Synthesis of MIL-101(Cr) metal-organic framework material and research photocatalytic capability of material for nitrate removal in water

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ABSTRACT

This paper presents the research results on some characteristics of the metal-organic framework material MIL-101(Cr) and its ability to remove nitrate from aqueous environments based on photocatalytic reactions. This material was synthesized using the hydrothermal method in the laboratory. Techniques such as FE-SEM, XRD, FT-IR, and BET were employed, demonstrating that the porous structure of the material is highly developed, with a specific surface area of up to 3017 m²/g and characteristic octahedral crystal size ranging from approximately 100 to 300 nm. Experimental results show that under UV light conditions at a wavelength of 365 nm and a power of 250 W, the MOF MIL-101(Cr) can catalyze nitrate removal under UV light, achieving a maximum removal efficiency of up to 99% after 180 minutes of reaction. The nitrate removal efficiency of MIL-101 is significantly improved, and reaches nearly 100% within a reaction time of 40 minutes when formic acid (HCOOH 46 mM) is used as a hole scavenger.

Keywords: MOF; MIL-101; Nitrate removal efficiency; Hole scavenger.

1. INTRODUCTION

Nitrogen is the main element in fuels, and explosives and primarily exists in the form of nitrite (NO_2^-) and nitrate (NO_3^-). Nitrate is a stable ion in water and not toxic to humans, but when absorbed into the body, it is converted into nitrite by gut bacteria [1]. Nitrite oxidizes hemoglobin in red blood cells, transforming it into methemoglobin, which inhibits oxygen transport, leading to oxygen deficiency in the blood. Moreover, research has shown that nitrate and nitrite can cause cancer in humans because nitrite can combine with amino acids in food to form nitrosamines, a family of precancerous compounds [2]. Nowadays, nitrate treatment can be attributed to three main methods: chemical, physical, biological, and combined methods. In recent years, scientists have studied and developed nitrate treatment through photocatalysis both domestically and internationally due to its advantages, such as high treatment efficiency, low cost, environmentally friendly, no secondary pollution, and the ability to handle high concentrations of nitrate [3].

Porous materials with large surface structure and capillary pores provide a large contact surface area to enhance the performance of the photocatalyst. Traditional porous materials have mainly been studied as either inorganic or organic substances [4]. Among them, the most common organic material is activated carbon, which has a large surface area and high adsorption capacity but lacks an orderly structure. Meanwhile, inorganic porous materials have highly ordered structures (like zeolites), but their frameworks easily collapse and are not diverse [5]. To combine the advantages of organic and inorganic porous materials, Yaghi et al. first introduced metal-organic frameworks (MOFs) in 1995 [6]. MOF materials, due to their highly porous structure and chemical tunability, can absorb light and participate in photocatalytic reactions [6]. Among the MOF material systems, MIL(Materials of Institut Lavoisier)-based materials have a significant advantage over other photocatalytic materials due to their porous structure, large surface area, and diverse active sites. MIL-101(Cr) is a chromium-based metal-organic framework, and this material has fixed, uniform

porous cavities and a very large surface area compare with other MOF such as MOF-5, ZIF-8, UiO-66, MOF-74, etc. leading to numerous active sites and good mass transfer properties [7]. Previous studies on MOF materials have mainly focused on the treatment of organic pollutants, while research on the treatment of inorganic pollutants remains limited. Therefore, studying nitrate removal using MOF materials helps explore another aspect of MOF materials [8].

This study introduces the fabrication of MIL-101(Cr) by hydrothermal methods and the characteristics of synthesized MIL-101(Cr) were evaluated using several analysis methods Field Emission Scanning Electron Microscope (FE-SEM), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), Brunauer-Emmett-Teller (BET). The nitrate removal performance of the MOF was investigated through several experiments, including MIL-101 dosage, hole scavenger and kind of hole scavenger.

