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Fe/Zelite Beads: An Innovative Strategy for Photocatalytic Degradation of *N*-Methyl-2-Pyrrolidone

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ABSTRACT

The widespread use of *N*-methyl-2-pyrrolidone (NMP) in semiconductor, petrochemical, and pharmaceutical industries has raised environmental concerns due to its toxicity, high water solubility, and resistance to conventional treatment methods. Its persistence in wastewater highlights the need for effective and sustainable remediation strategies. This study aims to develop a reusable, magnetically recoverable photocatalyst for the degradation of NMP under visible light. Fe/Zelite catalysts were embedded in a sodium alginate matrix and chemically crosslinked with epichlorohydrin to form stable bead-type photocatalysts. This immobilization approach enhances structural durability, hydrophilicity, and mechanical strength while preventing nanoparticle aggregation. The photocatalytic performance of the beads was investigated under varying pH levels (3–11), catalyst dosages (20–100 g L⁻¹), and initial NMP concentrations (20–100 mg L⁻¹). Optimal degradation efficiency of 83.28% was achieved at pH 7 with a catalyst loading of 40 g L⁻¹ and an NMP concentration of 20 mg L⁻¹ after 120 minutes of visible light irradiation. Kinetic studies confirmed that the degradation followed pseudo-first-order kinetics, aligning with the Langmuir–Hinshelwood model. The reaction rate constant (K_R) and adsorption equilibrium constant (K_{LH}) were determined to be 0.776 mg L⁻¹ h⁻¹ and 0.026 L mg⁻¹, respectively, indicating a balanced interaction between adsorption and surface reactivity. The Fe/Zelite bead catalyst also demonstrated excellent reusability, magnetic separability, and operational stability, supporting its potential for practical application in wastewater treatment systems. Overall, the synthesized hybrid photocatalyst offers a cost-effective, environmentally friendly, and efficient solution for the removal of recalcitrant organic pollutants like NMP from industrial effluents.

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1. Introduction

The rapid expansion of industrial sectors particularly semiconductor, petrochemical, and pharmaceutical industries has led to the increased release of persistent organic pollutants (POPs) into aquatic ecosystems. Among these, N-methyl-2-pyrrolidone (NMP), a high-boiling-point polar aprotic solvent, has garnered significant environmental concern due to its extensive use and poor biodegradability [1]. NMP is widely employed in applications such as microelectronics cleaning, surface coatings, lithium battery electrolytes, and synthetic resins, owing to its strong solvency, low volatility, and chemical stability [2]. However, its environmental persistence, high water solubility, and resistance to conventional biological treatment systems make it a recalcitrant pollutant [3]. Furthermore, NMP poses acute and chronic toxicity risks to aquatic organisms and is associated with adverse human health effects, including reproductive toxicity, neurotoxicity, and dermal irritation. In recognition of these risks, the European Chemicals Agency (ECHA) has classified NMP as a substance of very high concern (SVHC) under Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) regulations [4].

The removal of such stable organic compounds necessitates advanced treatment methods that are both effective and sustainable. Among emerging technologies, photocatalysis has received considerable attention due to its reliance on renewable solar energy and its potential to degrade a wide range of organic pollutants [5]. Photocatalytic processes, initiated by light-induced excitation of electrons in semiconductor materials, generate reactive oxygen species (ROS), such as hydroxyl radicals ($\bullet\text{OH}$), which can non-selectively oxidize organic contaminants into less harmful or mineralized end-products [6].

Iron-based materials, particularly magnetite (Fe_3O_4) and hematite ($\alpha\text{-Fe}_2\text{O}_3$), have emerged as promising visible-light-responsive photocatalysts due to their narrow band gaps, strong redox activity, and natural abundance [7,8]. Fe_3O_4 nanoparticles, in particular, offer the added advantage of magnetic separability, facilitating easy recovery and reusability, which reduces secondary pollution and operational costs. However, bare Fe-based nanoparticles suffer from aggregation, surface oxidation, and rapid electron-hole recombination, which limit their photocatalytic efficiency and long-term stability.

