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Photocatalytic Performance Of Fe/Zeolite Bead Catalyst For The Removal Of N-Methyl-2-Pyrrolidone

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ABSTRACT

The increasing presence of N-methyl-2-pyrrolidone (NMP) in industrial wastewater poses a significant environmental challenge due to its toxicity, chemical stability, and resistance to conventional treatment methods. This research presents the synthesis and application of a novel 3wt% Fe/Zeolite bead catalyst for the efficient photocatalytic degradation of NMP under visible light irradiation. The beads were fabricated by incorporating Fe/Zeolite into a sodium alginate polymer matrix, followed by crosslinking with epichlorohydrin to improve their mechanical strength, hydrophilicity, and structural stability. The photocatalytic performance of the synthesized beads was systematically evaluate d under varying conditions, including pH levels (3–11), catalyst dosages (20–100 g L^{-1}), and initial NMP concentrations (20–100 mg L^{-1}). Optimal degradation efficiency was achieved at pH 7, with a catalyst dosage of 40 g L^{-1} and an initial NMP concentration of 20 mg L^{-1}, resulting in a maximum removal rate of 86.41% within 120 minutes of visible light exposure. The 3wt% Fe/Zeolite bead catalyst exhibited excellent reusability, magnetic recoverability, and structural integrity, making it a promising candidate for scalable and sustainable wastewater treatment. This study demonstrates the potential of magnetically recoverable 3wt% Fe/Zeolite beads as a cost-effective and environmentally friendly approach for the remediation of organic pollutants in industrial effluents.

Keywords : *Magnetic zeolite bead catalyst, N-methyl-2-pyrrolidone, photocatalytic degradation, visible light irradiation, wastewater treatment.*

1. INTRODUCTION

The intensification of industrial development, particularly in the semiconductor, petrochemical, and pharmaceutical sectors, has resulted in the growing release of persistent organic pollutants into the aquatic environment. Among these contaminants, N-methyl-2-pyrrolidone (NMP) is of particular concern due to its widespread use as a high-boiling-point, polar aprotic solvent (Sliz et al., 2022). NMP is commonly employed in various manufacturing processes including microelectronics cleaning, surface coatings, synthetic resins, and lithium battery electrolytes, owing to its excellent solvency, low volatility, and chemical stability (Chattopadhyay et al., 2023). However, the environmental fate of NMP is problematic. NMP is not only highly soluble in water but also exhibits high resistance to biodegradation, making it a recalcitrant pollutant in conventional biological wastewater treatment systems. Its acute and chronic toxic effects on aquatic organisms, along with potential human health risks such as reproductive toxicity, skin irritation, and neurotoxicity, have led to regulatory concerns (Li et al., 2025; Ramsperger et al., 2023). For instance, the European Chemicals Agency (ECHA) has classified NMP as a substance of very high concern (SVHC) under REACH regulations (Li et al., 2025). This underscores the urgent need for advanced treatment technologies capable of degrading such pollutants effectively and sustainably.

A variety of technology-driven solutions are being explored today to address these environmental challenges. The rapid growth of technology over the past few decades, one of the main areas of concern in modern times is the search and utilisation of renewable energies (Pulli et al., 2020). The renewable nature, ease of use, and mobility of solar energy have made it a highly regarded choice for sustainable technology such as photocatalysis. The photocatalysis is a unique technique that is widely used in many interdisciplinary study disciplines. These days, photocatalysis shows great promise for recovering ecological principles and advances in sustainability. Since the process can be influenced by light or photon sources, photocatalysis theoretically requires radiation to activate electrons to shift to begin the reaction. These processes produce highly reactive species, such as hydroxyl radicals (•OH), which can engage with various organic compounds via electrophilic addition to double bonds or electron-transfer reactions. The intermediates formed can subsequently react with dissolved molecular oxygen, boosting degradation efficiency and transforming contaminants into less harmful or non-toxic substances (Dostanić, et al., 2024).

