Acoustic cavitation Coupled with Advance Oxidation Process for Treatment of Dairy Industry Wastewater

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ABSTRACT: - *This paper investigates the reduction of COD of dairy industry waste water by individual and combined process of acoustic cavitation,* H_2O_2*, Fenton and Photo Fenton process. All experiments were performed on a laboratory scale setup. The effect various parameters such as sonication power, duty cycle, initial pH, hydrogen peroxide concentrations and Fenton reagents on the reduction of COD of dairy waste water have been assessed. Effective system conditions were found to be sonication power of 800W, duty cycle of 60 %, pH of 3, 200 µl/L of hydrogen peroxide concentration and 78 mg/L of Fenton reagents. The results show that the % of COD reduction of dairy industry waste water after 180 min reaction time follows the decreasing order: Acoustic Cavitation + Photo Fenton Process (88.78 %) > Acoustic Cavitation + Fenton Process (66.22 %) > Acoustic Cavitation +* H_2O_2 *(40.02 %) > Acoustic Cavitation (13.53 %).*

KEYWORDS:- *Dairy Industry Waste Water, Acoustic Cavitation, Acoustic Cavitation +* H_2O_2*, Acoustic Cavitation + Fenton Process, Acoustic Cavitation + Photo Fenton Process, Chemical Oxygen Demand (COD).*

1. INTRODUCTION

Among the food industries, the dairy industry is the most polluting in volume in regards to its large water consumption. Water is used throughout all steps of the dairy industry including cleaning, sanitization, heating, cooling and floor washing and naturally the requirement of water is massive [1].In the dairy industry, starting, equilibrating, interrupting and stopping any of the processing units generate large volumes of effluent [2].Dairy wastewater generally does not contain conventional toxic chemicals like those listed under EPA's Toxic Release Inventory. However, it has high concentration of dissolved organic components like fats, oils and grease, nutrients such as ammonia or minerals and phosphates and therefore require proper attention before disposal[3].

In Advance Oxidation Processes (AOPs) oxidation is based on intermediate reactions in which the hydroxyl radical ($HO^{\bullet}$) is present. These processes are able to degrade a large number of organic compounds by reduction–oxidation and free-radical reactions to carbon dioxide (CO_2) and water (H_2O).Advanced oxidation processes (AOP) such as Acoustic cavitation, Fenton and Photo Fenton process are useful for the treatment of high initial concentration of COD [4].The sonochemical effect is cavitational phenomena based on ultrasound interaction with aqueous liquid medium, when ultrasound passed through the liquid medium cycles of compression and rarefaction are developed. These cycles induce the cavitational bubble which expands and compresses before the collapse at million locations in the reactor. The higher temperature (4000–5000 K) and pressure (1000–50,000 bars) conditions reach inside the bubble cavity before collapsing. These conditions are highly useful for enhancing the rate of chemical processing. During cavitation cycle, reactions are occurring at the three zones: (a) inside the cavity of the bubble, (b) at the interface of gas–liquid, and (c) radicals generated after the collapse of the cavity [5,6]**.** The application of ultrasound alone is not efficient for the degradation of the target organic pollutant because of the fact that the ultrasound alone requires more time and high amount of energy for an acceptable degradation [7,8].

When applied individually cavitation often gives lower rates of the degradation, but the efficiency of cavitation can be significantly enhanced by combining it with other advanced oxidation processes (AOP) such as Fenton [9,10] and Photo Fenton [11,12]. Combination of acoustic cavitation with other AOPs often leads to an intensification of the degradation of organic pollutants due to the enhanced generation of the hydroxyl radicals [13].

The present work deals with the study of reduction of COD of dairy industry waste water by using Acoustic cavitation and Acoustic cavitation based hybrid techniques such as Acoustic cavitation + Fenton and Acoustic cavitation + Photo Fenton process and also to study the effect of key operating conditions on the reduction of COD of dairy industry waste water.

2. MATERIALS AND METHODS

2.1 Materials

Raw wastewater was collected from local dairy industry, Jalgaon, Maharashtra, India at an interval of 15 days and stored in the refrigerator at 4^oC, in order to maintain its characteristics throughout the research.

