



Study the influence of experimental conditions on the removal of organic compound in the Fenton process

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Abstract

The oxidation of cyclohexylamine (CHA) was carried out in a batch reactor, using Fenton's reagent. The effects of the following parameters were investigated: residence time, pH level, ferrous ion concentration, and hydrogen peroxide concentration. Residence time ranged from 30 min to 2 h, and its effect was found to be significantly positive. A pH of 3 for the medium was observed to be optimum. Ferrous ion and hydrogen peroxide concentrations ranged from 0.1 g/l to 0.265 g/l and from 0.5 g/l to 1.3 g/l, respectively. The influence of hydrogen peroxide was significant at 0.16 g/l of ferrous ions and a pH of 3. CHA removal efficiency was measured by calculating chemical oxygen demand (COD). The maximum COD removal efficiency was 62%, and the minimum yield of ammonia was 14%. The yields of nitrite and nitrate were negligible. Overall, the results proved the effectiveness of the Fenton process for the treatment of CHA.

Keywords: Cyclohexylamine, Fenton, Chemical oxygen demand, ammonia, Removal

1. Introduction

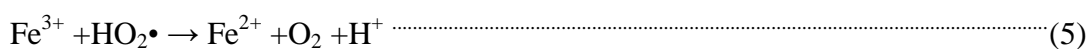
Industrialization and rapid urbanization are contributing to an increase in wastewater volume; this negatively affects public health and the environment. In several cases, wastewater treatment requires new methods. These methods must possess the ability to mineralize organic wastewater into non-harmful by-products. Organic wastewater is considered a real problem as it requires an integrated solution to reduce its impacts on the environment. The governments as well as environmental and health establishments have created laws and policies that regulate the disposal of organic wastes. Consequently, the process of disposal must undergo a series of steps to avoid legal issues (Bianco et al., 2011; Ebrahiem et al., 2017; Güneş et al., 2018; İlhan et al., 2019; Martini et al., 2021; Tony et al., 2016).

Numerous technologies are employed to deal with different organic wastewater streams. Traditional treatment methods such as bio-filtration, adsorption, chemical flocculation, and pre-treatment with ion-exchange resins, and according to the literature review, organic waste removal efficiency is limited and using these technologies as a first step without pre-treatment is not favorable. Industries employ different techniques to dispose of different types of organic wastes. Organic waste is considered dangerous for several reasons, which include, but are not limited to, its non-biodegradability, chemical inertness, and bioaccumulation in the human body (Ahmed et al., 2011; Papić et al., 2009; Singa et al., 2018).

Advanced oxidation processes have the efficiency to destroy various types of wastes. The main principle of these processes is the generation of free radicals under various reaction conditions. Several advanced oxidation processes are used to treat organic wastes, for example, Fenton's reagent ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$), photo-Fenton ($\text{H}_2\text{O}_2/\text{UV}/\text{Fe}^{2+}$), UV/ H_2O_2 ,

Ti₂O/H₂O₂, ozone/H₂O₂, ozone/H₂O₂/UV, ozone/TiO₂/H₂O₂. The Fenton process attracted a lot of attention in the last decade. Numerous researchers demonstrated the efficiency of the Fenton process in treating organic wastewater. In addition, the applications of the Fenton technology were applied extensively in the treatment of wastewater (Chu et al., 2012; Nidheesh, 2015; Nidheesh and Gandhimathi, 2012).

The Fenton reaction was described for the first time by H.J. Fenton in 1894 (Ameta et al., 2018). Fenton's reagent has many advantages such as its high removal efficiency, easy operation, and ability to facilitate non-hazardous sludge treatment and to be used in pre-treatment to destroy organic wastes and convert their compounds into simple products before injecting them in the biological treatment. These advantages make the Fenton technology an interesting one. In the process, the ferrous cation in Fenton's reagent decomposes hydrogen peroxide catalytically to generate free radicals that contribute to the destruction of organic wastes. The products of the Fenton process are carbon dioxide, water, and inorganic salts (da Silva et al., 2015; Mokhena et al., 2016; Rodrigues et al., 2009; Zazouli et al., 2012). The following chemical equations present the main reactions of the Fenton process as well as additional ones (Lucas and Peres, 2009):



The final products of the Fenton reaction are highly reactive and contribute to the continuation of the oxidation.