To overcome these challenges, integration with structural supports such as zeolites has been explored. Zeolites are microporous aluminosilicates with high surface area, ion-exchange capacity, and thermal stability, making them suitable hosts for dispersing and stabilizing metal oxides [9]. The incorporation of iron oxides into zeolite frameworks not only enhances catalyst dispersion and pollutant adsorption but also facilitates improved charge carrier separation, thereby boosting photocatalytic activity. Additionally, the porous architecture of zeolites allows for the pre-concentration of pollutants near active sites especially beneficial for treating trace organic contaminants such as NMP.

Despite their photocatalytic advantages, powdered catalysts like Fe/Zeolite composites often present operational difficulties in water treatment, including poor mechanical stability, particle agglomeration, and challenges in post-treatment recovery. To address these limitations, immobilization techniques have been developed, particularly using natural polymers such as sodium alginate. Alginate, a biodegradable polysaccharide derived from brown algae, can form hydrogel beads via ionic crosslinking with divalent cations (e.g., Calcium ion (Ca^{2+})). These beads serve as environmentally friendly, mechanically stable matrices for encapsulating photocatalytic materials.

Embedding Fe/Zeolite catalysts within alginate matrices offers several key advantages. Firstly, alginate prevents nanoparticle aggregation and improves the mechanical stability and durability of the catalyst. Secondly, the beads' magnetic properties enable facile separation and recycling using

external magnetic fields, addressing the challenges of catalyst loss and secondary contamination [10]. Moreover, alginate's functional groups (e.g., hydroxyl and carboxyl) promote pollutant adsorption, enhancing the local concentration of target molecules near the catalytic sites and thereby improving degradation efficiency. Compared to conventional photocatalysts such as titanium dioxide and zinc oxide, alginate-encapsulated magnetic catalysts also offer improved environmental compatibility and lower toxicity [11].

However, despite promising advances in hybrid photocatalyst development, there is a lack of research specifically targeting the efficient degradation of highly recalcitrant contaminants like NMP under visible light using bead-immobilized systems. Furthermore, few studies combine photocatalytic performance with mechanical stability, magnetic recovery, and environmental safety in a single composite material. This gap hinders the practical scalability of such technologies for industrial wastewater treatment. The significance of this study lies in its attempt to integrate Fe_3O_4 , zeolite, and sodium alginate into a multifunctional bead-type photocatalyst that is magnetically recoverable, structurally stable, environmentally benign, and highly effective under visible light conditions. By doing so, this work contributes toward bridging the gap between laboratory-scale photocatalyst design and real-world environmental applications. Therefore, the objective of this study is to synthesize, characterize, and evaluate the photocatalytic performance of Fe/Zeolite bead catalysts encapsulated in sodium alginate for the degradation of NMP in aqueous systems. The catalyst's activity was investigated under varying operational parameters including pH, catalyst loading, and initial NMP concentration. The kinetic behavior was also examined to understand the interaction mechanisms between the catalyst and NMP.

2. Methodology

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 ($\text{C}_2\text{H}_5\text{OH}$) (> 99% purity) from HmbG Chemicals, calcium chloride (CaCl_2) was obtained from R&M Chemicals and sodium hydroxide (NaOH), hydrochloric acid (HCl), were obtained from QReCTM. Zeolite ($\text{C}_5\text{H}_8\text{O}_2$), epichlorohydrin and sodium alginate ($\text{NaC}_6\text{H}_7\text{O}_6$) were purchased from Sigma-Aldrich (Malaysia). N-methyl-2-pyrrolidone ($\text{C}_5\text{H}_9\text{NO}$) (>99% purity) was obtained from JAEWON Co. Ltd.