2.2 Chemicals and reagents

Hydrogen peroxide (30% W/V H_2O_2), Ferrous sulfate heptahydrate ($FeSO_4 .7H_2O$), Sulfuric acid (H_2SO_4) and Sodium hydroxide (NaOH) etc. of S. D. fine were purchased from the local supplier, Jalgaon, India. All reagents and chemical were used as received from vendors without any purification or further treatment.

2.3. Methods

2.3.1 Characterization of dairy industry waste water

pH, Turbidity, Conductivity, Hardness, COD, BOD, TDS and TSS of dairy industry waste water were determined using APHA, Standard Methods for the Examination of Water and Waste-Water [14].

2.3.2 Cavitation Reactor

The cavitation reactor (Figure 1) used in the present study was an ultrasonic horn manufactured by Johnson Plastosonic Pvt. Ltd., Pune. Ultrasonic processor was operated at a constant frequency of 20 kHz with maximum power 800W. The height of the ultrasonic horn was adjusted using metal stand in such way that the tip of the probe was immersed 2 cm inside the liquid. The source of UV radiations were four UV lamps emitting light of 8 Watt at wavelength 254 nm, manufactured by Phillips Company. UV lamps were placed parallel to the each other at the top of photolytic reactor. UV lamps were cooled with fan incorporated in setup. At the beginning of the treatment, 400 ml of dairy industry waste water taken in a glass beaker was put up on the stand to adjust height of the beaker and then ultrasonic processer and UV light was immediately turned on, that accounted for time = 0; i.e. the start of the reaction. The effect of different process parameters like sonication power (200, 400, 600 and 800 Watts), duty cycle (80%, 60%, 40% and 20%), pH (3, 6.5, and 9), H_2O_2 loading (50, 100, 150, 200 and 150 µl/L) and Fenton reagent ($FeSO_4 .7H_2O$ at optimum H_2O_2 concentration) loading (26, 39, 52, 65 and 78 mg/L) were determined at constant operating temperature of 28 ± 3 °C and 20 kHz constant sonication frequency.

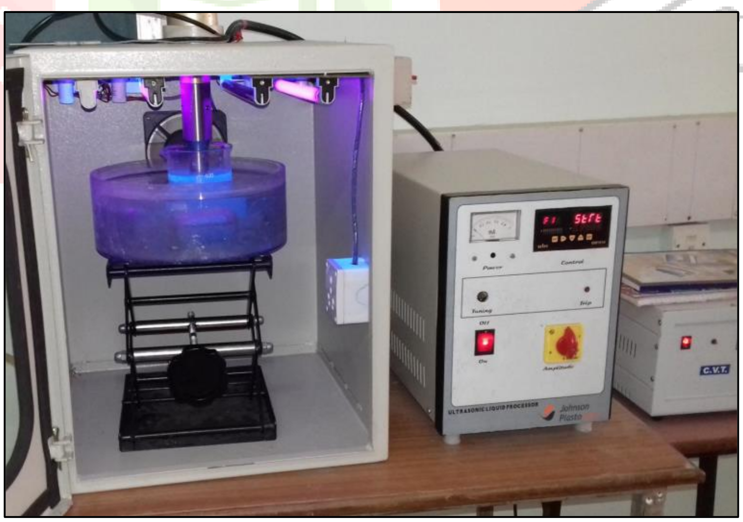

Figure 1. Experimental Setup of Acoustic cavitation

2.3.3. Analysis

Required amount of sample dairy industry waste water were taken from the reactors at regular time intervals and analyzed for Chemical Oxygen Demand (COD). It was determined by APHA, Standard Methods for the Examination of Water and Waste-water [14].

3. RESULTS AND DISCUSSION

3.1 Characterization of dairy industry waste water

The main characteristics of the dairy industry effluent used in this work were presented in Table 1.

