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Comparative Studies on Colour and COD Removal of Reactive Dyes by a Novel Steel Scrap as a Catalyst with Conventional Fenton Process

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Abstract

Aim: *To remove the colour and COD of two reactive dyes namely reactive blue and yellow by Fenton and modified Fenton processes.* ***Materials and Methods****: The total number of groups used in this study is two (Control group N = 16 and Experimental group N = 16).The sample size was taken as 16 after being calculated for a pre-test power of 80% with an alpha value of 0.05 in clinical.com. The removal of colour and COD of two reactive dyes by Fenton process and modified fenton process was carried out****. Results and Discussion****: Colour removal upto 90% and 95% by Fenton and modified Fenton Processes respectively. The best COD removal efficiencies were 65.54 %, and 57.92% in the case of reactive yellow and reactive blue dyes in the Modified Fenton Process respectively. In the modified fenton process steel scrap is replaced instead of* $FeSO_4$*.* ***Conclusion:*** *Compared to the Fenton process, the modified Fenton process has more percentage of colour and COD removal within the limits of the study.*

Key-words: Environmental Engineering, Colour, COD, Reactive dyes, Novel Steel Scrap, Modified Fenton Process.

1. Introduction

There is an ever increasing demand for fabric in the world due to population growth. More than 7,00,000 tonnes of ten thousand different dyes are produced and used for dyeing of fabric annually worldwide (Prakash 1996). Textile industry plays an important role in the industrial development of India and is the second largest sector of Indian economy, next to agriculture.

(Ramachandran and Kumarasamy 2013). A large number of textiles mills have mushroomed in India to meet the demand for fabric. These mills are located mainly in Gujarat, Maharashtra, Delhi, Tamil Nadu and Karnataka. These mills consume large volumes of water for various processes. It is estimated that 100 to 200 litres of water are required for processing one kilogram of fabric. The wastewater of the dyeing process is highly coloured by the release of unfixed dye. It is estimated that 10-15 percent of the dye is lost during dyeing process(Ge and Qu 2003) A promising way to perform the degradation of these types of recalcitrant pollutants is by the application of Advanced Oxidation Processes (AOPs) are well known strong technologies for wastewater treatment purposes. (Zainah, Zainah, and Saksono 2017)

The total number of articles published in this topic over the past 5 years in google scholar and sciencedirect is 48 and 35 respectively. The term Fenton reagent refers to aqueous mixtures of ferrous ion (II) and hydrogen peroxide (Fenton 1894). The effective oxidative agent in the Fenton reaction is the hydroxyl radical as proposed by Haber Weiss ("The Catalytic Decomposition of Hydrogen Peroxide by Iron Salts" 1934). Since then, some groups have tried to explain the whole mechanism (Walling 1975) and many authors have reviewed the Fenton method and the photo Fenton method (Feng and Nansheng 2000; Safarzadeh-amiri et al 1996 a; Zepp et al 1992). Carneiro et al (2006) investigated the oxidation of C.I. Reactive Blue 4 (RB4) by photo-Fenton process mediated by ferrioxalate under artificial and solar irradiation. The influence of ferrioxalate and H_2O_2 concentration RB4 dye initial concentration and solar light intensity on the dye degradation was studied

The Fenton reaction can be classified into two broad categories homogeneous and heterogeneous processes. In homogeneous processes, iron species are in the same phase as the reactants and there is no limitation for mass transfer. Sludge formation with high iron contents, the deactivation of iron because of complex formation and a specific pH range (2.0–4.0) dependency are considered as the significant shortcomings of the homogeneous process(Shah 2015) (Abdullah 2020)All these drawbacks can be conquered by the use of the heterogeneous catalytic approach (Nakagawa, Sakakibara, and Gotoh 2016).

Previously our team has a rich experience in working on various research projects across multiple disciplines (Sathish and Karthick 2020; Varghese, Ramesh, and Veeraiyan 2019; S. R. Samuel, Acharya, and Rao 2020; Venu, Raju, and Subramani 2019; M. S. Samuel et al. 2019; Venu, Subramani, and Raju 2019; Mehta et al. 2019; Sharma et al. 2019; Malli Sureshbabu et al. 2019; Krishnaswamy et al. 2020; Muthukrishnan et al. 2020; Gheena and Ezhilarasan 2019; Vignesh et al.

