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Fenton's Oxidation of Phenalkamine Condensate Using Aluminium Dross And Laterite Iron Nanoparticle As A Catalyst

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Abstract—The use of low cost and eco-friendly catalysts in heterogeneous Fenton for the treatment of an actual Phenalkamine condensate (having COD varying from 83000 to 150000 mg/L) was investigated. Phenalkamineis a curing agent which is produced by the reaction of cardanol, and amines. The Phenalkaminecondensatehashigh pH, high COD, and low BOD/COD ratio. The efficiency of Aluminium dross and Iron nanoparticle as catalysts instead of the conventional Fe (II) salts were evaluated.Aluminium dross is a waste product which is obtained during aluminium smelting process and the usage ofthis waste also reduces the disposal issuesrelated to Al dross. Iron nanoparticle (FeNP) was synthesized using laterite soil as the Iron source and cashew apple extract as the reducing agent. The main operating parameters for heterogeneous Fenton's reaction were pH, H_2O_2: COD ratio and H_2O_2: Catalyst ratio. The optimal conditions were obtained as pH=3, H_2O_2: COD=1.25:1, H_2O_2: Al^{3+}=25:1 with a maximum COD removal efficiency of 75.83%, for Aluminium dross and pH=3.5, H_2O_2: COD=1.67:1, H_2O_2: FeNP=20 with a maximum COD removal efficiency of 88.91%, for Iron nanoparticle as the catalyst. Comparable efficiencies were obtained for both the catalysts, which proves that they can be used as an alternative to conventional Fe (II) salts in Fenton's process

IndexTerms—Fenton's oxidation, HeterogeneousFenton oxidation, Phenalkamine industry, Aluminium dross, Iron nanoparticle.

I. INTRODUCTION

Due to industrialization, the generation of wastewater is continuously growing up and this is an environmental problem that must be taken into account. In recent years due to more stringent about environmental laws and regulations and also because of increasing awareness about the environment, treatment of industrial wastewater has always been a key aspect of research.Phenalkamines are the curing agent formed by the Mannich based reaction of cardanol, and amines as shown in Figure 1 [1]. The amines generally used are ethylene diamineorDiethylenetriamine.Phenalkamines are generally used in coatings for ships and offshore constructions,marineand industrial maintenance coatings, tank linings due to various advantages it has like: extremely fast cure, low temperature cure (even below 0°C), good surface appearance, good moisture &surface tolerance, excellent water and salt water resistance.Phenalkamines produced from cardanol serve as epoxy hardeners for high-end applications. They are also well known for their anticorrosive properties, mainly in piping and onshore applications [2].

Duringprocessing of Phenalkamine, 10% of water comes out as waste which consists of phenols, aldehyde, and amines which are highly reactive compounds and toxic to the environment and it is surrounding and also to marine life so it needs to be treated before discharge. The major pollutants in the Phenalkaminewastewater are high pH, color, high COD, high BOD, and low BOD/COD ratio.As the Phenalkamine condensate have low BOD/COD ratio, it is more advisable to go for chemical methods of treatment than the biological methods. Advanced oxidation has gained more attention these days, which involves oxidation of the pollutant by reaction with hydroxyl radical. There are several advanced oxidation processes such as homogenous Fenton, heterogeneous Fenton, photo Fenton, photocatalysis and ozonation. Among these heterogeneous Fenton is an interesting alternative for wastewaters with high organic content. Heterogeneous Fenton is an oxidation process involving solid iron-based catalyst and hydrogen peroxide. The reaction seems to be simple but many reaction mechanisms have been proposed based on different active intermediates. The reaction largely depends on pH, the concentration of hydrogen peroxide and the catalyst. Compared to conventional Fenton, heterogeneous Fenton has the advantage of less sludge production, easy recovery and reuse of catalyst and the process can even be carried out at nearly neutral pH, reducing the need for acidification and subsequent neutralization [3].