Table 1. Characteristics of the dairy industry effluent

Sr. No.	Parameter	Values
1	pH	6.5
2	Turbidity	12 NTU
3	Hardness	640 ppm
4	COD	900 ± 50 mg/L
5	BOD	580 mg/L
6	TDS	1922 mg/L
7	TSS	651 mg

3.2 Calculation of Percentage of Reduction of COD

The percentage of reduction of COD of dairy industry waste water was calculated according to equation.

$$\%\ \boldsymbol{of\ COD\ Reduction} = [\frac{(\boldsymbol{COD0 - CODt})}{\boldsymbol{COD0}}] \times \mathbf{100}$$

COD0: initial COD of the dairy industry waste water (mg/l)

CODt: COD of the dairy industry waste water after time ‘t’ (mg/l)

3.3 Acoustic Cavitation

3.3.1. Effect of sonication power on the reduction of COD of dairy industry waste water

The effect of sonication power on reduction of COD was studied at different operating powers (200, 400, 600 and 800 Watts) with 400 ml of dairy industry waste water, pH 6.5, 60 % duty cycle, at constant operating temperature of 28 ± 3 °C and 20 kHz sonication frequency. Sonication was carried out for 180 minutes. Figure 2 shows the effect of sonication power on reduction of COD of dairy industry waste water at different operating powers (200, 400, 600 and 800W) using acoustic cavitation.

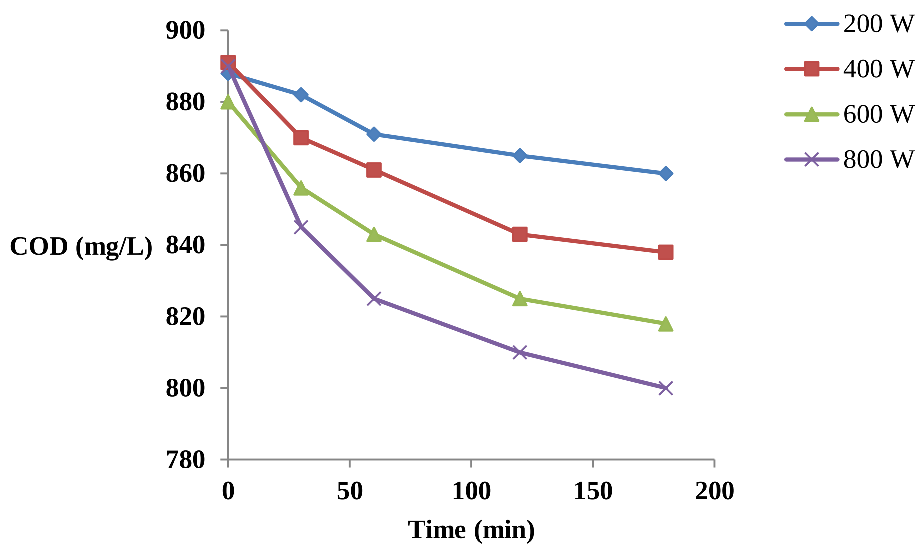

Figure 2. Effect of sonication power on the reduction of COD of dairy industry waste water

It has been observed that the reduction of COD of dairy industry waste water increases with an increase in the operating power. The maximum COD reduction was observed at power dissipation of 800 W whereas minimum COD reduction was observed at power dissipation level of 200 W. The increase in % of COD reduction of dairy industry waste water with increasing power can be due to the enhancement in the number of cavities leading to higher cavitation activity and hence enhanced production of the hydroxyl radicals [15].

Table 2 shows the effect of sonication power on the % of COD reduction of dairy industry waste water using acoustic cavitation. It has been observed that as sonication power is increased from 200 W to 800 W and % of COD reduction of dairy industry waste water increases from 3.15 % to 10.11 %. Thus 800 W sonication power was considered as optimum for further studies.

Table 2.Effect of sonication power on the % of COD reduction of dairy industry waste water

Process	Power (Watts)	% of COD Reduction
Acoustic Cavitation	200	3.15
	400	5.95
	600	7.50
	800	10.11

3.3.2. Effect of duty cycle on the reduction of COD of dairy industry waste water

The effect of different duty cycles (20%, 40%, 66% and 80%) on the treatment of dairy industry wastewater was investigated by varying ON and OFF time of ultrasonic irradiation.These experiments were conducted for 400 ml dairy industry waste water, sonication power 800 W, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time.Figure 3 shows the effect of duty cycle on reduction of COD of dairy industry waste water.

