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Treatment of Organic Pollutant by Advanced Oxidation Processes

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Abstract:- The investigation involved the oxidation of urea (UR) in a batch reactor, employing Fenton's reagent. Various parameters, namely reaction time, pH level, ferrous ion dose, and hydrogen peroxide dose, were scrutinized. The reaction time spanned from 30 minutes to 3 hours, revealing a notably positive impact. An optimal pH of 3 was identified for the medium. The concentrations of ferrous ions ranged from 0.2 g/l to 0.53 g/l, with hydrogen peroxide levels ranging from 1 g/l to 2.65 g/l. The impact of hydrogen peroxide was notably significant at a ferrous ion concentration of 0.3 g/l and a pH of 3. Evaluating urea removal efficiency through chemical oxygen demand (COD) calculations showed a maximum efficiency of 86.8%, with a minimum ammonia yield of 6%. Overall, the outcomes underscored the efficacy of the Fenton process in urea treatment.

***Keywords**:- Urea, Fenton oxidation, Ammonia, treatment, Chemical oxygen demand.*

I. INTRODUCTION

The rapid growth of industrialization and urbanization has resulted in a significant increase in wastewater volume, which poses serious risks to both public health and the environment. Tackling this problem requires the creation of advanced wastewater treatment techniques that can transform organic wastewater into safe by-products. The key challenge is to develop comprehensive solutions that reduce the environmental footprint of organic wastewater. To address this, governments, environmental agencies, and health organizations have established regulations to oversee the disposal of organic wastes, ensuring a thorough process to avoid legal issues.[1–4].

Various technologies are employed to manage different types of organic hazardous effluent. Traditional methods, including chemical flocculation, adsorption, ion-exchange resin pre-treatment and bio-filtration, often have limited success in removing organic waste, making pre-treatment a crucial step. Industries utilize diverse techniques for disposing of specific types of organic waste, which are considered hazardous due to their non-biodegradability, chemical stability, and potential to bioaccumulate in the human body [5], [6].

Advanced oxidation processes (AOPs), which generate free radicals under specific conditions, are highly effective in eliminating various types of waste. These methods include the use of Fenton's reagent, photo-Fenton, UV/H_2O_2, TiO_2/ H_2O_2, ozone/ H_2O_2, ozone/ H_2O_2/UV, and ozone/TiO_2/ H_2O_2, all of which are employed to treat organic waste. Notably, the Fenton process has gained significant attention for its efficiency in treating organic wastewater, leading to widespread application.[7,8]. Introduced by H.J. Fenton in 1894, The Fenton reaction has several advantages, including ease of operation, a high removal rate, and the absence of hazardous sludge generation. It is particularly useful in pre-treatment, breaking down organic wastes into simpler compounds before biological treatment. This process involves the catalytic decomposition of hydrogen peroxide by ferrous ions, generating free radicals that degrade organic wastes into carbon dioxide, water, and inorganic salts. [9–11]. The key reactions of the Fenton process, along with supplementary ones, are detailed in the chemical equations by Lucas and Peres (2009).[12].

The Fenton process, although effective in oxidizing a wide range of organic compounds, encounters resistance with certain organic groups. Various studies have documented the application of Fenton’s reagent in treating different types of wastewater, including those from laboratories, landfills, pesticides, textiles, pulp mills, pharmaceuticals, green table olives. The perfect performance of this process depends on factors such as the concentration of hydrogen peroxide and Fe^{2+}, pH level, temperature, and the concentration of the organic matter. [4].

Urea, denoted by the chemical formula $CO(NH_2)_2$, plays a pivotal role in both agricultural and industrial contexts. Acting as a fertilizer, it serves as an easily accessible nitrogen source for plants, fostering robust growth and heightened crop yields. Its distinct molecular arrangement, characterized by a carbonyl group flanked by two amine groups, enables its effective solubility in water and absorption by plant roots. Beyond its agricultural applications, urea is employed in diverse industrial processes. It functions as a primary component in the production of plastics, adhesives, and resins. Furthermore, its elevated nitrogen content adds value to the manufacture of pharmaceuticals and dyes.

Urea (CH_4N_2O) holds significance as a crucial organic intermediate used in industries for the synthesis of plasticizers, insecticides, and dyes. Its properties, including toxicity, have garnered attention, and it is categorized as a weak carcinogen. Urea serves as an osmotic agent to manage conditions such as elevated intraocular pressure (glaucoma). Additionally, it is utilized as a diuretic and as a topical treatment for skin conditions like psoriasis and other dry, scaly disorders [13]. The production and use of UR contribute to environmental pollution, necessitating the protection of human health from its effects. Although research works indicates the use of biological treatment for UR removal, it proved insufficient.