Aluminium dross is a waste product obtained during aluminium smelting process and it consists of various metals, metal oxides,andsalts [4].The worldwide aluminium industry produces more than five million tonnes of furnace waste each year known as dross, and above 95% of these waste are being disposed in the landfill adding cost to the industries [4]. Various methodsweredeveloped to treat such dross materials, but most of the dross is still used as landfill material [5,6,8]. However, their safe disposal is a problem because of its negative impact on the ecosystem, groundwater, and surface [4].Waste Aluminium dross can be recycled and it can be

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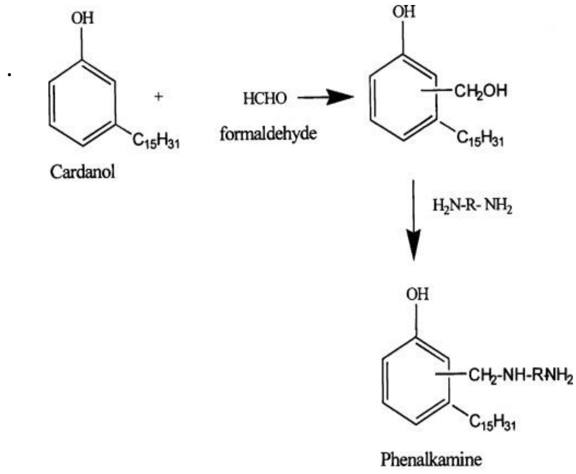
utilized as a useful engineering material.Recycling 1 kg of aluminium can save about 4 kg of bauxite, 2 kg of chemicals, and 7.5 kWh of electricity.[7] It can be used to make bricks or can be used in concrete as filler, catalyst, control microcracking and also used as an additive for de-sulphurizing steel slag.[4, 8, 9]

Iron nanoparticles are now widely used in wastewater treatment due to its high surface area, low cost and eco-friendly nature. There are many chemical and physical methods to produce iron nanoparticle. But in these methods chemicals are used as reducing and stabilizing agents, which are non-biodegradable[10,11]. Green methods of synthesis are an alternative for this, which uses plant extracts as the reducing and stabilizing agents.

Therefore, in this study, aluminium dross and Iron nanoparticleare introduced as a Fenton's catalyst for the removal of organic pollutants from Phenalkaminecondensate.



R= CH_2-CH_2 (Ethylene diamine)

CH_2-CH_2-NH-CH_2-CH_2 (Diethylene triamine)

CH_2-CH_2-NH-CH_2-CH_2-NH-CH_2-CH_2 (Triethylene tetraamine)

Figure 1: Structure of Phenalkamine

II. MATERIAL AND METHODOLOGY

A. Materials:

- **Wastewater**

The Phenalkaminecondensateused in this Fenton's oxidation process were collected from a nearby Cashew nut processing industry and is stored at a low temperature (4^oC).The Phenalkamine condensate characteristics are presented in table 1.

- **Chemicals**

Chemical reagents used in this study were Hydrogen peroxide (30%, 50% w/w), Hydrochloric acid, sulphuric acid (98%), Sodium Hydroxide (98%) available in the purest grade and used without further purification. Potassium dichromate ($K_2Cr_2O_7$), Ferrous Ammonium Sulphate (FAS), Mercuric sulphate ($HgSO_4$), Ferroin indicator, Potassium Iodide (KI), Sodium thiosulphatethese chemicals were used as standard chemicals to determine COD and BOD concentration of the effluent.

B. Catalyst Preparation:

- **Iron Nanoparticle**

The iron required for synthesizing iron nanoparticle was extracted from laterite soil by using acid leaching method. The soil was collected from Surathkal, which is a highly laterite saturated area. The soil was dried, crushed to powder and sieved through 105μm sieve. 3g of the sieved sample was taken in a glass beaker and about 15 mL of 35% HCl was added to it and this solution was mixed and grinded until dissolved. The beaker was heated on a hot plate, till a residue was formed at the bottom. The residue was then baked in an oven for 1 hour. Again, to this baked residue 15 mL of 35% HCl was added and it was heated for 1 minute. 20ml of distilled water was heated and then it was added. The above solutions were filteredthrough whatman no: 42 filter paper. The filtrate was transferred to a Nessler's cylinder and diluted up to 100ml. The solution thus obtained is the laterite soil extract (Figure 2(a)).