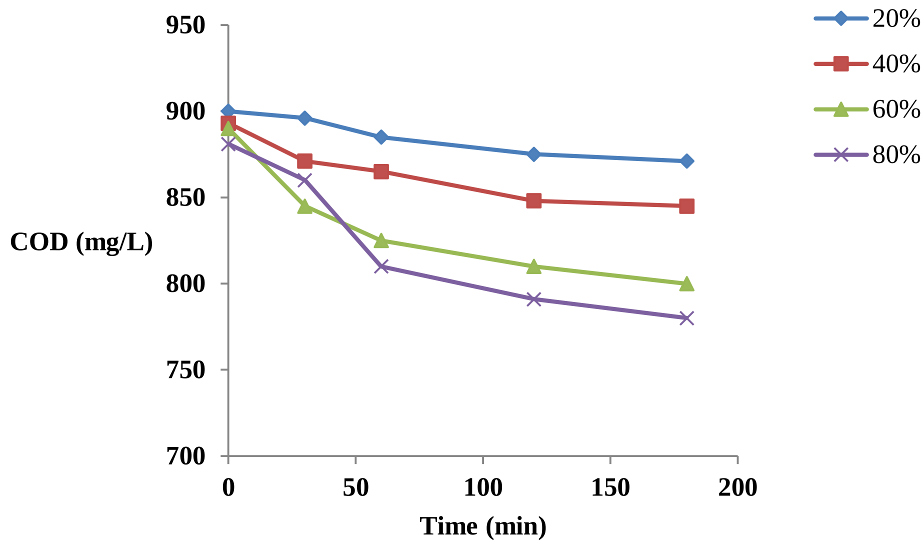

Figure 3. Effect of duty cycle on the reduction of COD of dairy industry waste water

Table 3 shows that the % of COD reduction of dairy industry wastewater increased from 3.22% to 11.46% with an increase in duty cycle from 20% to 80%. The increase in % of COD reduction of dairy waste waterdue to increase inthe time of exposure to the sound wave more is attributed to the bubble formation and implosion more is the reduction. But, operating the ultrasonicator for long period of time (ON time) is not recommended due to excessive heating and also it may creates problem to transducers [16,17]. Additionally, only marginal increase of % COD of reduction of dairy waste water was observed at 80% duty cycle and thus accordingly 60% duty cycle was set as optimum duty cycle.

Table 2.Effect of duty cycle on the % of COD reduction of dairy industry waste water

Process	Duty cycle (%)	% of COD Reduction
Acoustic Cavitation	20	3.22
	40	5.38
	60	10.11
	80	11.46

3.3.3. The effect of pH on the COD reduction of dairy industry waste water

Initial pH is an important parameter influencing the treatment efficiency waste water. In this study, in order to investigate the effects of pH on reduction of COD of dairy industry waste water using acoustic cavitation, experiments were conducted at different pH values 3, 6.5 and 9. These experiments were conducted for 400 ml dairy industry waste water, sonication power 800 W, 60 % duty cycles, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. Figure 3 shows the effect of pH on the reduction of COD of dairy industry waste water using acoustic cavitation.

