RESEARCH ARTICLE

Phenol Photodegradation over Ni/MCM-41: Effect of the Ni Incorporation Method

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Abstract

This study explores the synthesis and application of Ni-based photocatalysts supported on MCM-41 and Al-MCM-41 for phenol degradation. Three preparation methods were evaluated and compared: Chemical Vapor Deposition (CVD), Incipient Wet Impregnation (IWI), and Template Ion Exchange (TIE). The influence of the Si/Al ratio of 5 and 60 on phenol photodegradation was also examined. The results showed that the Ni/MCM-41 photocatalyst prepared by CVD achieved the highest degradation efficiency (97.28%), while the TIE method proved to be the most versatile overall. Notably, the TIE method was the only synthesis method capable of exhibiting photocatalytic activity in phenol degradation on all supports at different Si/Al ratios, unlike the materials prepared by CVD and IWI, which failed to degrade phenol in the presence of aluminum. These results indicate the TIE method as a novel synthesis method for the development of mesoporous photocatalysts with high photocatalytic activity in the degradation of phenol with different Si/Al ratios in the MCM-41 structure.

Keywords: Ni/MCM-41, photodegradation of phenol, TIE, CVD, IWI.

1 Introduction

Water pollution is a major environmental problem worldwide caused by human activities that generate the discharge of wastewater containing organic and inorganic compounds, heavy metals, dyes, and pharmaceutical waste [1]. Among these compounds, phenol is a persistent organic pollutant (POP) that is resistant to degradation through conventional treatment processes [2].

Heterogeneous photocatalysis is an advanced

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oxidation process (AOP) that has emerged as an alternative technology for wastewater treatment with the ability to degrade and mineralize a wide range of contaminants, including phenol [3]. NiO has proven to be one of the best semiconductors for the photocatalytic degradation of pollutants, due to its properties of high photoactivity, stability, non-toxicity, accessibility, and low cost [4]. Several strategies have been developed to improve visible light absorption and/or reduce e^-/h^+ recombination. Among these, doping and impregnation with transition metal ions and/or the use of mesoporous supports lead to improved photocatalytic activity [5]. The MCM-41 material has been of great interest as a photocatalytic support due to its characteristics [6–9]. The development of new efficient materials beyond TiO2 and the modification of the photocatalyst are of great current interest [7]. The choice of synthesis method plays a crucial role in incorporating Ni over MCM-41, as it has a direct impact on the physicochemical properties of the photocatalyst, including particle dispersion, surface area, and photocatalytic activity. The IWI method is the most widely used method for catalyst preparation due to its simplicity, low cost, and effectiveness. However, it can result in poorly dispersed particles due to limited interaction between the support and the precursor [10]. Novel methods, such as TIE and CVD, offer greater dispersion, enhanced support-precursor interaction, and a larger surface area [11].

The use of Ni/MCM-41 as a photocatalyst for the photodegradation of phenol has been scarcely explored in the literature, making this material novel for this application. This study evaluates the influence of the synthesis method on the preparation of Ni/MCM-41 photocatalysts by comparing a conventional method - IWI - with two novel and/or understudied methods in the field of photocatalysis: TIE and CVD.

2 Methodology

2.1 Materials

Fumed silica (SiO_2, 99.8%, Sigma Aldrich), tetrabutylammonium hydroxide (TBAOH, 40 wt.%, Sigma Aldrich), cetyltrimethylammonium bromide (CTAB, $\geq$ 97%, Sigma Aldrich), sodium aluminate ($NaAlO_2$, technical grade, Sigma Aldrich), nickel nitrate (Ni $(NO_3)_2 \cdot 6H_2O$, ACS reagent, 99%, Sigma Aldrich), nickel acetylacetonate (Ni $(acac)_2$, 95%, Sigma Aldrich), deionized water (DIW), oxygen (O_2), nitrogen (N_2) and phenol (C_6H_5OH, 99.5%, Sigma Aldrich).

2.2 Synthesis of MCM-41 and AlMCM-41 supports

The MCM-41 and Al-MCM-41 supports were synthesized following a modified method described by Perea et al. [12]. The molar composition obtained was 1 SiO_2 : 0.35 CTAB : 0.31 TBAOH : 0–0.2 $NaAlO_2$: 55 H_2O. $NaAlO_2$ was used as the aluminum source, and its amount was adjusted to obtain different Si/Al ratios 60 and 5.

