



Simultaneous removal of COD and Ammoniacal Nitrogen from dye intermediate manufacturing Industrial Wastewater using Fenton oxidation method

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Abstract

The untreated wastewater from the dye industry having high ammoniacal nitrogen and chemical oxygen demand (COD) significantly pollutes the receiving water, such as a river, having major ill implications on the aquatic environment. The present study is undertaken to address this problem. It investigates the effectiveness of Fenton reagent in simultaneous treatment (removal) of ammoniacal nitrogen and COD present in the wastewater by varying the parameters like pH, concentration of Fe^{2+} and H_2O_2 and their molar ratio. The reaction time for all the experiments was kept 60 min. The experimental analysis in terms of calculating the residual % of ammoniacal nitrogen and COD revealed that the maximum reduction occurred at pH 3. At this pH, further set of experiments were performed by varying the molar doses of $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ as 1:1, 3:3 and 5:5, and the results reveal that the maximum reduction in both ammoniacal nitrogen and COD happens at molar dose of 3:3. To understand the effect of the concentration of Fe^{2+} and H_2O_2 on the respective removal efficiency, set of experiments were performed by taking the molar ratio of $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ as 2:1, 3:1, 5:1, 1:2, 1:3 and 1:5. The experimental analysis reveals that the maximum reduction happened at molar ratio 1:3. Fenton process can mineralize the pollutants up to 75.8% and 78.6% for ammoniacal nitrogen and COD removal. The findings of the study would be of great use for planning and optimizing the wastewater treatment facility for the dye intermediate industry.

Keywords Ammoniacal nitrogen · COD · Dye intermediate wastewater · Fenton process · Molar dosage · pH

Introduction

In India, the production of dyestuff and pigments is more than 80,000 tonnes per annum (Nidheesh et al. 2018). Textile industries are the main users of the dyestuff. Gujarat is textile hub of India with more than 1500 textile industries (Bhatia et al. 2018; Paździor et al. 2018). These industries contribute more than 40% of the Indian dyestuff production and more than 60% of Indian exports of dyestuff. The typical wastewater generation from these industries is between

125 and 150 L per kg of finished dye products (Rathi 2003; Markandeya et al. 2018).

Industrial wastewater is generally characterized by extensive contamination with organic compounds, halogenated aliphatic and aromatic compounds, agrochemicals, high concentrations of heavy metals, sulphur and nitrogen-containing compounds, high COD, TDS and TSS (Bolong et al. 2009; Dükkanci et al. 2014; Gonzalez-Merchan et al. 2016). The wastewaters from the dye intermediate industry are highly acidic and contain nitrogenous pollutants (various compounds of nitrate, nitrite and ammoniacal nitrogen), carbonaceous pollutants and other toxic compounds which are hazardous to the human health and to the aquatic environment (Bhatia et al. 2018; Markandeya et al. 2018; Kaur et al. 2019). Nitrogenous and carbonaceous compounds present in the wastewater not only deplete the DO of the receiving water but also cause eutrophication (Camargo and Alonso 2006; Huang et al. 2014). Eutrophication favours the growth of toxin-producing cyanobacteria. Exposure to such toxins causes high health risks like liver damage, skin irritation

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and liver cancer (Akpors and Muchie 2011). Ammoniacal nitrogen when converted to nitrate nitrogen causes high nitrogenous oxygen demand (NOD) which leads to methemoglobinemia. Ammoniacal nitrogen also affects the chlorine disinfection efficiency (Oturam and Aaron 2014; Karri et al. 2018).

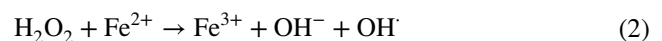
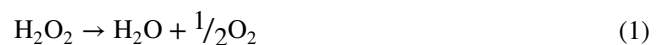
Many technologies are available for the treatment of some primary pollutants and their derivatives present in the wastewater from the dye industry; however, the issue of removal of nitrogen from wastewater is now a matter of immediate concern. Degradation/removal of ammoniacal nitrogen includes physical, chemical and biological methods. Various chemical methods such as breakpoint chlorination, precipitation and ion exchange; physical methods like ammonia stripping and membrane separation; and biological methods such as nitrification, denitrification and advanced oxidation processes such as ozonation, sonolysis, wet air oxidation and UV-based oxidation processes are used for the degradation and removal of ammoniacal nitrogen from textile industrial wastewater (Yuan et al. 2016; Nancharaiah et al. 2016; Katheresan et al. 2018). The disadvantages include high retention time, low removal rate, high reaction time, high sludge formation, etc. Chemical process includes ion exchange and breakpoint chlorination for treatment of low-strength ammoniacal nitrogen (Wang et al. 2006); however, ion exchange process is more expensive than the conventional processes (Lotito et al. 2012; Buthiyappan and Abdul Raman 2019). The physical processes for ammoniacal nitrogen removal, i.e. air stripping, is applicable to concentration levels of 10–100 (mg/l). Higher ammonia content requires alternative removal technique (Karri et al. 2018). Biological process in particular nitrification and denitrification methods is poor in removing ammoniacal nitrogen due to longer treatment time and low rate of removal efficiency (Sabumon 2007; Ge et al. 2014).