2. MATERIALS AND METHODS

2.1. Chemicals and equipment

All the chemicals used for analysis were of analytical grade and used as received without further purification. Chromic nitrate nonahydrate ($Cr(NO_3)_3.9H_2O$, 99%), Hydrofluoric acid (HF, 40%), 1,4-benzene dicarboxylic acid (H_2BDC, 99%), N,N′-dimethylformamide (DMF,99%) was obtained from Macklin, China. Potassium nitrate (KNO_3, ≥ 99% Macklin ACS reagent) was used as the model nitrate source and pollutant, acid Formic (HCOOH, 99%), acid oxalic ($H_2C_2O_4.2H_2O$, 99%), was supplied by Aladin, China.

2.2. Material synthesis

The MIL-101(Cr) was synthesized using the hydrothermal method in the laboratory through the following steps [9]: 1.64 g Terephthalic acid, 4.0g Cr $(NO_3)_3 \cdot 9H_2O$, 0.25 ml 40% HF and de-ionized water (24 mL) were placed in a 40 mL Teflon-lined autoclave and heated 230 ^oC for 8 h. After cooling, wash the solid with distilled water 5 times, hot ethanol 3 times, and DMF 3 times, then dry the material at 80 °C for 8 hours.

2.3. Mechanism and data analysis

The mechanism for treating nitrate through photocatalytic reactions and when adding hole scavengers as follows [11]:

$$MIL\text{-}101 + hv \rightarrow h_{(VB)}^{+} + e_{(CB)}^{-} \quad (1)$$

$$2NO_3^- + 10e^- + 12H^+ \rightarrow N_2(gas) + 6H_2O \quad (2)$$

$$2NO_3^- + 2e^- + 2H^+ \rightarrow NO_2^- + 6H_2O \quad (3)$$

$$2NO_2^- + 6e^- + 8H^+ \rightarrow N_2 + 4H_2O \quad (4)$$

$$h_{(VB)}^{+} + OH^- \rightarrow \bullet OH^- \quad (5)$$

$$HCOOH + h_{(VB)}^{+} \rightarrow CO_2 + H_2 \quad (6)$$

$$H_2C_2O_4 + h_{(VB)}^{+} \rightarrow 2CO_2 + H_2 \quad (7)$$

When stimulated by light, the photocatalyst generates electron-hole pairs, and there is an electron exchange between the adsorbed substances via a semiconductor bridge, as described in equation (1). The photogenerated electron-hole pairs move to the surface and interact with adsorbed nitrate, where nitrate ions (NO_3^-) can either be directly reduced to nitrogen gas (N_2) (equation (2)), or only reduced to nitrite (NO_2^-) and released from the material's surface as in equation (3). Then, the nitrite ions in the aqueous solution can continue to be reduced to the final product, nitrogen gas (N_2), as per equation (4). The photogenerated holes ($h_{(VB)}^{+}$) interact with H_2O to produce hydroxyl radicals ($\bullet OH^-$), which are strong oxidizing agents (equation (5)), and the recombination of electron-hole pairs reduces the material's nitrate reduction efficiency. Therefore, to prevent electron-hole recombination, hole scavengers must be added to eliminate the photogenerated holes (equations (6), (7)), enhancing the reduction efficiency of the material [10].

The nitrate reduction process and the reduction of nitrate to nitrogen are calculated using the following formulas:

$$C(NO_3^-)\ (\%) = \frac{[NO_3^-]_0 - [NO_3^-]_t}{[NO_3^-]_0} \text{ x } 100\%$$

While $[NO_3^-]_0$ and $[NO_3^-]_t$ (mg/L) are the concentrations of NO_3^- at the initial and at time t, respectively.

2.4. Photocatalytic experiment for nitrate treatment in water

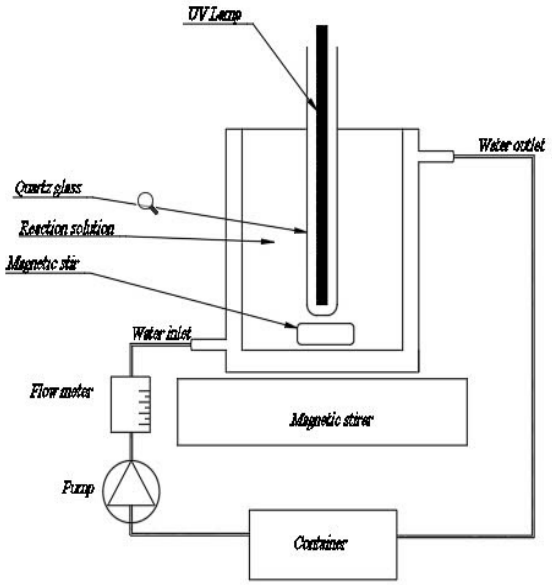

Figure 1. *Schematic Diagram of the photocatalytic experiment for nitrate treatment in water.*

