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Reduction of COD of Textile Industry Waste Water by Using Acoustic Cavitation Coupled with Advanced Oxidation Processes

Yogendra D. Thakare[1], Kishor S. Wani[2]
[1]Research Scholar, SSBT's College of Engineering & Technology, Bambhori, Jalgaon, MS, India
[2]Professor and Principal, SSBT's College of Engineering & Technology, Bambhori, Jalgaon, MS, India

***Abstract:** The textile and dye industries require ahuge amount of water for processing and produce a large volume of wastewater. Generated wastewater had potential hazards and a threat to the aquatic life.In the present study, reduction of chemical oxygen demand (COD) of textile industry waste water has been carried out using acoustic cavitation, acoustic cavitation + H_2O_2, acoustic cavitation + Fenton process and acoustic cavitation + Photo Fenton process. The process parameters such as sonication power, duty cycle, solution pH, loading of H_2O_2 and loading of $FeSO_4 .7H_2O$ were investigated in detail to evaluate their effects on the reduction of COD of textile industry waste water. Effective optimum conditions were found to be 800W of sonication power, 60 % duty cycle, pH of 3, loading of H_2O_2, 75 µl/Land loading of $FeSO_4.7 H_2O$, 65 mg/L. The results showed that the percentage of COD reduction of textile industry waste water was maximumfor Acoustic cavitation + Photo Fenton process as compared to Acoustic cavitation + Fenton process, acoustic cavitation + H_2O_2 and the conventional processes. It was also observed that percentage of COD reduction of textile industry waste water was more effective in acoustic cavitation based combined techniques as compared to individual process.*

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

I. INTRODUCTION

Textile and dye industries are considered as the second largest water consuming industries among all industries. The high amount of water was consumed during the processing, finishing and cleaning operations in textile industry[1, 2]. In textile industries, the massive amounts of wastewater are generated during production and application of dye. Generated wastewater has high initial chemical oxygen demand (COD) and is a major threat to ecology and aquatic life[3, 4]. From an environmental point of view, the discharge of textile wastewaters into the aqueous environments can obstruct light penetration, preventing the photosynthesis phenomenon. In general, wastewaters generated by the textile industries are characterized with the presence of low biodegradable organic dyes, which are not suitable for conventional biological wastewater treatment technologies. Conventional biological methods have limitations to treat the high level of COD [5-7]. Advanced oxidation processes (AOP) were found to be more efficient for the treatment of elevated levels of COD[7-8].Advanced oxidation processes (AOP) such as Acoustic cavitation, Fenton and Photo Fenton process are useful for the treatment of high initial concentration of COD [9]. 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 induced 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 [10-13].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 [14-15].

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 [15,16]and Photo Fenton [17,18]. 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 [19].

In recent year, attention has been focused on photochemical advanced oxidation processes using Fenton reagent with UV light for the treatment of wastewater. Fenton reagent had been found to be effective in degrading dye pollutants [20-23]. The oxidation power of Fenton reagent is due to the generation of hydroxyl radical ($OH^{\bullet}$) during the iron catalyzed decomposition of hydrogen peroxide in acid medium [24].The hydroxyl radical with a high oxidation potential attacks and completely destroys the pollutants in Fenton process. The degradation of pollutants can be considerably improved by using UV-radiation. This is due to the generation of additional hydroxyl radicals [25,26]. This Photo Fenton process had been effectively used to degrade the pollutants.

Various AOPs such as acoustic cavitation [28], H_2O_2[29,30], Fenton [4,29,31], Photo Fenton [31], Photolytic (UV) [30,32], Photolytic with H_2O_2 (UV + H_2O_2) [33], Photo catalytic [34-36], Electrochemical [37]and Electro Fenton [38]degradation processes have been reported in the literature for the degradation of synthetic textile industry waste water, not studied for real textile industry waste water. However, the use of acoustic cavitation combination of Fenton and Photo Fenton process for the treatment of textile industry waste water has not yet been explored. The present work deals with the study of reduction of COD of textile industry waste water by using of Acoustic cavitation and Acoustic cavitation based hybrid techniques such as Acoustic cavitation + Fenton and Acoustic cavitation + Photo Fenton process and observe the effect of key operating conditions on the reduction of COD of textile industry waste water.