Various technologies have been employed in several studies to remove UR. This study specifically utilized a catalyst for urea removal. Experiments were carried out in a batch reactor to find an effective way to remove UR from wastewater using α-Al_2O_3 and η-Al_2O_3 catalysts. The η-Al_2O_3 catalyst proved to be more efficient than α-Al_2O_3 and thermal hydrolysis, with its performance improving with higher temperatures, increased catalyst amounts, and longer reaction times. It reduced UR concentration to below 1 mg/L at 165°C after 150 minutes[14]. This study assessed granular activated carbon and granular activated alumina for UR removal from wastewater. The pH impact on UR removal was studied, finding points of zero charge at 8.8 for alumina and 7.1 for carbon. Maximum removal efficiencies were 24% for alumina and 31% for carbon at pH 9.0. Results confirmed that adsorption occurred, with increased efficiency at higher pH levels [15]. Another study evaluated the electrocoagulation technology for UR removal from synthetic and real waste water using zinc electrodes. Testing various conditions, it found a maximum removal rate of 66% at pH of 7.0 and magnesium chloride as the electrolyte. Results were compared to chemical coagulation using zinc sulfate [16]. The effectiveness of electrocoagulation (EC) for removing UR from wastewater was assessed using iron and copper electrodes. Results showed UR removal efficiencies of 40.15% at 6 V for copper and 51% at 12 V for iron after 90 minutes. Analysis revealed higher iron electrode consumption compared to copper[17].

This research focuses on investigating the impact of various operating conditions on the performance of Fenton's reagent in eliminating nitrogenous wastes and by-products. Key parameters under consideration include reaction time and the concentration of H_2O_2, Fe^{2+} concentration and pH level. The research primarily focuses on applying Fenton's reagent to degrade UR, a chemical compound characterized by the presence of two amino groups.

II. MATERIALS AND METHODS

UR (CH_4N_2O) is a nitrogen-containing chemical compound. Featuring amino groups, it possesses a density of 1.33 g/ml and a molecular weight of 60.06 g/mol. In contrast, ferrous sulfate heptahydrate presents itself as a powder. Sulfuric acid is a colorless, highly corrosive, and dangerous substance known for its role as a potent electrolyte. When introduced to a solution, it significantly lowers the pH, making the environment highly acidic. It has a density of 1.840 g/ml and a molecular weight of 98.08 g/mol. Sodium hydroxide, existing in powder form, is a chemical substance with a molecular weight of 40 g/mol.

➢ *Experimental Method*

In the laboratory, we conducted all experiments using one liter batch reactor placed on a hot plate magnetic stirrer. The procedure started with the preparation of a UR solution, which was then added to the reactor. Next, ferrous sulfate heptahydrate was introduced, and the pH of the solution was adjusted to 3 using sulfuric acid. Initially, the mixing speed was set to 40 rpm before hydrogen peroxide was added. The residence time varied between 30 minutes and 3 hr, with samples collected at 30-minute intervals. To assess the effectiveness of the Fenton process, we tested various operating conditions, determining the initial hydrogen peroxide concentration based on the initial COD levels. All experiments were performed at room temperature, and different instruments were used to measure COD removal efficiency, pH, and the concentrations of ammonia, nitrate, and nitrite ions.

III. RESULTS AND DISCUSSION

➢ *Effect of Reaction Time*

Residence time plays a crucial role in the Fenton reaction, as highlighted in previous studies [18,19]. This investigation focused on the impact of reaction duration on the conversion rate, spanning from 30 minutes to 3 hr in 30-minute intervals. The removal rate of COD exhibited a positive trajectory, escalating from 21% at 30 minutes to 76% at 3 hours. Consequently, the influence of reaction time on COD removal rate was affirmative. Figure 1 illustrates the correlation between reaction time and removal rate.