The experiment to investigate the photocatalytic ability of the material for nitrate treatment in water was conducted using the photocatalytic experimental setup described in figure 1. A 250 W high-pressure mercury lamp was utilized as the light source, primarily emitting at a wavelength of 365 nm. The reaction vessel, with a capacity of 100 ml, was made of borosilicate glass, and the temperature inside the reaction vessel was maintained at room temperature (20 - 25 °C) using a continuous water cooling circulation system. The working solution with a nitrate concentration of 100 mg/L (calculated as nitrogen) was prepared, and then experiments were conducted to investigate the nitrate treatment efficiency with catalyst weights of 0.01 g, 0.02 g, 0.05 g, 0.08 g, 0.1 g, and 0.15 g of MIL-101. The nitrate treatment efficiency was also assessed with a hole scavenger, formic acid (HCOOH), at molar concentrations of 23 mM and 46 mM. During the reaction process, samples were periodically collected to analyze nitrate, nitrite, and ammonium concentrations using a UV-Vis spectrophotometer (Aligent 8453) at wavelengths of 415 nm, 540 nm, and 690 nm, respectively.

3. RESULT AND DISSCUSION

3.1 Properties of the material

3.1.1. Morphology of the material

The morphology of the fabricated material was determined using Scanning Electron Microscopy (SEM) on a HITACHI S-4800 device. FE-SEM image (figure 2) has shown that the synthesized material has the characteristic octahedral structure of MIL-101, with relatively high crystal intensity, particle size ranging from 100-300 nm, and a fairly uniform and porous structure.

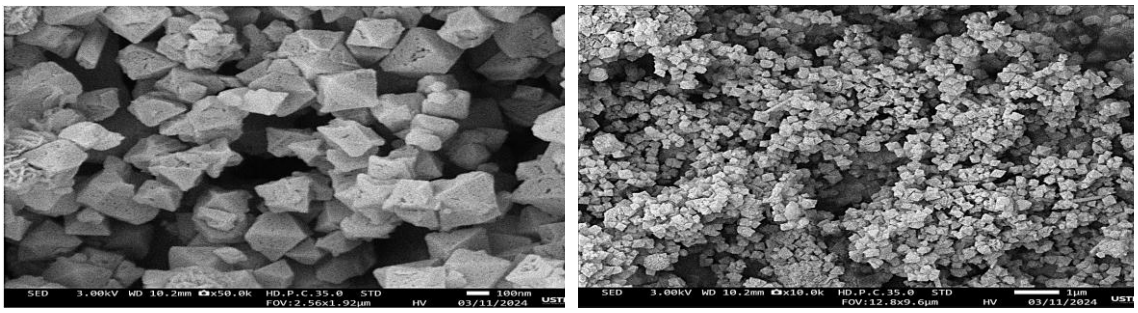

Figure 2. *FE-SEM image of MIL-101 material.*

3.1.2. Crystal structure and bonding characteristics of the material

The D8-Advance X-ray diffractometer (Bruker) was used with a CuKα radiation source (λ = 1.5406 Å) operating at a power of 1000 W (40 kV, 25 mA) at room temperature, with a 2θ scan range from 5º to 50º and a scan speed of 0.02º/0.2 seconds to measure the XRD of the material. Figure 3 shows the peaks at 8.3º, 10.3º, and 16.6º corresponding to the Miller indices (020), (220), and (240) of MIL-101 material, with high and distinct XRD peaks, indicating that the synthesized MIL-101 material has a good crystalline structure and high order within the framework structure.