II. MATERIALS AND METHODS

1) *Materials:* Raw wastewater was collected from Textile Industry, Burhanpur, Madhya Pradesh, India at an interval of 15 days and stored in the refrigerator, in order to maintain their physical and chemical characteristics throughout the research.
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 venders without any purification or further treatment.
3) *Methods*

A. Characterization of textile industry waste water

pH, Turbidity, conductivity, hardness, COD, BOD, TDS and TSS of textile industry waste water were determined using APHA, Standard Methods for the Examination of Water and Wastewater, 2005 **[39]**. The main characteristics of the textile industry effluent used in this work were presented in **Table 1**.

Table 1. Characteristics of the textile industry effluent

Sr. No.	Parameter	Values
1	pH	7.5
2	Turbidity	11 NTU
3	Conductivity	3.0
4	Hardness	640 ppm
5	COD	750 ± 50 mg/L
6	BOD	680 mg/L
7	TDS	2362.25 mg/L
8	TSS	40 mg/L

B. Cavitation Reactor

The cavitation reactor 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 UV radiation is 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 textile industry waste water was taken in glass beaker; put it 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. Different process parameter like effect of sonication power (200, 400, 600 and 800 Watts), duty cycle (20%, 40%, 60%, and 80%), pH (3, 7.5, and 9), H_2O_2 loading (25, 50, 75, 100 and 150 µl/L) and Fenton reagent ($FeSO_4$. 7H_2O at optimum H_2O_2 concentration) loading (13, 26, 39, 52 and 65 mg/L) were determined at constant operating temperature of 28 ± 3 °C and 20 kHz constant sonication frequency.

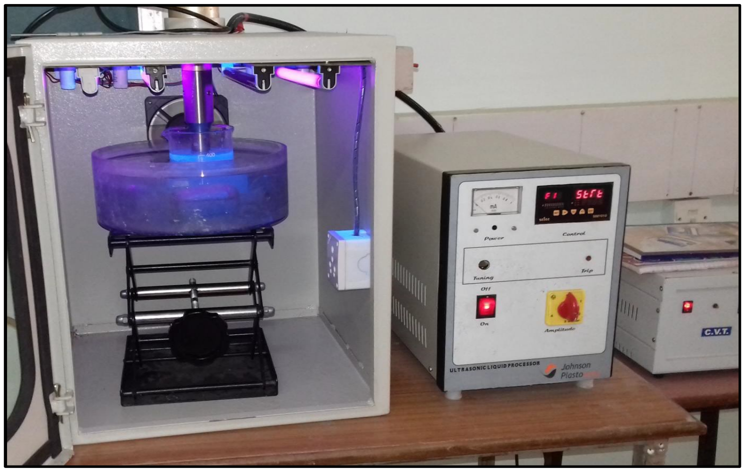

Figure 1.Experimental Setup of Acoustic cavitation

C. Analysis

Required amount of sample textile 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 Wastewater [39].

III. RESULT AND DISCUSSION

A. Calculation of Percentage of Reduction of COD:-

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

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

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

CODt: COD of the textile industry waste water after time 't' (mg/l)

B. Acoustic Cavitation

1) *Effect of sonication power on the reduction of COD of textile industry waste water:* The effect of sonication power on reduction of COD was studied at different operating powers (200, 400, 600 and 800W) with 400ml of textile industry waste water, 60% duty cycle, pH 7.5, at constant operating temperature of 28 ± 3 °C and 20 kHz sonication frequency. Sonication was carried out for 180 minutes. Figure 2shows the effect of sonication power on reduction of CODat different operating powers (200, 400, 600 and 800W) using acoustic cavitation. It has been observed that the reduction of COD of textile industry waste water increases with an increase in the operating power. The maximum COD reduction was observed at power dissipation of 800W whereas minimum COD reduction was observed at power dissipation level of 200W. The increase in % of COD reduction of textile 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 [40].

