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### Original article

## Survey efficiency of dairy wastewater treatment by combined chemical coagulation and fenton oxidation process

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#### ABSTRACT

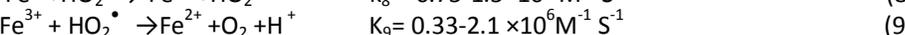
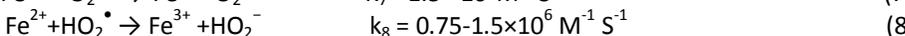
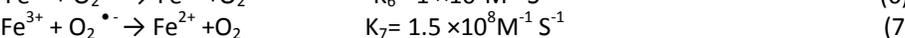
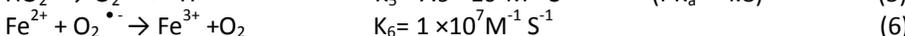
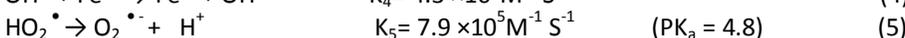
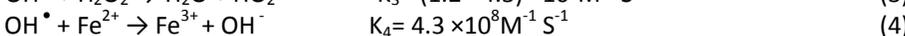
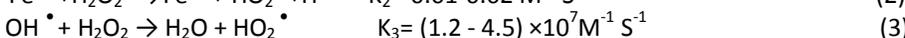
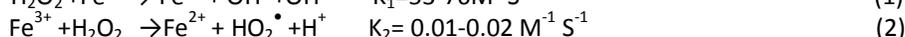
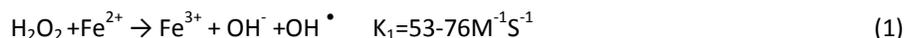
A large amount of wastewater is produced in the dairy industry. This effluents creates major environmental problems when discharged to surface water. The combined chemical coagulation and Fenton oxidation process was applied for the treatment of dairy wastewater. In the first stage, poly aluminum chloride (PAC) was used as coagulant and optimum dose of coagulant was determined. After that, the effluent of coagulation process was used in Fenton oxidation process. In Fenton process, the influence of pH, reaction time, Fe<sup>2+</sup> and hydrogen peroxide on the removal efficiency of COD and BOD from dairy wastewater were studied, and subsequently the optimal values for each parameter were determined. In The combined chemical coagulation and Fenton a huge decrease in COD and BOD concentrations were achieved. Based on the result of this study, coagulation/Fenton as an effective method can be used for dairy wastewater treatment.