The task of providing proper treatment facility for the removal of ammoniacal nitrogen ($\text{NH}_4\text{-N}$) from the wastewaters from the dye intermediate industry is not only difficult but also expensive too; hence, there is a strong need of

a new or modified technology which overcomes the limitations of the conventional methods (Hasanoglu et al. 2010; Karri et al. 2018). Advanced oxidation processes (AOPs) drew the attention of researchers for its advantages as it works in ambient temperature and pressure, it has less reaction time and is more economical and feasible than conventional methods (Guedes et al. 2003; Khatri et al. 2018; Zhang and Zhou 2019). Fenton oxidative process is used for the oxidation of organic pollutants by using nascent hydroxyl ion resulted from the reaction of hydrogen peroxide by ferrous ion (Quan et al. 2010; Gonzalez-Merchan et al. 2016; Nidheesh 2015). The various advantages of Fenton reagent over other methods are given in Table 1.

Among the common oxidants, the hydroxyl radicals, which are second only to fluorine, can react rapidly and non-selectively with nearly all-organic pollutants (da Silva Brito et al. 2019).

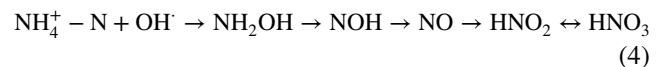
Equations (1) and (2) symbolize main reactions of the system.



The reaction of organic species with $\text{OH} \cdot$ radical is represented in Eq. (3).



The $\text{OH} \cdot$ radical reacts with $\text{NH}_4^+ \text{-N}/\text{NH}_3 \text{-N}$ to give the following products and is represented in Eq. (4).



In the present study, investigations were done on the application of Fenton oxidation process for simultaneous degradation of COD and ammoniacal nitrogen from dye intermediate industrial wastewater. The various operating parameters like pH, concentration of Fe^{2+} and concentration of H_2O_2 were taken into consideration, and their effects on

Table 1 Advantages of Fenton reagent

Advantages	References
They can oxidize organic contaminants and metals, contributing to their degradation and transformation	Barbusiński and Majewski (2003), Moussavi and Matavos-Aramyan (2016) and Shao et al. (2016)
The main advantage is the complete destruction of contaminants to harmless compounds, e.g. carbon dioxide, water and inorganic salts	Eren and Acar (2006) and Nidheesh and Gandhimathi (2012)
It allows high mineralization level at room temperature and pressure conditions	Tony et al. (2016)
No energy input is necessary to activate hydrogen peroxide	Bautista et al. (2007)
It is a cost-effective source of hydroxyl radicals, using easy-to-handle reagents and commonly requires a relatively short reaction time	Barbusiński (2005) and Zhang et al. (2016)
These are relatively reasonably priced, and the process is easy to operate and maintain	Nidheesh and Gandhimathi (2012) and Singa et al. (2018)

the removal efficiencies of COD and ammoniacal nitrogen were examined.

Materials and methodology

Materials used

The dye manufacturing industrial raw wastewater samples were collected in a 20 L can from GIDC, Ankleshwar, Gujarat, and characterization of wastewater was done as per standard methods of American Public Health Association and is given in Table 2.

H₂O₂ used for the study was of Merck brand of 30% W/W purity, FeSO₄ was of Finar Analytical grade with 99% purity, and NaOH was of Finar Brand of Analytical grade with 96% purity. Boric acid, borate buffer, methyl red and methylene blue of Analytical grade and Merck brand were used for the analysis of ammoniacal nitrogen. For the analysis of COD, potassium dichromate (Merck brand) of Analytical grade, concentrated sulphuric acid of 98% purity (Finar brand), mercury sulphate, silver sulphate and ferrous ammonium sulphate (Merck brand) of Analytical grade were used.