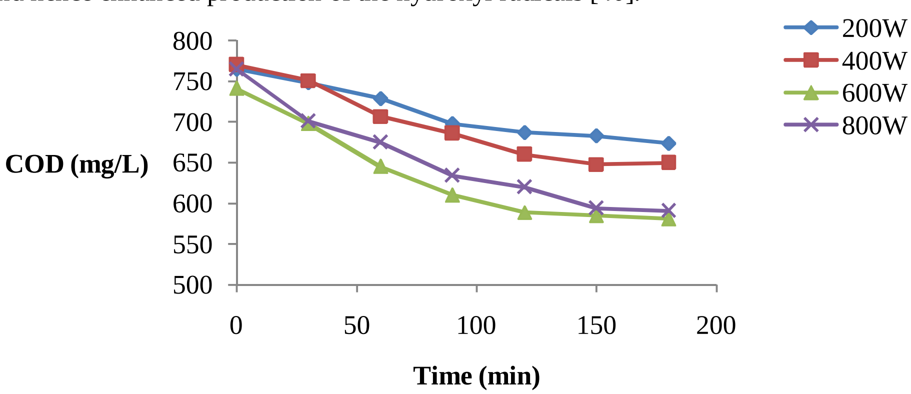

Figure 2. Effect of sonication power on the reduction of COD of textile industry waste water

Table 2shows the effect of sonication power on the % of COD reduction of textile industry waste water using acoustic cavitation. It has been observed that as sonication power is increased from 200W to 800W and % of COD reduction of textile industry waste water increases from 11.90 % to 22.75 %.

Table2.Effect of sonication power on the % of COD reduction of textile industry waste water

Process	Power (Watts)	% of COD Reduction
Acoustic Cavitation	200	11.90
	400	15.58
	600	21.59
	800	22.75

2) *Effect of duty cycle on the reduction of COD of textile industry waste water:* The effect of different duty cycles (20%, 40%, 60% and 80%) on the treatment of textile industry wastewater was investigated by varying ON and OFF time of ultrasonic irradiation. These experiments were conducted for 400 ml textile industry waste water, sonication power 800 W, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. Figure 3shows the effect of duty cycle on reduction of COD of textile industry waste water.

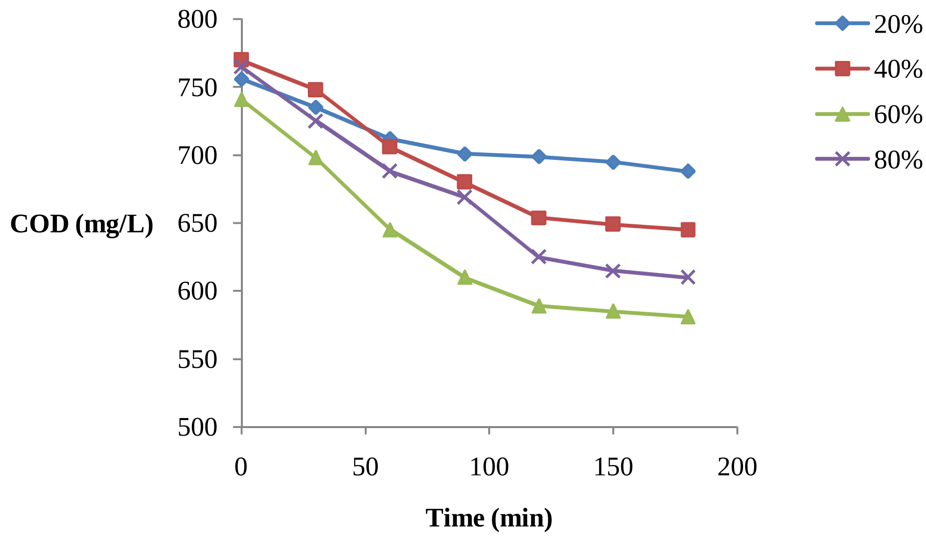

Figure3. Effect of duty cycle on the reduction of COD of textile industry waste water

Table 3 shows that the % of COD reduction of textile industry wastewater increased from 8.99% to 21.59% with an increase in duty cycle from 20% to 60%. The decrease in % of COD reduction of textile waste water at 80% duty cycle due to 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 [41,42].At maximum duty cycle also increase in the number of cavities & chances of coalescence of the bubbles which increases the size of bubble and erosion of transducer due to vibration cause during collapse of large size bubbles [43].