The cashew apple collected was washed thoroughly to remove sand and grit. About 200g of cashew apple was taken and it was grinded using little distilled water, in order to extract the pulp. This juice was then allowed to settle in a beaker for 30 minutes and the supernatant was filtered using Whatman no: 42 filter paper. This filtrate was then diluted up to 1L (Figure 2(b)). The cashew apple extract thus obtained, have high polyphenol content, which acts as a reducing agent for iron nanoparticle synthesis.

The iron nanoparticles are synthesized by adding the laterite soil extract and the cashew apple extract in different volume proportions into a conical flask kept on a magnetic stirrer with hot plate maintained at 540rpm and 80^0C. The immediate appearance of black color indicated the presence of iron nanoparticles[12].The solution was then centrifuged using a high-speed centrifuge at 2000rpm for 30 minutes. The liquid was removed carefully and the nanoparticles were washed using distilled water and dried in an oven at about 50^0C. The synthesized nanoparticles were preserved for further use (Figure 2(c)).

(a) (b) (c)

Figure 2: (a) Iron extract from laterite soil; (b) Cashew apple extract; (c) Synthesised Iron nanoparticle

The surface morphology and elemental composition of the iron nanoparticles were determined using Field Emission Scanning Electron microscopy X-ray energy dispersive

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Spectro-photometer (FESEM-EDX)[13,14]. In order to study the functional groups on the surface of nanoparticles which acts as capping and stabilizing agents, Fourier Transform Infrared spectroscopy (FTIR) was done. The concentration of Iron in the laterite soil was determined by using UV-Visible Spectrophotometer. The presence of polyphenol content in the cashew apple extract was done by Ferric chloride test. The presence of phenol was indicated by red, blue, green or purple color on the addition of the few drops of Ferric chloride to the sample and the polyphenol content was confirmed by FTIR analysis[14, 15].

- **Aluminium Dross**

Aluminium dross sample was collected from a nearby supplier. The sample was in powder form and the color of the dross is black. Aluminium dross samples were produced when the raw aluminium material was burnt at very high temperature. Aluminium dross sample was sieved by a 75 µm sieve and then used in the treatment process. The sample is shown in the figure 3.

Figure 3:Aluminium dross sample.

Aluminium dross sample has the highest percentage of Al_2O_3 followed by $SiO_2$and small amounts of oxides of Fe, Mg, Ca along with small amount of salts in the form of NaCl and KCl. The catalyst was characterized using the following techniques:-

i. The surface morphology of dross sample was found by scanning electron microscope (SEM).
ii. The elemental composition of the dross sample was determined by energy dispersive X-ray spectroscopy (EDS)
iii. To identify the various phases in the sample, X- ray powder diffraction (XRD)techniques were conducted.

C. Experimental Methodology:

Fenton's oxidation generally depends on three factors: effect of pH, H_2O_2 concentration, Catalyst concentration[16].COD reduction of this high strength Phenalkaminescondensate was done by heterogeneous Fenton process using iron nanoparticle and Aluminium dross as catalysts. Experiments have been conducted at ambient temperature (27±3^{0}C) in batch reactors for the Phenalkaminescondensate.At first, the experiments have been conducted for pH optimization for 100ml sample in a 500ml of a conical flask with varying pH from 2 to 4 for aluminium dross and for iron particle pH was varied from 2 to 7. The pH of the solution was adjusted using 0.1NNaOH and 5N H_2SO_4. Then, Aluminium drossand iron nanoparticles were added to the desired $Al^{3+}$and Fe^{2+}concentration. Finally, H_2O_2 carefully added to start the Fenton's reaction. Experiments were conducted at optimum pH with different dosages of hydrogen peroxide and catalyst and final dosage have been optimized to get maximum COD removal (%).

All the experiments were done in duplicate sets for a reaction period of 24 hours so that hydrogen peroxide decomposes completely (zero residual hydrogen peroxide). After the reaction time, the samples were filtered using Whatman no: 42 filter paper for further analysis. COD of the treated sample was done to determine the COD removal efficiency. The experimental set up for Fenton's process is shown in Figure 4.

$$\% \text{ COD removal efficiency} = ((COD_i - COD_f)/COD_i) \times 100$$

Where,
$COD_i$is the initial COD (mg/L) and COD_f is the final COD (mg/L) after the reaction time.