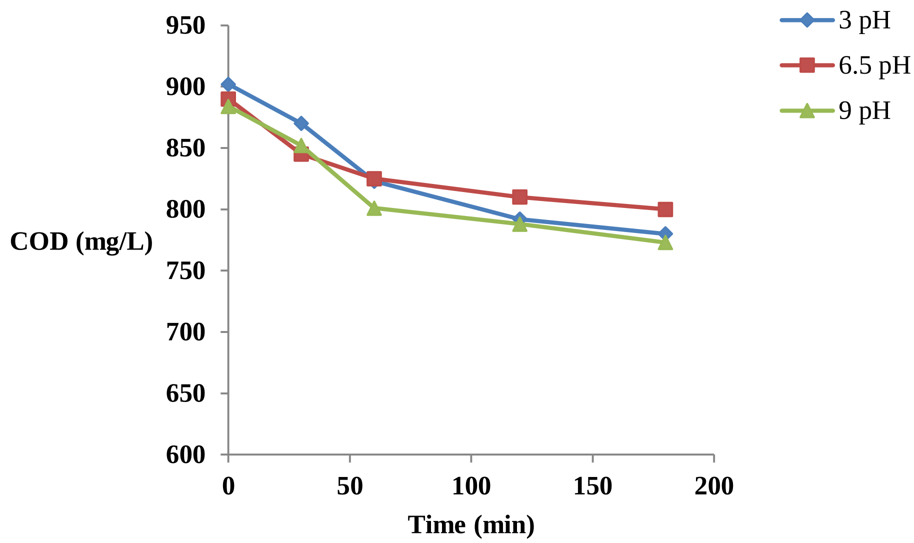

Figure 3. Effect of pH on the reduction of COD of dairy industry waste water

Table 3 shows that the % of COD reduction of dairy industry waste water is very rapid under acidic pH 3, 13.53 % reduction of COD of dairy industry waste water was obtained. At lower pH (acidic), the oxidation capacity of hydroxyl radicals is high and generates the maximum amount of hydroxyl radicalto oxidize organic compounds [18].The value of pH is one of the crucial factors affecting the performance of the Fenton-like process.The activity of $Fe(OH)^+$ in Fenton's and photo- Fenton's reaction and the decomposition of H_2O_2 in acidic medium was found to be very fast in producing hydroxyl radical ($OH^\bullet$) [19,20].Thus pH 3 was considered as optimum pH for further studies.

Table 3. Effect of pH on the % of COD reduction of dairy industry waste water

Process	pH	% of COD Reduction
Acoustic Cavitation	3	13.53
	6.5	10.11
	9	12.56

3.4 Acoustic cavitation + H_2O_2

3.4.1 Effect of H_2O_2 on the reduction of COD of dairy industry waste water

The amount of H_2O_2 is considered as one of the most important factors in the Fenton's and photo-Fenton's Process. To increase the generation of free radicals, the combination of ultrasound with hydrogen peroxide looks to be a promising option. The concentration of hydrogen peroxide plays a crucial role in deciding the extent of degradation obtained for the combined process. On one side where it acts as a source of free radicals by the dissociation process, also it acts as the scavenger of the generated free radicals, as indicated by the following reaction scheme:

$$H_2O_2 \rightarrow 2OH^{\bullet}$$

$$OH^{\bullet} + H_2O_2 \rightarrow H_2O + O_2H^{\bullet}$$

The effect of addition of H_2O_2(50 to 250µl/L) on the reduction of COD of dairy industry waste water is shown in Figure 4. These experiments were conducted for 400 ml dairy industry waste water, sonication power 800W, 60 % duty cycle, 3 pH, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. In Acoustic cavitation + H_2O_2 process, the addition of H_2O_2 from 50 to 200µl/L increases the reduction of COD from 27.40 % to 40.02 % for 180 minutes. Further increase from 200 to 250µl/L, decrease in reduction of COD from 40.02% to 37.53%.