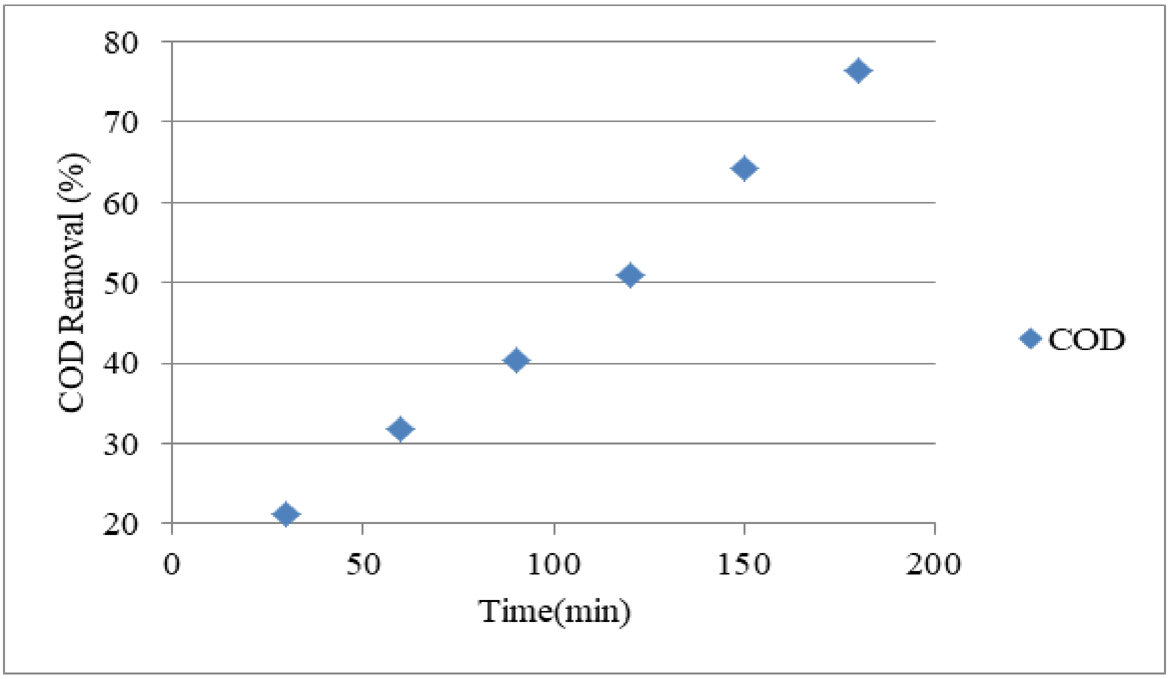

Fig. 1 Influence of Reaction Time on the Removal Rate of COD at pH of 3, Fe^{2+} 0.3 g/l and H_2O_2 of 1.6 g/l

Simultaneously, the study identified a significant effect of reaction time on ammonia yield. As time increased, the ammonia yield demonstrated a decrement, declining from 60% at 30 minutes to 12% at 3 hours. In essence, the prolonged reaction time contributed to a reduction in ammonia concentration. It is noteworthy that, despite the observed positive trends in this investigation regarding the impact of extended reaction time, some researchers have reported no noteworthy effect.

Figure 2 showcases the variation in ammonia yield at different reaction times, with ammonia identified as the primary product. The concentrations of other products remained minimal and fell below the detection limit of the photometer. Consequently, their concentrations were not examined. The Fenton process operated under specific conditions, including a pH of 3, laboratory temperature, Fe^{2+} dose of 0.3 g/l, and H_2O_2 concentration of 1.6 g/l. Altering these laboratory conditions could potentially optimize COD removal and minimize ammonia amount.

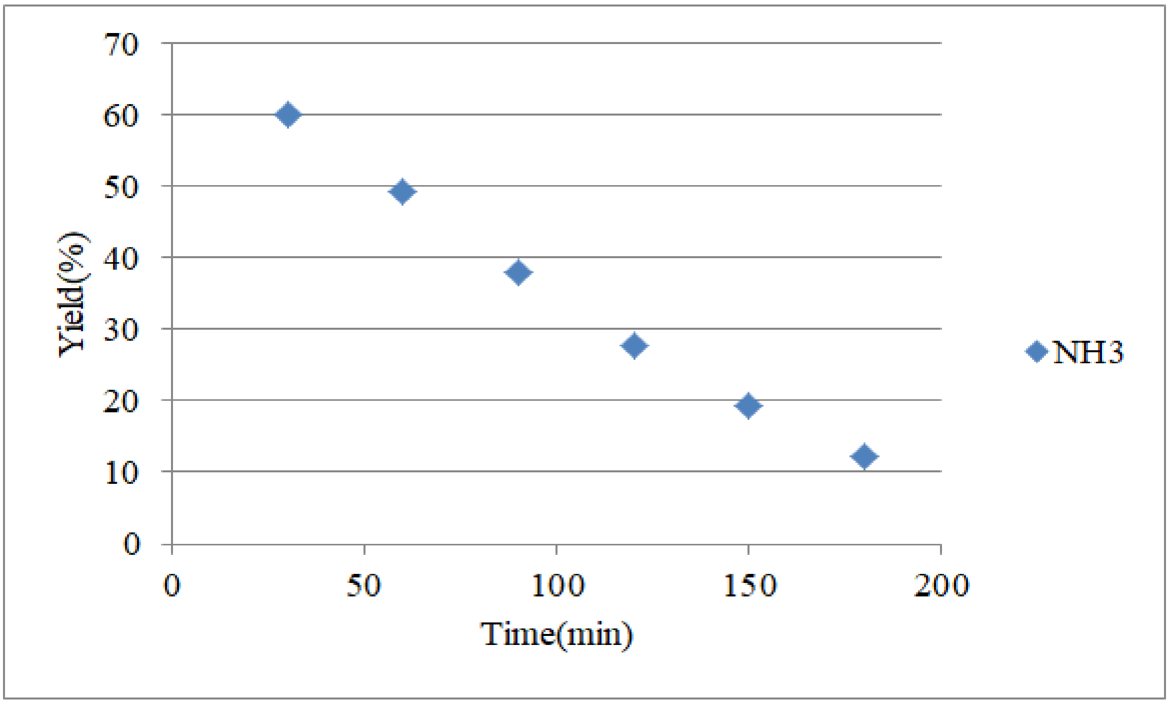

Fig. 2 Influence of Reaction Time on the Yield of Ammonia at pH of 3, Fe^{2+} of 0.3 g/l and H_2O_2 of 1.6 g/l