In photocatalysis, magnetite and maghemite are both often used. Iron oxide nanoparticles clump together because of their high surface to volume ratio, demonstrating an important proportion of surface energy ron-based oxides, such as magnetite (Fe_3O_4) and hematite (α-Fe_2O_3), have shown potential as visible-light-active photocatalysts due to their narrow band gap and strong redox properties (Kumar et al., 2022; Saied et al., 2022). Fe_3O_4, in particular, offers the added advantage of magnetic separability, which enables easy recovery and reuse of the catalyst, thereby reducing secondary pollution and operational costs. Iron oxide nanoparticles' dispensability and magnetic function are diminished by their strong reactivity, leading to them promptly oxidised in the environment. Therefore, it is imperative to maintain the iron oxide equilibrium, and this is easily achieved by carefully encapsulating the nanoparticles with organic ligands, polymers, and monomers. Deposition of Fe_3O_4 nanoparticles lowers surface-active regions, a feature essential for improved diffraction of light (Kumar et al., 2024). The photocatalysts may be easily retrieved from the method of processing in the aid of a magnetic external field, catalysts that integrate catalytic properties with magnetism allow photocatalysts to be regenerative. The enhanced optical, chemical-based magnetic, electrical, and thermal stability of magnetic iron oxide nanoparticles has allowed them to be used in a variety of applications, such as dyes, data storage, electronic products, catalysis, ferrofluids, and medical treatments (Imran et al., 2022). However, standalone Fe-based catalysts often suffer from drawbacks such as fast electron-hole recombination and limited surface area, which restrict their photocatalytic efficiency.

To enhance the performance of Fe-based catalysts, zeolites have been introduced as structural and functional supports. Zeolites are microporous crystalline aluminosilicates known for their high surface area, thermal stability, and ion-exchange capacity (Liaquat et al., 2024). When combined with iron oxides, zeolites can improve catalyst dispersion, promote pollutant adsorption, and facilitate charge carrier mobility, ultimately leading to enhanced photocatalytic activity. Moreover, their porous framework aids in concentrating pollutants near active sites, which is especially beneficial for low-concentration contaminants like NMP. Nevertheless, the application of powdered photocatalysts poses practical limitations in water treatment, including particle agglomeration, low mechanical stability, and challenges in recovery and reuse. To address these issues, researchers have turned to immobilization techniques, especially the fabrication of bead-type catalysts using biopolymers such as sodium alginate. Alginate, derived from brown algae, can form hydrogel beads in the presence of divalent cations like $Ca2^+$ and serves as a robust and environmentally benign matrix for encapsulating photocatalytic materials. The resulting composite beads combine the benefits of photocatalysis, adsorption, and easy recovery, while maintaining high reactivity and stability under aqueous onditions.

The optimal photocatalytic degradation can be achieved in this study by incorporation of magnetic zeolite catalyst with sodium alginate. This integration boosts the material's surface area, improving its attraction to hydroxyl groups and promoting the formation of hydroxyl radicals. Sodium alginate, an anionic polysaccharide, has gained popularity as a polymeric adsorbent due to its long shelf life, cost-effectiveness, and adsorption-regeneration capabilities (Wang & You, 2021). Composed of β-D-mannuronic acid and α-L-guluronic acid, sodium alginate can be formed into hydrogel beads and chemically modified through cross-linking of its α-L-guluronic acid units with poly- or divalent cations (Malektai et al., 2023). Widely utilized as a support framework for polymeric catalysts, alginate offers numerous

advantages as a bio sorbent. Its hydrophilic nature, natural abundance, active binding sites, and sustainability contribute to its superior performance.