Table 3.Effect of duty cycle on the % of COD reduction of textile industry waste water

Process	Duty cycle (%)	% of COD Reduction
Acoustic Cavitation	20	8.99
	40	16.23
	60	21.59
	80	20.26

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3) *The effect of pH on the COD reduction of textile industry waste water:* Initial pH is an important parameter influencing the treatment efficiency. In this study, in order to investigate the effects of pH on reduction of COD of textile industry waste water using acoustic cavitation, experiments were conducted at different pH values 3, 7.5 and 9. These experiments were conducted at 400 ml textile industry waste water, sonication power 800W, 60 % duty cycles, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. Figure 4shows the effect of initial pH on the reduction of COD of textile industry waste water using acoustic cavitation.

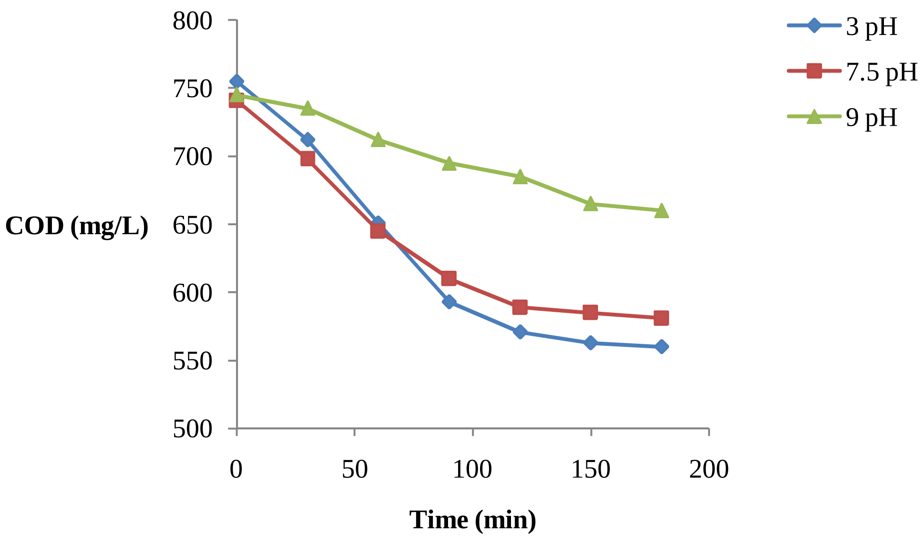

Figure 4. Effect of pH on the reduction of COD of textile industry waste water using acoustic cavitation

Table 4 shows that the reduction of COD of textile industry waste water is very rapid under acidic pH 3, 25.83 % reduction of COD of textile industry waste water was obtained at constant reaction temperature (28 ± 3 °C), constant sonicationfrequency 20 kHz, 800W sonication power, 60 % duty cycle and 400 ml of textile industry waste water.At lower pH (acidic), the oxidation capacity of hydroxyl radicals is high as compared to the higher pH (basic)[44-46].

Table4.Effect of pH on the % of COD reduction of textile industry waste water

Process	pH	% of COD Reduction
Acoustic Cavitation	3	25.83
	7.5	21.59
	9	11.41

C. Acoustic cavitation + H_2O_2:-

1) *Effect of H_2O_2 on the reduction of COD of textile industry waste water:* 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(25 to 125µl/L) on the reduction of COD of textile industry waste water is shown in Figure 5. These experiments were conducted at 400 ml textile 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

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addition of $H_2O_2$from 25 to 75µl/L increases the reduction of COD from 55.38 % to 66.80 % at 180 minutes. Further increase from 100 to 125 µl/L, decrease in reduction of COD from 64.61 % to 61.72 %. 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[47, 48].