(a) (b)

Figure4: Experimental set up for Fenton's process: (a)Using Aluminium dross as the catalyst; (b) Using iron nanoparticle as the catalyst

D. Analytical Procedure

Analytical procedures were conducted as per standard methods [17]-Chemical oxygen demand (Closed reflux titrimetric method, Method 5220C), Biological oxygen demand (BOD,Method 5210B), Suspended solids (SS, Method 2540D). pH was measured by HI763100: Digital 4 ring conductivity probe with an integrated temperature sensor

Table 1:Characteristics of the Phenalkamine condensate, from a Cashewnut processing industry

pH	11.5-12.5
COD(mg/L)	83000-150000
BOD (mg/L)	3600- 5800 mg/L
TSS (mg/L)	8500-9720 mg/L

III. RESULTS AND DISCUSSION

A. Catalyst Characterization:

- Aluminium Dross

In the present study, the finer particles (<75 µm) taken for the Fenton's oxidation study were subjected to SEM, EDAX and

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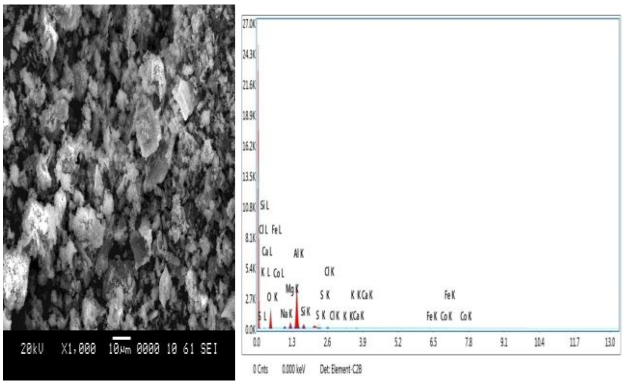
XRD analysis. The morphology of aluminium dross wasimagined using SEM (scanning electron microscope) and the results as illustrated in Fig 5 (a) shows the dross has an irregular shape, and there are no voids or cavities observed in the image indicate that, the particles are well dispersed.Energy Dispersive X-Ray Analysis was done to identify the elemental composition of the dross sample. The chemical composition of Aluminium dross (express in weight percentage) as illustrated in Figure 5 (b).Aluminium percentage was found 40% by weight.Figure 5 (c) shows the XRD pattern of the aluminium dross and the figures reveals the various phases present in the sample



(a) (b)

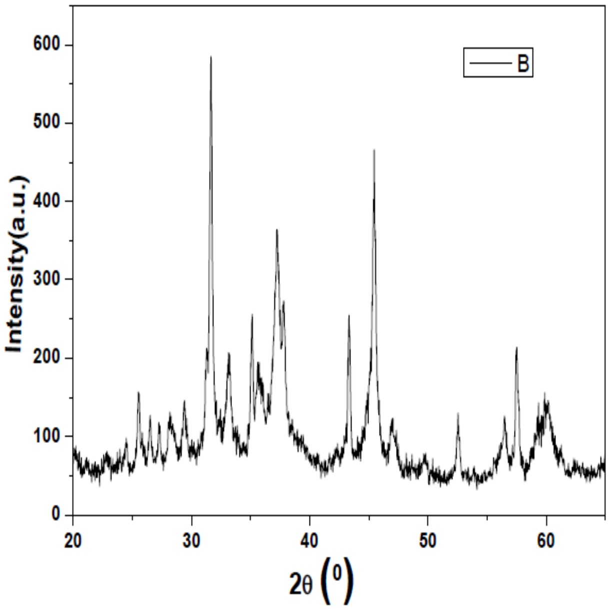


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Figure 5: Characterization of aluminium dross using (a) SEM, (b) EDAX, (c) XRD

Those main peaks show the existence of Al_2O_3, AlN, $MgAl_2O_4$, SiO_2 and probably also KCl, NaCl, MgF_2, etc.