- *pH Influence*

The sensitivity of pH as a crucial operational parameter cannot be overstated. It exerts a significant impact on the activity of H_2O and Fe^{2+} ions. The specific pH range is pivotal for the effectiveness of free radical generation. Thus, maintaining an optimal pH is imperative for peak performance [19]. The Fenton process, influenced by pH, is vital for various applications [20]. The Fenton reaction happens in an acidic environment, and the density of radicals diminishes as pH rises. The Fenton oxidation process is inherently tied to pH, influencing the generation of radicals. The potency of HO• drops with increasing pH. While an acidic environment is favorable for Fenton process, an alkaline medium proves inefficient in enhancing the oxidation rate. The optimal pH range for Fenton process is established as 2.0–4.0 [21]. Deviating from this range, a pH below 2 reduces radical production, and a pH above 4.5 diminishes Fenton oxidation. Consequently, the removal rate of organic waste and by-products experiences a decline at extreme pH values, underscoring the necessity to maintain the pH at an optimum level for optimal free radical generation.

In the context of the literature reviewed, a pH range of 2 to 6 is recommended to ensure the effective removal of hazardous waste. However, in the current study, the pH range explored extended from 2 to 6. Notably, the efficiency of COD removal exhibited an increase at pH 3, followed by a decline at pH 4, 5 and 6. The minimum removal rate, at a reaction time of 3 hours and pH of 6, was recorded at 42.5%. Removal efficiency correlated negatively with increased acidity of the medium. Figure 1 shows the changes in COD removal rate in relation to increasing pH levels.

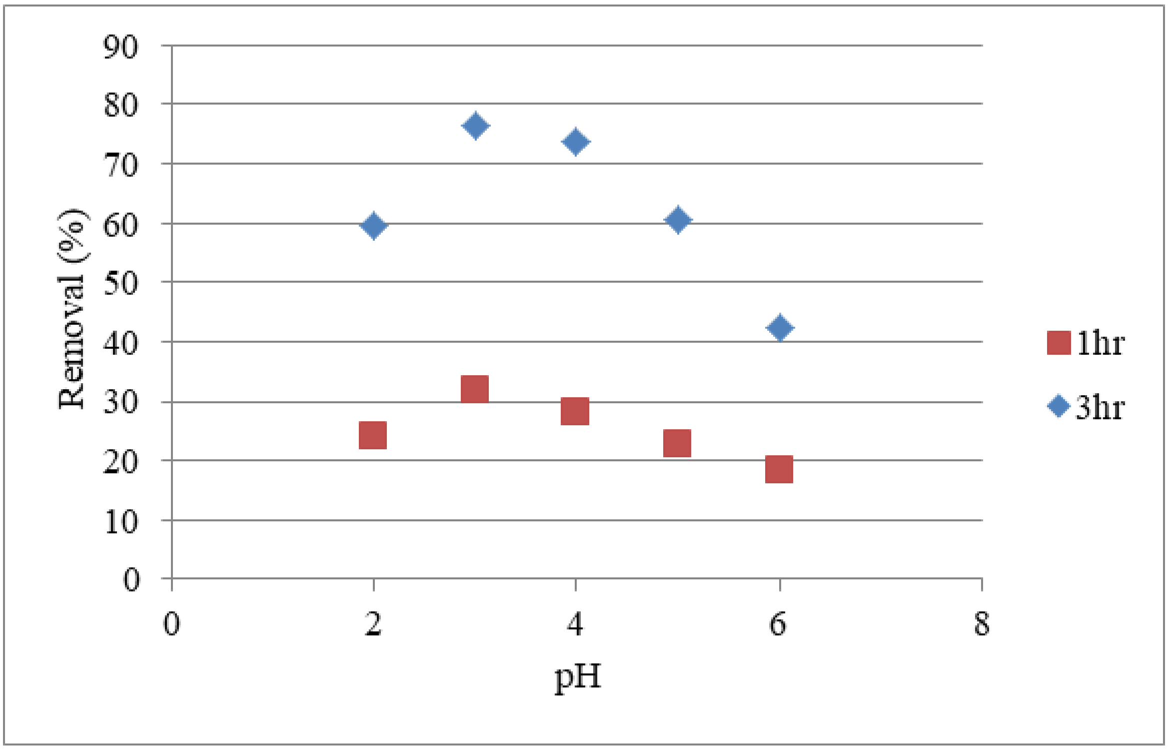

Fig. 3 Influence of pH on the Yield of Ammonia at Fe^{2+} of 0.3 g/l and H_2O_2 of 1.6 g/l

Examining the impact of pH on ammonia, its yield decreased at pH of 3 and then increased at pH of 4, 5 and 6, reaching a highest yield of 37%. Results affirmed a diminishing effect of pH at its highest value. Although other products such as nitrate ions and nitrite ions were examined, their low yields led to their exclusion from consideration. Figure 4 depicts the impact of pH level on the production of ammonia.