Magnetic zeolite catalyst combined with alginate were selected for their ability to enhance photocatalytic performance in several keyways. First, alginate serves as an excellent stabilizer, preventing the aggregation of magnetic particles and improving the stability and durability of the catalyst. This ensures more consistent performance over time, which is often a challenge with bare magnetic beads. Additionally, the magnetic zeolite beads allow for easy recovery using an external magnetic field, making them highly recyclable. The alginate coating helps protect the beads from degradation during repeated cycles, addressing the common issue of catalyst loss seen with other material (Sanakal et al., 2025). This contributes to the cost-effectiveness of the system. The synergistic effect of the magnetic beads and alginate further improves photocatalytic performance. The alginate matrix contains functional groups such as hydroxyl or carboxyl groups, which can interact with and adsorb pollutants, increasing their concentration at the catalyst's active sites. This interaction enhances the efficiency of the photocatalytic process. Moreover, alginate is biodegradable and environmentally friendly, making it a more sustainable option compared to non-biodegradable catalysts like TiO_2, Fe_3O_4 or ZnO (Wan et al., 2022).

Overall, the combination of Fe/Zeolite catalyst and alginate is expected to provide superior photocatalytic performance by offering improved stability, recyclability, and cost-effectiveness, while also being environmentally friendly. These attributes make magnetic beads a promising alternative in catalytic applications, offering both practical and economic benefits. Current work on the advancement of Fe/Zeolite bead catalysts and nanomaterials holds great promise, especially for purifying water processes. Given its porous architecture and greater surface area, the nanoparticles in magnetic bead catalyst possess the ability to respond swiftly. They may further be functionalised with various chemical groups to boost their selectivity or reactivity for a specific molecule. The process for developing Fe/Zeolite bead catalyst strengthened the shape and arrangement of the iron particles, resulting in turn boosted the catalyst's capabilities and accelerated the breakdown of NMP in water. The activity of the catalyst for the photodegradation of NMP was studied under various conditions to determine the effect of pH, catalyst dosage, and initial phenol concentration.

2. MATERIALS AND METHODS

2.1 Materials

All chemicals were brought in analytical grade and used without further purification. Iron (II, III) oxide magnetic (Fe_3O_4) was obtained from Bendosen Laboratory Chemicals, ethanol absolute (C_2H_5OH) (> 99% purity) from HmbG Chemicals, calcium chloride ($CaCI_2$) was obtained from R&M Chemicals and sodium hydroxide (NaOH), hydrochloric acid (HCl), were obtained from QReC^{TM}. Zeolite ($C_5H_8O_2$), epichlorhydrine and sodium alginate ($NaC_6H_7O_6$) were purchased from Sigma-Aldrich (Malaysia). N-methyl-2-pyrrolidone (C_5H_9NO) (>99% purity) was obtained from JAEWON Co. Ltd.

2.2 Preparation of catalyst

To prepare the precursor mixture, the powdered catalyst was thoroughly blended into a 3% (w/w) sodium alginate solution made with deionized water, resulting in a homogeneous and viscous suspension. This mixture was then dispensed dropwise using a syringe into a coagulation bath containing 0.5 M calcium chloride ($CaCl_2$) solution, where it solidified into wet beads through ionic gelation. These beads were subsequently subjected to a chemical crosslinking process using epichlorohydrin at ambient temperature. Initially, the Fe/Zeolite beads were soaked in three sequential ethanol–water baths (60% v/v ethanol, 400 mL each) for 2 hours per bath to facilitate the replacement of water with ethanol. Following this, the beads were immersed in 400 mL of an ethanol–water mixture (60% v/v) containing 6.109 g of epichlorohydrin. The pH was then adjusted to approximately 13 by the slow addition of 1 mol/L sodium hydroxide solution, initiating the crosslinking reaction, which proceeded for 4 hours. After the reaction, the beads were rinsed in three separate baths of distilled water (600 mL each) for 2 hours to remove residual chemicals. During the final rinsing stage, concentrated nitric acid (53.7% w/w) was introduced to neutralize the system, adjusting the pH to around 7.