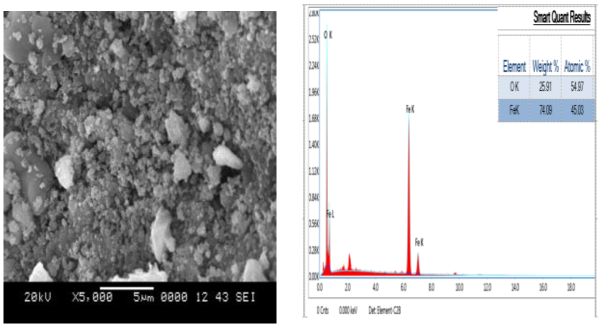
- **Iron nanoparticle**

The concentration of Iron in the laterite soil was determined by using UV-Visible Spectrophotometer as 62.234 mg/L. Ferric chloride test for the cashew apple extract gave a positive result, indicating the presence of polyphenol and the polyphenol content was confirmed by FTIR analysis.

The morphology and elemental composition of synthesizedIron nanoparticles were analyzed by usingFESEM-EDX. The SEM micrograph (Figure 6 (a)) shows small sphericalparticles in the nanometer range.

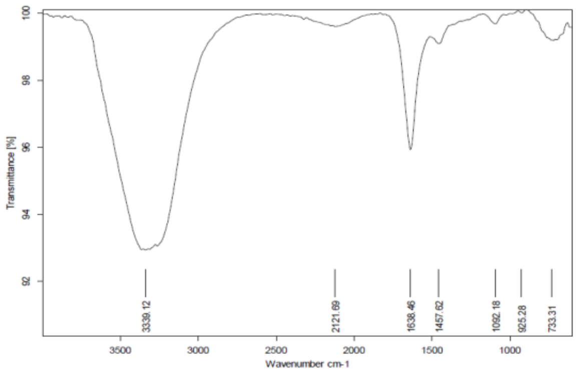
The elemental composition of the synthesized iron nanoparticle was determined by EDAX analysis. The EDAX spectra show strong peaks of Oxygen and Iron, with a chemical composition of 74.09% Fe and 25.91% O(Figure 6 (b)).FTIR analysis provides the information on the functional groups present in the cashew apple extract as well as the Iron nanoparticle. Briefly, a broad peak at 3339.12 cm-1 in Figure 7(a) and 3333.74 cm-1in Figure 7(b)corresponds tothe stretching vibration of –OH groups, peaks 1638.46cm-1 (Figure 7(a)) and 1636.58 cm-1 (Figure 7(b)) assigned to thestretching vibrations of C=O and C=C group, peaks 1092.18(Figure 7(a)) and 1097.83 cm-1 (Figure 7(b)) correspond to C–Ostretching of alcohols

andpeaks925.28 (Figure 7(a)) and 922.54 cm-1 (Figure 7(b)) correspond to the stretchingvibration of C–C group.Peaks at around 570 cm-1 in Figure 7(b) indicates the stretching vibrations of Fe-O. Similar peaks in the FTIR spectra of the cashew apple extract and the synthesized nanoparticle indicates that the nanoparticle was successfully stabilized by the cashew apple extract



6 (a) 6(b)

Figure 6: FESEM-EDX analysis of Iron nanoparticle; (a) SEM micrograph; (b) EDAX spectra



7(a)

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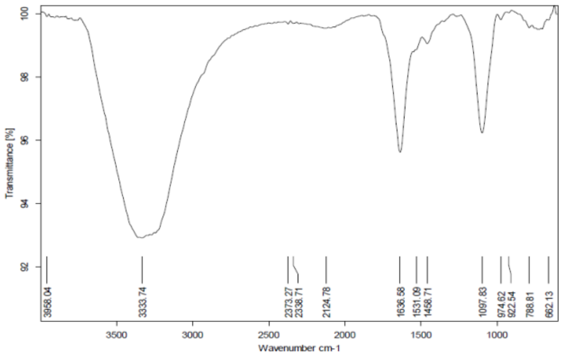

7(b)

B. Fenton's oxidation of Phenalkaminecondensate

- **Effect of pH**

ThepH of the solution is an important parameter for Fenton's oxidation process, which controls the production rate of hydroxyl radical thus the oxidation efficiency[18, 19]. A series of preliminary batch experiments were conducted using different H_2O_2 and Al^{3+} concentrations at a pH ranging from 2.0 to 4.0for aluminium dross and for iron nanoparticle pH was varied from 2 to 7using different H_2O_2 and FeNPconcentrationto determine the optimal condition for the initial pH.