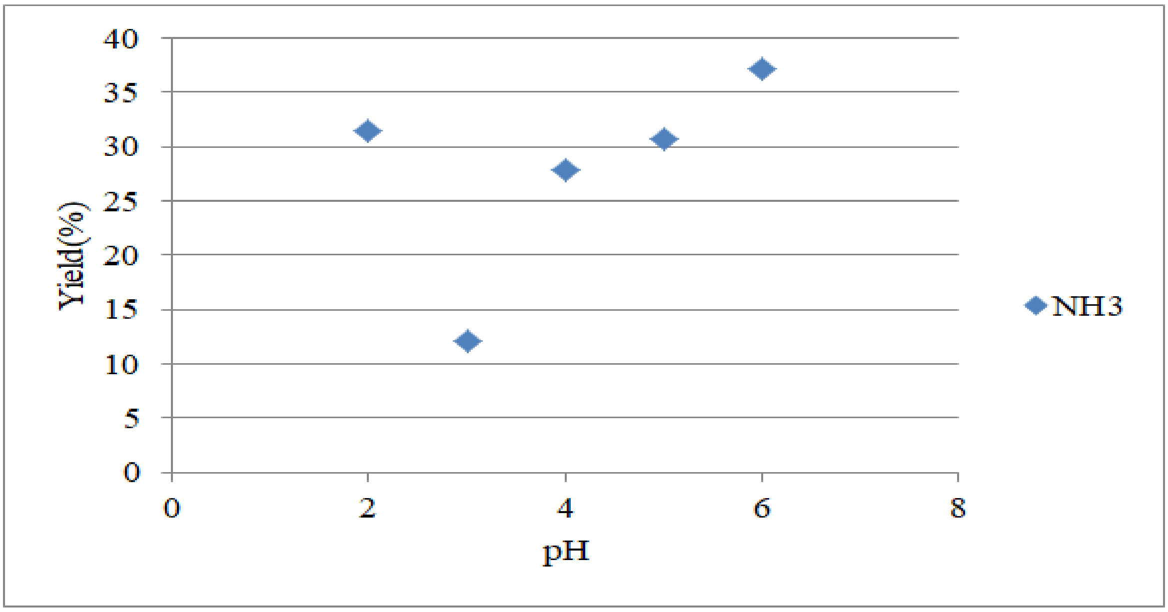

Fig. 3 Effect of pH on the Yield of Ammonia at Fe^{2+} of 0.3 g/l, Reaction Time of 3 hr and H_2O_2 of 1.6 g/l

- *Impact of Ferrous Ions (Fe^{2+})*

The concentration of Fe^{2+} plays an essential role in the Fenton reaction and the generation of free radicals [22]. This significance arises from the fact that an excessive amount of Fe^{2+} results in the consumption of radicals before the complete degradation of hazardous pollutants, while a low amount leads to a decrease in the production of (•OH). In the Fenton oxidation, Fe^{2+} catalytically decomposes H_2O_2; generating radicals that facilitate the breakdown of organic hazardous wastes into desired organic and inorganic compounds, as well as water.

The Fenton process exhibits both positive and negative aspects. Noteworthy advantages include high removal efficiency and straightforward operation, making it a valuable tool for improving the breakdown of organic hazardous wastes and potential application in wastewater treatment. However, drawbacks include sludge formation, the acidic nature of the reaction medium, and the need for an effluent reactor for neutralization [23].

In Figure 5, the influence of Fe^{2+} dose on the rate of COD removal is depicted. The findings highlight the essential effect of Fe^{2+} ions at the optimal dose. A minimum removal rate of 56% was observed at a Fe^{2+} dose of 0.2 g/l, reaction time of 3 hr, and pH of 3. The impact of Fe^{2+} ions diminished at concentrations of 0.2 g/l and 0.53 g/l, and the reaction time exhibited a significant positive effect on the conversion rate. Under optimal conditions and at 1 hr, the COD removal rate was 31.8%.