2.2 Preparation of Samples

The catalyst beads were prepared by dispersing the 15 wt% Fe/Zeolite powder into a 3% (w/w) sodium alginate solution prepared with deionized water, forming a homogeneous and viscous suspension. This mixture was introduced dropwise into a 0.5 M CaCl_2 coagulation bath using a syringe, leading to immediate bead formation via ionic gelation. The resulting wet beads were subjected to chemical crosslinking at ambient temperature. Initially, the beads were immersed in three consecutive ethanol–water baths (60% v/v ethanol, 400 mL each) for 2 h per bath to replace water with ethanol. They were then transferred to 400 mL of an ethanol–water solution (60% v/v) containing 6.109 g of epichlorohydrin. The pH of the mixture was adjusted to ~13 by the gradual addition of 1 mol/L NaOH , initiating the crosslinking reaction, which proceeded for 4 h. Post-reaction, the beads were rinsed in three separate distilled water baths (600 mL each) for 2 h to remove residual reactants. During the final rinse, concentrated HNO_3 (53.7% w/w) was added to neutralize the system, adjusting the pH to approximately 7.

2.3 Photocatalytic Performance Study

The photocatalytic performance of the Fe/Zeolite bead catalyst for the degradation of *N*-methyl-2-pyrrolidone (NMP) was assessed under visible light irradiation. The study investigated the influence of three key operational parameters: solution pH (3, 5, 7, 9, and 11), catalyst dosage (20, 40, 60, 80, and 100 g L⁻¹), and initial NMP concentration (20, 40, 60, 80, and 100 mg L⁻¹). All experiments were conducted over a 2 h period with continuous magnetic stirring to ensure uniform suspension. The pH of the reaction medium was adjusted as required using 0.1 M NaOH or 0.1 M HCl. For the standard degradation test, 2.0 g L⁻¹ (wet weight) of the Fe/Zeolite bead catalyst was added to 100 mL of a 20 mg L⁻¹ NMP solution in a glass beaker. The suspension was stirred throughout the experiment and irradiated using a fluorescent light source placed 15 cm above the liquid surface. At predetermined time intervals (0, 30, 60, 90, and 120 min), 1.5 mL aliquots were withdrawn and filtered for analysis. The residual concentration of NMP was determined using a Shimadzu UV-2600i UV-Vis spectrophotometer by monitoring absorbance at 197 nm.

3. Results and Discussion

3.1 Effect of pH on Photocatalytic Degradation of NMP

The photodegradation of *N*-methyl-2-pyrrolidone (NMP) in aqueous solution was carried out to evaluate the catalytic performance of the synthesized Fe/Zeolite bead catalysts. A series of batch experiments were conducted under ambient conditions, with a total contact time of 2 hours to account for both adsorption equilibrium and photocatalytic activity. The influence of initial solution pH, catalyst dosage, and starting NMP concentration was systematically examined.

As semiconductor-based catalysts exhibit amphoteric surface behavior, the surface charge and thus catalytic activity can vary significantly with pH. The percentage of photodegradation of NMP for 2 hours is listed in the Table 1 and depicted in Figure 1. In this study, the maximum NMP degradation efficiency was observed at pH 7, achieving a removal rate of 77.69% (Figure 1). This enhanced activity under neutral conditions is primarily attributed to the optimal formation of hydroxyl radicals ($\bullet\text{OH}$), which serve as the dominant reactive oxygen species (ROS) responsible for oxidizing and decomposing NMP molecules.

At alkaline conditions (pH 11), a noticeable decline in photocatalytic efficiency was observed. This reduction is likely due to the instability and rapid decomposition of hydrogen peroxide and hydroxyl radicals in basic environments, resulting in a decreased concentration of ROS available for effective degradation [12]. Conversely, under strongly acidic conditions (pH 3), the protonation of the catalyst surface by excess H⁺ ions imparts a positive charge, leading to electrostatic repulsion between the catalyst and the similarly polar NMP molecules. This repulsion inhibits effective adsorption and limits access to catalytic active sites, thereby reducing overall degradation efficiency [13].

