

Ni(OH)₂ and Co(OH)₂ modified g-C₃N₄ for photocatalytic CO₂ reduction

Angelina V. Zhurenok^{1,a}, Alina E. Ermoshkina^{1,2,b}, Alexey E. Zazulya^{2,3,c}, Denis D. Mishchenko^{1,4,d}, Evgeny Yu. Gerasimov^{1,e}, Ekaterina A. Kozlova^{1,f}

¹Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, Russia

²Novosibirsk National Research State University, Novosibirsk, Russia

³A.V. Nikolaev Institute of Inorganic Chemistry SB RAS, Novosibirsk, Russia

⁴Multiaccess Center “SKIF” Boreskov Institute of Catalysis, Novosibirsk, Russia

^aangelinazhurenok@gmail.com, ^ba.ermoshkina@g.nsu.ru, ^ca.zazulya@g.nsu.ru, ^dq14999@yandex.ru,

^egerasimov@catalysis.ru, ^fkozlova@catalysis.ru

Corresponding author: Angelina V. Zhurenok, angelinazhurenok@gmail.com

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ABSTRACT This study presents the synthesis and characterization of composite photocatalysts based on g-C₃N₄ modified with Ni(OH)₂ and Co(OH)₂ for CO₂ reduction under visible light ($\lambda = 410$ nm). XRD, UV-Vis spectroscopy, and high-resolution TEM analyses revealed that the deposition of hydroxides did not disrupt the g-C₃N₄ structure but caused a slight increase in the interlayer spacing and band gap; Ni(OH)₂ formed nanoparticles of ~ 7 nm, whereas Co(OH)₂ crystallized as larger particles of ~ 24 nm. TEM showed that after the photocatalytic CO₂ reduction, partial agglomeration of Ni(OH)₂/NiO occurs. The catalyst containing 0.1 wt.% Ni(OH)₂ exhibited the highest activity – $7.04 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$, achieving a CO₂ reduction rate 1.9 times higher than that of pristine g-C₃N₄. The 0.5 wt.% Co(OH)₂/g-C₃N₄ photocatalyst was achieved a 1.3-fold increase in activity compared to pristine g-C₃N₄. This enhancement is attributed to the formation of a II type heterojunction between g-C₃N₄ and Ni(OH)₂, which facilitates charge transfer and suppresses recombination.

KEYWORDS g-C₃N₄, photocatalysis, Ni(OH)₂, Co(OH)₂, CO₂ reduction

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1. Introduction

In today’s world, the intensification of the greenhouse effect and climate change are global problems that the entire international scientific community is interested in solving [1–3]. The concentration of carbon dioxide (CO₂) in the atmosphere continues to increase due to the use of fossil fuels in modern energy production [4–7]. Therefore, it seems reasonable not only to capture CO₂ but also to convert it into valuable products in order to achieve a balance between energy demands and environmental sustainability [8, 9]. Converting CO₂ into valuable chemicals not only decreases its atmospheric concentration, but also helps to store solar energy in the form of stable chemical bonds [10–12]. This is important because CO₂ is available in huge volumes and can serve as a raw material for various materials. Among various CO₂ conversion approaches, photocatalysis is particularly attractive as it utilizes solar energy to transform CO₂ into CO, CH₄, CH₃OH, and other products and is carried out at room temperature and atmospheric pressure [13, 14], unlike industrial processes [15].

Efficient CO₂ conversion into chemical compounds requires catalytic systems with high activity and selectivity for the target products, and resistance to photodegradation. Current challenges in this area include selecting efficient and durable materials, reducing the energy required for CO₂ activation, increasing the proportion of target products, and minimizing side reactions such as H₂ evolution and H₂O splitting. Photocatalytic systems based on semiconductors, in particular graphitic carbon nitride (g-C₃N₄), doped with metals or metal oxides are of particular interest [16–18]; g-C₃N₄ has a favorable band gap, the ability to absorb visible light, and great potential as a photocatalyst [19–21]. However, pristine g-C₃N₄ has limitations: a relatively small surface area, rapid recombination of photogenerated charge carriers, and low selectivity in CO₂ reduction reactions [20, 22, 23]. To overcome these limitations, g-C₃N₄ modification through doping, heterostructure formation, and active phase deposition is widely used [20, 24, 25]. In this regard, transition metal hydroxides such as Ni(OH)₂ and Co(OH)₂ act as effective cocatalysts. Introducing them into the g-C₃N₄ system improves the separation of photogenerated charge carriers, more effectively adsorbs CO₂ molecules on the material surface, and reduces side reactions, thereby paving the way for increased activity and selectivity [26–28].

This study aims to compare the effect of $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ supported on $\text{g-C}_3\text{N}_4$ on the photocatalytic activity and selectivity in CO_2 reduction. The results will provide recommendations for the optimal ratio of $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ phases deposited onto $\text{g-C}_3\text{N}_4$, which will increase the yield of target products.