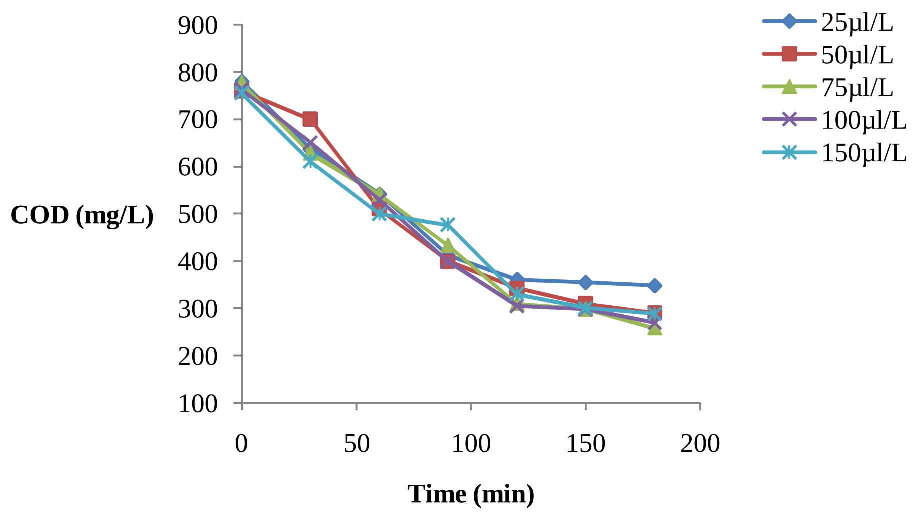

Figure 5. Effect of H_2O_2 on the reduction of COD of textile industry waste water using acoustic cavitation

Table 5 shows the effect of H_2O_2on % of COD reduction of textile industry waste water. It has been observed that 22.75 % and 3.87 % reduction of COD was obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively. Further the COD reduction was significantly increased to 66.80 % and 13.11 % by applying the combination of Acoustic Cavitation with H_2O_2 and Aeration with H_2O_2 respectively. A similar observation was also reported by Meric, S., et al (2004)[49], Benitez, F.J., et al (2001) [50], Kang, S.F., et al(2000) [25]and Azbar, N., et al (2004)[51] for study the effect of H_2O_2 on synthetic textile industry wastewater.

Table5.Effect of H_2O_2 on the % of COD reduction of textile industry waste water

Process	Loading of H_2O_2 (µl/L)	% of COD Reduction (180 min)
Acoustic Cavitation only	-	22.75
Aeration only	-	3.87
	25	55.38
	50	61.84
	75	66.80
Acoustic Cavitation + H_2O_2	100	64.61
	125	61.72
	25	5.89
	50	8.54
Aeration + H_2O_2	75	13.11
	100	11.98
	125	12.01

D. Acoustic Cavitation + Fenton Process:

1) Effect of $FeSO_4.7\ H_2O$ on the reduction of COD of textile 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 processes. More hydroxyl radicals are produced with the increase in the concentration of ferrous ions (Fe^{2+}). Effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water has been investigated for various concentration of Ferrous sulfate heptahydrate ($FeSO_4$ $.7H_2O$) in the presence of H_2O_2 (75 µl/L) and Acoustic Cavitation (800W).

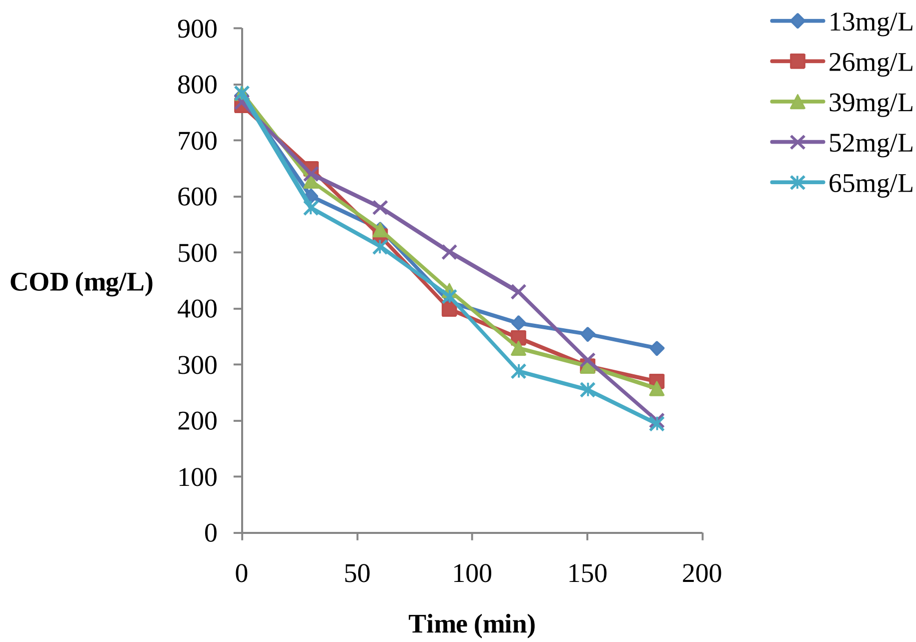

Figure 6. Effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water using acoustic cavitation and H_2O_2.