2019; Ke et al. 2019; Vijayakumar Jain et al. 2019; Jose, Ajitha, and Subbaiyan 2020). Now the growing trend in this area motivated us to pursue this project.

By using these methods we can control the water pollution and we can convert the waste materials into usable things. In this research, The major aim of this current study is to analyse and compare the Colour and COD removal of two reactive dyes using a noval catalyst namely the steel scrap used in the Fenton process with Conventional Fenton Process.

2. Materials and Methods

Fenton and Modified Fenton experiments were carried out in the Water quality Laboratory, Saveetha School of Engineering, Chennai. The total number of groups involved in this project is two (Control group and Experimental group). In this Fenton process is the control group and the Modified Fenton process is set as an experimental group. The sample size was taken as 16 after being calculated for a pretest power of 80% with an alpha value of 0.05 in clinical.com. The input for the sample size calculation is the mean and standard deviation of the parameters from previous established studies. This resulted in a total of 32 samples.

Chemicals and Reagents

Samples were collected in accordance with the American Public Health Association (APHA) standard procedure. The chemicals used throughout this study was analytical grade (make: Merck) hydrogen peroxide (30 % w/v, Merck). The double distilled water was used to prepare experimental solutions. The pH of the solution was adjusted using 1N H_2SO_4 and 1N NaOH. For analysis of Chemical Oxygen Demand (COD) estimation, analytical grade ammonium ferrous sulphate, concentrated sulphuric acid, silver sulphate, potassium dichromate, mercuric sulphate and ferroin indicator from Merck Chemicals were used.

Fenton Reagent

Fenton reagent was prepared by using ferrous sulphate ($FeSO_4$ 99.5%) hydrogen peroxide (30% w/v)Merck.

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Modified Fenton Reagent

Modified Fenton process is a solution of hydrogen peroxide(30% w/v)Merck with steel scrap as a catalyst. As a deviation from the conventional catalysts used in the Fenton process, it was proposed to evaluate the potential of steel scrap as a catalyst. The steel scrap is the waste during the metal sawing operation in steel workshops. The scrap was washed with double distilled water to remove the dust.

Testing Procedure

The colour of the dye solution was measured using a Digital spectrophotometer at the wavelength corresponding to maximum light absorption for two dyes (yellow and blue) as shown in Fig.1 The colour measurements were done at two wavelengths for yellow 400 nm and blue 600 nm are used to measure the colour. The COD of the dye solution was measured using COD Digester. The samples were collected at regular intervals of time and analysed for colour and COD as per the standard methods (APHA 2005). The degradation of the dyes was monitored for 60 minutes.

