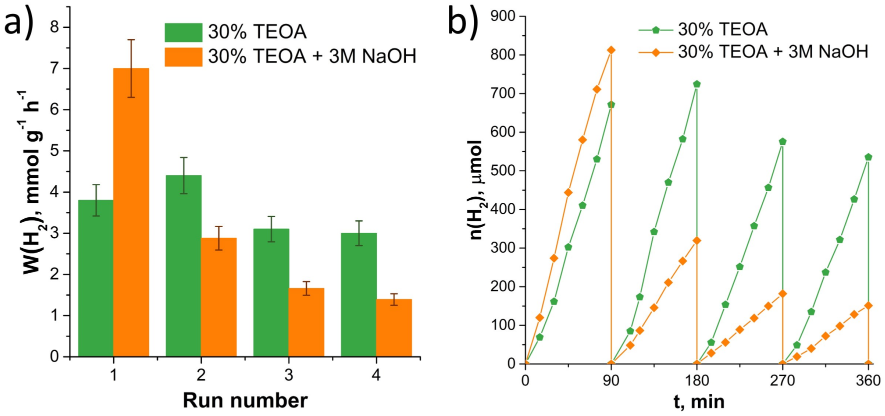

FIG. 4. Dependence of hydrogen evolution rate on the reaction cycle (a) and kinetic curves of hydrogen formation (b) for Pt–CN catalyst. Experimental conditions: $C(\mathrm{TEOA}) = 30$ vol.% / $C(\mathrm{TEOA}) =$ 30 vol.% + C(NaOH)=3M, $t_{run} = 90$ min, $m_{cat} = 25$ mg, $V_{susp} = 50$ mL, $\lambda = 430$ nm

4. Conclusion

In this work, the dependences of the rate of photocatalytic hydrogen evolution under the visible light irradiation (wavelength 430 nm) on the concentration of TEOA and NaOH in the presence of 0.1 wt.% Pt/g-C_3N_4 catalyst were obtained. The kinetic dependences of hydrogen evolution rate on TEOA concentration were approximated by Langmuir–Hinshelwood model with high accuracy, the optimum electron donor concentration was found, which was 30 vol.% TEOA. In alkaline medium, a linear increase in rate was observed with increasing NaOH concentration, the maximum activity was observed at 30 vol.% TEOA + 3M NaOH and was 7.24 mmol·g^{-1}h^{-1}, AQE = 2.1 %. The obtained reaction rate is currently very high for the photocatalytic hydrogen evolution process in the presence of a catalyst with such a low platinum content of 0.1 wt.% Pt/g-C_3N_4. The stability of 0.1 wt.% Pt/g-C_3N_4 catalyst was studied in long-term photocatalytic hydrogen evolution experiments. The catalyst is shown to be a stable material in the absence of sodium hydroxide. On the contrary, upon addition of alkali, a decrease in activity by a factor of almost 5 is observed, which is associated with carbonization of the 0.1 wt.% Pt/g-C_3N_4 surface.

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