Figure 6 shows the effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water. These experiments were conducted at 400 ml textile industry waste water, sonication power 800W, 60 % duty cycle, 3 pH, 75 µl/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 textile industry waste water increases with an increase in the loading of $FeSO_4.7H_2O$ from 13mg/L to 65 mg/L under acoustic cavitation with optimum concentration of hydrogen peroxide(75µl/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 [53]. 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 **[52].**

Table 6 shows the effect of $FeSO_4.7H_2O$ on % of COD reduction of textile industry waste water. It has been observed that 22.75 % and 3.87 % reduction of COD obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively which significantly increased to 75.16 % and 22.98 % by applying the combination of Acoustic Cavitation with Fenton Process and Aeration with Fenton Process respectively. A similar observation was also reported for Fenton process by Meric, S., et al (2004)[49], Neamtu, M., et al (2003)[53] and Arslan, I., et al (2000) [54]for study the effect of $FeSO_4$ $.7H_2O$ on synthetic textile industry wastewater.

Table 6. Effect of $FeSO_4.7H_2O$ on the % of COD reduction of textile industry waste water

Process	Loading of $FeSO_4.7H_2O$(mg/L)	% of COD Reduction (180 min)
Acoustic Cavitation only	-	22.75
Aeration only	-	3.87
Acoustic Cavitation + Fenton process	13	57.69
	26	64.61
	39	67.13
	52	73.96
	65	75.16
Aeration + Fenton process	13	12.01
	26	15.89
	39	17.54
	52	19.89
	65	22.98

E. Acoustic Cavitation + Photo Fenton Process (Sonophotofenton Process)

1) *Effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water :* The reduction of COD of textile industry waste water was investigated using different concentration of $FeSO_4.7H_2O$ ranging from 13 mg/L to 65 mg/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 at 400 ml textile industry waste water, sonication power 750W, 60 % duty cycle, 3 pH, 75 µl/L of optimum H_2O_2 loading, constant temperature of 28 ± 3 °C, constant sonication frequency 20 kHz for 180 minutes of time. Figure 7 shows the effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water. It has been observed that the reduction of COD of textile 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 (75 µl/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 [55-57].

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

Also hydrogen peroxide readily decomposes into hydroxyl radicals in the presence of acoustic cavitation and UV lights**[58, 59].**

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

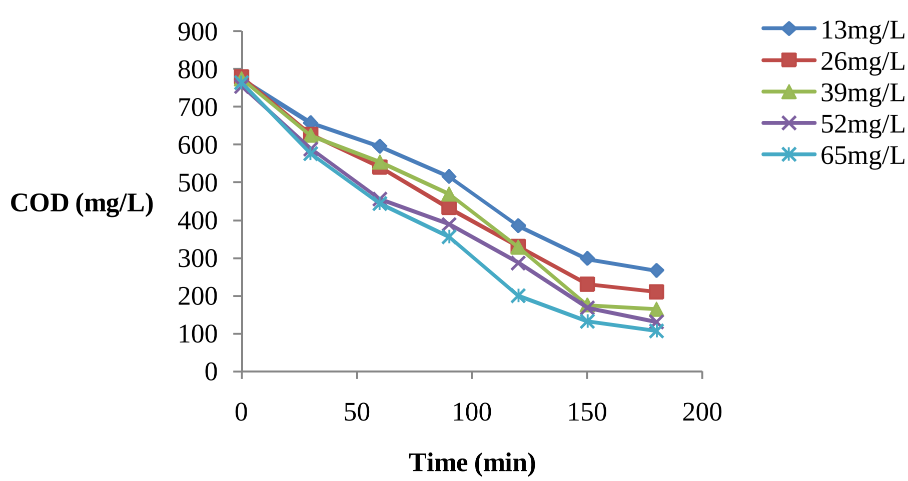

Figure 7. Effect of $FeSO_4.7H_2O$ on the reduction of COD of textile industry waste water using acoustic cavitation, UV lights and H_2O_2

Table 7 shows the effect of $FeSO_4.7H_2O$ on % of COD reduction of textile industry waste water. It has been observed that 22.75 % and 3.87 % reduction of COD obtained after 180 min of operation using Acoustic Cavitation and Aeration respectively which significantly increased to 86.01 % and 26.78 % for the combination of Acoustic Cavitation with Photo Fenton Process and Aeration with Photo Fenton Process. For Photo Fenton process, similar observation was reported by Prajisha, V.P., et al (2016)[60], Sahunin, C., et al (2006)[61] and Liu, R.,et al (2006) [62] for study the effect of $FeSO_4$ .7 H_2O on synthetic textile industry wastewater.