2.3 Photocatalytic performance study

The 3wt% Fe/Zeolite bead catalyst was evaluated for its photocatalytic efficiency in degrading N-methyl-2-pyrrolidone (NMP) under visible light irradiation. The investigation focused on the impact of key operational parameters, including solution pH levels (3, 5, 7, 9, and 11), catalyst loading (20, 40, 60, 80, and 100 g L^{-1}), and initial NMP concentrations (20, 40, 60, 80, and 100 mg L^{-1}). All experiments were carried out over a 2-hour period under constant stirring to maintain homogeneity. To adjust the pH of the solutions, 0.1 M sodium hydroxide (NaOH) or 0.1 M hydrochloric acid (HCl) was added as needed. Approximately 2.0 g L^{-1} (wet weight) of the Fe/Zeolite bead catalyst was introduced into 100 mL of a 20 mg L^{-1} NMP solution contained in a beaker. The reaction mixture was continuously stirred using a magnetic stirrer while being exposed to a fluorescent light source, positioned 15 cm above the solution surface. At selected time intervals (0, 30, 60, 90, and 120 minutes), 1.5 mL of the solution was withdrawn and analyzed using a UV–Vis spectrophotometer (Thomas Edison LSS-U2900 Double Beam). The concentration of NMP was quantified at a detection wavelength of 197 nm.

3. RESULTS AND DISCUSSION

3.1 Photocatalytic performance of bead catalyst

Photocatalytic degradation of N-methyl-2-pyrrolidone (NMP) is a promising hybrid approach that integrates adsorption and photocatalysis to effectively eliminate this recalcitrant organic pollutant from water. NMP, commonly found in industrial effluents, is known for its environmental persistence and toxicological impact. In this study, the degradation of NMP was investigated using different bead-based catalyst systems: 3wt% Fe/Zeolite magnetic bead catalyst, Fe bead catalyst, zeolite bead catalyst, and alginate bead catalyst. Batch experiments were conducted under ambient temperature conditions, involving a two-hour adsorption phase followed by a two-hour photocatalytic process. Optimal conditions for the experiments were maintained at pH 7, a catalyst loading of 40 g L^{-1}, and an initial NMP concentration of 20 mg L^{-1}. As shown in Fig. 1, the 3wt% Fe/Zeolite bead catalyst exhibited the highest photocatalytic degradation efficiency, achieving 86.41% removal of NMP. This performance was substantially higher than that of the Fe bead (56.67%), zeolite bead (53.44%), and alginate bead (23.24%) catalysts. The superior activity of the 3wt% Fe/Zeolite bead catalyst is attributed to the synergistic interaction between Fe_3O_4 nanoparticles and the zeolite framework. Iron oxide contributes active photocatalytic sites that generate ROS upon light irradiation, while zeolite enhances adsorption, promotes better dispersion of iron particles, and facilitates charge separation. The encapsulation of these active materials in a sodium alginate matrix provides mechanical stability and allows for easy recovery of the beads without impeding mass transfer or catalytic efficiency. The adsorption process involves the accumulation of NMP molecules onto the surface of a solid material, effectively concentrating them in proximity to the photocatalyst's reactive sites. This enhances the subsequent photocatalytic reactions by increasing the local pollutant concentration and facilitating greater interaction with photogenerated reactive oxygen species (ROS). These ROS, including hydroxyl radicals (•OH) and superoxide anions (O_2•–), initiate oxidative degradation of NMP, ultimately breaking it down into less harmful compounds such as CO_2 and H_2O.

In contrast, the Fe bead and zeolite bead catalysts demonstrated moderate degradation efficiencies, indicating that each component alone lacks the multifunctionality required for highly efficient NMP removal. The Fe bead likely suffered from issues such as nanoparticle agglomeration and rapid electron–hole recombination, limiting its ROS generation. The zeolite bead, although beneficial for adsorption, lacks intrinsic photocatalytic activity. The Alginate bead, used as a control, showed the lowest degradation efficiency, confirming its role solely as an inert support material with negligible contribution to NMP degradation. These findings underscore the effectiveness of the composite 3wt% Fe/Zeolite bead catalyst, which integrates both adsorption and photocatalysis in a robust, magnetically recoverable system. Its high degradation efficiency, combined with structural integrity and ease of reuse, suggests strong potential for application in the treatment of industrial wastewater containing persistent organic contaminants like NMP.