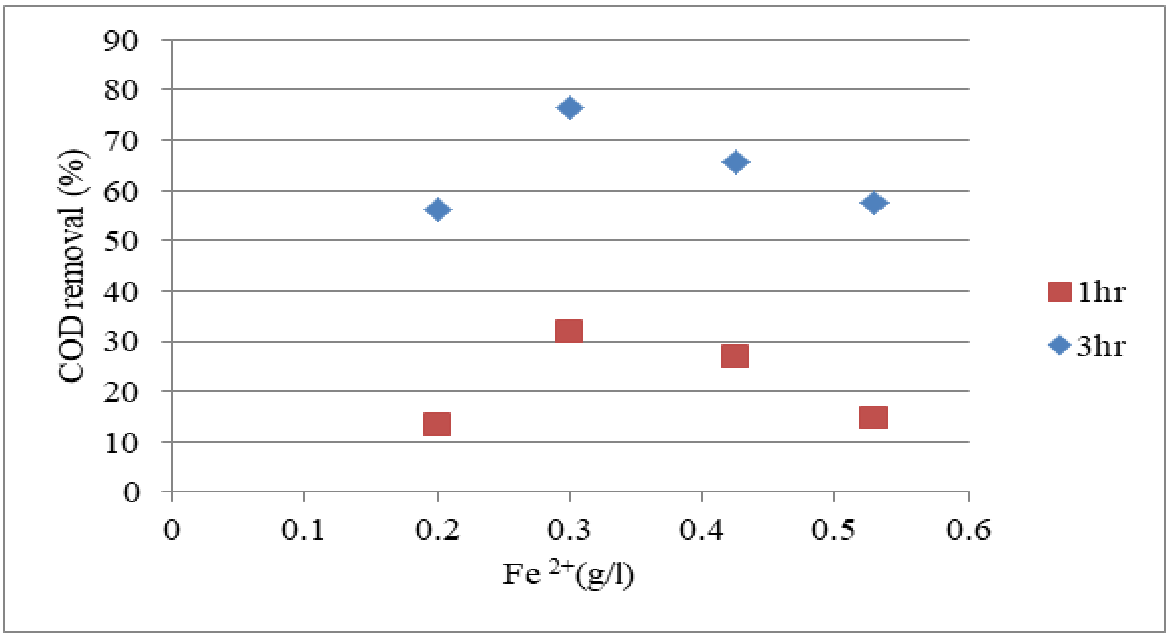

Fig.5 Influence of Different Doses of Fe^{2+} ions on the Removal Rate of COD at pH of 3, and H_2O_2 of 1.6 g/l

Figure 6 represents the effect of ferrous ions on ammonia yield. Various studies suggest a reduction in ammonia yield in the presence of abundant free radicals [24,25]. In this study, the Fenton process led to a decrease in ammonia yield at a ferrous ion concentration of 0.3 g/l. Fe^{2+} ions. Both low and high concentrations of Fe^{2+} ions had a little effect on an ammonia yield. The lowest recorded ammonia yield was 12% at a pH of 3, ferrous ion of 0.3 g/l and H_2O_2 concentration of 1.6 g/l. This maximum yield was achieved under the same pH and H_2O_2conditions at a Fe^{2+} concentration of 0.2 g/l and 0.265 g/l. Notably, even higher concentrations of ferrous ions did not lead to a significant reduction in ammonia yield, and no observations of nitrate and nitrite ion yields were noted.

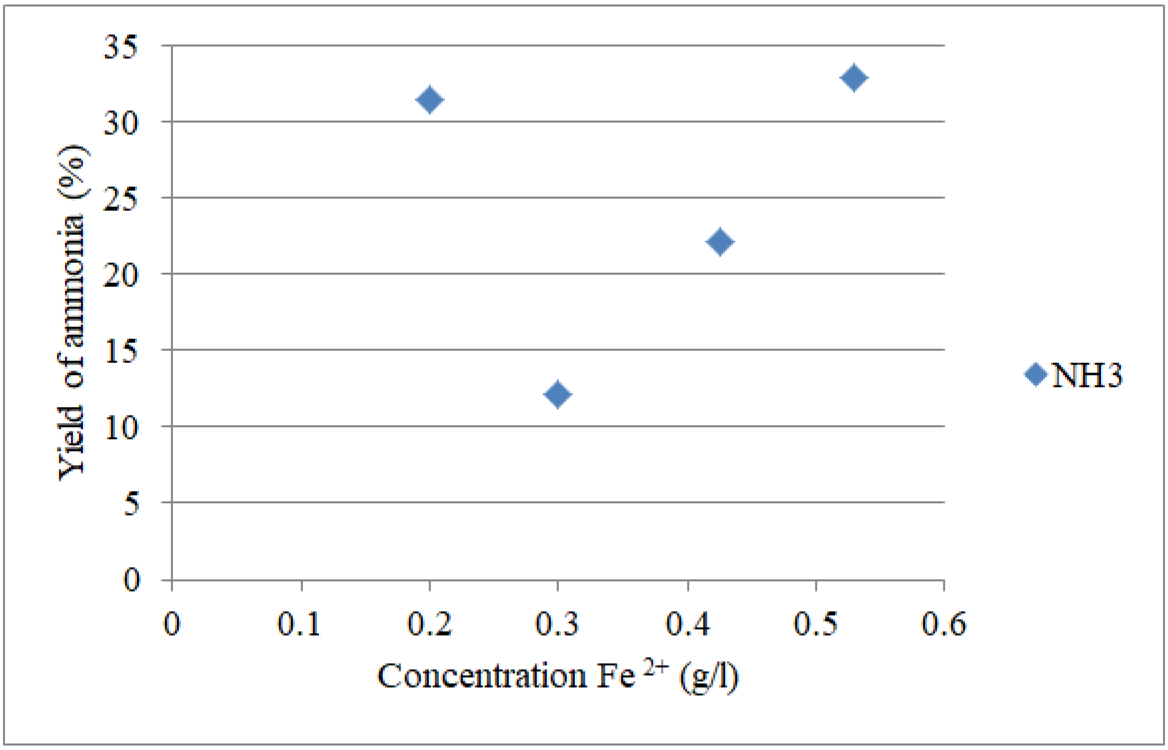

Fig.6 Influence of Different Doses of Fe^{2+} ions on the Yield of Ammonia at pH of 3, Residence Time of 3 hr and H_2O_2 of 1.6 g/l