Although this process can oxidize different organic compounds, some organic groups are not easily removed through the Fenton process; these groups include chlorinated alkanes (e.g., tetrachloroethane, trichloroethane, and n-paraffins) and carboxylic acids (e.g., malonic, acetic, maleic, and oxalic acid). The applications of Fenton's reagent have been reported in the literature review, such as laboratory wastewater, landfill leachate, pesticides, textile wastewater, pulp mill effluents, pharmaceutical wastewater, green table olive wastewater, phenol degradation, MTBE degradation, and petroleum refinery. This demonstrates the efficiency of the Fenton process, which depends on operating conditions such as hydrogen peroxide concentration, Fe^{2+} concentration, pH level, temperature, and organic concentration (Bianco et al., 2011).

Cyclohexylamine ($\text{C}_6\text{H}_{13}\text{N}$) is considered an important organic intermediate that is employed as an antiseptic in various industries to make plasticizers, insecticides, and dyes (Shen et al., 2008). Its properties such as toxicity have received consideration. Moreover, it is classified as a weak carcinogen. The making and consumption of CHA contribute to water, air, and soil pollution. Therefore, human health and the environment must be protected from CHA contamination. According to literature review, biological treatment was used to remove this compound, but the removal was inadequate. Al-Kaabi and Al-Duri (Hadi Al-Kaabi and Al-Duri, 2022) investigated the influence of supercritical conditions on the removal of CHA in both the absence and presence of a co-fuel. Their laboratory results were significant.

The objective of this study is to examine how different operating conditions affect the efficiency of Fenton's reagent in the removal of nitrogen-containing wastes and by-products. Specifically, the parameters investigated include residence time, hydrogen peroxide

concentration, Fe^{2+} concentration, and pH level. The experimental focus of this research is the application of Fenton's reagent to address the degradation of CHA, a chemical substance that contains an amino group.

2. Materials and Methods

CHA ($\text{C}_6\text{H}_{13}\text{N}$) is a colorless chemical compound with a fishy odor at environment temperature. The compound contains an amino group. Its density and molecular weight are 0.867 g/ml and 99.17 g/mol, respectively. As for ferrous sulfate heptahydrate, it is a green powder, and its density and molecular weight are 1.898 g/ml and 278.01 g/mol, respectively. Sulfuric acid is a colorless hazardous substance and a strong electrolyte. Its density and molecular weight are 1.840 g/ml and 98.08 g/mol, respectively. Sodium hydroxide is a chemical substance and exists in the powder form. Its molecular weight is 40 g/mol.

2.1 Experimental Procedure

All experiments conducted in the laboratory utilized a 1 liter batch reactor placed on a hot plate magnetic stirrer. The CHA solution was prepared and introduced into the reactor, followed by the addition of ferrous sulfate heptahydrate. The solution's pH was adjusted to 3 using sulfuric acid. The mixing speed was set at 40 rpm at the start of the experiment. Subsequently, hydrogen peroxide was added to the solution. The residence time varied between 30 minutes and 2 hours, with samples collected every 30 minutes. To evaluate the effectiveness of the Fenton process, the impact of various operating conditions was examined. The initial concentration of hydrogen peroxide was determined based on the initial concentration of COD. All experiments were conducted at the laboratory's ambient temperature. Distinct instruments were employed to measure different parameters, including the removal efficiency of chemical oxygen demand (COD), pH level, and concentrations of ammonia, nitrate ion, and nitrite ion.

3. Results and Discussion

3.1 Effect of Residence Time

Residence time is an important parameter in the Fenton reaction (Ahmadian et al., 2013; Babuponnusami and Muthukumar, 2012; Mirzaei et al., 2017). In this study, the influence of reaction time on the rate of conversion was investigated. The time ranged from 30 min to 2 h with an increment of 30 min. The COD removal rate increased from 28% at 30 min to 60% at 2 h. Thus, the effect of residence time on COD removal efficiency was positive. Figure 1 shows the effect of residence time on removal efficiency.