2.3 Ni/MCM-41 and Ni/Al-MCM-41 photocatalysts synthesis

The Ni/MCM-41 and Ni/Al-MCM-41 photocatalysts prepared by the TIE method were synthesized following the procedure described by Yonemitsu et al. [13]. Obtaining Ni loads of 4.0, 4.6 and 2.8 wt.% for MCM-41, MCM-41 Si/Al of 60 and MCM-41 Si/Al of 5, respectively. The photocatalysts prepared by the IWI method were synthesized using $Ni(NO_3)_2 \cdot 6H_2O$ as a precursor to obtain nominal Ni loadings of 4.0, 4.6, and 2.8 wt.%. The impregnated solids were dried at 105 °C for 24 h and calcined at 600 °C for 6 h at a heating rate of 5 °C min^{-1}. The photocatalysts prepared by the CVD method were synthesized in an L-shaped APCVD reactor. $Ni(acac)_2$ was used as the precursor; the precursor was sublimated at 220 °C and decomposed at 350 °C for 180 min and calcined at 600 °C for 6 h at a heating rate of 5 °C min^{-1}.

2.4 Photodegradation of phenol

The photocatalytic experiments were performed using 200 mL solutions at a concentration of 40 mg/L of phenol in a batch reactor, in which 0.2 g of photocatalyst was suspended. The reactor has a double wall that allows cooling by water flow, maintaining constant temperature, pressure, and air bubbling during the reaction. Each photoreaction was monitored for a total of 7 h, under continuous stirring at 300 rpm and using a 254 nm UV lamp suspended vertically, centered, and submerged in the reactor within a quartz immersion tube.

3 Experiments

Figure 1 shows the results of the C/C_0 ratio as a function of time during the photodegradation of phenol using Ni/MCM-41 photocatalysts with Si/Al ratios of 60 and 5, prepared by TIE, CVD, and IWI methods.

The results indicate different material behavior depending on the synthesis method and the Si/Al

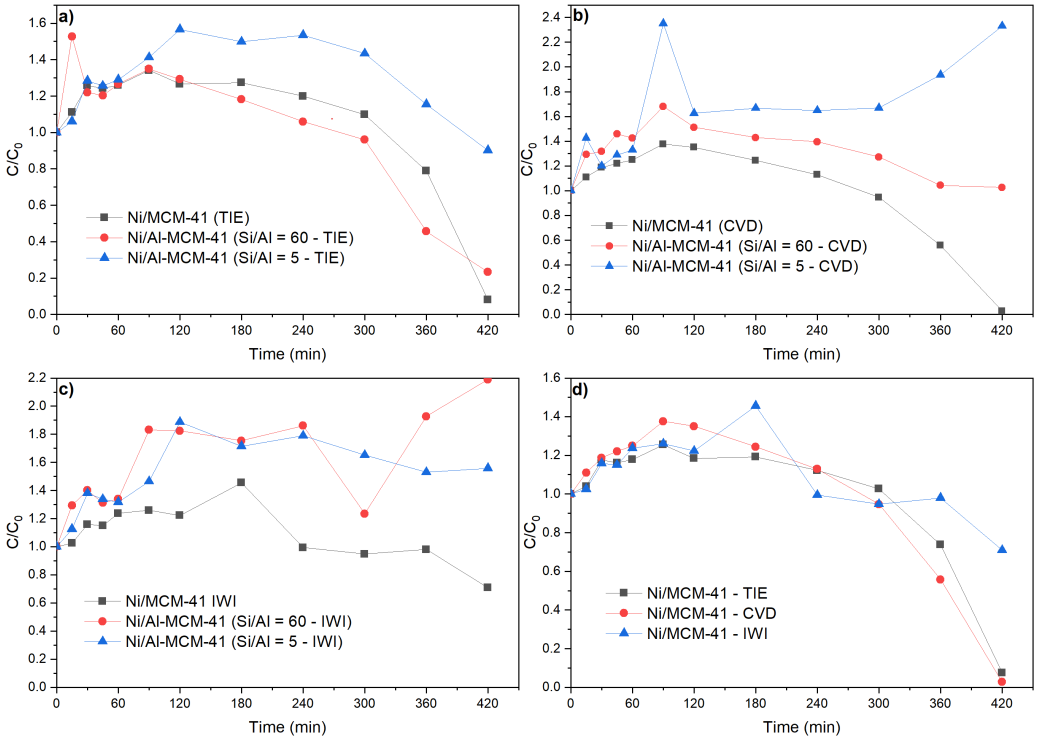

Figure 1. Variation of C/C_0 during the photodegradation of phenol as a function of time using Ni/MCM-41 and Ni/Al-MCM-41 with Si/Al ratios of 60 and 5 prepared by the methods a) TIE, b) CVD, c) IWI and d) comparison of the preparation methods.

ratio. Figure 1(a) shows that the Ni/MCM-41 and Ni/Al-MCM-41 materials prepared by the TIE method exhibit photocatalytic activity in the degradation of phenol. However, the Ni/MCM-41 material shows the greatest phenol degradation, reaching 92.45%, followed by the Ni/MCM-41 material with a Si/Al ratio of 60, which achieves a degradation of 76.69%. Finally, the photocatalyst with a Si/Al ratio of 5 exhibits the lowest photocatalytic activity, with a degradation of only 9.79%.