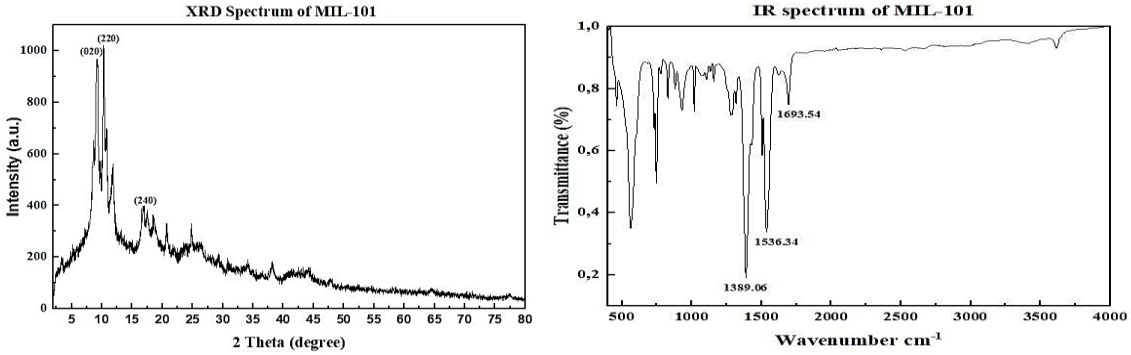

Figure 3. *XRD and FT-IR of MIL-101 material.*

The bonding characteristics were determined using FTIR spectroscopy on the TENSOR II device from Bruker, Germany, with a wavelength range of 400 to 4000 cm^{-1}. The FT-IR spectrum of the MIL-101 sample shown in figure 4 reveals a prominent absorption band at 1389 cm^{-1}, which is associated with the symmetric stretching vibration of the O-C-O group, characteristic of the dicarboxylate group in the MOF framework. A characteristic absorption peak at 1536 cm^{-1} corresponds to the C=C or C=O bond in organic molecules bound to the metal. Additionally, the absorption band at 1693.54 cm^{-1} indicates the presence of -OH groups within the porous structure of the material. These characteristics confirm that the synthesized material exhibits all the characteristic properties of MIL-101 [6].

3.1.3. Specific surface area of the material

The specific surface area was determined based on the nitrogen adsorption isotherm at 77K using the TriStar II Plus surface area analyzer. The results of the porosity analysis have shown that the MIL-101 material has a specific surface area of 3017 m^2/g, a pore radius of 1.68 nm, a pore volume of 0.396 cm^3/g, and a pore surface area of 230.65 m^2/g. Compared to previously published studies [6, 9], the MIL-101 material synthesized in this study exhibits relatively high porosity. MIL-101, with a larger surface area, provide more active sites for molecules nitrate to interact and undergo photocatalytic reactions and increases the material's ability to absorb light, as more particles are exposed to illumination. This promotes the generation of reactive radicals (•OH, $•O_2^-$), which are essential for degrading pollutants.

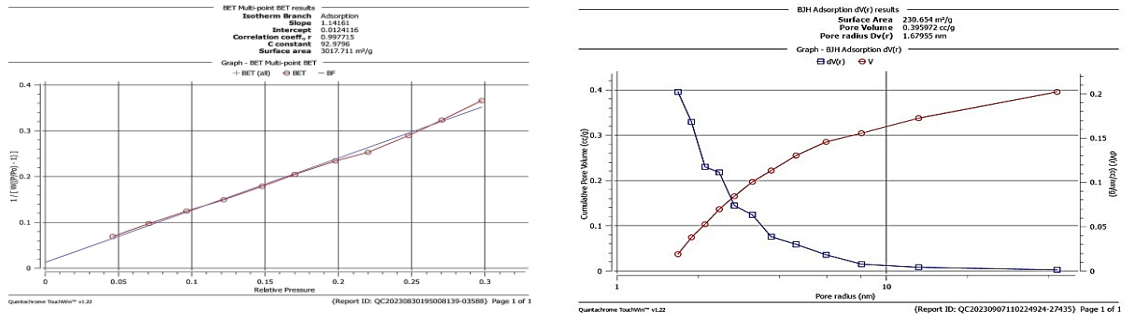

Figure 4. *Specific surface area and porosity of MIL-101 material.*

3.2. Performance of nitrate removal using MIL-101 material in aqueous solution

a) The nitrate adsorption capability of MIL-101

The nitrate adsorption experiment of MIL-101 was carried out in the dark at room temperature using a YIHDER TS 520D shaker. 0.08 g of MIL-101 was added to 100 ml of a 100 mg/l nitrate solution and shaken at 150 rpm for 12 hours. At the end of the experiment, the solution was centrifuged and the nitrate concentration was analyzed, resulting in a measured concentration of 98.7 mg/l, indicating that the nitrate adsorption capacity of MIL-101 is negligible.