2. Experimental

2.1. Photocatalyst synthesis

2.1.1. $\text{g-C}_3\text{N}_4$ synthesis. A suspension was prepared from a mixture of melamine, cyanuric acid and distilled water (300 ml) and heated with constant stirring for 12 h at 90 °C [29]. The resulting supramolecular complex of melamine-cyanuric acid was washed with water, dried in vacuum, and calcined in air at 550 °C for 1 h at a heating rate of 1 °C/min.

2.1.2. $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ deposition. A suspension was prepared by dispersing 500 mg of $\text{g-C}_3\text{N}_4$ in 20 ml of acetone. The required amount of nickel or cobalt nitrate ($\text{M}(\text{NO}_3)_2$) was then added to the suspension. The mixture was sonicated for 1 min, after which acetone was removed using a rotary evaporator. To convert the metal species into the hydroxide form on the $\text{g-C}_3\text{N}_4$ surface, small portions of the dried $\text{M}(\text{NO}_3)_2/\text{g-C}_3\text{N}_4$ samples were added to a 0.5 M NaOH solution with intensive stirring. After 10 min, the samples were filtered through a PTFE membrane, washed twice with water and dried under vacuum at 40 °C for 10 hours.

2.2. Photocatalyst characterization

The synthesized catalysts were studied using a set of physicochemical analysis methods: X-ray diffraction (XRD), diffuse reflectance spectroscopy (DRS), and high-resolution transmission electron microscopy (HRTEM). The phase composition and particle size were determined by XRD. XRD patterns were recorded on a Bruker D8 Advance diffractometer in the 2θ range from 10° to 65° using Cu $K\alpha$ radiation. DRS in the UV- and visible spectral regions were obtained on a Shimadzu UV-2501 PC spectrophotometer with an ISR-240A diffuse reflectance attachment in the 200 – 800 nm wavelength range. The optical band gap was calculated from a linear fit to the Tauc plot for indirect allowed transitions. Surface morphology was studied using HR TEM on a ThemisZ microscope (Thermo Fisher Scientific) with an accelerating voltage of 200 kV. To prepare for analysis, samples were dispersed in an ultrasonic bath and applied to perforated carbon substrates.

2.3. Photocatalytic CO_2 reduction experiment

The photocatalytic CO_2 reduction was performed in a static glass reactor ($V = 170$ ml) with a quartz window (16 cm^2) (Fig. 1a). Typically, 30 mg of photocatalyst dispersed in 500 μl of ultrapure water (NuZar Q water purification system), after that this suspension was applied to a glass support (8 cm^2). The photocatalyst was then dried and illuminated with LED-410 nm for 30 min to remove organic contaminants from the photocatalyst surface. The glass support with the deposited photocatalyst was then placed in a reactor containing 1 ml of ultrapure water. The reactor was purged with Ar for 20 min to remove O_2 , followed by CO_2 (99.995 %) purging also for 20 min, and then irradiated with 410-nm LED (Fig. 1b). The gas phase was analyzed by Chromos GKh-1000 gas chromatograph equipped with a flame ionization detector with methanator and a thermal conductivity detector. Samples were collected every hour, and the photocatalytic experiment lasted 4 hours. The production rates (CO , CH_4) were calculated by linear approximation of the kinetics curves. The total electron absorption rates were calculated by the following formula:

$$W_{e-}(\text{CO}_2\text{RR}) = 2 \times W(\text{CO}) + 8 \times W(\text{CH}_4), \quad (1)$$

where 2 and 8 are coefficients for electron balance, and $W(\text{CO})$ and $W(\text{CH}_4)$ are the production rates of CO and CH_4 , respectively.

3. Results and discussion

The photocatalysts $\text{g-C}_3\text{N}_4$, 2.5 % $\text{Ni}(\text{OH})_2/\text{g-C}_3\text{N}_4$, and 2.5 % $\text{Co}(\text{OH})_2/\text{g-C}_3\text{N}_4$ were analyzed by XRD. Fig. 2 shows the XRD patterns of these photocatalysts. The diffraction peaks at 13.6° and 27.7° correspond to the (210) and (002) reflections, respectively [29]. In addition to the two characteristic peaks of the $\text{g-C}_3\text{N}_4$ structure, several weaker peaks are observed, including a peak near 57° related to the (004) plane. This confirms the formation of $\text{g-C}_3\text{N}_4$ with an orthorhombic structure [30, 31].

Furthermore, the impact of the supported $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ on the coherent scattering region (CSR) of the intraplanar structure of $\text{g-C}_3\text{N}_4$ and the interplanar spacing is evident. The CSR of $\text{Co}(\text{OH})_2$ is significantly higher than that of $\text{Ni}(\text{OH})_2$, which indicates crystallization. The deposition of $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$ does not lead to the appearance of new reflections in the XRD patterns. This indicates the absence of bulk crystalline hydroxide phases and their high dispersion on the $\text{g-C}_3\text{N}_4$ surface.