These results underscore the significance of pH control in photocatalytic processes. Neutral pH conditions not only promote favorable catalyst-pollutant interactions but also facilitate efficient generation and utilization of oxidative species, making pH 7 the optimal condition for the effective degradation of NMP using Fe/Zeolite bead catalysts.

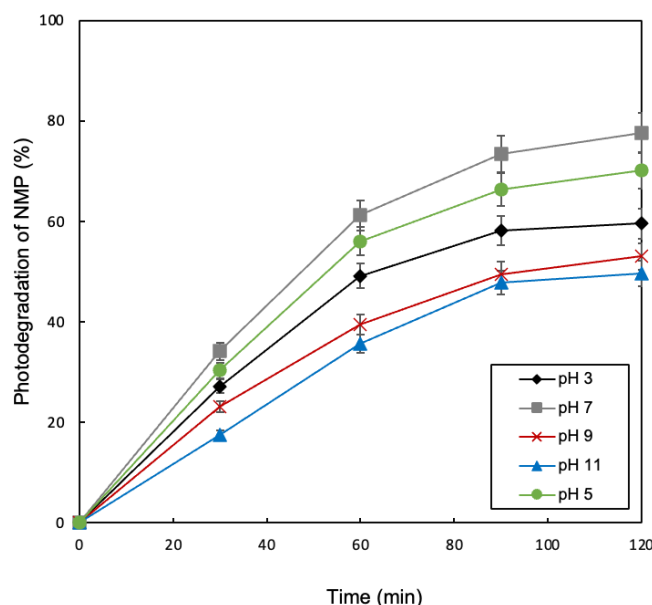


Fig. 1. Effect of pH solution for Fe/Zelite bead catalyst [W = 20.0 g L⁻¹; C = 20 mg L⁻¹; t = 2 h; T = 303.15 K]

Table 1

The percentage degradation of NMP at different pH levels

Time, t	pH 3	pH 5	pH 7	pH 9	pH 11
0	0	0	0	0	0
30	27.16	30.35	34.16	23.14	17.53
60	49.15	56.07	61.23	39.52	35.66
90	58.17	66.38	73.51	49.53	47.84
120	59.60	70.15	77.69	53.06	49.65

3.2 Effect of Catalyst Dosage on NMP Degradation

Optimizing catalyst dosage is a critical factor in enhancing photocatalytic efficiency. The percentage of photodegradation of NMP for 2 hours is listed in the Table 2. As illustrated in Figure 2, the degradation efficiency of NMP increased proportionally with the Fe/Zelite bead catalyst loading up to 40 g L⁻¹, beyond which further increases resulted in a negligible improvement. The highest observed degradation rate was 83.28%, achieved at this optimal dosage. Further increases in catalyst concentration led to a plateau in performance, indicating that the photocatalytic system had reached a saturation point or equilibrium.

This plateau effect is commonly associated with a reduction in photon utilization efficiency at higher catalyst concentrations. As previously reported by Ansari *et al.*, [14], excessive catalyst loading can induce light scattering and internal shielding effects, which hinder light penetration and reduce the number of photons reaching the active catalytic sites. Consequently, the effective surface area exposed to irradiation decreases, limiting ROS generation and suppressing further degradation despite the increased catalyst mass.

The initial improvement in photocatalytic activity with increased catalyst dosage is attributed to the greater availability of active sites on the Fe/Zelite bead surface. This enhances the generation of reactive oxygen species (ROS), such as hydroxyl radicals ($\bullet\text{OH}$) and superoxide anions ($\text{O}_2\bullet^-$), which are responsible for initiating the oxidative degradation of NMP. These observations are

consistent with previous studies, such as Ahmad *et al.*, [15], which demonstrated a similar trend of performance enhancement up to an optimal dosage, followed by a plateau or slight decline due to agglomeration or light attenuation effects.