In addition, the effect of reaction time on the yield of ammonia was found to be significant. The yield of ammonia decreased with increase in time. The yield of ammonia lowered from 80% at 30 min to 35.7% at 2 h. In other words, the increase in reaction time contributed to lowering ammonia concentration. However, although positive trends were observed in this study with regard to the effect of increasing reaction time, some studies reported no significant influence.

Figure 2 presents the yield of ammonia at various residence times. Ammonia was the main by-product. The concentrations of other by-products were small and less than the photometer's limit of detection; therefore, the photometer did not investigate their concentrations. The conditions of the Fenton process were pH of 3; laboratory temperature, ferrous ion concentration of 0.16 g/l, and oxidant concentration of 0.8 g/l. Varying some of these operating conditions could enhance the removal of COD and reduce the yield of ammonia.

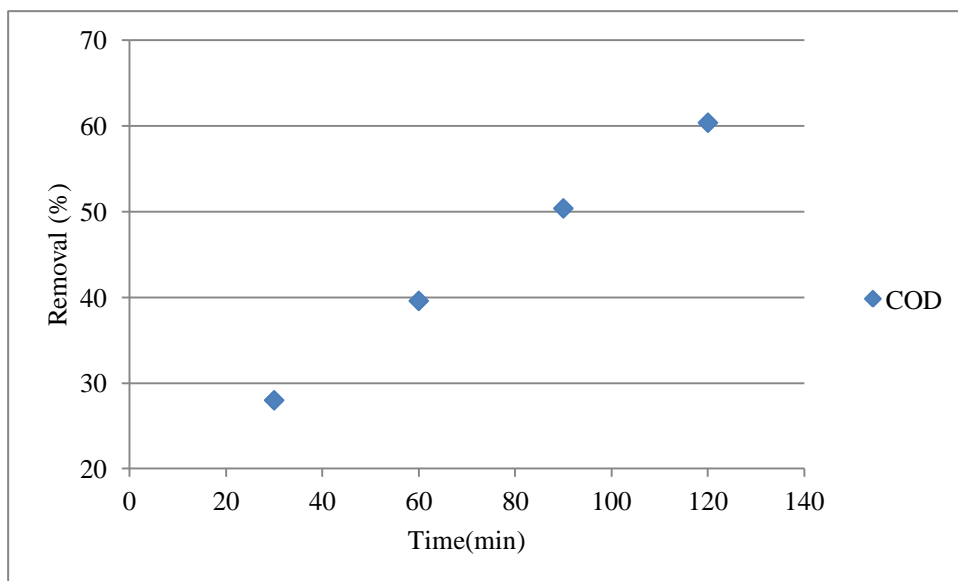


Fig 1: Effect of residence time on the removal efficiency of COD at pH of 3, H_2O_2 of 0.8 g/l, Fe^{2+} of 0.16 g/l and COD of 250 mg/l