However, quantitative analysis of the XRD patterns (see Table 1) revealed the influence of the modifying additives on the $\text{g-C}_3\text{N}_4$ structural parameters. Specifically, the interplanar spacing d_{002} increases from 3.217 Å for unmodified $\text{g-C}_3\text{N}_4$ to 3.244 and 3.236 Å for composites with 2.5 % $\text{Ni}(\text{OH})_2$ and $\text{Co}(\text{OH})_2$, respectively. This indicates the successful interaction between the deposited hydroxides and the $\text{g-C}_3\text{N}_4$ lattice. Calculating the CSR sizes using the Scherrer

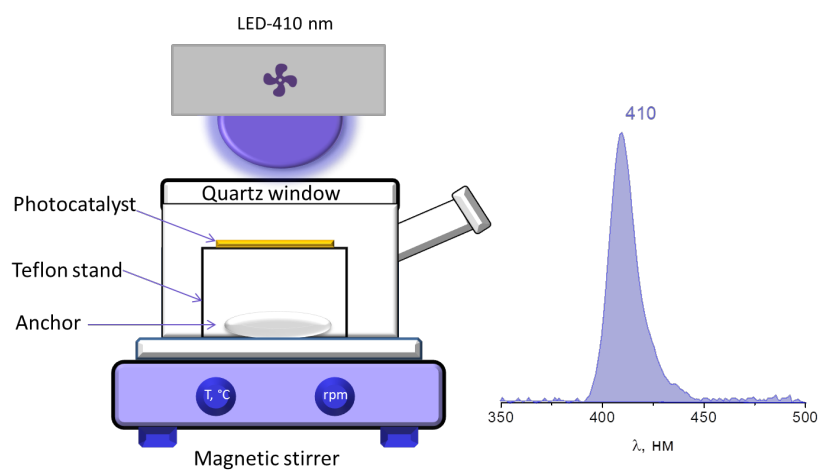


FIG. 1. Photocatalytic reactor for CO_2 reduction and the emission spectra of LED-410 nm

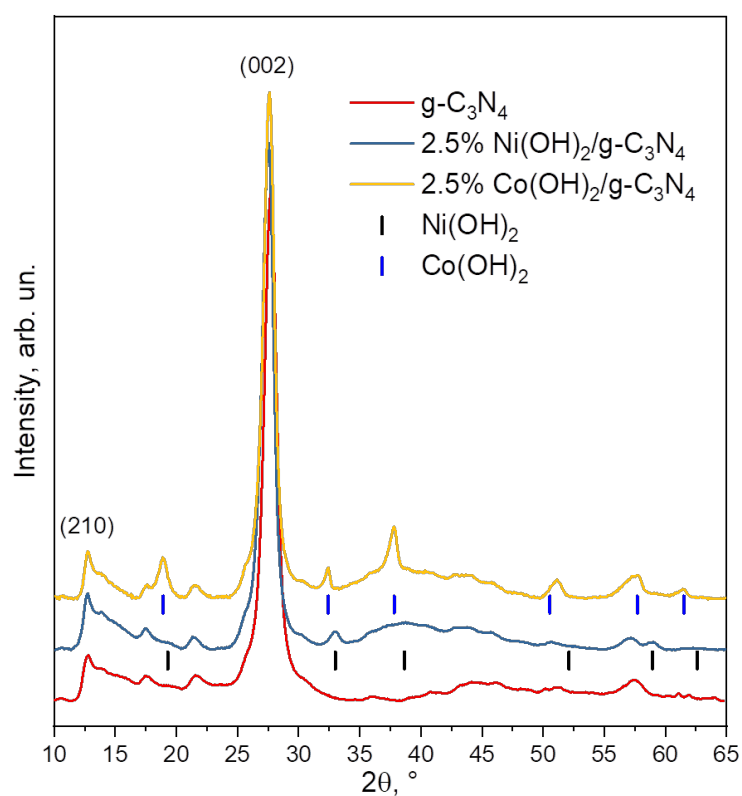


FIG. 2. XRD patterns of $\text{g-C}_3\text{N}_4$, 2.5 % $\text{Ni}(\text{OH})_2/\text{g-C}_3\text{N}_4$ and 2.5 % $\text{Co}(\text{OH})_2/\text{g-C}_3\text{N}_4$

TABLE 1. Structural characteristics of $\text{g-C}_3\text{N}_4$, 2.5 % $\text{Ni}(\text{OH})_2/\text{g-C}_3\text{N}_4$ and 2.5 % $\text{Co}(\text{OH})_2/\text{g-C}_3\text{N}_4$ photocatalysts calculated from XRD data