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## 1. Introduction

The dairy industry is one the largest industrial water consumer and industrial wastewater producer (Andrade et al., 2014; Shete and Shinkar, 2013). It generates about 0.2–10 liters of effluent per liter of processed milk (Tchamango et al., 2010; Kushwaha et al., 2010). The dairy waste water is produced from production line, pasteurization and homogenization tanks, clean in place equipment and cooler system (Shete and Shinkar, 2013; Tawfik et al., 2008). The dairy effluents is characterized by high chemical oxygen demand (COD) and biological oxygen demand (BOD) (Shete and Shinkar, 2013; Karadag et al., 2014). This effluent contains fat, lactose, protein and residual cleaning agents (Kushwaha et al., 2010; Authority, 1997; Tikariha and Sahu, 2014). Dairy Waste water volume and that's chemical Characteristic is fluctuated widely from day to day due to differences in the type of product being processed (Farizoglu and Uzuner, 2011; Authority, 1997; Hassan and Nelson, 2012; Tikariha and Sahu, 2014). In addition, climate of the area is other major reason, responsible for changing waste water character from one industry to another dairy industry and from season to season (Tikariha and Sahu, 2014). The dairy effluent is discharged into rivers, causing significant environmental problems when it is discharged without treatment (Tchamango et al., 2010). This effluent results in water is eutrophication by phosphorus and nitrogen compounds and hazardous to aquatic life and soils (Amini et al., 2013; Danalewich et al., 1998). Up to now, many methods have been used for the treatment of dairy wastewater. Conventional treatment systems for these wastewaters include biological (aerobic and anaerobic) and chemical process (Moulik et al., 2014; Luo and Ding, 2011). Whereas most biological treatment method being used at the external temperature, their efficiency is strongly dependent on the season and on climate of the area where they are installed (Danalewich et al., 1998; Castillo and Zapico, 2007; Metcalf et al., 1972). Moreover, in these methods several problems have been observed, such as high production of sludge and scum, extensive energy requirements for oxygen supply, sludge poor settleability, sensibility to organic shock, difficulties in removing nutrients (nitrogen and phosphorus) and problems in the degradation of fats (Andrade et al., 2014; Karadag et al., 2014; Tikariha and Sahu, 2014; Danalewich et al., 1998; Mohapatra et al., 2010). It is necessary to develop novel and cost-effective technologies to treat this wastewater. Technical and economical feasible options must be obtained by appropriate treatment systems. Combined Fenton and coagulation process is known as effective method to remove most organic pollutants from wastewater (Tisa et al., 2014; Kabita et al., 2001). This method have been widely studied in the degradation of organic pollutants (Perdigón-Melón et al., 2010; Boumechhour et al., 2013). Fenton oxidation process is performed by combination ferrous ion ( $\text{Fe}^{2+}$ ) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) in acid conditions which can generate Hydroxyl radicals ( $\text{OH}^\bullet$ ) with powerful oxidizing ability. Hydroxyl radicals ( $\text{OH}^\bullet$ ) generated by this process is non-selective and exhibit oxidation potentials of approximately 2.80V at pH of 3.0 (Mohapatra et al., 2010; Liu et al., 2013; Xu et al., 2004).

The Fenton process is more explained by flowing reactions (Burbano et al., 2005; Xu et al., 2004; Wolfgang Gernjak, 2006):



The reactions are conducted dependent on their reaction rate constant (k) and the concentration of each reactants (Burbano et al., 2005).

Several advantages about Fenton oxidation process has been reported, such as generation of harmful byproducts with this process is lower than other AOPs (advanced oxidation process),  $\text{H}_2\text{O}_2$  is environment friendly (it is harmless and slowly decomposed to water and oxygen), the simplicity of operation and design of Fenton process, complete destruction of contaminants to  $\text{CO}_2$ , water and inorganic salts (Santos et al., 2014; Burbano et

al., 2005; Neyens and Baeyens, 2003; Riaño et al., 2014). In addition costs of application of Fenton's reagents is lower than other AOPs (Kabita et al., 2001).

The main goal of this study was examination of feasibility and efficiency of combined chemical coagulation and Fenton oxidation process for treatment of dairy wastewater.

## 2. Materials and methods

Dairy industry Wastewater samples were obtained from equalization tank and stored at +2 to +6 °C in refrigerator. The samples were analyzed for Chemical Oxygen Demand (COD), Biochemical Oxygen Demand (BOD), Total Kjeldahl Nitrogen(TKN), Total Phosphorus(TP) and pH values according to standard methods (Lenore et al., 2005). Main Characteristics of dairy industry wastewaters effluents used in experiments are given in table1. Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 30.0% w/w), iron (II) sulfate heptahydrate (FeSO<sub>4</sub>·7H<sub>2</sub>O≥99.0%), sodium sulfite (Na<sub>2</sub>SO<sub>3</sub>) and other chemical reagents used in experiments were purchased from Merck. Chemical coagulation and Fenton oxidation experiments were performed in jar test apparatus. All reaction were performed in batch reactor.

**Table1**

Main characteristics of dairy wastewater effluent.