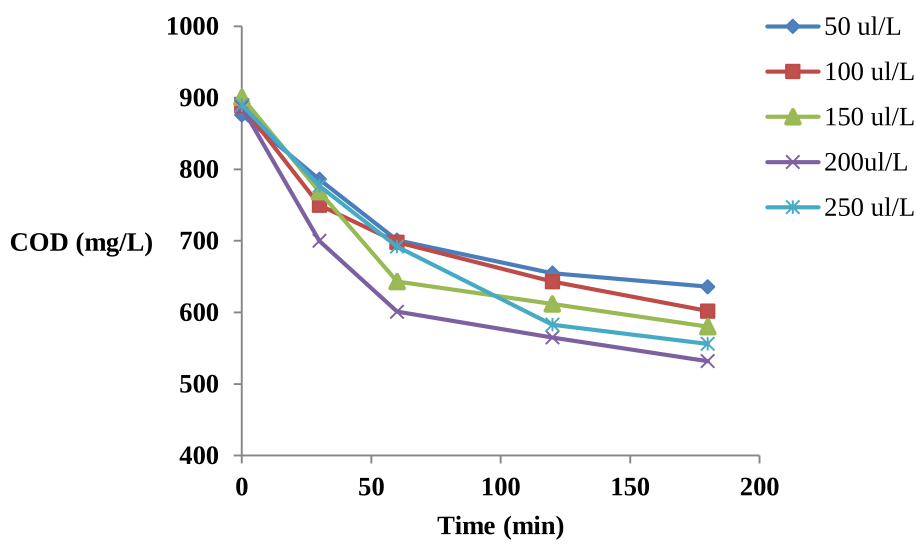

Figure 4. Effect of H_2O_2 on the reduction of COD of dairy industry waste water

The increase in the reduction of COD is due to the increase in hydroxyl radical concentration by the addition of H_2O_2. This adverse effect after optimum concentration is due to scavenging effect. On increasing concentration of H_2O_2 rate of hydroxyl radical also increases but beyond certain concentration some of H_2O_2 remains in excess. This excess H_2O_2 recombines with $OH^{\bullet}$ radicals and form H_2O and $O_2H^{\bullet}$ radical that has low oxidation potential compared to that of $OH^{\bullet}$ radical, which results into decrease in rate of degradation [21,22].

Table 4 shows the effect of H_2O_2on % of COD reduction of dairy industry waste water. It has been observed that 13.53% and 2.89% reduction of COD was obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively. Further the COD reduction was significantly increased to 40.02% and 6.11% by applying the combination of Acoustic Cavitation with H_2O_2 and Aeration with H_2O_2 respectively.

Table 4.Effect of H_2O_2 on the % of COD reduction of dairy industry waste water

Process	H_2O_2 (μl/L)	% of COD Reduction
Acoustic Cavitation	-	13.53
Aeration	-	2.89
Aeration + H_2O_2	200	6.11
	50	27.40
	100	32.28
Acoustic Cavitation + H_2O_2	150	35.63
	200	40.02
	250	37.53

3.5 Acoustic Cavitation + Fenton Process

3.5.1 Effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water

Fenton process utilizes the reactivity of hydroxyl radicals generated in acidic conditions by iron catalyzed decomposition of hydrogen peroxide for the degradation of organic pollutant.Amount of ferrous ion is one of the main parameters to influence the Fenton process. More hydroxyl radicals are produced with the increase in the concentration of ferrous ions (Fe^{2+}) [24]. Effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water has been investigated for various concentration of Ferrous sulfate heptahydrate ($FeSO_4 .7H_2O$) in the presence of H_2O_2 (200μl/L) and Acoustic Cavitation (800W). Figure 5 shows the effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water**.** These experiments were conducted for 400 ml dairy industry waste water, sonication power 800W, 60% duty cycle, 3 pH, 200μl/L of optimum H_2O_2 loading, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. It has been observed that the reduction of COD of dairy industry waste water increases with an increase in the loading of $FeSO_4.7H_2O$ from 26mg/L to 78mg/L under acoustic cavitation with optimum concentration of hydrogen peroxide(200μl/L). The reduction of COD is faster in the early stage of the reaction than in the later stage. Most of the hydrogen peroxide dose was consumed in the early stage of the Fenton reaction. Since ferrousion catalyzes hydrogen peroxide to form hydroxyl radical quickly in the first stage of reaction, more reduction occurs in the early stage of reaction [25]**.** Fenton reaction can be described using equation,

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet}$$

Resulting Fe^{3+} ions can react with H_2O_2 to produce the intermediate complex $Fe\text{-}O_2OH^{2+}$, which can easily get converted into Fe^{2+} and $HO_2^\bullet$ under cavitation.

$$Fe^{3+} + H_2O_2 \rightarrow Fe\text{-}O_2OH^{2+} + H^+$$

$$Fe\text{-}O_2OH^{2+} \rightarrow Fe^{2+} + HO_2^\bullet$$

Generated Fe^{2+} ions can again react with H_2O_2 to generate even more number of hydroxyl radicals. In addition to this, some part of H_2O_2 directly decomposes to hydroxyl radicals in presence of acoustic cavitation as shown in below,

$$H_2O_2 \rightarrow OH^\bullet + OH^\bullet$$

Hence, the combination of Acoustic cavitation and Fenton process accelerates the rate of generation of hydroxyl radicals [25-27].

Table 5 shows the effect of $FeSO_4.7H_2O$ on % of COD reduction of dairy industry waste water. It has been observed that 13.53% and 2.89% reduction of COD obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively which significantly increased to 66.22% and 29.12% by applying the combination of Acoustic Cavitation with Fenton Process and Aeration with Fenton Process respectively.