Photocatalyst	$d_{210}/\text{\AA}$	L_a , nm	$d_{002}/\text{\AA}$	L_c , nm	$\text{Ni}(\text{OH})_2/\text{Co}(\text{OH})_2$ CSR, nm
$\text{g-C}_3\text{N}_4$	6.95(1)	8(1)	3.217(5)	7(1)	—
2.5 % $\text{Ni}(\text{OH})_2/\text{g-C}_3\text{N}_4$	6.98(1)	11(1)	3.244(5)	8(1)	7(1)
2.5 % $\text{Co}(\text{OH})_2/\text{g-C}_3\text{N}_4$	6.94(1)	11(1)	3.236(5)	8(1)	24(1)

formula showed that Ni(OH)_2 forms nanosized particles measuring ~ 7 nm, while Co(OH)_2 crystallizes as significantly larger particles (~ 24 nm). Such difference in crystallite size may significantly affect the photocatalytic properties of the synthesized materials.

The optical properties of all synthesized photocatalysts were investigated. Fig. 3(a and c) presents the diffuse reflectance spectra, showing that all photocatalysts absorb visible light in the 400 – 800 nm range. The absorption edge is located in the 425 – 460 nm region. Fig. 3(b and d) shows the plots in Tauc coordinates for calculating the optical band gap. Upon the addition of Ni(OH)_2 and Co(OH)_2 , the optical band gap slightly increases from 2.68 to 2.87 eV. This is due to the fact that Ni(OH)_2 and Co(OH)_2 are wider-bandgap semiconductors [32, 33].