Experimental reactor and procedure

A measured quantity of the sample was taken in a batch reactor with a working volume of 1000 mL. In order to see the impact of pH on the removal efficiency for COD and ammoniacal nitrogen and subsequently workout the optimum pH value, the pH of the solution was varied between 1.5 and 3.5 with 1 mL H₂O₂ and 0.4928 g of Fe²⁺ (molar ratio as 1:1). Another set of experiments were done by taking different molar doses of Fe²⁺/H₂O₂ which were 1:1, 3:3, 5:5, 1:2, 1:3, 1:5, 2:1, 3:1 and 5:1. All the experiments were done at room temperature. The COD analysis was done using COD digester with closed reflux apparatus, and ammoniacal nitrogen analysis was carried out by conventional distillation apparatus for ammonia.

Table 2 Characteristics of raw dye intermediate wastewater

Parameters	Concentration in mg/L (except pH)
pH	1.53
TSS	250
TDS	32,600
TS	32,850
COD	5632
Ammoniacal nitrogen	1372

Results and discussion

Effect of pH on COD and ammoniacal nitrogen removal

The Fenton oxidation process is influenced by the initial pH of the sample. The production of OH[·] radical and concentration of Fe in Fe²⁺ or Fe³⁺ form are controlled by pH; hence, it is an important parameter in Fenton process (Kang and Hwang 2000; Meng et al. 2019). The oxidation potential of OH[·] radicals decreases with increasing pH. The Fenton oxidation is effective in acidic medium and slow in alkaline medium and works pre-eminent at pH 2–4.

Higher pH conditions (> 4.5) retard the formation of OH[·] radicals due to decomposition of H₂O₂ into O₂ gas and lose its oxidation ability. On the other hand, extreme acidic conditions are also not conducive for the generation of OH[·] radical. Under highly acidic conditions, solvation of H₂O₂ occurs and it becomes more stable, which reduces its reactivity with ferrous ions to generate OH[·] radicals (Deng and Zhao 2015). Oxonium (H₃O₂)⁺ ion is formed at higher concentration of H⁺ which enhances the stability of H₂O₂ and prevents it from reacting with ferrous ion. The efficiency of the Fenton process to degrade ammoniacal nitrogen is reduced at both high and low pH (Wang et al. 2016). Moreover, the degradation was also slower, which was due to the formation of iron species capable of inhibiting the reaction between Fe²⁺ and H₂O₂. Therefore, to improve the overall degradation efficiency for efficient generation of OH[·] radical, pH value needs to be optimized (Barros et al. 2006; Dükkanci et al. 2014).

In the present study, the effect of pH on removal of COD and ammoniacal nitrogen was investigated. The pH was varied between 1.5 and 3.5, and another experiment was performed without disturbing the initial pH of the sample which was 1.53. Concentration–pH profile on degradation of COD and ammoniacal nitrogen is shown in Fig. 1. The fraction remaining (C/C_0) at pH 1.53, 2.5, 3.0 and 3.5 was found as 0.352, 0.284, 0.227 and 0.272 for COD removal and 0.363, 0.342, 0.251 and 0.279 for ammoniacal nitrogen removal, respectively. It can be seen from Fig. 1 that COD and ammoniacal nitrogen removal increased up to pH 3 and dropped at pH 3.5. The C/C_0 values for COD and ammoniacal nitrogen at original pH of 1.53 were found as 0.352 and 0.363, respectively.

The maximum COD and ammoniacal nitrogen removal (lowest value of C/C_0) was achieved at pH 3, and it was found to be decreasing by increasing the pH to 3.5. At lower pH, when the initial pH was lower than the optimum pH value (pH 3), complex species such as (Fe(II)(H₂O)₆)²⁺ and (Fe(II)(OH)(H₂O)₅)⁺ are formed by Fe²⁺, which in turn reacted very slowly with H₂O₂ and produced less OH[·] radicals. OH[·] radical was scavenged by H⁺ at low

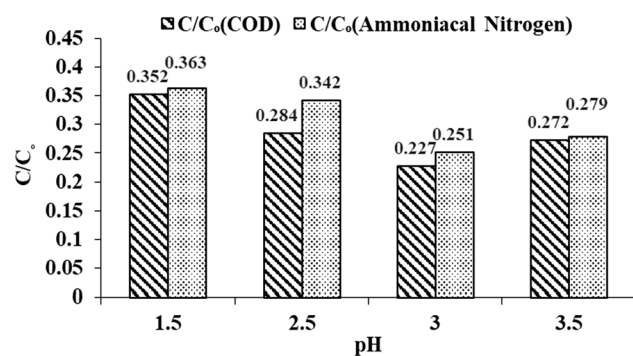


Fig. 1 Profile of fraction remaining set against pH on degradation of COD and ammoniacal nitrogen by Fenton process using molar ratio of $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ as 1:1

pH and resulted in inhibition reaction of Fe^{2+} and H_2O_2 as shown in Eq. (5) (Gogate and Pandit 2004; Nidheesh and Gandhimathi 2014).



