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Treatment of the pesticide industry effluent using hydrodynamic cavitation and its combination with process intensifying additives (H₂O₂ and Ozone)

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1   *Abstract*

2   Hydrodynamic cavitation (HC) and its combination with H_2O_2 and ozone have been

3   applied in the present work for the treatment of industrial pesticide effluent. Initially, the

4   effect of dilution of the effluent on the efficacy of hydrodynamic cavitation has been studied

5   using circular venturi as a cavitator. Although an increase in the extent of dilution has not

6   shown any beneficial effect on the actual moles of pollutant degraded, hybrid processes have

7   been studied using 1:5 dilution due to very high TDS content of the effluent. Treatment of the

8   industrial pesticide effluent using HC+Ozone (3 g/h) process has demonstrated that the

9   biodegradability index (BI) of the effluent increases from 0.123 to 0.324 after 2 h of

10   operation. The rate of COD and TOC reduction has also increased by many folds by using

11   HC in combination with ozone. In addition this, the treatment of industrial pesticide effluent

12   using HC+H_2O_2 has also indicated that the rate of COD and TOC reduction increases

13   significantly by using HC in combination with various loadings of H_2O_2. The study of

14   interference of added H_2O_2 on the COD analysis has exhibited that the COD equivalence is

15   0.441 mg/L for 1 mg/L of H_2O_2. The energy efficiency and operating cost of various hybrid

16   processes have been compared based on the cavitational yield and the cost of electricity. The

17   combined process of HC and H_2O_2 has observed to be the most cost-effective one due to its

18   higher cavitational yield and lower power consumption.

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20   **Keywords:** Hydrodynamic cavitation, Venturi, Biodegradability index, Industrial pesticide

21   effluent, Hydrogen peroxide, Ozone

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1. Introduction

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27   Contamination of surface and groundwater resources by the pesticides is mainly due

28 to industrial discharges and extensive agricultural activities [1].Wastewater generated from

29 the pesticide manufacturing industries poses significant pollution problems due to the high

30 values of chemical oxygen demand (COD) and reduced biodegradability [2]. Most of the

31 pesticide compounds are very toxic and hazardous in nature, even at low concentrations.

32   Although conventional biological treatments are widely used for the degradation of

33 organic pollutants in wastewater, they are not very effective for the degradation of

34 biorefractory compounds [3]. Since last decade, advanced oxidation processes (AOPs) have

35 proved to be very effective in improving the overall degradation efficiency of biorefractory

36 compounds such as pesticides [4, 5]. The degradation mechanism of AOPs is primarily based

37 on the generation of highly reactive and nonselective hydroxyl radicals (OH$^{\bullet}$) and its

38 subsequent attack on the organic pollutant to convert them into CO_2, water and inorganic ions

39 [6]. However, the application of AOPs is not very cost-effective if they are applied

40 individually to obtain complete mineralization of recalcitrant compounds in wastewater.

41 Hence, instead of replacing the cost-effective biological processes with AOPs, the effective

42 and proper combination of both systems is very essential [7]. AOPs such as Fenton,

43 ozonation, cavitation etc. can be used for the pre-treatment of industrial wastewater to

44 improve its biodegradability index (BOD_5:COD ratio) and thus enhancing the probability of

45 degradation using microbial action [8].

46   Cavitation is one of the emerging AOP which is capable of reducing the toxicity and

47 enhancing the mineralization of the wastewater [9]. It is the phenomena of formation and

48 growth of the millions of micro cavities under the controlled conditions and their subsequent

49 violent collapse due to the pressure variations created [10]. Although cavitation can be

50 induced by many ways, hydrodynamic cavitation has been reported to be the most cost

3

effective and efficient way of inducing the cavitation [11]. The degradation of pollutants using hydrodynamic cavitation occurs through the pyrolysis/thermal decomposition of the pollutant molecules entrapped inside the cavity and the oxidation of pollutant molecule using $OH^{\bullet}$ radicals at cavity water interface [12]. Hydrodynamic cavitation has attracted the attention of many researchers since it can be easily scaled up [13]. However, it has a serious limitation of low degradation or mineralization rates when applied individually for the treatment of complex wastewater [14]. The efficacy of hydrodynamic cavitation can be enhanced by using the process intensifying additives such as hydrogen peroxide and ozone since all such processes have similar degradation mechanism i.e the generation and subsequent attack of $OH^{\bullet}$ radicals on the pollutant molecule [15].

The work done so far on the application of combination of hydrodynamic cavitation and other AOPs is primarily based on the degradation of synthetic wastewater containing single pollutant and the reports on the treatment of real industrial effluent are very few. Chakinala et al. [16] have explored the combined process of hydrodynamic cavitation (HC) and advanced Fenton process for the treatment of real industrial wastewater and reported that HC is very effective as a pre-treatment to biological oxidation. Padoley et al. [9] have also reported the improvement in the biodegradability index (BI) of complex biomethanated distillery wastewater (B-DWW) by using the hydrodynamic cavitation.