Fig. 1 - Digital Spectrophotometer

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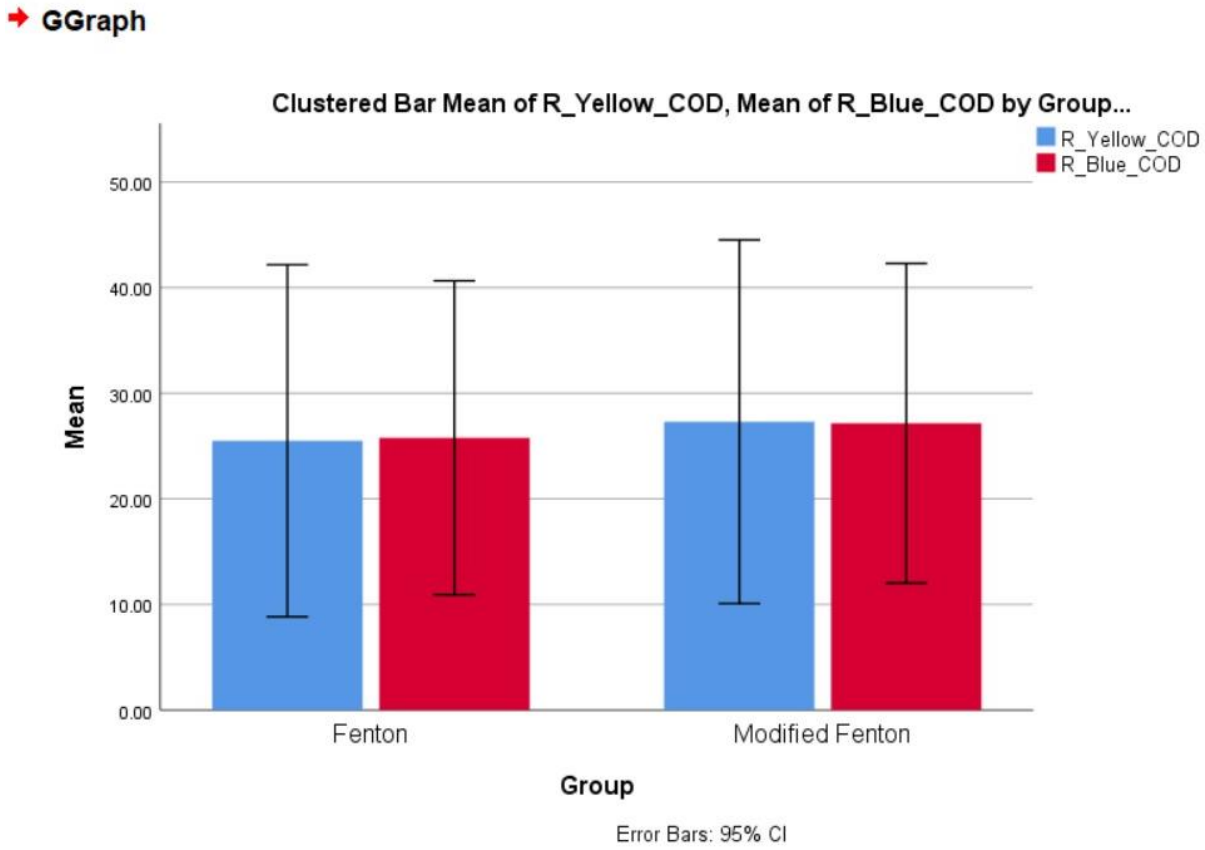
Fig. 2 - Comparison in terms of mean accuracy of Fenton and Modified Fenton process values by SPSS Version 25 with SD±1.The mean accuracy of The error bars suggest that the values are around the mean. Both Fenton and Modified Fenton processes are observed, Modified Fentonis slightly better than Fenton Process.



3. Results

In Table 1. it was observed that all the two dyes have the same pH value about 6. The most significant characteristic is absorbance which is an indication of colour intensities. Higher the absorbance, higher will be intensity of colour. Therefore reactive yellow has a high colour intensity. The maximum COD is present in the Reactive blue.

Table 1 - Characteristics of Dye solutions, the colour of the dye solution was monitored using a Digital spectrophotometer at the wavelength corresponding to maximum light absorption. Reactive yellow dye more than blue dye

Types of dye	Maximum wavelength (nm)	Absorbance	pH	COD (mg/L)
Reactive Yellow	600	1.181	6.2	148
Reactive Blue	400	0.988	5.9	164

Table 2 - Effect of $FeSO_4$ dosage on decolourisation of Reactive yellow dye maximum % of colour removal is at 25mg/L

Effect of $FeSO_4$ Dosage on decolourisation of reactive yellow dye				
Reaction time in minutes	**FeSO4 (mg/L)**			
	0	**25**	**50**	**100**
	Absorbance(400nm)			
0	1.181	1.181	1.181	1.181
5	1.181	0.901	1.229	1.043
10	1.181	0.697	0.963	0.944
20	1.181	0.412	0.717	0.787
30	1.181	0.208	0.582	0.612
40	1.181	0.125	0.488	0.474
50	1.181	0.095	0.391	0.316
60	1.181	0.054	0.242	0.152

From Table 2. the effect of dosage of $FeSO_4$ on decolourisation of reactive yellow dye was studied for concentration of H_2O_2 dosage of 8 mL/L and pH 3.0 are constant and varying $FeSO_4$ dosage of 25 mg/L, 50 mg/L and 100 mg/L. The absorbance has a direct relationship with colour of the dye. The maximum intensity of the colour is, the maximum will be the absorbance. Hence, the reduction in the absorbance value indicates the process of decolourisation. It was observed that the least absorbance was 0.054 compared to pH of 3 and $FeSO_4$ dosage of 25 mg/L after 60 minutes of reaction time. This is equivalent to 95.42 % colour removal.