b) Photodegradation efficiency of nitrate by mass of MIL-101

The nitrate removal efficiency of MIL-101 material based on mass is shown in figure 5a with a reaction time of 60 minutes, 100 mL solution, and an initial nitrate concentration of 100 mg/L. The bar chart comparing the nitrate removal efficiency with different masses of MIL-101 material indicates that MIL-101 can effectively remove nitrate under light irradiation. Figure 5a shows that as the catalyst mass increases, the nitrate removal efficiency rate rises from 52.5% to 67.3%, with the highest efficiency rate observed at a MIL-101 mass of 0.08 g, corresponding to a nitrate removal efficiency rate of 67.3%. However, when the material mass is increased to 0.10 g and 0.15 g, the efficiency rate gradually decreases to 61.0% and 58.5% within the same period of time, respectively. The decrease in efficiency rate could be because when the material mass increases, a large portion of the material's surface area may not be exposed to light, reducing the ability to activate photocatalytically. The areas not illuminated do not participate in the photocatalytic reactions, leading to a decrease in catalytic effectiveness.

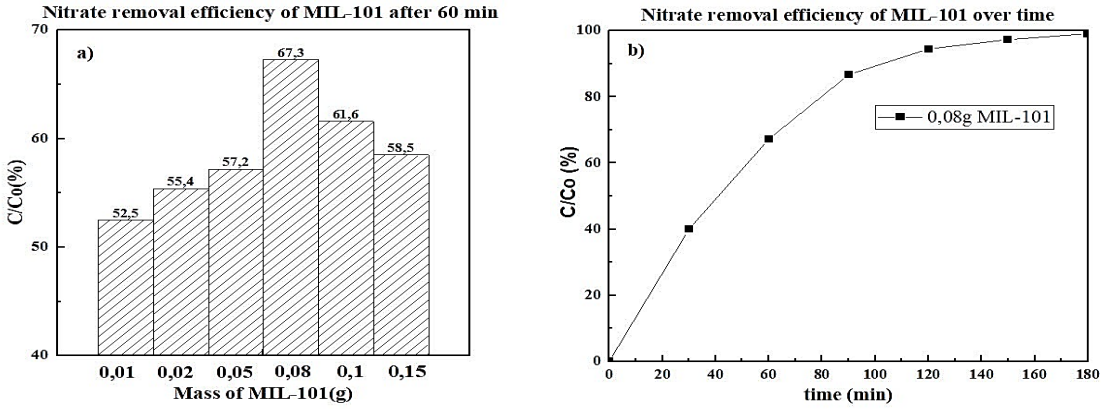

Figure 5. *Nitrate removal efficiency of MIL after 60 mins and contact time.*

Figure 5b shows the nitrate removal efficiency over time with the optimal MIL-101 mass is 0.08 g. It can be observed that as the reaction time increases, the removal efficiency also increases. After 2 hours of reaction, the nitrate removal efficiency reaches 94.5%. In the first 30 minutes, the efficiency may increase rapidly due to the chemical reactions between the free electrons generated

and the nitrate, removal efficiency reaches 40%. After each subsequent 30-minute interval, the removal efficiency decreases compared to the first 30 minutes. The nitrate removal efficiency reaches 94.5% after 2 hours and reaches 99% after 3 hours, and the chemical reactions slow down due to the decrease in the concentration of nitrate ions in the solution, resulting in a slower rate of removal efficiency.