Parameter	Unit	Minimum	Maximum	Average	Standard deviation
COD	mg/L	3924	5395	5002	370.4829
BOD	mg/L	2260	3107	2871	211.86
TKN	mg/L	50	203	148	41.97
TP	mg/L	26	47	38	7.4
COD/BOD <sub>5</sub>	-	1.73	1.78	1.74	0.015
PH	-	6.3	8.9	7.53	0.84

### 2.1. Coagulation

The coagulation process was performed at natural pH of dairy industry wastewater (pH=6.3-8.9) by the jar test apparatus. In this experiments poly aluminum chloride (PAC) was used as a coagulant. The coagulant concentration of 10, 25, 50, 75 or 100 mg was added to 1 L wastewater sample. The operating parameters were adopted as rapid mixing speed 120 rpm for 2 min, slow mixing speed 30 rpm for 15 min and settling time for 30 min. After the settling stage, the supernatant was collected from a point located about 2 cm below the surface and then analyzed. The effluent of coagulation stage was used in Fenton stage.

### 2.2. Fenton

The Fenton process was carried out with 1 L of the supernatant, which obtained from coagulation process. The pH values were adjusted by using concentrated H<sub>2</sub>SO<sub>4</sub> or 1.0M NaOH. Subsequently, Fenton reaction was initiated by the addition of ferrous sulfate (FeSO<sub>4</sub>, 7H<sub>2</sub>O) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 30%) under vigorous stirring at 150 rpm. After this process, the supernatant was analyzed. Potential reaction of hydroxyl radical in the collected effluent was quenched by using 10% Na<sub>2</sub>SO<sub>3</sub>.

## 3. Results and discussion

### 3.1. The effect of coagulant dose on coagulation

Poly aluminum chloride (PAC) was added to desired values and experiments were conducted. The effect of different doses of PAC on the removal of COD and BOD are shown in Figure. 1. The results showed that higher COD and BOD removal were achieved with increasing dose of PAC. Consequently, when the dosage of PAC increased from 25 mg/l to 100 mg/l, the COD and BOD removal increased from 17% to 49.5% and 10.72% to 44.28% respectively. Poly aluminum chloride contains relatively large molecules that can carry the cationic charge to the solution(Can et al., 2006). The isoelectric pH of dairy wastewater effluent is about 4.5-4.6 (Gong et al., 2012; Seesuriyachan et al., 2009). The colloidal proteins presented in dairy wastewater are negatively charged at (pH> pH<sub>iso</sub>) and can be removed by PAC (Kushwaha et al., 2010; Ratnaweera and Selmer-Olsen, 1996). The increasing COD removal efficiency is due to increasing concentration of positively charged species which destabilize the colloidal

particles. Regarding the COD removal efficiency and economic considerations, the concentration of 100 mg was chosen as the optimum concentration of coagulant.

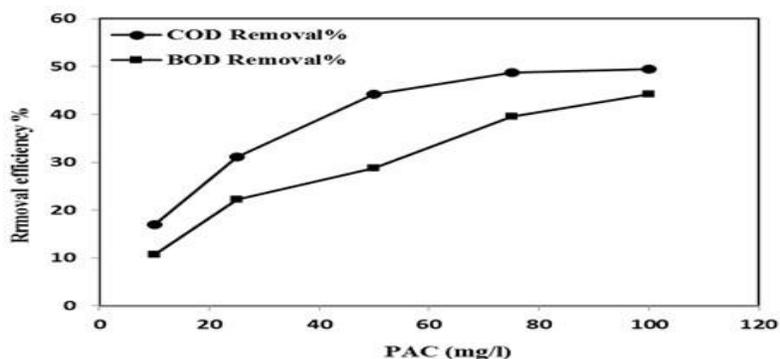


Fig. 1. Effect of different doses of PAC on the removal of COD and BOD.