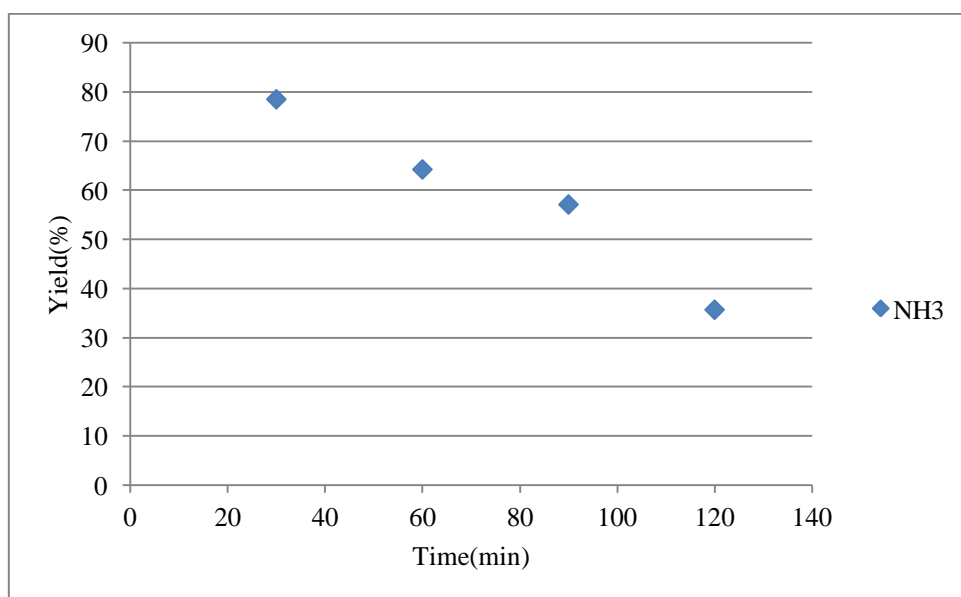


Fig 2: Effect of the residence time on the yield of ammonia at pH of 3, Fe^{2+} of 0.16 g/l, H_2O_2 of 0.8 g/l and COD of 250 mg/l

3.2 Effect of pH

The pH level holds significant importance as a highly sensitive operating parameter. It plays a crucial role in influencing the activity of ferrous ions and hydrogen peroxide. The effectiveness of free radical generation also relies heavily on the specific pH range. Thus, maintaining the appropriate pH is essential for optimal performance (Alalm et al., 2015; Mirzaei et al., 2017). Consequently, the optimum value of pH is essential in the Fenton process (Mohajeri et al., 2010). The Fenton reaction takes place in an acidic environment. The concentration of free radicals drops with increase in pH level. The process of Fenton oxidation is based on the value of pH due to the essential influence of the generation of free radicals. The effect of HO· decreases when pH increases. The acidic medium is favored for the Fenton oxidation, while the alkaline medium is not efficient for the enhancement of oxidation rate. The suitable pH range for the Fenton oxidation is 2.0–4.0 (Aramyan, 2017; Tercero et al., 2020). A pH of less than 2 leads to reduction in free radical generation, and a pH of higher than 4.5 lowers the Fenton oxidation. Consequently, organic waste and by-product removal rate will be less at higher and lower values of pH, and the conversion rate must be kept at the optimum value of pH to improve the generation of free radicals.

According to the literature review, pH should range from 2 to 4 to ensure the removal of high amount of organic waste. In this study, pH ranged from 2 to 5. COD removal efficiency increased at pH 3 and then decreased at pH 4 and 5. The minimum removal efficiency was 44.8% at residence time of 2 h and pH of 2. Removal efficiency decreased with increase in the acidic nature of the medium. Figure 3 shows the variations in COD removal efficiency with respect to increase in pH.

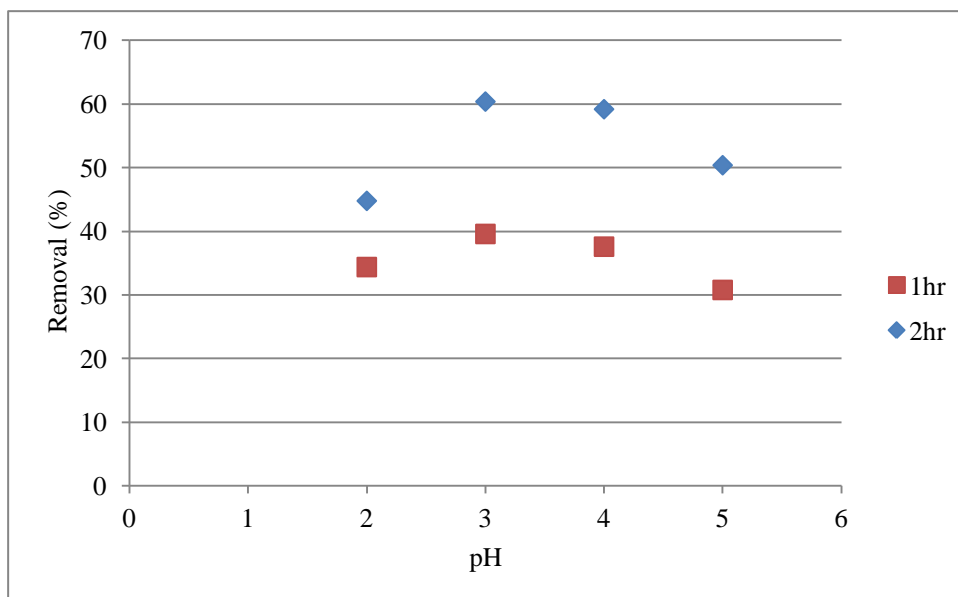


Fig 3: Effect of removal efficiency of COD at various pH, various residence time, 250 mg/l of COD, 0.8 g/l H_2O_2 and 0.16 g/l Fe^{2+}