Table 3 - Effect of $FeSO_4$ dosage on decolourisation of Reactive blue dye maximum % of colour removal is at 25mg/L

Effect of FeSO4 Dose on decolourisation of reactive blue dye				
Reaction time in minutes	**FeSO4 (mg/L)**			
	0	**25**	**50**	**100**
	Absorbance(600nm)			
0	0.988	0.988	0.988	0.988
5	0.988	0.887	0.751	0.737
10	0.988	0.799	0.611	0.641
20	0.988	0.515	0.513	0.407
30	0.988	0.469	0.425	0.343
40	0.988	0.370	0.371	0.298
50	0.988	0.268	0.292	0.112
60	0.988	0.126	0.112	0.076

From Table 3,the effect of dosage of $FeSO_4$ on decolourisation of reactive blue dye was studied for concentration of H_2O_2 dosage of 8 mL/L and pH 3.0 are constant and varying $FeSO_4$ dosage of 25 mg/L, 50 mg/L and 100 mg/L. The experimental results are shown in Table.3. It was observed that the least absorbance was 0.076 comparing to pH of 3 and Fe SO_4 dosage of 100 mg/L after 60 minutes of reaction time. This is equivalent to 92.30 % colour removal.

Table 4 - Effect of H_2O_2 Dose on decolourisation of reactive yellow dye maximum % of colour removal is at 2 mL/L

Table.4. Effect of H_2O_2 Dose on decolourisation of reactive yellow dye				
Reaction time in minutes	**H2O2 (mL/L)**			
	2	**4**	**6**	**8**
	Absorbance(400nm)			
0	1.173	1.154	1.236	1.257
5	1.047	1.095	1.103	1.148
10	0.909	0.932	0.981	0.988
20	0.797	0.789	0.843	0.783
30	0.612	0.638	0.695	0.637
40	0.454	0.506	0.427	0.503
50	0.291	0.347	0.392	0.485
60	0.128	0.212	0.246	0.238

Table 5 - Effect of concentration of H_2O_2 on decolorization of reactive blue dye maximum % of colour removal is at 2 mL/L

Table 5. Effect of H2O2 Dose on decolourisation of reactive blue dye				
Reaction time in minutes	**H2O2 (mL/L)**			
	2	**4**	**6**	**8**
	Absorbance(600nm)			
0	1.342	1.342	1.342	1.342
5	1.093	1.109	1.121	1.289
10	0.963	0.985	0.993	1.143
20	0.825	0.836	0.852	1.091
30	0.607	0.695	0.708	0.845
40	0.478	0.427	0.587	0.687
50	0.235	0.372	0.332	0.432
60	0.101	0.257	0.187	0.285

From Table.4& 5 the effect of concentration of H2O2 on decolourisation of two reactive dyes namely reactive yellow, and reactive blue were studied by keeping the FeSO4dosage and pH as constant at 2 mL/L, 4 mL/L 6 mL/L, and 8 mL/L respectively and absorbance was observed at different H2O2 dosage. The experimental results are shown in Table.4 &5 it was observed that the least absorbance was 0.128 at H2O2 dosage of 2 mL/L after 60 minutes of reaction time. This is equivalent to 89.80 % colour removal. Hence, the H2O2 dosage of 2 mL/L is an optimum dosage for reactive yellow dye. The increase in H2O2 dose there was no significant increase in the colour removal.

Table 6 - COD Removal by Fenton Process Maximum % of COD removal is reactive yellow

Reaction time in minutes	**Dye name**			
	Reactive yellow		**Reactive blue**	
	COD mg/L	**% COD Removal**	**COD mg/L**	**% COD Removal**
0	148	0	164	0
10	139	6.0	156	4.8
20	134	9.4	140	14.6
30	127	14.1	134	18.2
40	119	19.5	128	21.9
50	104	29.7	118	28.0
60	98	33.7	102	37.8
90	67	54.3	82	50.0
120	55	62.8	71	56.7

Table 7 - COD Removal by Modified Fenton Process Maximum % of COD removal is reactive yellow

Reaction time in minutes	**Dye name**			
	Reactive yellow		**Reactive blue**	
	COD mg/L	**% COD Removal**	**COD mg/L**	**% COD Removal**
0	148	0	164	0
10	135	8.78	154	6,09
20	129	12.83.	138	15.85
30	125	14.1	132	19.51
40	118	20.27	125	23.78
50	103	30.40	115	29.87
60	95	35.81	99	39.63
90	62	58.10	79	51.82
120	51	65.54	69	57.92

From Table 6 & 7 the best COD removal efficiencies were 65.54 %, and 57.92% in the case of reactive yellow and reactive blue dyes in the Modified Fenton Process respectively. The reason for slow COD removal when compared to decolourisation, could be the formation of stable intermediate products, which require longer time for further oxidation. Arslan et al (2001).