### 3.2. Fenton process

#### 3.2.1. The effect of pH on Fenton oxidation

The initial pH has been observed to be a significant parameter in Fenton process (Burbano et al., 2005). The experiments were performed in the initial pH range of 2–6 and fixed  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio of 8. The effect of pH on the removal of COD and BOD are shown in Figure 2. As shown in Fig. 2, the oxidation degradation rate increased when the initial pH increased from 2.0 to 3.0, subsequently it kept constant when the initial pH was raised from 3.0 to 5.0, and then dropped off sharply with the increase of pH higher than 5.0. At a pH below 2, oxidation removal decreased due to the formation of complex species  $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$  which reacted slower with  $\text{H}_2\text{O}_2$  (Xu et al., 2004; Farrokhi et al., 2003). In addition, the scavenging effect of  $\text{OH}^\bullet$  by  $\text{H}^+$  is important at lower pH, In the other hand, the lower efficiency at pH above 4 is due to decrease of the dissolved iron species in the solution. Moreover, the oxidation potential of  $\bullet\text{OH}$  radical decreases with increasing pH (Burbano et al., 2005; Xu et al., 2004; Farrokhi et al., 2003; Zhang et al., 2012). The oxidation potential of  $\bullet\text{OH}$  radical is between 2.65–2.80V at pH 3.0 and 1.90V at pH 7.0 (Burbano et al., 2005; Farrokhi et al., 2003). Therefore, the optimum pH for Fenton reaction was 3.0. The maximum removal of COD and BOD at pH 3.0 were obtained 71% and 51% respectively.

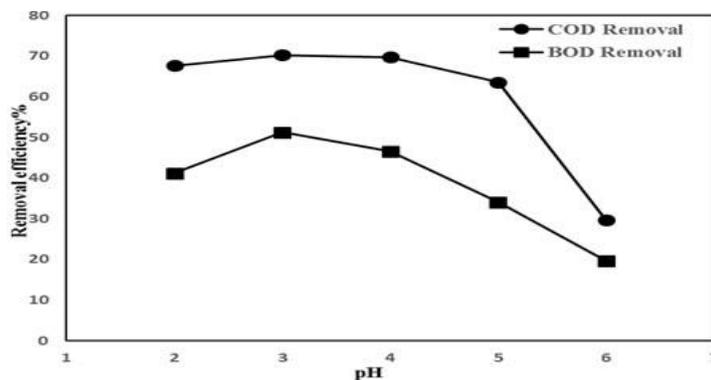


Fig. 2. Effect of pH on the removal of COD and BOD by Fenton process ( $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio=8, reaction time = 120min).

#### 3.2.2. The effect of reaction time on Fenton oxidation

In this experiment, COD and BOD removal were measured at 5, 15, 30, 45, 75 and 120 min. figure3 shows the effect of time on the removal of COD and BOD. The results show that the removal efficiency was increased by the increasing reaction time. The oxidation kinetics is fast until 30 min reaction but then becomes slow. The COD removal efficiency by oxidation achieved 70.79% during the first 30 min reaction at  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio of 8, while the next 90 min reaction resulted in only 7.6% more oxidation removal of COD. The BOD removal efficiency in a reaction times of 30 min and 120 min were 69% and 70% respectively. In the first stage,  $\text{Fe}^{2+}$  reacted very

quickly with  $\text{H}_2\text{O}_2$  to produce large amount of  $\text{OH}^\bullet$  (Eq.1). The organic substances can be degraded by  $\text{OH}^\bullet$  rapidly (Wu et al., 2010; Burbano et al., 2005; Xu et al., 2004). Therefore the first part of reaction is very fast. On the other hand, the main reaction in the second stage is the reaction between hydrogen peroxide and ferric ion (Eq.2) produced in the first stage (Eq.1). However this reaction is very slow ( $K_2 = 0.001\text{--}0.01\text{M}^{-1}\text{S}^{-1}$ ). Moreover, the formed hydroperoxyl radical ( $\text{HO}_2^\bullet$ ) is distinctly less oxidant than  $\text{OH}^\bullet$  (Galano, 2011). Therefore, the oxidation kinetics in second stage is much slower than the first stage (Wu et al., 2010; Burbano et al., 2005; Xu et al., 2004). In this study, the optimal time was 120 minutes.