The combined application of hydrodynamic cavitation and the process intensifying additives for the degradation of industrial pesticide effluent is not yet reported in the literature to the best of our knowledge. The present work aims at reducing the toxicity and increasing the biodegradability of industrial pesticide effluent by the application of hydrodynamic cavitation in combination with process intensifying additives such as hydrogen peroxide (H_2O_2) and ozone (O_3). The change in the toxicity of industrial pesticide effluent after treatment has been evaluated by measuring the COD, BOD and TOC values of the effluent.

4

76 However, H_2O_2 interferes with COD analysis by consuming certain amount of potassium

77 dichromate and lead to overestimation of the COD values [17-19]. Due to this, the

78 interference of H_2O_2 on COD estimation during HC+ H_2O_2 has been evaluated to obtain the

79 corrected COD values. In addition to this, the cost estimation of HC, HC+ H_2O_2 and HC +

80 Ozone processes has also been included in the present work to obtain the most effective and

81 economical process for the treatment of industrial pesticide effluent.

82 **2. Materials and methods**

83 *2.1 Materials*

84 Hydrogen peroxide (30 % w/v) and sodium hydroxide (NaOH) both of AR grade

85 were obtained from S D Fine Chemicals Ltd., Mumbai, India. A sample of industrial

86 pesticide effluent was collected from the pesticide manufacturing industry in Mumbai, India.

87 The effluent collected was filtered before use, to remove any suspended solids and the

88 supernatant obtained has been used in the degradation study after making the desired dilution

89 and pH adjustment. The characteristics of the effluent have been summarized in Table 1.

90 *2.2 Experimental set-up*

91 Hydrodynamic cavitation set-up used in the present work is as shown in the Fig .1.

92 The set-up is a closed circuit assembly which essentially consists of holding tank (Max.

93 capacity-15L), cavitator, positive displacement pump (1.1 kW), control valves (V_1, V_2 and

94 V_3) and pressure gauges (P_1 and P_2). Geometrical specifications of the cavitator used in the

95 present work (circular venturi) have been mentioned in the Fig. 2. The dimensions of the

96 cavitator are based on the optimized parameters obtained by Bashir et al. [20] using the CFD

97 analysis. The provision was made in the cavitator for insertion of the ozone at the throat of

98 the venturi.

99 The holding tank is surrounded by the cooling jacket to remove the heat generated

100 during cavitation. The solution temperature was maintained at $25 \pm 5 \ ^{\circ}C$ by circulating water

101 through the cooling jacket. The base of the holding tank is connected to the suction side of

102 the pump through a pipe and the discharge line of pump is branched further into two lines, the

103 main and a bypass line. The control valve in the bypass line was used for regulating the inlet

104 pressure to the cavitating device by regulating the flow through the main line. The pressure

105 gauges have been provided before and after the cavitator to obtain the inlet pressure to the

106 cavitating device and fully recovered downstream pressures. Both the mainline and bypass

107 line have terminated well inside the holding tank, to avoid the possible induction of air into

108 the liquid due to the plunging jet.

109 *2.3 Experimental methodology*

110   All the experiments have been performed for the duration of 120 min by using 6 L of

111 industrial pesticide effluent with pH adjusted to 7 by adding 5 N NaOH. The holding tank

112 was filled with the effluent to be treated and the reciprocating pump was used to circulate the

113 effluent continuously in the experimental set-up. The control valve in the bypass line was

114 used to adjust the desired inlet pressure to the cavitating device by controlling the flow in the

115 main line. The samples were collected after regular interval of time to determine the COD,

116 BOD and TOC values of the samples. Initially, during HC pre-treatment the effect of dilution

117 of effluent on COD reduction was studied by using effluent without any dilution and with 1:5

118 and 1:10 times dilution with pH adjusted to 7. The inlet pressure to the cavitating device was

119 6 bar in all such experiments.

120   The effect of loading of ozone on the performance of combined process of

121 hydrodynamic cavitation and ozone has been evaluated by varying loading of ozone from 0.5

122 to 3 g/h (0.5, 0.75, 1 and 3 g/h). The flow rate ratios of effluent to ozone corresponding to the

123 loadings of 0.5, 0.75, 1 and 3 g/h of ozone are 0.0000525, 0.000787, 0.001050 and 0.00315

124 respectively. These values are based on the liquid flow rate of 445 LPH at the chosen inlet

125 pressure of 6 bar to the cavitator. Ozonator with a capacity of 180 W (make- Eltech

126 Engineers, India) was used for generating ozone with a maximum ozone producing capacity

127 of 10 g/h. Oxygen generator (capacity – 420 W and oxygen output - 1–5 LPM) was used to

128 produce the oxygen (94% purity) since it is a feed to the ozonator for producing the ozone.