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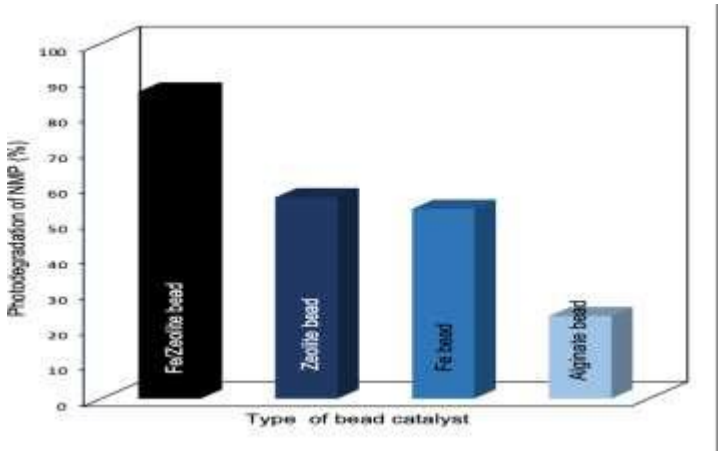

Fig. 1. Photocatalytic performance on NMP degradation validation of 3wt% Fe/Zeolite bead catalyst, zeolite bead catalyst and Fe bead catalyst, alginate bead [pH = 5; W = 40 g L^{-1}; C_0 = 20 mg L^{-1}; T = 303.15 K]

3.2 Effect of pH

Photodegradation of N-methyl-2-pyrrolidone (NMP) in aqueous solution was conducted to evaluate the photocatalytic activity of the synthesized bead catalysts. The effects of key operational parameters including solution pH, catalyst dosage, and initial NMP concentration were systematically investigated. All batch experiments were performed at ambient temperature, with a contact time of two hours for both adsorption and subsequent photocatalytic degradation. Semiconductor-based photocatalysts typically exhibit amphoteric behavior, meaning their surface charge varies with pH, directly affecting the availability and reactivity of active sites. In this study, the 3wt% Fe/Zeolite bead catalyst achieved its highest NMP removal efficiency at pH 7, with a degradation rate of 80.68% as displayed in Fig. 3.2. The enhanced performance under neutral conditions is attributed to the optimal generation of hydroxyl radicals (HO•), which are the primary oxidative species responsible for breaking down NMP molecules. At alkaline pH (pH 11), a notable decrease in degradation efficiency was observed. This reduction is likely due to the rapid decomposition of hydrogen peroxide and hydroxyl radicals under basic conditions, which diminishes the availability of ROS for effective photodegradation. On the other hand, under acidic conditions (pH 3), the excess H^+ ions in solution can protonate the surface of the photocatalyst, imparting a positive charge. This protonation limits NMP adsorption due to electrostatic repulsion, thereby reducing access to active sites and lowering the degradation efficiency. These findings highlight the importance of maintaining neutral pH 7 conditions to achieve optimal photocatalytic degradation of NMP using 3wt% Fe/Zeolite bead catalysts, as it ensures a favorable balance between surface interactions and reactive species generation.

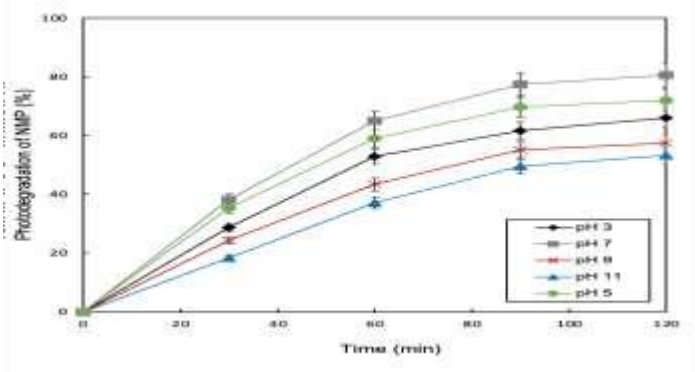

Fig. 2. Effect of pH solution for 3wt% Fe/Zeolite bead catalyst [W = 20.0 g L^{-1}; C = 20 mg L^{-1}; t = 2 h; T= 303.15 K]