Regarding the influence of pH on ammonia, the yield of ammonia dropped at pH of 3 and then increased at pH of 4 and 5. The maximum yield was 92.8%. The results confirmed the effect of pH decreased at the maximum value of pH. In addition, the other by-products such as nitrate and nitrite were also investigated, but the findings were neglected due to their low yields. Figure 4 shows the influence of pH value on the yield of ammonia.

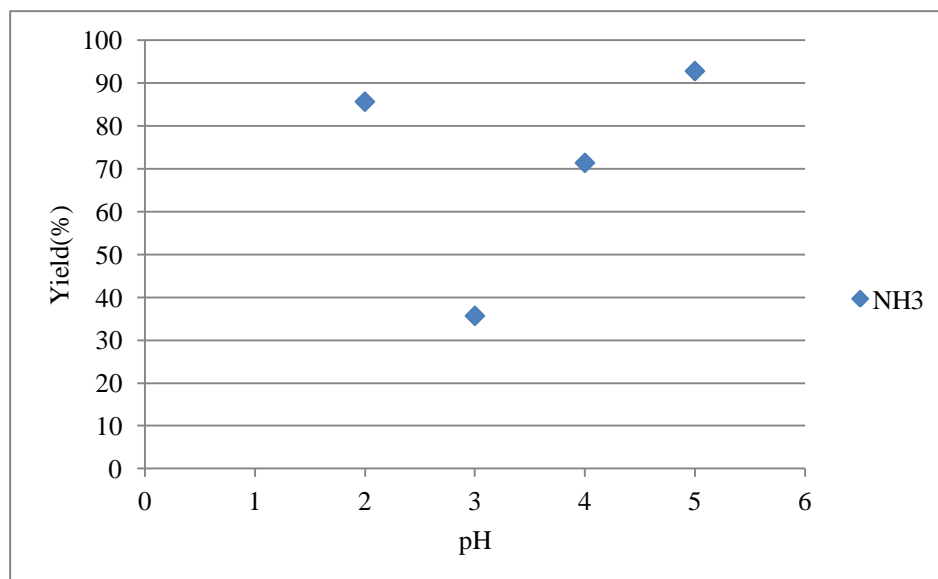


Fig 4: Effect of pH on the yield of ammonia at 2 hr, Fe^{+2} of 0.16 g/l, H_2O_2 of 0.8 g/l and COD of 250 mg/l

3.3 Effect of Ferrous Ions (Fe^{2+})

Fe^{2+} concentration is considered an important parameter for Fenton reaction and free radical generation (Miklos et al., 2018). This is because a high dosage of Fe^{2+} leads to free radical consumption before the complete destruction of organic pollutants, and a low dosage of Fe^{2+} causes a reduction in the generation of ($\cdot\text{OH}$). Fe^{2+} in Fenton's reagent decomposes hydrogen peroxide catalytically to generate free radicals that help destroy organic wastes and mineralize them to wanted organic and inorganic compounds and water.

The Fenton process has both advantages and disadvantages. The main advantages of this technology include high removal efficiency and easy operation. It is considered significant in enhancing the biodegradability of organic wastes, and it has the potential to be used for wastewater treatment. The disadvantages include the formulation of sludge, the acidic medium reaction, and the requirement of an effluent reactor for neutralization (Domingues et al., 2018).

Figure 5 shows the effect of Fe^{2+} concentration on COD removal efficiency. The results demonstrate the positive influence of ferrous ions at the optimum concentration. The minimum COD removal rate was found to be 44% at Fe^{2+} concentration of 0.1 g/l, 2 h, and pH of 3. The influence of ferrous ions decreased at 0.1 g/l and 0.265 g/l. In addition, the positive impact of residence time was significant on the rate of conversion. COD removal rate under optimum conditions and at 1 h was 39.6%.