- *Impact of Hydrogen Peroxide (H_2O_2)*

The impact of hydrogen peroxide (H_2O_2) is evident in the Fenton reaction, where it serves as the primary source of free radicals and functions as an oxidant. The presence of Fenton's reagent is crucial for the release of free radicals, and an acidic medium proves conducive to the decomposition of H_2O_2, leading to the generation of hydroxyl radicals (HO•) [25,26]. Particularly, the efficiency of the oxidant experiences fluctuations under varying pH conditions, being less effective at both low and high pH levels. The oxidation rate exhibits an upward trend with an increase in H_2O_2 concentration. The Fenton oxidation involves a sequence of intermediate reactions, necessitating the continuous addition of oxidant to eliminate organic contaminants.

Within the scope of this study, the impact of the oxidant on chemical oxygen demand removal efficiency and products was investigated. The influence of radicals on the conversion rate proved to be significant. Laboratory experiments, conducted at a pH of 3, Fe^{2+} dose of 0.3 g/l, and diverse reaction times, revealed that H_2O_2 dosage ranging from 1.06 g/l to 2.65 g/l positively influenced the process, albeit with diminishing returns at higher concentrations. The highest COD removal rate reached 86.8% at 2.65 g/l of the oxidant and a residence time of 3 hr. At 1 hour, a positive effect on COD removal efficiency was observed, with the highest removal efficiency recorded at 53% under identical conditions. An increase in reaction time from 1 hour to 3 hours significantly enhanced the conversion rate, leading to improved UR removal. Figure 7 visually illustrates the effect of the H_2O_2 on COD removal rate.

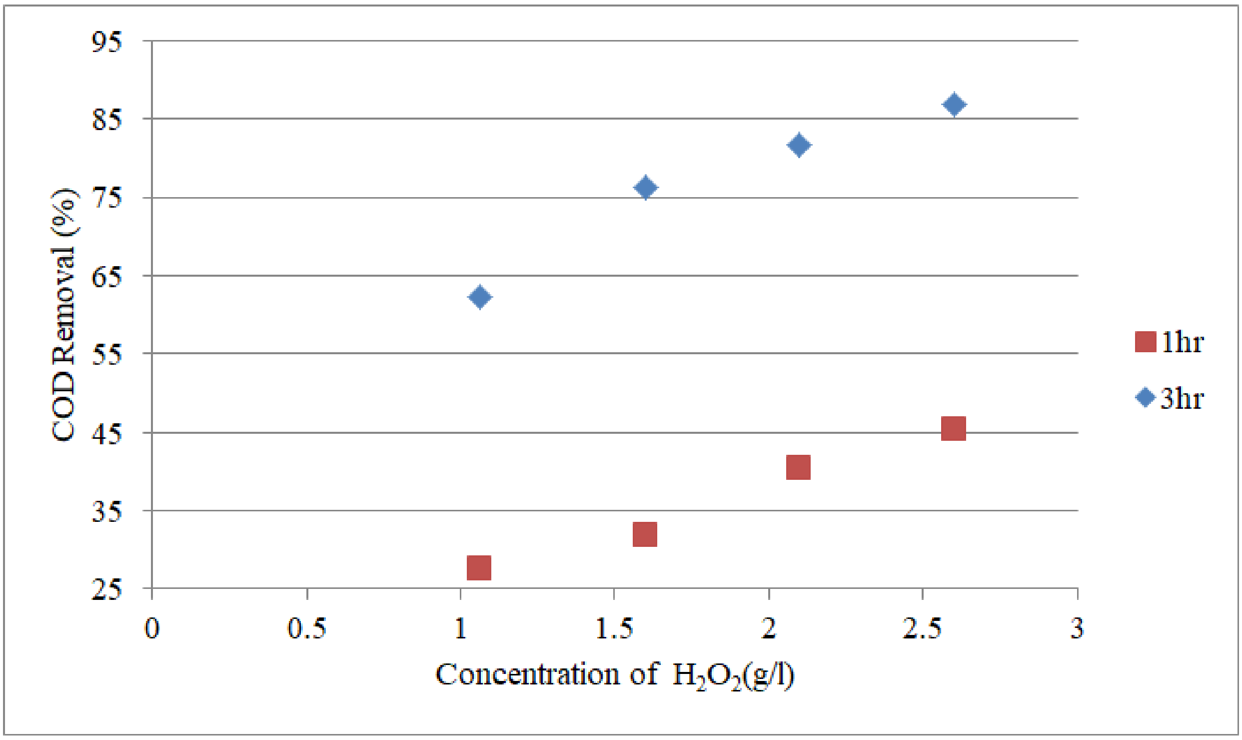

Fig. 7 Influence of Different Doses of H_2O_2 on the Removal of COD at pH of 3, and Fe^{2+} of 0.3 g/l

The primary by-product formed during UR oxidation was ammonia, and its production was heavily dependent on the type of oxidant used. Notably, the yield of ammonia diminished as the concentration of hydrogen peroxide increased. The heightened presence of hydrogen peroxide correlated with an augmented generation of hydroxyl radicals (•OH), contributing to increased ammonia removal.