c) Photodegradation efficiency of nitrate when adding hole scavengers

The influence of the hole scavengers on the nitrate treatment efficiency is illustrated in figure 6a. The results show that when adding 2.3 mmol of HCOOH (23 mM), the nitrate concentration significantly decreased over time, and after 30 minutes of reaction, the nitrate concentration reduced from 100 mg/L to 11.2 mg/L, corresponding to an efficiency of 88.8%. In contrast, without adding HCOOH and only using 0.01 g of MIL-101, the treatment efficiency reached only 30% after 30 minutes, indicating a significant increase in nitrate treatment efficiency with the presence of the hole scavengers. When the amount of hole scavengers was doubled to 4.6 mmol of HCOOH (46 mM), the treatment efficiency reached 92% after 30 minutes of reaction, an increase of nearly 4%. After 1 hour, no nitrate was detected in the solution with the addition of formic acid, and the nitrate removal efficiency reached 100%. It can be observed that formic acid donates electrons to quench the photogenerated holes through reaction (6), preventing the recombination of free electrons and photogenerated holes, thereby significantly increasing the nitrate removal efficiency. Besides formic acid and oxalic acid, there are many other hole scavengers in photocatalysis. However, both acids have simple structures and are easily oxidized completely to CO_2 and H_2O, without leaving any by-products that cause pollution, making them suitable for environmental treatment.

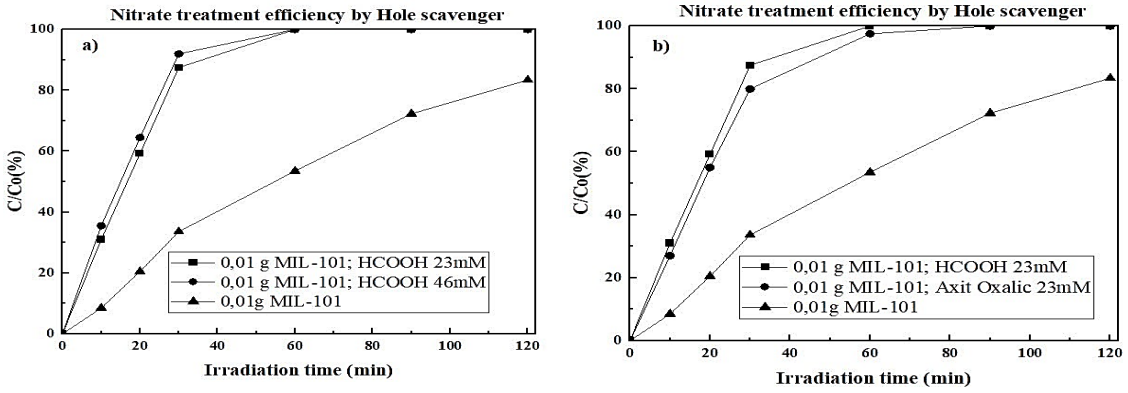

Figure 6. *Treatment efficiency of the material in the presence of a hole scavengers.*

Figure 6b show that when both sacrificial agents are added at the same concentration of 23 mM, the nitrate concentration decreases considerably over time. After 30 minutes of reaction, the nitrate concentration drops from 100 mg/L to 11.2 mg/L, corresponding to an efficiency of 88.8%, and reaches 100% removal efficiency after 1 hour of reaction. In contrast, when oxalic acid 23 mM is added, the nitrate removal efficiency reaches 80.2% after 30 minutes and 96.5% after 60 minutes of reaction. Both formic acid and oxalic acid can act as sacrificial agents, providing electrons to minimize the recombination process of electron-hole pairs in the photocatalyst, thus improving the reaction efficiency according to reactions (6) and (7). However, at the same concentration, formic acid exhibits higher nitrate reduction efficiency, because formic acid has a simpler structure and smaller molecular size than oxalic acid, leading to faster decomposition under light irradiation, thereby providing electrons more quickly and continuously during the photocatalytic process.

3.3. Comparison of nitrate removal efficiency for different types of catalysts

Table 1 summarizes the results attained in this work with those reported in the bibliography.

As can be observed, regardless of the hole scavenger, the use of MIL-101 compares favorably with all of them in nitrate removal efficiency. Similar results with other solids require the presence of noble and expensive metals (e.g., Au, Pd, and Pt).