Therefore, while higher catalyst dosages can initially boost degradation efficiency by increasing active surface area and ROS production, surpassing the optimal concentration may hinder light-driven reactions due to optical interference and particle crowding. These findings underscore the importance of dosage optimization in scaling photocatalytic systems for practical wastewater treatment applications.

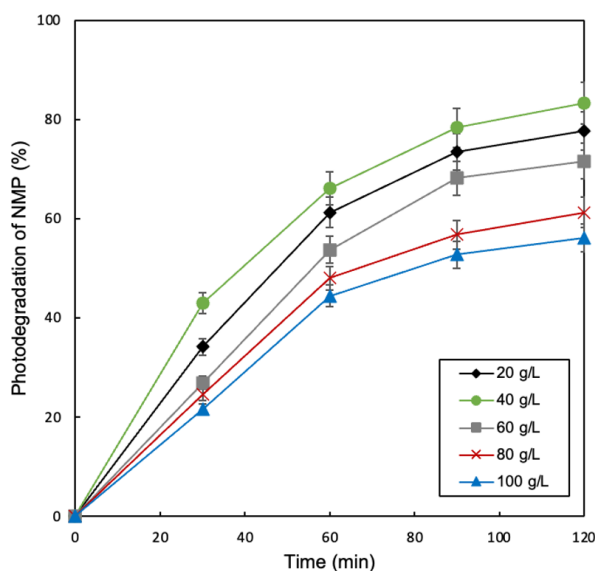


Fig. 2. Effect of catalyst dosage for Fe/Zelite bead catalyst [pH = 7; C = 20 mg L⁻¹; t = 2 h; T = 303.15 K]

Table 2

The percentage degradation of NMP at different catalyst dosage

Time, t	20 g L ⁻¹	40 g L ⁻¹	60 g L ⁻¹	80 g L ⁻¹	100 g L ⁻¹
0	0	0	0	0	0
30	34.16	42.95	26.92	24.54	21.62
60	61.23	66.16	53.77	48.04	44.43
90	73.51	78.31	68.22	56.76	52.72
120	77.69	83.28	71.57	61.26	56.18

3.3 Effect of Initial NMP Concentration on Photocatalytic Degradation

The efficiency of photocatalytic degradation is governed by both the adsorption behavior of the catalyst and the initial concentration of the target pollutant. In this study, the interaction between *N*-methyl-2-pyrrolidone (NMP) and the Fe/Zelite bead catalyst was investigated to evaluate how varying initial concentrations influence photocatalytic performance. The percentage of photodegradation of NMP for 2 hours is listed in the Table 3 and depicted in Figure 3. As shown in Figure 3, the highest degradation efficiency (83.28%) was achieved at an initial NMP concentration of 20 mg L⁻¹ after 2 hours of visible light irradiation.

However, as the initial NMP concentration increased beyond this point, a gradual decline in degradation efficiency was observed. This trend is primarily attributed to the finite number of active sites available on the catalyst surface. At elevated pollutant concentrations, an increased number of NMP molecules compete for these limited sites, reducing the likelihood of effective photon-catalyst

interaction and limiting the generation and activity of reactive oxygen species (ROS), particularly hydroxyl radicals ($\bullet\text{OH}$) [16]. Since ROS production is determined by a fixed quantity of catalyst and hydrogen peroxide in the system, the excess of pollutant molecules at higher concentrations surpasses the oxidative capacity of the system, thereby diminishing degradation efficiency. These results are consistent with the observations of Ahmad *et al.*, [15], who reported that excessive initial contaminant concentrations lead to surface saturation and a corresponding decline in photocatalytic activity due to insufficient ROS generation.

An additional advantage of the Fe/Zelite bead catalyst is its magnetic recoverability. Unlike conventional powdered photocatalysts that require filtration or centrifugation for separation, the magnetic nature of the Fe-based beads enables rapid and efficient recovery using an external magnetic field. This not only simplifies the post-treatment process but also minimizes catalyst loss, making the system more practical and sustainable for repeated use in wastewater treatment applications.