3.3 Effect of catalyst dosage

An essential aspect of optimizing photocatalytic efficiency is determining the optimal catalyst dosage. As shown in Fig. 3, increasing the dosage of 3wt% Fe/Zeolite bead catalyst significantly enhanced the photodegradation of NMP up to a certain point, after which the degradation rate plateaued. The maximum degradation efficiency of 86.41% was achieved at a catalyst loading of 40 g L^{-1}. Beyond this dosage, further increases in catalyst concentration did not lead to additional improvements in degradation performance, indicating the onset of a saturation phase where the system reaches photocatalytic equilibrium. This plateau effect can be attributed to a decline in photon absorption efficiency once the catalyst concentration exceeds a critical threshold. According to the findings of Ansari et al. (2025), excessive catalyst loading can cause light scattering and internal shielding, reducing the penetration of light into the suspension and consequently lowering the number of photons reaching active sites. This light-shielding effect diminishes the effective photocatalytic surface area exposed to irradiation, thereby limiting further increases in degradation despite the availability of additional catalyst. The initial enhancement in degradation with increasing catalyst dosage is due to the greater number of active sites available on the Fe/Zeolite bead surface, which facilitates the generation of reactive oxygen species (ROS), such as hydroxyl radicals (•OH) and superoxide anions (O_2•$^-$). These radicals play a pivotal role in oxidizing and breaking down NMP molecules. The findings align with those of Ahmad et al. (2020), who reported that catalyst performance improves with increasing dosage up to an optimal point, after which the rate stabilizes or slightly declines due to particle agglomeration or light attenuation. In essence, while increasing the 3wt% Fe/Zeolite bead catalyst dosage enhances photocatalytic activity by promoting radical generation and pollutant interaction, exceeding the optimal dosage can impair performance due to optical interference and particle crowding, thus emphasizing the need for dosage optimization in practical applications.

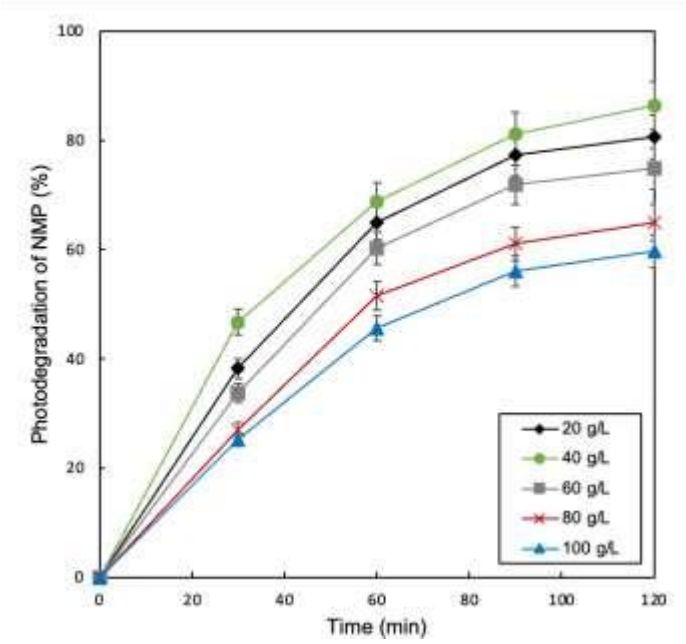

Fig. 3. Effect of catalyst dosage for 3wt% Fe/Zeolite bead catalyst [pH = 5; C = 20 mg L^{-1}; t = 2 h; T = 303.15 K]