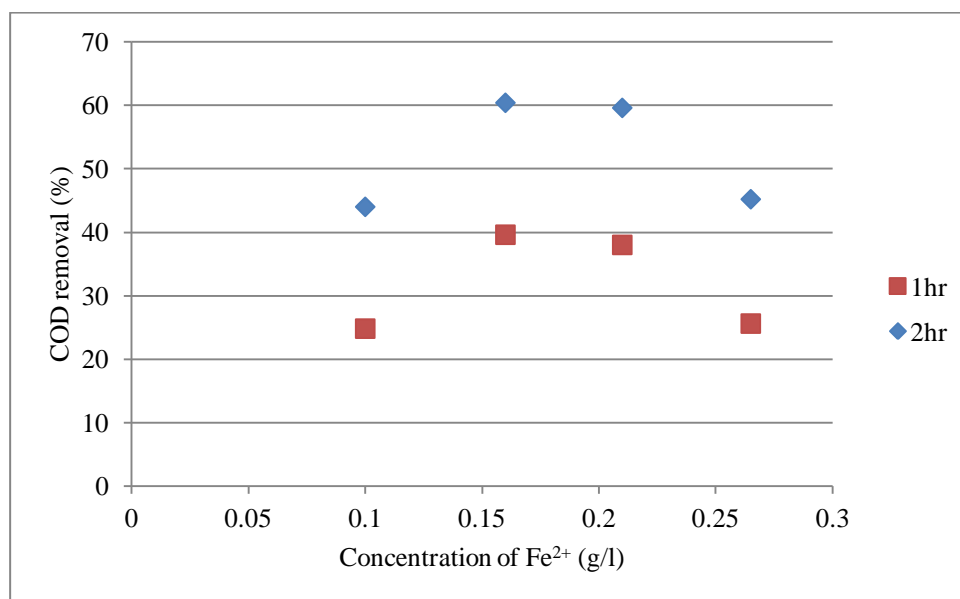


Fig 5: Effect of ferrous ion on the removal efficiency of COD at various residence times, H_2O_2 of 0.8 g/l and pH of 3.

Figure 6 illustrates the effect of ferrous ions on the yield of ammonia. According to various studies, the yield of ammonia decreased in the abundant presence of free radicals (Das and Adak, 2022; Pani et al., 2020). In this study, during the Fenton process, the yield of ammonia dropped at 0.16 g/l of the ferrous ion. The low and high concentrations of ferrous ions had a low influence on ammonia. The maximum yield of ammonia was recorded to be 78.5% at pH of 3 and H_2O_2 concentration of 0.8 g/l. The highest yield was recorded at the same conditions of pH and H_2O_2 but with Fe^{2+} concentration of 0.1 g/l and 0.265 g/l.

However, higher concentrations of ferrous ions did not lead to significant decrease in the yield of ammonia. The yields of nitrate and nitrite ions were not observed.