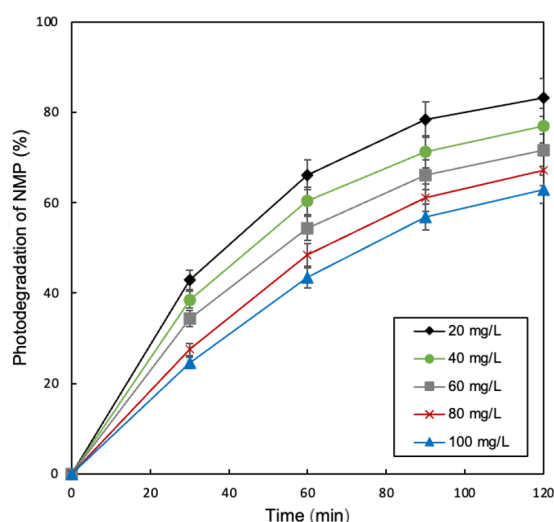


Fig. 3. Effect of initial concentration for Fe/Zelite bead catalyst [pH = 7; W = 40.0 g L⁻¹; t = 2 h; T = 303.15 K]

Table 3

The percentage degradation of NMP at different initial concentration

Time, t	20 mg L ⁻¹	40 mg L ⁻¹	60 mg L ⁻¹	80 mg L ⁻¹	100 mg L ⁻¹
0	0	0	0	0	0
30	42.95	38.56	34.40	27.54	24.66
60	66.16	60.36	54.33	48.45	43.39
90	78.31	71.25	66.16	61.10	56.90
120	83.28	77.01	71.55	67.09	62.92

3.4 Photocatalytic Kinetic and Catalyst Performance

The photocatalytic degradation kinetics of *N*-methyl-2-pyrrolidone (NMP) using Fe/Zelite bead catalysts were assessed using the pseudo-first-order Langmuir–Hinshelwood model. Key kinetic parameters including the apparent rate constant (K_{app}), coefficient of determination (R^2), initial reaction rate, and overall degradation efficiency were evaluated across a range of initial pollutant concentrations (20–100 mg L⁻¹) and Fe loadings (15 wt%).

In heterogeneous photocatalysis, the Langmuir–Hinshelwood (LH) kinetic model is widely recognized as the most suitable framework for describing reaction mechanisms and rate expressions

[17]. Photodegradation of organic pollutants, including phenol and *N*-methyl-2-pyrrolidone (NMP), typically follows pseudo-first-order kinetics, which is often validated by an adapted LH model that accounts for reactions occurring at the solid–liquid interface [18]. This implies that the active sites on the catalyst surface drive the degradation reactions in aqueous phase conditions.

The pseudo-first-order kinetic expression used to model the photodegradation rate is given by Eq. (1), which upon integration yields Eq. (2):

$$\ln C_t = -kt + \ln C_0 \quad (1)$$

$$\ln \left(\frac{C_0}{C_t} \right) = kt \quad (2)$$

where k is the pseudo first-order rate, C_0 and C_t are the concentrations of NMP at initial and time t , respectively.

The straight line drawn with $\ln (C_0/C_t)$ as a function of time in Figure 5a theoretically represents the photodegradation of NMP, accordingly with introduction of Fe/Zeolite bead catalysts using a pseudo first-order kinetic model. The gradient of the line depicts the apparent first-order rate constant (K_{app}).

The NMP photocatalytic activity may be an interface process that follows the LH model (Eq. (3-4)).

$$r_0 = -\frac{dC}{dt} = \frac{K_R K_{LH} C_0}{1 + K_{LH} C_0} = k_{app} C_0 \quad (3)$$

$$\frac{1}{k_{app}} = \frac{1}{K_R K_{LH}} + \frac{C_0}{K_R} \quad (4)$$

where K_R is the reaction rate constant and K_{LH} is the Langmuir-Hinshelwood adsorption equilibrium constant.