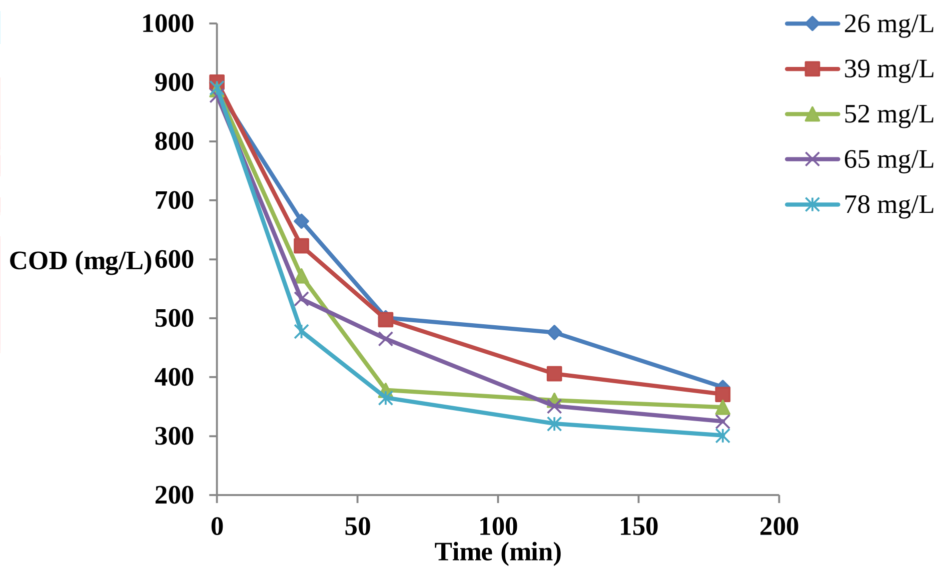

Figure 5. Effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water

Table 5. Effect of $FeSO_4.7H_2O$ on the % of COD reduction of dairy industry waste water

Process	$FeSO_4$ $.7H_2O$(mg/L)	% of COD Reduction
Acoustic Cavitation	-	13.53
Aeration	-	2.89
Aeration +Fenton process	78	29.12

	26	56.97
Acoustic Cavitation +	39	58.82
Fenton process	52	60.70
	65	62.98
	78	66.22

3.6 Acoustic Cavitation + Photo Fenton Process (Sonophotofenton Process)

3.6.1 Effect of $FeSO_4.7\ H_2O$ on the reduction of COD of dairy industry waste water

The reduction of COD of dairy industry waste water was investigated using differentconcentration of $FeSO_4$ $.7H_2O$ ranging from 26mg/L to 78mg/L. Four 8 W low-pressure mercury UV lamps (Philips) were used at 254 wavelengths for UV irradiation and ultrasonic processer for cavitation. These experiment conducted for 400 ml dairy industry waste water, sonication power 800W, 60% duty cycle, 3 pH, 200µl/L of optimum H_2O_2 loading, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time.