3.4 Effect of initial concentration

The efficiency and frequency of photocatalytic degradation of pollutants are influenced by both the adsorption capacity of the catalyst and the initial concentration of the contaminant. In this study, the interaction between N-methyl-2-pyrrolidone (NMP) and the surface of the 3wt% Fe/Zeolite bead catalyst was examined to assess how initial pollutant concentrations impact photocatalytic performance. Among the tested conditions, the Fe/Zeolite bead

catalyst achieved its maximum degradation efficiency of 86.41% at an initial NMP concentration of 20 mg L^{-1} after 2 hours of light irradiation as shown in Fig. 4. As the initial concentration of NMP increased beyond this value, a decline in degradation efficiency was observed. This inverse relationship is primarily attributed to the limited number of active sites on the catalyst surface. At higher pollutant concentrations, more NMP molecules compete for adsorption on these active sites, which can inhibit the interaction between the catalyst surface and photon energy, as well as reduce the availability of reactive oxygen species (ROS) such as hydroxyl radicals (•OH). Since the generation of •OH radicals is dependent on the fixed quantity of 3wt% Fe/Zeolite catalyst and hydrogen peroxide (H_2O_2) in the system, an overabundance of pollutant molecules results in an insufficient concentration of ROS to maintain efficient degradation. Similar findings were reported by Ahmad et al. (2020), who noted a decline in degradation rate with increasing initial contaminant concentration due to site saturation and limited oxidant availability. A significant advantage of the 3wt% Fe/Zeolite bead catalyst lies in its magnetic separability. Unlike conventional photocatalysts that require filtration or centrifugation for recovery, the Fe-based beads can be conveniently retrieved using an external magnetic field, streamlining the post-treatment process, and reducing catalyst loss.

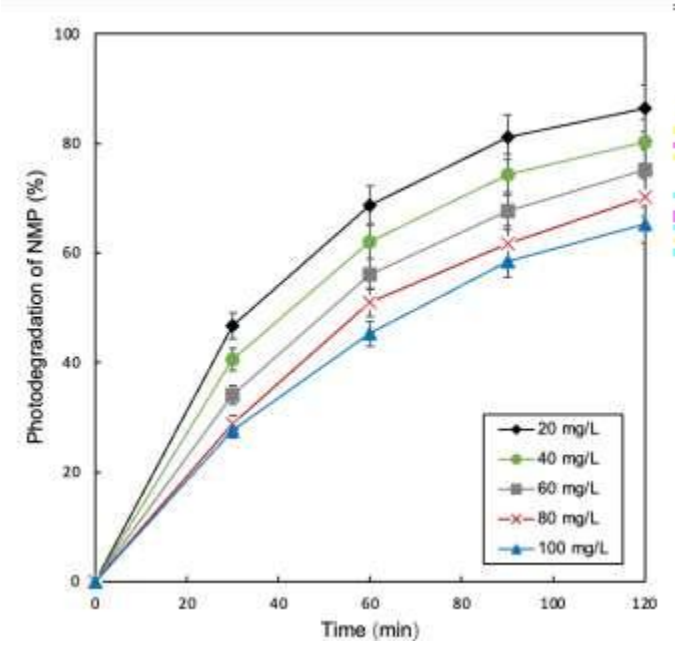

Fig. 4. Effect of initial concentration for 3wt% Fe/Zeolite bead catalyst [pH = 5; W = 40.0 g L^{-1}; t = 2 h; T = 303.15 K]

4. CONCLUSION

The developed 3wt% Fe/Zeolite bead catalyst demonstrated excellent photocatalytic activity for the degradation of N-methyl-2-pyrrolidone (NMP), achieving a maximum removal efficiency of 86.41% under optimal condition of pH 7, catalyst dosage of 40 g L^{-1}, and initial NMP concentration of 20 mg L^{-1} after 2 hours of light exposure. This process not only effectively breaks down NMP but also results in the formation of environmentally benign by-products, primarily CO_2 and H_2O. Structural modifications during synthesis led to significant improvements in the beads' pore structure, mechanical strength, chemical stability, hydrophilicity, and biocompatibility. These enhancements contributed to an increased number of active sites and improved surface morphology and selectivity toward target pollutants. As a result, the catalyst's overall efficiency in degrading NMP was significantly elevated. In summary, the findings highlight the potential of magnetic 3wt% Fe/Zeolite bead catalysts as a promising and efficient solution for addressing challenges in industrial wastewater treatment.

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