Table 1. *Comparison of nitrate removal efficiency for different types of catalysts.*

Catalysts	UV Source	$[NO_3^-]_o$	Hole scavenger	Time graduation	Treatment efficiency	Ref.
MIL-101	High-pressure Hg, 250 W	100 mg N/L	HCOOH 23 mM	40 min	100%	This work
P_{25}	Medium-pressure Hg, 150 W	50 mg/L	$H_2C_2O_4$ 20 mM	4 h	90%	[11]
Ag_2O/ P_{25}	High-pressure Hg, 300 W	100 mg N/L	HCOOH 40 mM	4 h	97,2%	[12]
Fe/TiO_2	High-pressure Hg, 110 W	100 mg/L	HCOOH 40 mM	3 h	100%	[13]
AgCl/TiO_2 nanotube nano-Cu	High-pressure Hg, 250 W	100 mg N/L	HCOOH 100 mM	30 min	94,5%	[14]
$CuFe_{0.7}Cr_{0.3}S_2$ $0.75_{wt\%}Pd$, $3_{wt\%}Au$	Hg 500W or Xe 300 W	100 mg N/L	$Na_2C_2O_4$ 10 mM	5 h	100%	[15]
2.5 Cu_2O/TiO_2-20AC	400 W UVlamp	100 mg N/L	$Na_2C_2O_4$ 5 mM	3 h	12,7%	[16]

4. CONCLUSIONS

The metal-organic framework material MIL-101(Cr) was successfully synthesized using the hydrothermal method. The synthesized material exhibits the characteristic octahedral structure of MIL-101 with relatively high crystallinity and a uniform particle size distribution between 100-300 nm. The material has a large specific surface area of up to 3017 m^2/g, a pore radius of 1.68 nm, a pore volume of 0.396 cm^3/g, and a pore surface area of 230.65 m^2/g, indicating a relatively high porosity. The material demonstrates photocatalytic activity in nitrate removal with an initial concentration of 100 mg/L (NO_3^-). Without hole scavengers, the material (0.08 mg/100 mL solution) achieved a maximum nitrate removal efficiency of 99% after 180 minutes of reaction. In the presence of hole scavengers, the solution reached a nitrate removal efficiency of 92% within 30 minutes of reaction with HCOOH 46 mM. Comparing the nitrate removal efficiency of this study with previously published reports shows that MIL-101 effectively treats high-concentration nitrate with a shorter treatment time. These results confirm the potential of MIL-101(Cr) as an effective photocatalyst for nitrate removal in water, paving the way for development in environmental treatment applications.

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TÓM TẮT

Nghiên cứu chế tạo vật liệu khung cơ kim MIL-101(Cr) và khả năng xúc tác quang của vật liệu để xử lý Nitrat trong nước

Bài báo này trình bày kết quả nghiên cứu một số đặc trưng của vật liệu khung cơ kim MIL-101(Cr) và khả năng xử lý nitrat trong môi trường nước của vật liệu trên cơ sở phản ứng xúc tác quang. Vật liệu này được chế tạo bằng phương pháp thủy nhiệt trong phòng thí nghiệm và các kỹ thuật FE-SEM, XRD, FT-IR, BET đã được sử dụng cho thấy cấu trúc xốp của vật liệu rất phát triển, diện tích bề mặt riêng lên đến 3017 m^2/g, kích thước tinh thể bát diện đặc trưng với kích thước hạt khoảng 100 - 300 nm. Kết quả thí nghiệm cho thấy trong điều kiện ánh sáng đèn UV bước sóng 365 nm, công suất 250 W, MOF MIL-101(Cr) có khả năng xúc tác xử lý nitrat dưới ánh sáng đèn UV bước sóng 365 nm, công suất 250 W với hiệu suất xử lý cao nhất lên đến 99% sau 180 phút phản ứng. Hiệu suất xử lý nitrat của MIL-101 đạt gần 100% trong thời gian phản ứng là 40 phút khi xuất hiện tác nhân loại bỏ lỗ axit fomic (HCOOH 46 mM).

Từ khóa: MOF; MIL-101; Hiệu suất xử lý nitrat; Tác nhân loại bỏ lỗ.