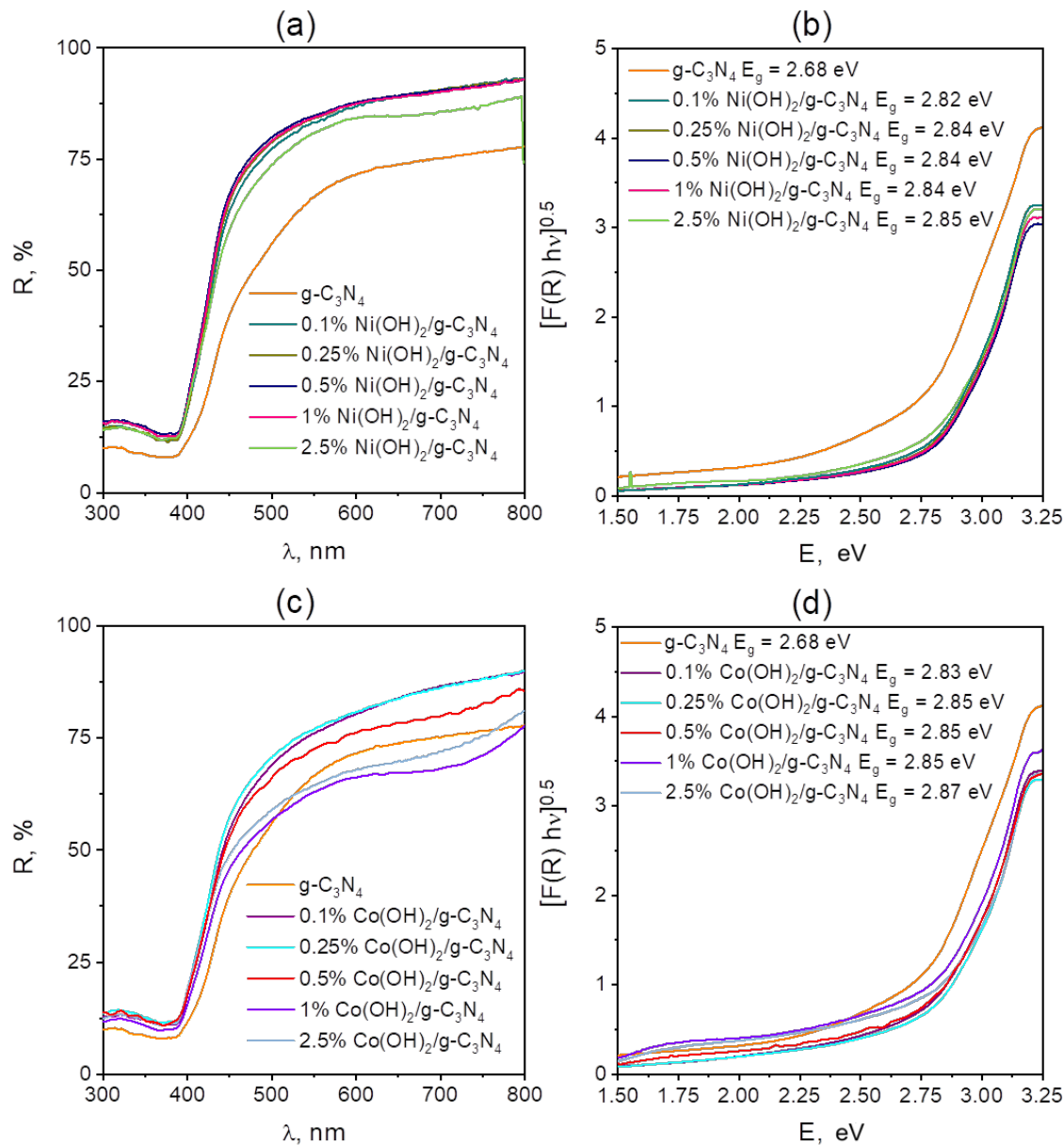


FIG. 3. (a, c) UV-Vis spectra of photocatalysts; (b, d) Tauc plots for calculating E_g of the x % $\text{Ni(OH)}_2/\text{g-C}_3\text{N}_4$ and x % $\text{Co(OH)}_2/\text{g-C}_3\text{N}_4$ ($x = 0 - 2.5$ wt.%) photocatalysts

TEM was used to study the 1 % $\text{Ni(OH)}_2/\text{g-C}_3\text{N}_4$ photocatalyst (Fig. 4(a–c)). It was shown that g-C₃N₄ is represented by rounded thin nanosheets. Following the deposition of 1 % Ni(OH)_2 , the surface became covered with uniformly distributed nanoparticles of $\sim 0.5 - 2$ nm thick, corresponding to 2 – 5 stacked Ni(OH)_2 layers, while preserving the layered structure (Fig. 4(a, b)). Ni(OH)_2 exhibits strong adhesion to the surface of g-C₃N₄. The (001) plane of Ni(OH)_2 is oriented parallel to the corresponding (002) (from XRD data (Fig. 2)) plane of g-C₃N₄ and is consistent with the observed lamellar morphology. The Ni(OH)_2 particles are in close contact with the g-C₃N₄ surface (Fig. 4(c)), ensuring effective interfacial interaction and the formation of local type II heterojunctions. This organization promotes the spatial separation of photogenerated charge carriers, reduces their recombination and directs electrons to the cocatalyst surface, where CO_2 reduction occurs.

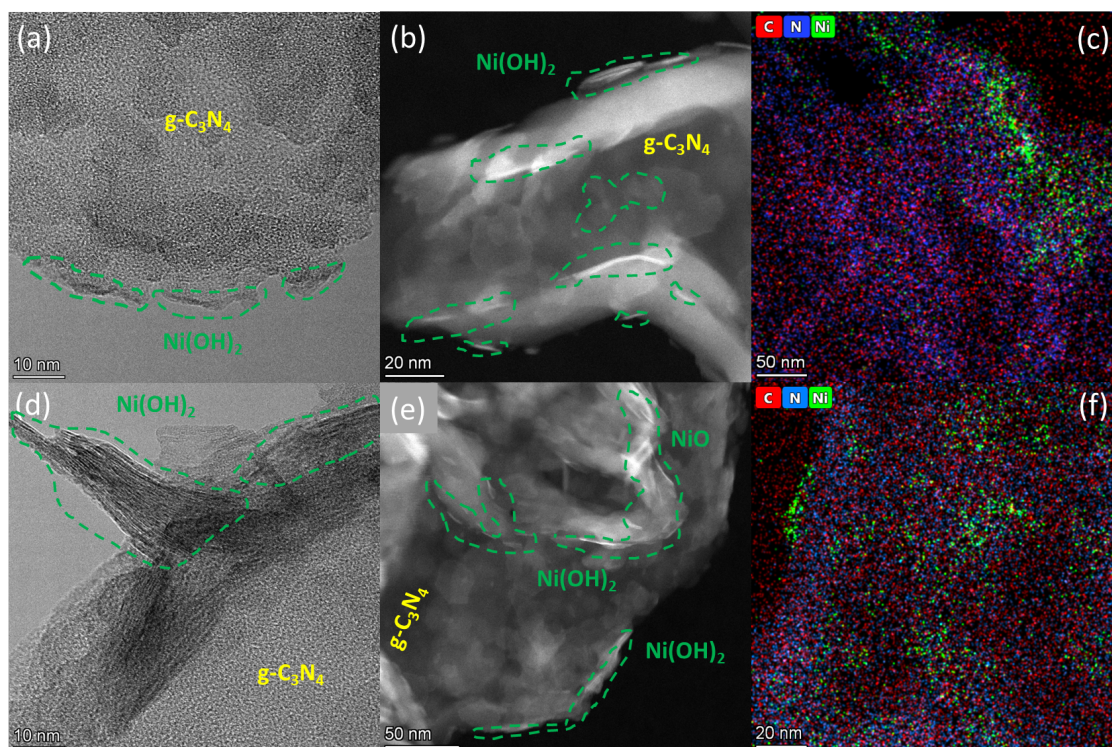


FIG. 4. (a) HR TEM, (b) HAADF STEM, (c) elemental mapping for 1 % Ni(OH)₂/g-C₃N₄ photocatalysts; (d) HR TEM, (e) HAADF STEM, (f) elemental mapping for 1 % Ni(OH)₂/g-C₃N₄ photocatalysts after CO₂ reduction