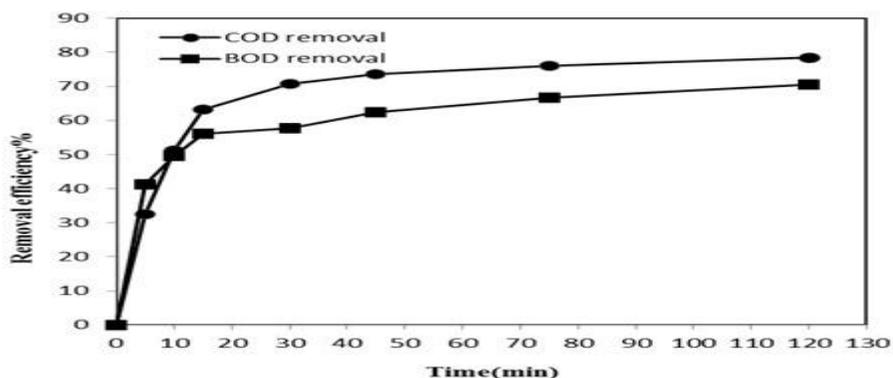


Fig. 3. Effect of Time on the removal of COD and BOD by Fenton process ( $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  molar ratio=8, pH = 3).

### 3.2.3. The effect of H<sub>2</sub>O<sub>2</sub> dosage

The oxidation potentials of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) are approximately 1.80 and 0.87V at pH 0 and 14, respectively (Liu et al., 2013; Xu et al., 2004; Neyens and Baeyens, 2003). However, the hydrogen peroxide is a strong oxidant but its oxidation potential is lower than hydroxyl radical. Therefore, the hydrogen peroxide is used alone cannot be effective in some oxidation processes (Xu et al., 2004). Since the main cost of Fenton oxidation process is due to  $\text{H}_2\text{O}_2$  consumption, it is important to optimize the amount of  $\text{H}_2\text{O}_2$  in the Fenton's oxidation process (Kabita et al., 2001; Wu et al., 2010). The process was performed in various dosages of  $\text{H}_2\text{O}_2$  at a fixed amount of 100mg/l ferrous. The pH was controlled at 3 and reaction time was 120min. As shown in Fig.4,  $\text{H}_2\text{O}_2$  doses significantly influenced the removal efficiency of COD and BOD. It was observed that at the  $\text{H}_2\text{O}_2$  doses 700 mg/l, the removal efficiency of COD and BOD was 79.56% and 74.8% respectively. When  $\text{H}_2\text{O}_2$  dosage was increased from 700 mg/l to 900mg/l, the COD and BOD removal decreased from 79.56% to 75.73% and 74.8% to 64.35% respectively. This is due to scavenging effect of  $\bullet\text{OH}$  by  $\text{H}_2\text{O}_2$  and generation of hydroperoxyl radical ( $\text{HO}_2^\bullet$ ) [Eq.3] (Kabita et al., 2001; Wu et al., 2010; Xu et al., 2004; Riaño et al., 2014). Therefore, the optimum dosage of  $\text{H}_2\text{O}_2$  was 700mg/l.

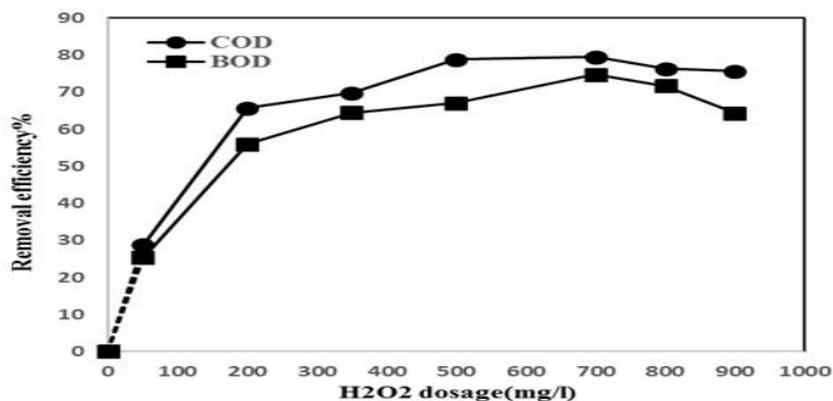


Fig. 4. Effect of H<sub>2</sub>O<sub>2</sub> dosage on the removal of COD and BOD by Fenton process ( $\text{Fe}^{2+}$  dosage=100mg/l, pH=3, reaction time = 120min).