The photocatalytic activities of Ni/MCM-41 and Ni/Al-MCM-41 materials prepared by CVD and IWI methods for phenol degradation are shown in Figures 1(b) and 1(c). The Ni/MCM-41 material prepared by CVD exhibited the highest phenol degradation, reaching 97.28%. In contrast, the Ni/MCM-41 material prepared by IWI only degraded 29.11%.

Photocatalysts with Si/Al ratios of 60 and 5, prepared by both methods, showed no phenol degradation. This indicates that the presence of aluminum reduces the photocatalytic efficiency of photocatalysts prepared by the CVD and IWI methods, though the TIE method retained activity even in the presence of aluminum. Initial increases in C/C_0 were observed throughout the reaction, suggesting the formation of intermediates that absorb closer to the UV-Vis region of the spectrum than phenol. This phenomenon has been widely reported in the literature since various photocatalysts have been used during the photodegradation of phenol, and intermediates such as hydroquinone, catechol, and benzoquinone are formed, which can be detected by UV spectrophotometry at wavelengths of 290 nm, 275 nm, and 255 nm, respectively [14, 15]. Furthermore, the initial increase in absorbance has been observed by UV-Vis spectroscopy and is attributed to the formation of these aromatic intermediates, whose characteristic UV-Vis peaks generate an increase in the signal during the reaction, as reported by Al-Hamdi et al. [14]. Although the literature reports that incorporating aluminum can improve the photodegradation of pollutants [16], in our Ni/MCM-41 materials with Si/Al ratios of 5 and 60, the presence of aluminum had a negative effect, preventing the complete degradation of phenol and causing the presence of intermediates during the reaction. Therefore, modification with aluminum does not always improve photocatalytic activity if it does not favor the generation of reactive species.

The superiority of the TIE and CVD methods over IWI method can be explained by their ability to enhance the interaction between the precursor and

the support, resulting in highly dispersed Ni particles and improved accessibility to the active sites, as shown in Figure 1(d). Furthermore, these methods contribute to maintaining high surface areas compared to the IWI method, which frequently produces agglomerated particles and less uniform Ni distribution on the mesoporous support surface.

The results of this study, in conjunction with the literature [15], indicate that CVD method is superior to IWI method in the synthesis of catalysts for advanced oxidation processes. This explains the higher degradation percentages obtained with Ni/MCM-41 prepared by CVD compared to a conventional method.

To support these results, the study will be complemented with characterization techniques such as XRD, BET, FTIR, SEM/TEM, and UV-Vis, which will allow us to correlate the photocatalyst's properties with the observed degradation performance. High-performance liquid chromatography (HPLC) will be used to identify the phenolic compounds present during the photocatalytic reactions. A Total Organic Carbon (TOC) analysis will be performed to verify that the mineralization process of the organic contaminant is taking place. This is because the formation of intermediate compounds during phenol photodegradation poses a risk when UV-Vis spectrophotometry alone is used to determine phenol concentration.

4 Conclusion

Ni/MCM-41 and Ni/Al-MCM-41 photocatalysts were prepared using TIE, CVD, and IWI methods. The study compares these synthesis methods to evaluate their influence on phenol photodegradation. The results demonstrated that both the Ni incorporation method and the Si/Al ratio significantly determine the photocatalytic efficiency for phenol degradation.

The TIE method is presented as a novel method in this field, standing out as the only synthesis method capable of achieving phenol degradation using Ni/MCM-41 and Ni/Al-MCM-41 with Si/Al ratios of 5 and 60. The Ni/MCM-41 photocatalyst prepared by CVD method achieved the highest phenol degradation (97.28%), while the TIE method obtained 92.45%. This is attributed to a greater dispersion of Ni particles and improved metal-support interaction compared to the material prepared by the IWI method. Conversely, no phenol degradation was observed during the reaction with the Ni/Al-MCM-41 photocatalysts prepared by the CVD and IWI methods; therefore, the Ni/MCM-41 photocatalysts prepared by the TIE method offer advantages for the development of mesoporous photocatalysts with superior photocatalytic performance.

Data Availability Statement

Data will be made available on request.

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Conflicts of Interest

The authors declare no conflicts of interest.

AI Use Statement

The authors declare that generative AI tools were used in the preparation of this manuscript. ChatGPT-5 was used for grammatical and stylistic refinement in the Introduction and Discussion sections. Grammarly was used for language checking and overall manuscript proofreading.

Ethical Approval and Consent to Participate

Not applicable.

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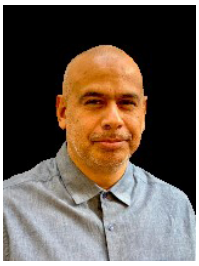

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