All synthesized x % Ni(OH)₂/g-C₃N₄ and x % Co(OH)₂/g-C₃N₄ photocatalysts were studied in the CO₂ reduction under 410-nm LED irradiation. Fig. 5(a) shows the rates of CO and CH₄ formation in the presence of x % Ni(OH)₂/g-C₃N₄ photocatalysts. As can be seen, the main product formed during the CO₂ reduction reaction is CO. Fig. 5(b) shows the overall rate of CO₂ reduction. The most active photocatalysts were 0.1 % and 0.25 % Ni(OH)₂/g-C₃N₄ with a CO₂ reduction rate of 7.04 and 7.08 $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$, respectively. The reaction rate of photocatalysts containing 1 and 2.5 % Ni(OH)₂ differed little from the rates of 0.1 % Ni(OH)₂/g-C₃N₄ and 0.25 % Ni(OH)₂/g-C₃N₄. Therefore, we will consider 0.1 % Ni(OH)₂ to be the most effective composition, since even a small addition of cocatalyst allows for a 1.9-fold increase in the CO₂ reduction rate.

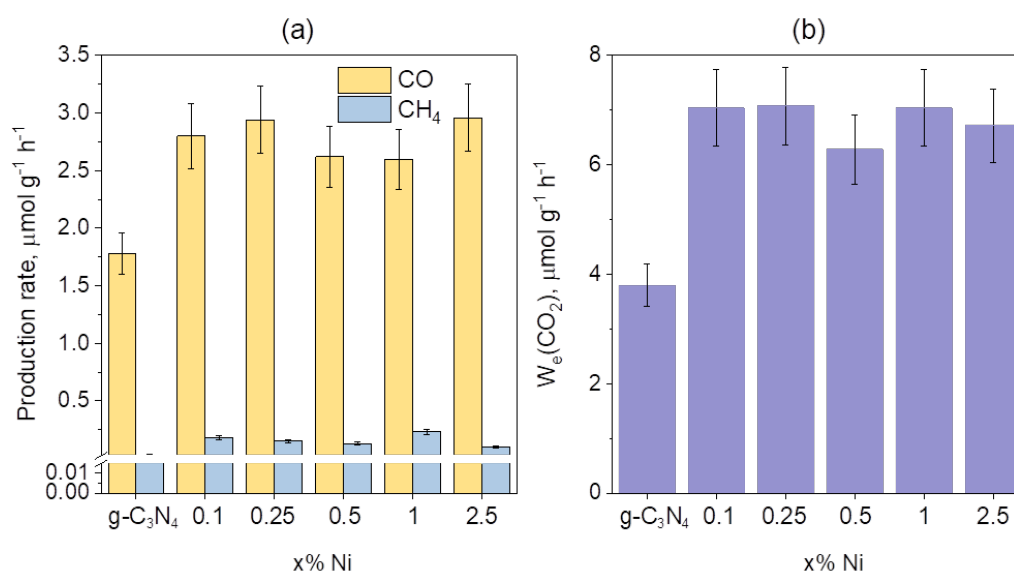


FIG. 5. (a) CO and CH₄ production rates; (b) total electron consumption rate during CO₂ reduction in the presence of x % Ni(OH)₂/g-C₃N₄ photocatalysts. Conditions: $m(\text{cat.}) = 30$ mg, $t(\text{reaction}) = 4$ h. Light source: LED with $\lambda = 410$ nm.

Figure 6(a) illustrates the CO and CH₄ formation rates in the presence of the x % Co(OH)₂/g-C₃N₄ photocatalysts. As can be seen, the main product formed during the CO₂ reduction is also CO. Fig. 6(b) shows the total rate of CO₂ reduction. The most active photocatalyst was 0.5 % Co(OH)₂/g-C₃N₄ with CO₂ reduction rate of $4.84 \mu\text{mol} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$. The rates of photocatalysts containing 1 % and 2.5 % Co(OH)₂ did not differ significantly from the rate of the 0.5 % Co(OH)₂/g-C₃N₄ photocatalyst within the measurement error. Unfortunately, only a slight increase in the photocatalytic activity is achieved for Co(OH)₂ deposition on the surface of g-C₃N₄.