From the preliminary batch experiments it showed that pH 3.0 was the optimal initial pHat the dosages of H_2O_2: COD= 1.66:1 [H_2O_2 concentration 7.3M] andH_2O_2: Al^{3+} = 20:1 [Al^{3+} concentration 0.36M] for removal of COD in Fenton's oxidation of PhenalkaminesCondensate. At pH 3 removal efficiency of COD is 67.2%. Figure 8 shows the effect of pH on the removal efficiency of COD of Phenalkamine wastewater

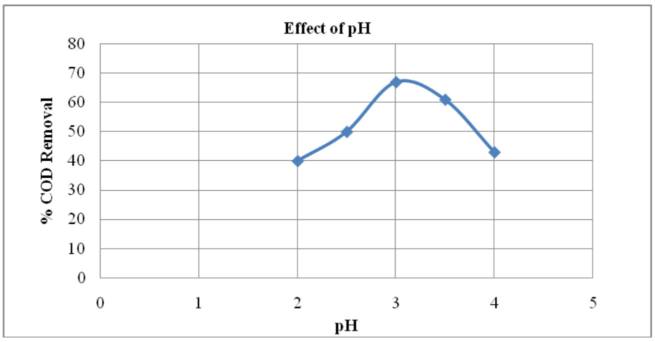

Figure 8: Effect of pH on removal efficiency (H_2O_2:Al^{3+} = 20:1)

For iron nanoparticle, Figure 9 shows the effect of pH on COD removal. It is observed that the efficiency increased initially and thereafter it decreased gradually. An optimum pH of 3.5 was obtained at H_2O_2: COD = 2:1 and H_2O_2:FeNP = 25:1, with a COD removal efficiency of 84.61%. Compared to conventional Fenton's process, where the removal efficiency is very low at pH>4, here, nearly 50% efficiency is obtained even at circumneutralpH

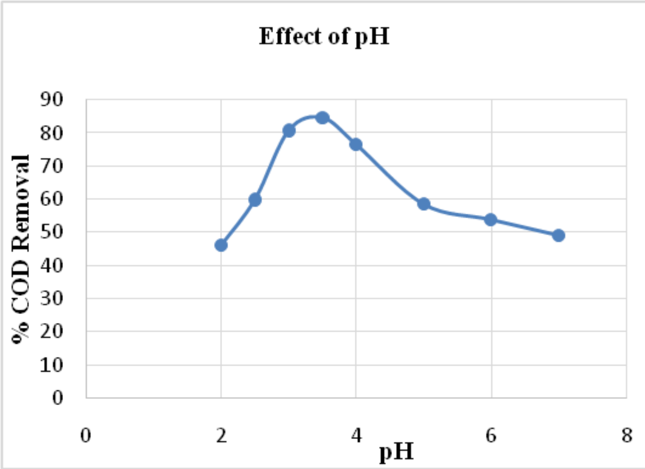

Figure 9: Effect of pH on removal efficiency (H_2O_2:FeNP = 25:1)

At higher pH, the removal efficiency of COD is lesser compared to optimum pH. This is because under alkaline conditions (Higher than optimum pH) dissociation and auto-decomposition of H_2O_2occurs.Also as the pH increases, there is a decrease in oxidation potential of hydroxyl radicals [20].

- **Effect of H_2O_2 concentration on removal efficiencies**

The concentration of hydrogen peroxide is one of the important parameters to influence the Fenton's processsince it is the source of OH radicals that cause the degradation of organic compounds.For aluminium drossthe experiments were conducted with varying H_2O_2: COD ratio maintaining all other parameters constant. The ratio was varied from 0.7 to 2.5 withH_2O_2: Al^{3+}= 20:1 [varying the H_2O_2 concentration from 3.1Mto 11M] and keeping Al^{3+} concentration 0.36 M] and time 24hours. The experiments were conducted at pH 3 which was the optimum pH value. It is observed that the maximum removal of COD is at H_2O_2: COD= 1.25:1 and maximum removal percentage is 71.3%.The percentage removal COD with COD:H_2O_2ratio as illustrated in Figure 10