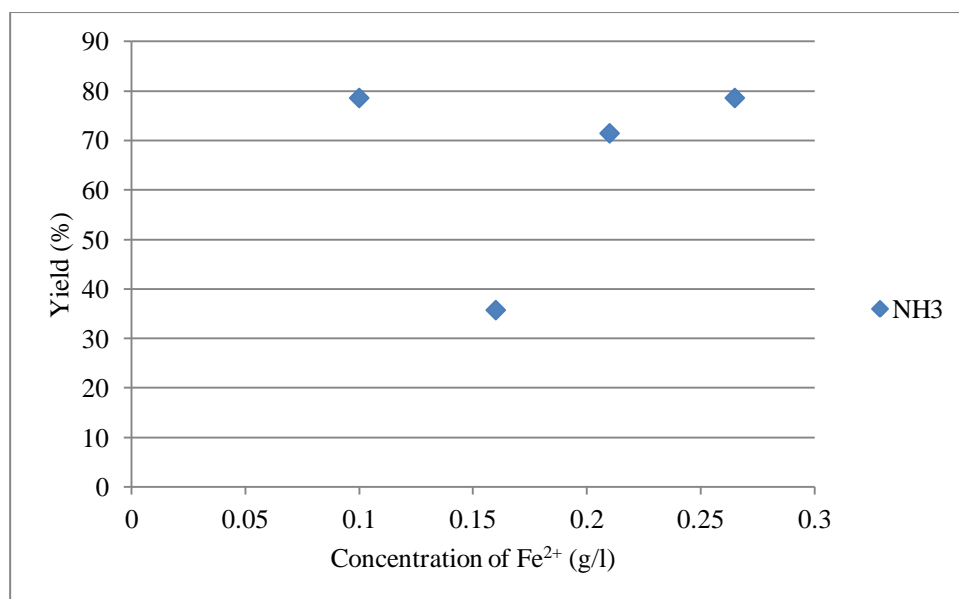


Fig 6: Effect of ferrous ion on the yield of ammonia at 2 hr, H₂O₂ of 0.8 g/l and pH of 3

3.4 Effect of Hydrogen Peroxide (H₂O₂)

In the Fenton reaction, the main source for free radicals is hydrogen peroxide, which acts as an oxidant. For the oxidant to help release free radicals, Fenton's reagent is essential (Naseem et al., 2019; Pani et al., 2020). The acidic medium is a suitable environment for the decomposition of H₂O₂ to generate HO. The efficiency of oxidant decreases at low pH and high pH. The rate of oxidation tends to enhance with increase in H₂O₂ concentration. A series of intermediate reactions occur during the Fenton oxidation; therefore, the oxidant must be continuously added to the reaction to destroy organic contaminants.

In this research, the effect of oxidant on COD and by-product removal efficiency was examined. The influence of free radicals on the rate of conversion was found to be significant. Laboratory experiments were carried out at pH of 3, Fe²⁺ concentration of 0.16 g/l, and various residence times, and the H₂O₂ dosage ranged from 0.5 g/l to 1.3 g/l; an

increase in the concentration of the oxidant had a positive influence, but this effect was not noteworthy with high concentration. The maximum COD removal rate was 62% at 1.3 g/l of the oxidant and 2 h. At the residence time of 1 h, the effect of oxidant on COD removal efficiency was positive. At 1 h, the highest removal efficiency was 53% under the same conditions. The enhancement of the conversion rate was found to be significant when the time of reaction was increased from 1 h to 2 h; hence, CHA removal rate improves with an increase in the residence time. Figure 7 presents the impact of the oxidant on COD removal efficiency.

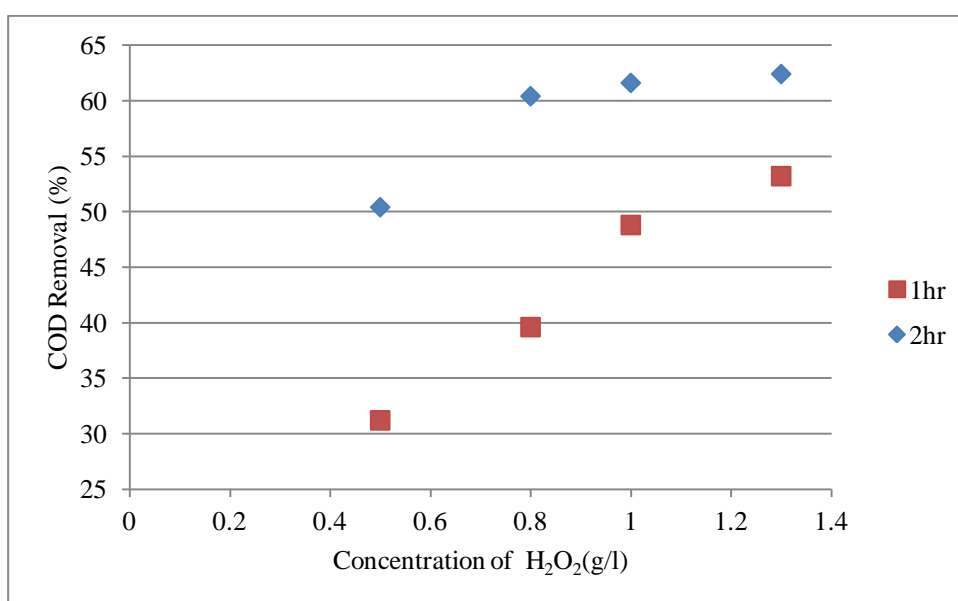


Fig 7: Effect of oxidant on the removal efficiency of COD at various residence times, pH of 3 and COD of 250 mg/l