The variation of the COD and ammoniacal nitrogen removal with pH observed in the present study converges with the previous studies (Kang and Hwang 2000; Wang et al. 2016).

Effect of Fe^{2+} dosage

Fe^{2+} dosage is an important factor to be considered for running Fenton reaction as it acts as a catalyst to decompose H_2O_2 to generate OH^\cdot radicals. Excessive Fe^{2+} results in self-consumption of free radicals and hinders the oxidation reaction and produces large amount of sludge (Papić et al. 2009). At a Fe^{2+} concentration higher than the optimum, the rate of OH^\cdot radicals originated from the decomposition of H_2O_2 is so high that most of the OH^\cdot radicals are consumed by the side reactions before they are utilized for the removal of the pollutant (Jiang et al. 2010; Chen et al. 2017).

To understand the proper dosages required to enhance removal efficiency, experiments were performed by taking molar ratio of $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ as 2:1, 3:1 and 5:1. The fraction remaining obtained for COD and ammoniacal nitrogen was 0.320, 0.298, 0.372 and 0.377, 0.331 and 0.411, respectively. As shown in Fig. 2, the highest removal efficiency of COD and ammoniacal nitrogen was found at $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ molar ratio 3:1 at pH 3 with reaction time of 60 min. Addition of excess $\text{Fe}(\text{II})$ resulted in brown-coloured sludge formation due to formation of $\text{Fe}(\text{III})$ radicals.

Effect of H_2O_2 dosage

H_2O_2 is the source of OH^\cdot radicals, and therefore, its concentration significantly affects the degradation of ammoniacal

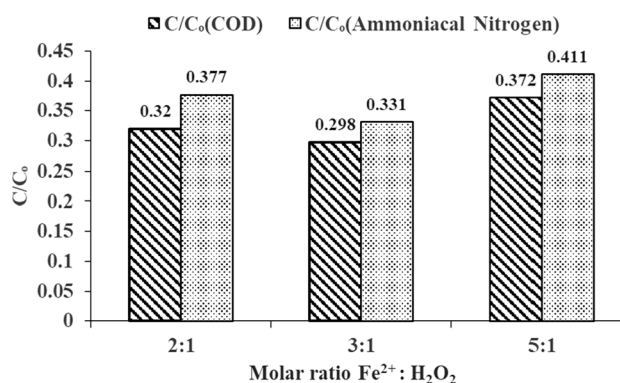


Fig. 2 Profile of fraction remaining set against molar ratio on degradation of COD and ammoniacal nitrogen by Fenton process at pH 3.0 and keeping H_2O_2 molar concentration constant and varying Fe^{2+} dosage (sample volume 1000 mL)

nitrogen and COD. The reaction rate tends to increase with increasing H_2O_2 concentration. As there are series of intermediate reactions which takes place in the process, sufficient H_2O_2 must be added in order to push the reaction beyond that point (Güneş et al. 2018).

In this study, the effect of the concentration of H_2O_2 was investigated on the removal of COD and ammoniacal nitrogen. Experiments were performed at $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ molar ratio 1:2, 1:3 and 1:5 at pH 3 with the reaction time of 60 min. Figure 3 shows the fraction remaining (C/C_0) for COD. From the figure, it can be seen that the maximum reduction in the COD was at molar ratio 1:3. Moreover, it can be seen that the higher H_2O_2 concentration had a scavenging effect on the OH^\cdot radicals resulting in the dip in COD removal. The same pattern was achieved for ammoniacal nitrogen removal with C/C_0 values of 0.265, 0.228 and 0.4 at $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ molar ratio of 1:2, 1:3 and 1:5 showing maximum removal at 1:3.