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

Process	Loading of $FeSO_4.7$ H_2O(mg/L)	% of COD Reduction (180 min)
Acoustic Cavitation only	-	22.75
Aeration only	-	3.87
	13	65.46
Acoustic Cavitation + Photo	26	73.08
Fenton process	39	78.71
	52	82.78
	65	86.01
	13	18.22
Aeration +	26	21.76
Photo Fenton process	39	23.34
	52	24.99
	65	26.78

IV. CONCLUSIONS

The present study has shown the efficiency of acoustic cavitation for the reduction of COD of textile 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 textile industry waste water was influenced by sonication power, duty cycle and solution pH using acoustic cavitation. Maximum percentage of COD reduction was 22.75% for sonication power of 800 W, 21.59 % of 60 % duty cycle and 25.83%of pH 3 respectively.
2) It was observed that acoustic cavitation coupled with H_2O_2 enhances the percentage of COD reduction of textile industry waste water, giving 66.80 % reduction of COD at an optimum concentration of H_2O_2 (75 µl/L) as compared 22.75 % obtained using acoustic cavitation alone.
3) The combination of Fenton process with acoustic cavitation gives 75.16 % reduction of COD of textile industry waste water at optimum concentration of $FeSO_4.7H_2O$ (65 mg/L)as compared 66.80 % and 22.75 % 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 86.01 % maximum reduction of COD of textile industry waste water at optimum concentration of $FeSO_4.7H_2O$ (65 mg/L) as compared 75.16 %, 66.80 % and 22.75 % 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 textile industry waste water.

Figure 7 shows the summarized graph containing % of COD reduction by individual methods and combination of the methods.

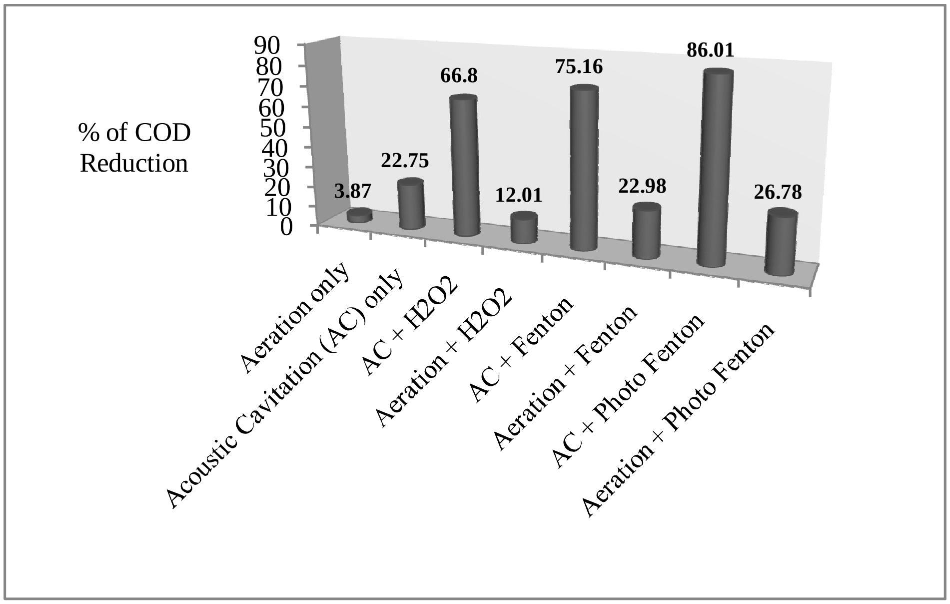

Figure 7.% of COD reduction by various methods

V. ACKNOWLEDGMENTS

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