Figure 8 represents the impact of various H_2O_2 concentrations on ammonia yields at a Fe^{2+} dose of 0.3 g/l and a pH of 3. Findings illustrate a decrease in ammonia yields with an increase in oxidant concentration, with the minimum yield recorded at 6% for 2.65 g/l of the oxidant after 3 hours. Despite investigating nitrite and nitrate ions, their negligible amounts led to the neglect of their results.

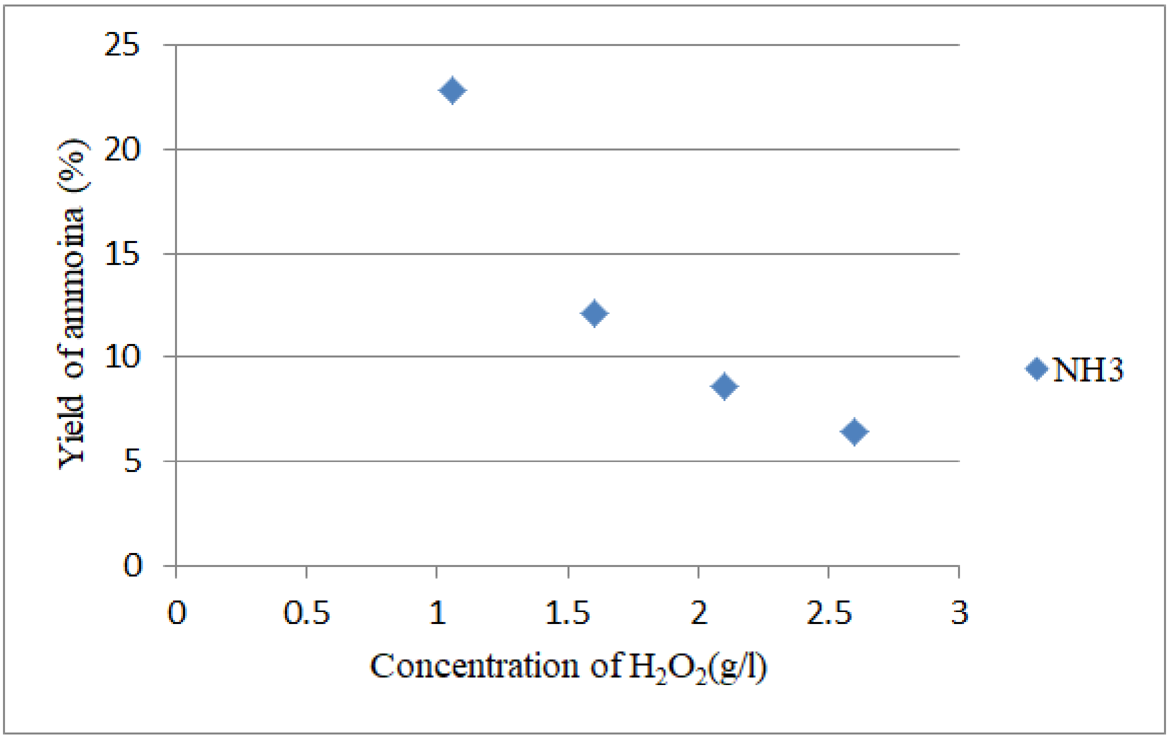

Fig.8 Impact of Different Doses of ferrous ions on the Yield of Ammonia at pH of 3, residence time of 3 hr and Fe^{2+} of 0.3 g/l

IV. CONCLUSIONS

The urea oxidation process was accomplished using Fenton's reagent. Improvements in COD removal efficiency were successfully achieved by implementing diverse experimental conditions. The results obtained were compelling, emphasizing the substantial impact of variables such as pH, Fe^{2+} concentration, H_2O_2 dosage, and reaction time. These elements played a pivotal role in shaping the observed outcomes. Fenton oxidation demonstrated the capability to transform organic hazardous compounds into simpler forms. At elevated oxidant concentration, a pH of 3, 3 hours of reaction time, and a Fe^{2+} concentration of 0.3 g/l, the maximum COD removal rate reached 86.8%, with a minimal ammonia yield of 6%. Remarkably, H_2O_2 concentrations exceeding 1.6 g/l did significantly enhance the COD removal rate. Additionally, the negligible concentrations of nitrite and nitrate ions were disregarded. In summary, the outcomes affirm the potential of Fenton's reagent for application in wastewater pre-treatment.

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