The main by-product of CHA oxidation was ammonia. The oxidant had a significant influence on ammonia. The yield of ammonia decreased with increase in hydrogen peroxide. The increase in hydrogen peroxide contributed to increase in the generation of free radicals (OH). The removal of ammonia increased in the presence of (OH), while the same amount of hydrogen peroxide did not have an important influence on COD removal at 1.3 g/l. In other words, the change of oxidant dosage from 0.5 g/l to 1.3 g/l did not have a remarkable

influence on COD removal. Figure 8 shows the effect of various concentrations of oxidant on the yields of ammonia at Fe^{2+} concentration of 0.16 g/l, and pH of 3. The results demonstrate that the yields of ammonia decreased with increase in the oxidant at Fe^{2+} concentration of 0.16 g/l and 2 h. The minimum yield was 14% at 1.3 g/l of the oxidant. The nitrite and nitrate ions were investigated, but the results were neglected due to their negligible amounts.

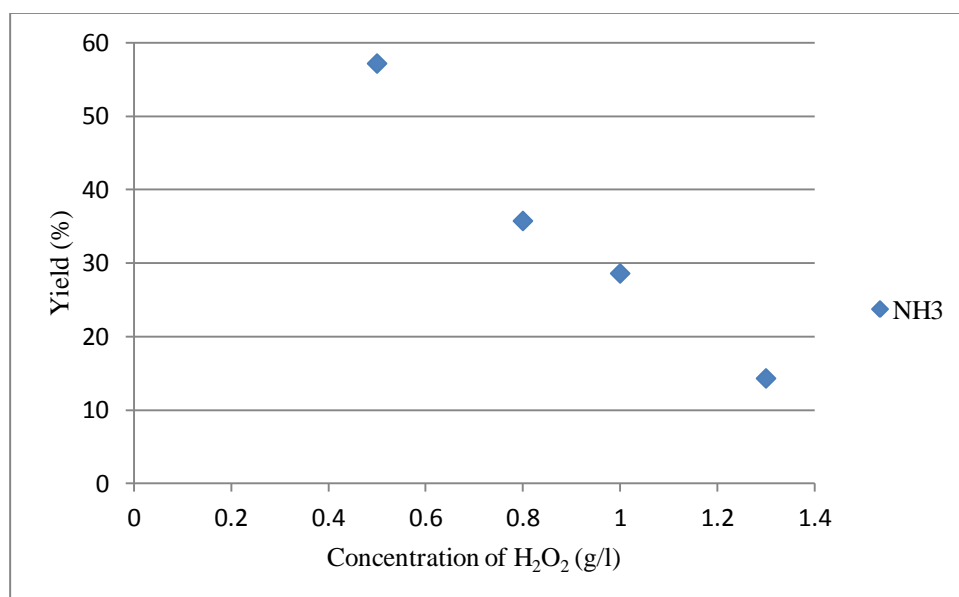


Fig 8: Effect of oxidant on the yield of ammonia at 2 hr, Fe^{2+} of 0.16 g/l, pH of 3, and COD of 250 mg/l

4. Conclusions

The oxidation of CHA could be achieved with Fenton's reagent. COD removal efficiency was successfully enhanced through various experimental conditions. The study yielded compelling results, underscoring the considerable influence of pH, ferrous ion, and hydrogen peroxide concentrations, along with residence time. These factors were found to play a crucial role in the observed outcomes. The Fenton oxidation has the power to mineralize organic compounds to simple organics. The maximum COD removal efficiency and the minimum ammonia yield were 62% and 14%, respectively, at a high concentration of

the oxidant, pH of 3, 2 h, and Fe²⁺ concentration of 0.16 g/l. However, hydrogen peroxide concentration beyond 0.8 g/l was not significant in enhancing COD removal rate. Further, the yields of nitrite and nitrate ions were neglected due to their very low concentrations. Overall, the findings confirm the potential of Fenton's reagent to be used in wastewater pre-treatment.

Conflict of Interest: no.

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