Table 8 - Statistical analysis of the two groups (Fenton and Modified Fenton Processes)

	Group	N	Mean	Std. Deviation	Std. Error Mean
Fenton Process	1	16	.7413	.30683	.07671
Modified Fenton Process	2	16	.6119	.28724	.07181

Group Statistics is tabulated in Table 8. The mean of Fenton value obtained is 0.7413, whereas in modified fenton is 0.6119.

Table 9 - Independent sample test; The Results of the independent sample test is shown in Table 9. The fischer value obtained from the study is 30 with a significance value of .0228 that is less than 0.05 which shows that the significance difference exists between the two processes

F	Sig.	t	df	Sig.(2-tailed)	Mean Difference	Std. Difference	94.5% Confidence Interval of the Difference Lower	Upper
.315	.579	1.231	30	.0228	.12938	.10507	-.08522	.34397
		1.231	29.870	.0228	..12938	.10507	-.08522	.34401

4. Discussion

The increase in $FeSO_4$ dose, there was no significant increase in the colour removal, and there was slight turbidity was observed while increasing $FeSO_4$dose.The treated dye samples were colourless and did not show any absorbance in the visible region indicating that colour removal could be achieved at low concentration of $FeSO_4$.Simillar studies have shown that during Fenton oxidation colour removal of dye was faster than COD removal (Kang and Chang 1997; Ertugay and Acar 2017)

The increase in $FeSO_4$ dose, there was no significant increase in the colour removal, and there was slight turbidity was observed while increasing $FeSO_4$dose$_4$.Simillar studies have shown that during Fenton oxidation colour removal of dye was faster than COD removal (Kang Chang 1997).The increase in H_2O_2 dose there was no significant increase in the colour removal. The decolourisation of dye wastewater was increased by increasing the concentration of H_2O_2. This can be explained by the effect of additionally produced hydroxyl radicals. The decolourisation above certain ranges improvement was not obvious. This may be due to recombination of hydroxyl radicals and also hydroxyl radical reaction with H_2O_2 contributing to the hydroxyl radical scavenging capacity (Modirshala et al 2006). Similar observation was made by others (Benitez et al ., 1996; Bossmann et al., 1998)

Our institution is passionate about high quality evidence based research and has excelled in various fields ((Vijayashree Priyadharsini 2019; Ezhilarasan, Apoorva, and Ashok Vardhan 2019; Ramesh et al. 2018; Mathew et al. 2020; Sridharan et al. 2019; Pc, Marimuthu, and Devadoss 2018; Ramadurai et al. 2019). We hope this study adds to this rich legacy.

Obviously, the inter particle spaces and free iron on the surfaces of steel scrap pieces help in accelerating the decomposition of H_2O_2. This increased catalytic activity helps to form $OH^{\cdot}$ radicals at an accelerated rate. These $OH^{\cdot}$ radicals attack the chromophoric groups thus improving the efficiency of the degradation process (Kril mert et al 2010). But when the dose of steel scrap was increased beyond 1 g/L, the efficiency of degradation was found to decrease. The. Similar studies have shown that during Fenton's oxidation, colour removal of dye was faster than COD removal (Kang and Chang, 1997).

5. Conclusions

The use of noval steel scrap as a catalyst in the fenton reaction greatly improves the Colour and COD removal without formation of sludge. Compared to the classical Fenton process reaction with alternative iron sources, the novel modified fenton process (H_2O_2 /Steel scrap) has clear advantages in the context of cost effectiveness.

Declarations

Conflict of Interests: No conflict of interest in this manuscript.

Authors Contributions

Author EPK was involved in data collection, data analysis, manuscript writing. Author RG was involved in conceptualization, data validation, and critical review of manuscript.

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