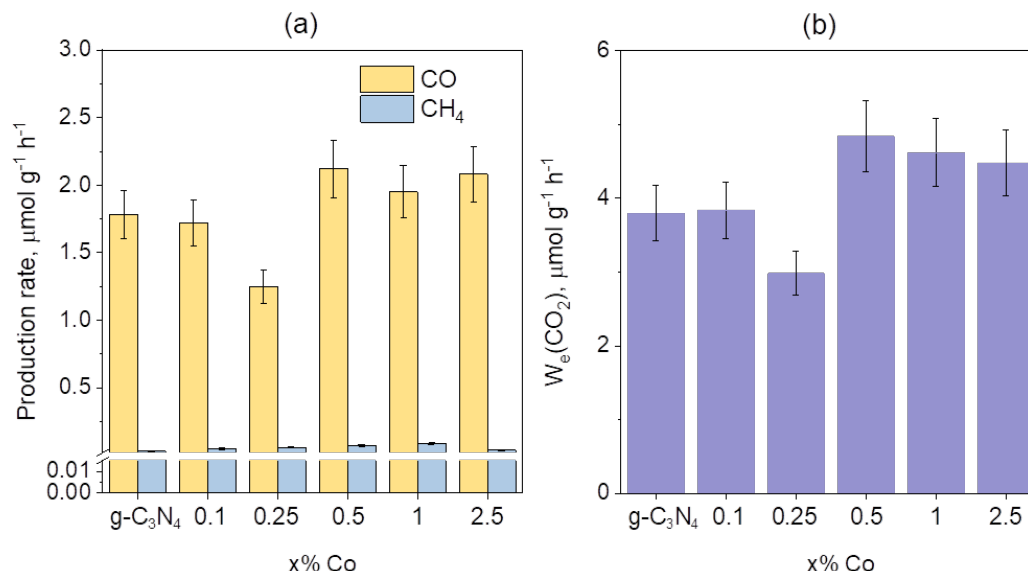


FIG. 6. (a) CO and CH₄ production rates; (b) total electron consumption rate during CO₂ reduction in the presence of x % Co(OH)₂/g-C₃N₄ photocatalysts. Conditions: $m(\text{cat.}) = 30 \text{ mg}$, $t(\text{reaction}) = 4 \text{ h}$. Light source: LED with $\lambda = 410 \text{ nm}$.

After the CO₂ reduction, the most active photocatalyst – 0.1 % Ni(OH)₂/g-C₃N₄ was studied by TEM (Fig. 4(d–f)). Some of the Ni(OH)₂ is converted into NiO, creating defects and oxygen vacancies that enhance photocatalytic activity. Adhesion is maintained, with the partial agglomeration of Ni(OH)₂/NiO particles on the g-C₃N₄ surface. A partial increase in particle size as a result of the CO₂ reduction reaction is observed in an increase in the thickness of layered particles (Fig. 4(d, e)), while the uniformity of the distribution of Ni(OH)₂ particles over the surface of g-C₃N₄ is maintained (Fig. 4(f)). After the photocatalytic CO₂ reduction, it is important to note that there are no signs of photocatalyst degradation: the layered g-C₃N₄ morphology and the Ni(OH)₂ dispersion are both preserved. This indicates the catalyst's structural stability under reaction conditions and confirms its high photocatalytic activity.

Also, the mechanism of photocatalytic CO₂ reduction on Ni(OH)₂/g-C₃N₄ (Fig. 7(a)) and Co(OH)₂/g-C₃N₄ (Fig. 7(b)) composites was proposed. Upon light irradiation, both semiconductors (g-C₃N₄ and Ni(OH)₂ or Co(OH)₂) are photoexcited. Electrons from the conduction band (CB) of g-C₃N₄ are transferred to the CB of the hydroxides, while holes from the valence band (VB) of the hydroxides migrate to the VB of g-C₃N₄, where they participate in the H₂O oxidation. This type II heterojunction scheme ensures the spatial separation of photogenerated charge carriers suppresses their recombination and directs electrons to the cocatalyst surface [34, 35], where activation and CO₂ reduction occur. The CB level of g-C₃N₄ provides sufficient potential for the two-electron reduction of CO₂ to CO, which explains why CO is the main reaction product. The comparatively low rate of CO₂ reduction in the presence of Co(OH)₂/g-C₃N₄ compared to Ni(OH)₂/g-C₃N₄ may be associated with the rather large size of cobalt hydroxide particles, three times larger than the size of nickel hydroxide particles (see Table 1).

4. Conclusion

This study presents the synthesis and characterization of composite photocatalysts based on g-C₃N₄, modified with Ni(OH)₂ and Co(OH)₂, for the photocatalytic reduction of CO₂ under visible light ($\lambda = 410 \text{ nm}$). The introduction of hydroxides did not disrupt the structure of g-C₃N₄, but rather to increase the interplanar spacing and optical band gap, indicating interaction between the phases. Note that Ni(OH)₂ forms nanoparticles with a CSR approximately 7 nm, whereas Co(OH)₂ crystallizes into larger particles of around 24 nm. TEM study showed that partial agglomeration of Ni(OH)₂/NiO particles observed after CO₂ reduction while adhesion was maintained, indicating morphological stability. The presence of cocatalysts promotes efficient charge separation and increases activity in the CO₂ reduction reaction. Catalysts containing 0.1 and 0.25 wt.% Ni(OH)₂ have comparable CO₂ reduction rates and exceed the activity of the unmodified g-C₃N₄ by 1.9 times. The 0.1 % Ni(OH)₂/g-C₃N₄ was selected as the optimal photocatalyst, as it provides