### 3.2.4. The effect of Fe<sup>2+</sup> dosage

Figure.5 shows the effects of Fe<sup>2+</sup> dosage on COD and BOD removal efficiency. Fe<sup>2+</sup> is the main species that can catalyze H<sub>2</sub>O<sub>2</sub> to produce •OH (Wu et al., 2010). The removal efficiency of COD and BOD were increased by increasing Fe<sup>2+</sup> concentration from 50mg/l to 150mg/l. However, higher concentration of Fe<sup>2+</sup> did not improve the reaction efficiency. The Fe<sup>2+</sup> can act as hydroxyl radical scavengers if not in the optimal dose, as shown in [Eq.4] (Xu et al., 2004; Riaño et al., 2014; Oturan et al., 2011). In addition, the sludge volume may be increased by increasing ferrous ions (Deng and Englehardt, 2006; Kavitha and Palanivelu, 2004). The maximum COD and BOD removal efficiency at H<sub>2</sub>O<sub>2</sub> dose of 700mg/l and Fe<sup>2+</sup> dose of 150mg/l were achieved 84.37% and 76.02% respectively. The Fe<sup>2+</sup> concentration of 150 mg was chosen as the optimum concentration of catalyst.

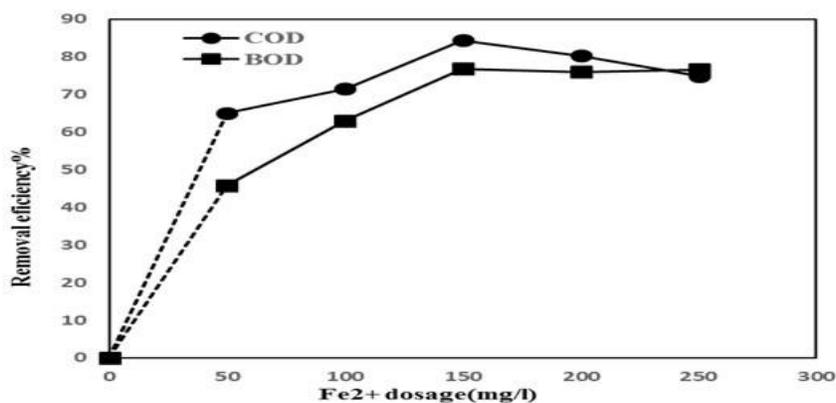


Fig. 5. Effect Fe<sup>2+</sup> dosage on the removal of COD and BOD by Fenton process (H<sub>2</sub>O<sub>2</sub> dosage=700mg/l, pH=3, reaction time = 120min).

## 4. Conclusion

Treatment of the dairy wastewater by combined chemical coagulation and Fenton oxidation process was performed in this study. In the first stage, poly aluminum chloride (PAC) was used as coagulant and optimum dose of coagulant was determined. After that, Fenton oxidation process was performed, on the effluent of coagulation process. In Fenton process, the influence of pH, reaction time, Fe<sup>2+</sup> and hydrogen peroxide on the removal efficiency of COD and BOD from dairy wastewater were studied, and subsequently the optimal values for each parameter were determined. Under optimum condition (dose of PAC=100mg/l, H<sub>2</sub>O<sub>2</sub>=700 mg/l, Fe<sup>2+</sup> =150mg/l, pH= 3, 120min reaction time and room temperature) the overall COD and BOD removal by combined chemical coagulation and Fenton's reaction was reached 91.5% and 86.22% respectively. Therefore, this method can be used as effective method for dairy wastewater treatment.

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