Previous studies reveal that due to the formation of hydro-peroxyl radical and auto-decomposition of hydrogen peroxide, the removal efficiency significantly decreases as

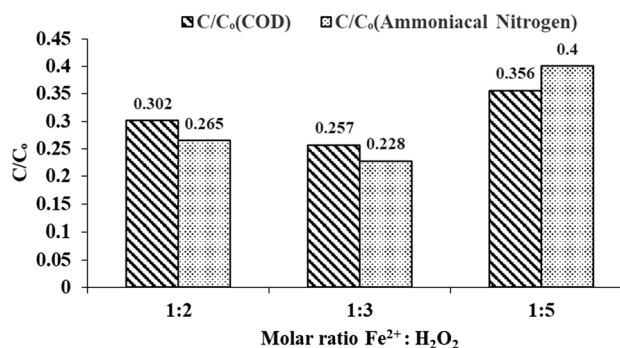


Fig. 3 Profile of fraction remaining set against molar ratio on degradation of COD and ammoniacal nitrogen by Fenton process at pH 3.0 and keeping Fe^{2+} molar concentration constant and varying H_2O_2 dosage

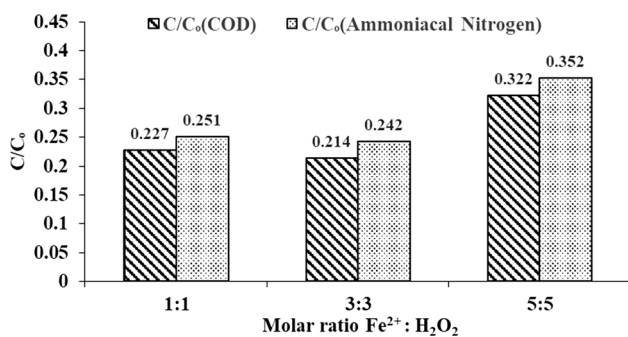
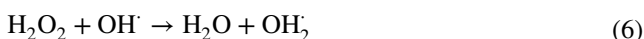


Fig. 4 Profile of fraction remaining set against molar ratio on degradation of COD and ammoniacal nitrogen by Fenton process at pH 3.0 and increasing the concentration of doses by 3:3 and 5:5 (sample volume 1000 mL)

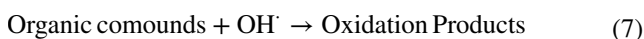
hydro-peroxyl radical has low oxidizing potential according to Eq. (6) (Wang et al. 2019; Nidheesh and Gandhimathi 2014).



Addition of excess H₂O₂ exceeding the optimum limit do not improve the degradation efficiency, which might be attributed to auto-decomposition of H₂O₂ to oxygen and water, and the recombination of OH[·] radicals (Mane et al. 2007; Xue et al. 2009). Moreover, higher concentrations of hydrogen peroxide act as free-radical scavenger itself, thereby decreasing the concentration of hydroxyl radicals and reducing compound elimination efficiency.

Effect of Fe²⁺/H₂O₂ molar ratio

As evident from Eq. (2), H₂O₂ and Fe²⁺ play an important role in the formation of OH[·] radical. The OH[·] radical combines with the organic compounds (pollutants) and oxidizes them, thereby degrading (treating) them (as shown in Eq (7)).



Thus, it is imperative to determine the optimum molar ratio of Fe²⁺/H₂O₂ (Mohajeri et al. 2010) for the maximum treatment efficiency in terms of removal of COD and ammoniacal nitrogen. In the present study, molar ratio of Fe²⁺/H₂O₂ was varied as 1:1, 3:3 and 5:5 at pH 3 with a reaction time of 60 min. As seen in Fig. 4, maximum removal for both COD and ammoniacal nitrogen occurred at molar ratio 3:3 which was 78.6% and 75.2%, respectively. At molar ratio 5:5, the treatment efficiency was 67.8% and 64.8% for COD and ammoniacal nitrogen, respectively. It is inferred that the reduction in the treatment efficiency could be because of the scavenging effect as shown in Eq. (6). These results showed that the reaction between excess Fe²⁺ and HO₂ radical leads to redox reaction which enhances the removal efficiency of

COD and ammoniacal nitrogen (Gulkaya et al. 2006; Chu et al. 2012; Couto et al. 2016).

Conclusions

The current study was found to be an efficient method for degrading (treating) ammoniacal nitrogen and COD from industrial wastewater. The Fenton oxidation process can be better used to treat the industrial wastewater which contains high COD and high ammoniacal nitrogen with very high TDS. The Fenton process can mineralize the pollutants up to 75.8% and 78.6% for ammoniacal nitrogen and COD at optimum conditions of pH 3 and the molar dosage of Fe²⁺ and H₂O₂ of 3:3 for 60-min reaction time. The Fenton process can be carried out in a batch process with detention time of 60 min to degrade the COD and ammoniacal nitrogen.

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Compliance with ethical standards

Conflict of interest All authors declare no conflict of interest.

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