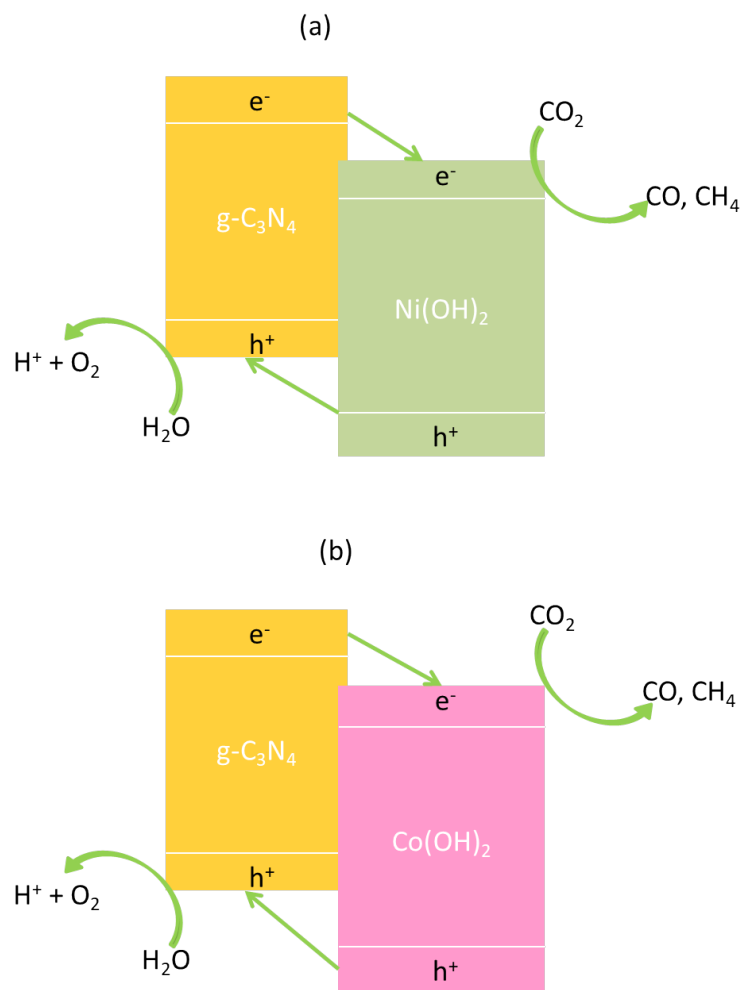


FIG. 7. Heterojunctions between (a) Ni(OH)₂ and g-C₃N₄; (b) Co(OH)₂ and g-C₃N₄

the same level of activity with minimal cocatalyst loading. For photocatalysts containing 0.5-2.5 % Co(OH)₂, the CO₂ reduction rate was almost the same and exceeded the activity of the pristine g-C₃N₄ by ~1.3 times. Therefore, 0.5 % Co(OH)₂/g-C₃N₄ was chosen as the active photocatalyst from all modified Co(OH)₂, as it provides the maximum effect with a minimum cocatalyst loading. This increase in activity is attributed to the formation of a type II heterojunction between g-C₃N₄ and the transition metal hydroxides, facilitating charge transfer and suppressing charge recombination. Therefore, modifying g-C₃N₄ with transition metal hydroxides is an effective strategy to improve the photocatalytic activity of CO₂ reduction under visible light irradiation.

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Information about the authors:

Angelina V. Zhurenok – Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, 630090, Russia; ORCID 0000-0003-3908-1963; angelinazhurenok@gmail.com

Alina E. Ermoshkina – Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, 630090, Russia; Novosibirsk National Research State University, Novosibirsk, 630090, Russia ORCID 0009-0003-8833-6819; a.ermoshkina@g.nsu.ru

Alexey E. Zazulya – A.V. Nikolaev Institute of Inorganic Chemistry SB RAS, Novosibirsk, 630090, Russia; Novosibirsk National Research State University, Novosibirsk, 630090, Russia; ORCID 0009-0008-2797-4557; a.zazulya@g.nsu.ru

Denis D. Mishchenko – Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, 630090, Russia; Multiaccess Center “SKIF” Boreskov Institute of Catalysis, Novosibirsk, 630559, Russia; ORCID 0000-0001-8822-5148; q14999@yandex.ru

Evgeny Yu. Gerasimov – Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, 630090, Russia; ORCID 0000-0002-3230-3335; gerasimov@catalysis.ru

Ekaterina A. Kozlova – Federal Research Center Boreskov Institute of Catalysis SB RAS, Novosibirsk, 630090, Russia; ORCID 0000-0001-8944-7666; kozlova@catalysis.ru

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