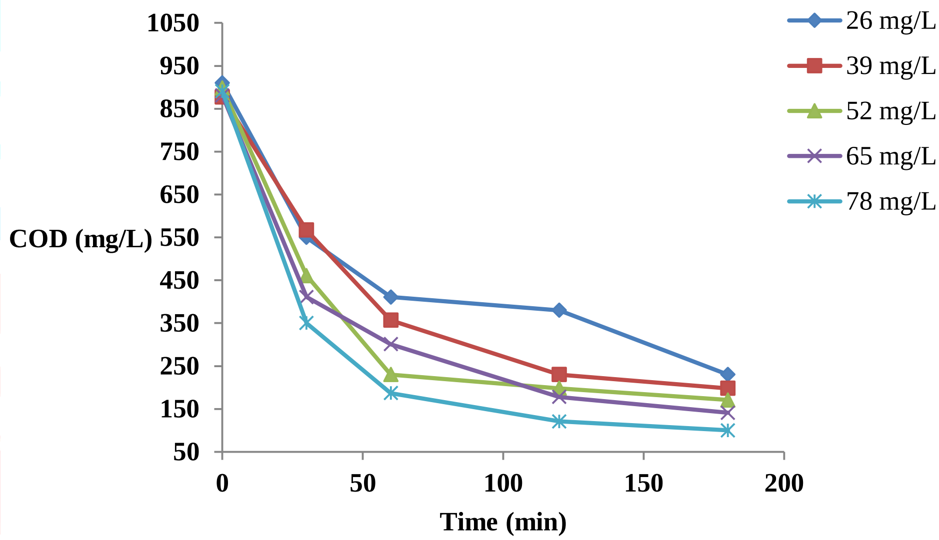

Figure 6. Effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water

Figure 6 shows the effect of $FeSO_4.7H_2O$ on the reduction of COD of dairy industry waste water**.** It has been observed that the reduction of COD of dairy industry waste water increases with increase in the concentration of $FeSO_4$ $.7H_2O$ under Acoustic Cavitation + Photo Fenton process with optimum concentration of H_2O_2 (200µl/L). In Fenton process ferrous salts react with hydrogen peroxide to generate the hydroxyl radicals as follows

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{-} + OH^{\bullet}$$

The rate of degradation can be considerably increased via photochemical reaction in the photo-Fenton's process. In this case, the regeneration of catalyst i.e. Fe^{2+} ions, with production of new $OH^{\bullet}$ radicals from Fe^{3+} / $Fe(OH)^{2+}$ by UV irradiation take place [25,26].

$$Fe^{3+} + H_2O \rightarrow Fe(OH)^{2+} + H^+$$

$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + OH^{\bullet}$$

Also hydrogen peroxide readily decomposes into hydroxyl radicals in the presence of acoustic cavitation and UV lights [27].

$$H_2O_2 + \text{US/UV Lights} \rightarrow 2OH^{\bullet}$$

Table 6 shows the effect of $FeSO_4.7H_2O$ on % of COD reduction of dairy industry waste water. It has been observed that 13.53% and 2.89% reduction of COD obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively which significantly increased to 88.78% and 48.89% for the combination of Acoustic Cavitation with Photo Fenton Process and Aeration with Photo Fenton Process.

Process	$FeSO_4.7H_2O$ (mg/L)	% of COD Reduction
Acoustic Cavitation	-	13.53
Aeration	-	2.89
Aeration + Photo Fenton process	78	48.89
Acoustic Cavitation + Photo Fenton process	26	74.73
	39	77.42
	52	80.96
	65	84.00
	78	88.78

4. CONCLUSIONS

The present study has shown the efficiency of acoustic cavitation for the reduction of COD of dairy industry waste water. Combined methods were found to be more efficient as compared to an individual process. The main conclusions drawn from present study can be summarized as follows:

1. The percentage of reduction of COD of dairy industry waste water was influenced by sonication power, duty cycle and solution pH using acoustic cavitation. Maximum percentage of COD reduction was 10.11% for sonication power of 800W, 11.46% for 80% duty cycle and 13.53% pH 3 respectively.
2. It was observed that acoustic cavitation coupled with H_2O_2 enhances the percentage of COD reduction of dairy industry waste water, giving 40.02% reduction of COD at an optimum concentration of H_2O_2 (200µl/L) as compared 13.53% obtained using acoustic cavitation alone.

3. The combination of Fenton process with acoustic cavitation gives 66.22% reduction of COD of dairy industry waste water at optimum concentration of $FeSO_4.7H_2O$ (78mg/L) as compared 40.02% and 13.53% reduction of COD obtained using acoustic cavitation + H_2O_2 and acoustic cavitation alone.
4. The combination of acoustic cavitation and Photo Fenton process gives 88.78% maximum reduction of COD of dairy industry waste water at optimum concentration of $FeSO_4.7H_2O$ (78mg/L) as compared 66.22%, 40.02% and 13.53% reduction of COD for acoustic cavitation + Fenton process, acoustic cavitation + H_2O_2 and acoustic cavitation alone.

Overall, the obtained results revealed that the combination of Acoustic Cavitation with Photo Fenton process can be a better method to maximum reduction of COD of dairy industry waste water.

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