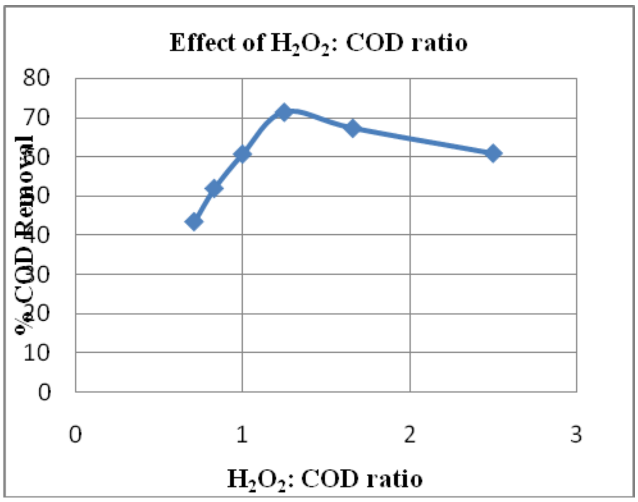

Figure 10:Effect ofH_2O_2: COD ratio on Removal efficiency (pH=3, H_2O_2:Al^{3+}=20:1)

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For iron nanoparticles,H_2O_2: COD ratio was varied from 0.8 to 2.5, with H_2O_2: FeNP = 25:1 and at the optimum pH of 3.5. The experiments were conducted for a reaction period of 24 hours and at the optimum ratio was determined corresponding to the maximum COD removal. An optimum H_2O_2: COD ratio of 1.67 was obtained with aCOD removal efficiency of 84.86%, Figure 11

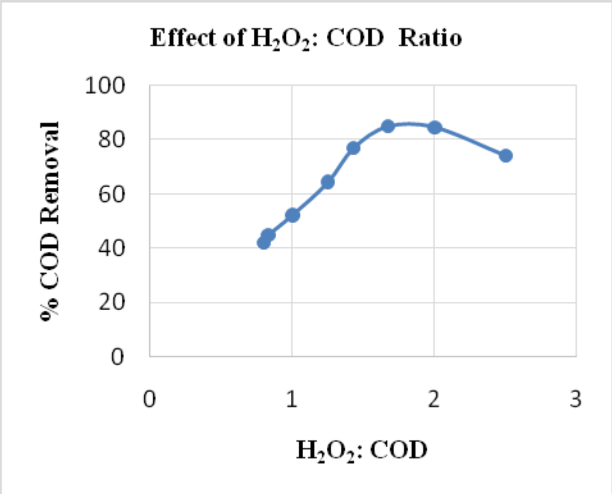

Figure 11:Effect of H_2O_2: COD ratio on Removal efficiency (pH=3.5, H_2O_2: Fe = 25:1)

From Figure 10and 11, it can be found that the removal efficiency increases with the increase of H_2O_2 concentrationtill optimal H_2O_2 [21]. This may be due to the fact that an increased amount ofH_2O_2reacts with more catalyst (iron nanoparticle and aluminium dross) and produces more amount of hydroxyl radical leading to more waste degradation. However, an excess of hydrogen peroxide contributes to the Chemical Oxygen Demand (COD) and is a harmful component for microorganisms.In addition, higher concentrations of hydrogen peroxide act as free radical scavenger itself, which decreases the concentration of hydroxyl radicals and thereby reducing degradation efficiency. Thus, the dosage of H_2O_2should be adjusted in such a way that the entire amount is utilized.