A linear plot was obtained by plotting $1/k_{app}$ as a function of C_0 (Figure 5b), indicating that the photodegradation by Fe/Zeolite bead catalyst is consistent with the Langmuir–Hinshelwood model. The reaction rate constant and the adsorption equilibrium constant were calculated to be $K_R = 0.776 \text{ mg L}^{-1} \text{ h}^{-1}$ and $K_{LH} = 0.026 \text{ L mg}^{-1}$, respectively. Since the value of K_R was larger than K_{LH} , these results suggest that the NMP adsorption was the controlling step of the process.

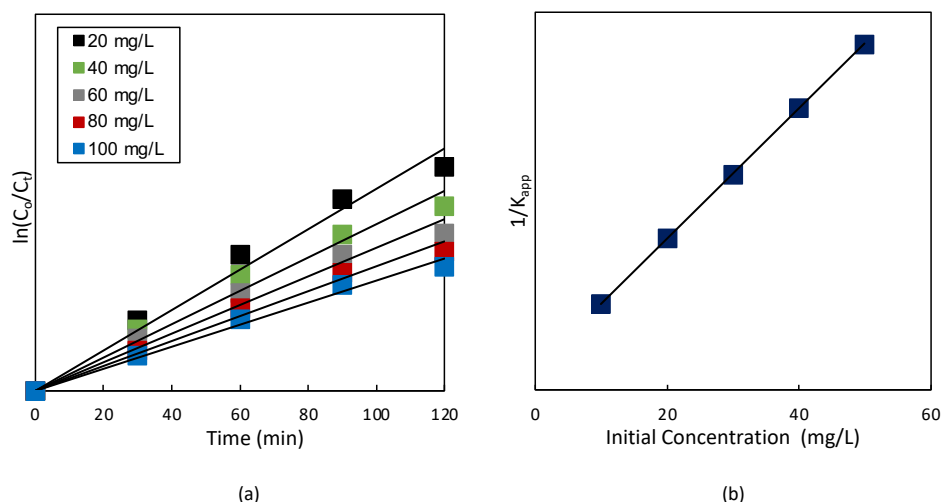


Fig. 5. (a) Photodegradation kinetics of NMP using Fe/Zelite bead catalyst at different NMP concentrations and (b) the relationship between $1/k_{app}$ and initial concentration of NMP

4. Conclusions

The synthesized Fe/Zelite bead catalyst exhibited outstanding photocatalytic performance for the degradation of *N*-methyl-2-pyrrolidone (NMP), achieving a maximum removal efficiency of 83.28% under optimized conditions namely, pH 7, a catalyst dosage of 40 g L⁻¹, and an initial NMP concentration of 20 mg L⁻¹ after 2 hours of visible light irradiation. Kinetic studies revealed pseudo-first-order behaviour, following the Langmuir–Hinshelwood model, with favourable rate constants and an effective balance between adsorption affinity and surface reactivity, with $K_R = 0.776 \text{ mg L}^{-1} \text{ h}^{-1}$ and $K_{LH} = 0.026 \text{ L mg}^{-1}$. This photocatalytic process effectively mineralizes NMP, yielding environmentally benign end-products such as CO₂ and H₂O. Structural modifications introduced during catalyst fabrication significantly enhanced the beads' pore architecture, mechanical robustness, chemical stability, hydrophilicity, and biocompatibility. These improvements collectively increased the density of active sites and optimized the surface morphology, thereby augmenting the catalyst's selectivity and interaction with target pollutants. Consequently, the overall photocatalytic efficiency was markedly improved. Collectively, these results demonstrate the potential of magnetic Fe/Zelite bead catalysts as a sustainable, efficient, and easily recoverable option for the remediation of recalcitrant organic contaminants in industrial wastewater treatment.

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