- **Effect of catalyst concentration on removal efficiencies**

Catalyst plays an important role in a reaction by providing a route that requires low activation energy, thereby increasing the rate of reaction. In Fenton's oxidation, the degradation efficiency increases with an increase in catalyst concentration, up to the optimal dosages.The ratios of H_2O_2:$Al^{3+}$is important for the degradation of pollutant in Fenton's process. For aluminium dross, after pH optimization set of experiments were conducted by varying H_2O_2 and Al^{3+} ratio for effective degradation by keeping all other parameters constant. A set of batch experiments were conducted by varying ratio from 5 to 35 [concentration of Al^{3+}1.1M to 0.16 M), pH=3, H_2O_2: COD=1.25:1.and from Figure 12 maximum removal efficiency was found 75.83% at H_2O_2: Al^{3+}= 25:1

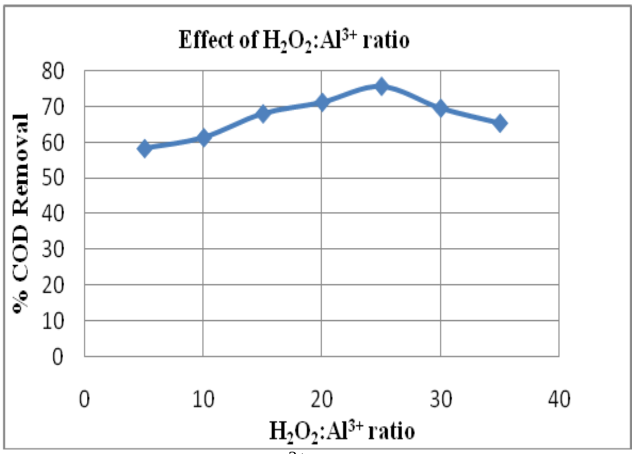

Figure12:Effect of H_2O_2: Al^{3+} ratio on Removal efficiency H_2O_2:Al^{3+} ratio (pH=3, H_2O_2: COD=1.25:1)

The ratio of H_2O_2: FeNP was varied from 5 to 100, with H_2O_2: COD ratio of 1.67 and pH of 3.5. An optimum H_2O_2: FeNP ratio of 20 was obtained with a COD removal efficiency of 88.91%. Iron acts as a catalyst for the initiation of OH radical generation. As the concentration of iron increases the efficiency also increases due to more degradation of H_2O_2, after reaching an optimum iron concentration the efficiency decreases due to the ferrous ion inhibition, Figure 13. As the iron used is in nanoparticle form the surface area is more compared to the conventional iron salts used for Fenton's process, as a result, the efficiency is high at the optimum iron concentration.

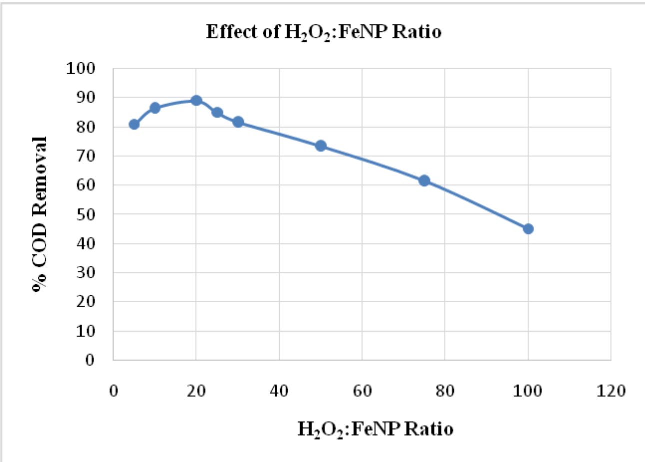

Figure 13:Effect of H_2O_2: FeNP ratio on Removal efficiency (pH=3.5, H_2O_2: COD=1.67:1)

From Figure 12and 13, it can be found that the efficiency of treatment is dependent upon the amounts of H_2O_2 and catalyst. It is because when either of them has overdosed, both react with hydroxyl radical and therefore inhibit the oxidation reaction. Therefore, for maximum removal efficiency, every parameter must be optimized. Maximum COD removal efficiency for aluminium dross and nano iron is 75.83% and 88.91 respectively

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IV. CONCLUSION

- Maximum COD removal efficiency of 75.83% was obtained at the optimised conditions (pH=3, H_2O_2: COD=1.25:1, H_2O_2:Al^{3+} = 25:1) for aluminium dross.
- Maximum COD removal efficiency of 88.91% was obtained at the optimised conditions (pH=3.5, H_2O_2: COD=1.67:1, H_2O_2: FeNP= 20:1) for iron nanoparticle.
- Thus, Aluminium dross and Iron nanoparticle can be used as an economical and efficient catalyst for Phenalkamine treatment

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