129 The generated ozone was then constantly injected at the throat of the venturi.

130 The effect of loading of H_2O_2 on the efficacy of combined process of hydrodynamic

131 cavitation and H_2O_2 has also been evaluated by using various concentrations of H_2O_2 such as

132 2, 5 and 10 g/L. All such experiments have been performed using 6 bar inlet pressure, 1:5

133 dilution and pH adjusted to 7. The decomposition of H_2O_2 during these experiments was also

134 evaluated by measuring the concentration of unreacted H_2O_2 in the effluent by using the

135 DMP method. The experiments of studying the interference of H_2O_2 on the standard COD

136 estimation have been carried out by using the various loadings of H_2O_2 such as 2, 5, 7 and 10

137 g/L. Initially, the effluent was diluted to different proportions and the COD was measured

138 using standard method [21]. Keeping the identical dilutions of the effluent, later the COD was

139 measured immediately after adding the fixed quantity of H_2O_2. Average increase in the COD

140 estimation for various loadings of H_2O_2 (2, 5, 7 and 10 g/L) was evaluated to determine the

141 correlation of COD overestimation.

142 *2.4 Analysis*

143 The samples withdrawn after pre-treatment were centrifuged at 4500 rpm (5660 g

144 relative centrifugal force) to remove the solid particles and clear supernatant was subjected to

145 the further physico-chemical analysis. The COD and BOD analysis was carried out as per the

146 standard methods [21]. The degree of mineralization of industrial effluent into end products

147 (such as CO_2 and H_2O) was analyzed using total organic carbon analyzer (make-Shimadzu

148 corporation, Japan) at various optimum conditions. The concentration of H_2O_2 was

149 determined by the spectrophotometric method (λ_{max} of $Cu(DMP)^{2+}$ complex- 454 nm) using

150 copper (II) ion and DMP (2,9-Dimethyl-1,10-phenanthroline) [22].

7

151 **3.   Results and discussion**

152 *3.1   Effect of dilution on COD reduction using hydrodynamic cavitation (HC)*

153   The effect of dilution of pesticide effluent on the rate and extent of COD reduction

154 has been evaluated by utilizing different dilutions of the effluent such as no dilution, 1:5

155 dilution and 1:10 dilution for the pre-treatment using hydrodynamic cavitation. All the

156 experiments have been carried out for the duration of 90 minutes using 6 L of the effluent

157 with desired dilution and by keeping the inlet pressure to the venturi as 6 bar. The initial pH

158 of the effluent was adjusted to 7 (by adding 5 N NaOH) in order to neutralize the effect of

159 chloride ions present in the effluent which may cause corrosion of the experimental setup

160 [23]. The samples withdrawn at the regular interval of time during these experiments have

161 been subjected to the COD analysis.

162   The results obtained have been summarized in the Fig. 3 which has indicated that the

163 rate of COD reduction obtained using pre-treatment of hydrodynamic cavitation follows first

164 order kinetics for all the dilutions studied. It has been observed that, with an increase in the

165 extent of dilution, the rate of COD removal also increases with the maximum COD reduction

166 of 21 % obtained using 10 times diluted effluent. However, an increase in the extent of

167 dilution has shown a negative impact on the actual moles of pollutant degraded (based on

168 COD values). It can be seen from the Table 2 that, with an increase in the extent of dilution,

169 although the extent of COD reduction is increasing, effective number of moles of pollutant

170 degraded have reduced. Hence, the use of much diluted effluent for the pre-treatment using

171 hydrodynamic cavitation is not a promising option for effective treatment of industrial

172 pesticide effluent.

173   The results obtained are consistent with the earlier reports indicating that, the rate of

174 removal of pollutants under cavitating conditions is inversely proportional to the initial

175 concentration of the pollutant. Chakinala et al. [16] have studied the effect of dilution of

176 industrial wastewater effluent on the extent of COD and TOC removal. The extent of COD

8

177 removal in 150 min of treatment time was 42 %, 62 % and 73 % for a dilution ratio of 10, 25

178 and 50 respectively. It was clearly observed that the extent of COD reduction does not

179 increase appreciably with an increase in the extent of dilution. Padoley et al. [9] have also

180 studied the effect of dilution of distillery waste water on the extent of mineralization obtained

181 using hydrodynamic cavitation. The original wastewater (34,000 mg/L COD) was diluted to

182 25% and 50% concentration (%, V/V) using tap water and was treated using hydrodynamic

183 cavitation. It was observed that the dilution has no significant effect on the mineralisation of

184 distillery waste water, since the COD and TOC reduction at 25 % and 50 % dilution was

185 lower as compared to undiluted waste water.

186 Although an increase in the extent of dilution has not shown any beneficial effect on

187 the actual moles of pollutant degraded, further experiments have been performed using 1:5

188 dilution of effluent in order to reduce the TDS content of the effluent. This is because, very

189 high TDS content of effluent along with very high temperature and pressure conditions at the

190 time of cavitation can damage the pump and the cavitator.

191 *3.2 Effect of loading of ozone on the mineralization of the effluent using HC + Ozone*

192 Ozonation process has attracted the attention of many researchers since it is capable

193 of oxidising the organic pollutants due to its high oxidation potential of 2.08 eV. However,

194 during conventional ozonation process, the rate of reaction of ozone with pollutant molecule

195 is not very significant due to high mass transfer resistances in aqueous solutions [24]. The

196 combination of HC and ozone can enhance the rate of reaction of ozone with pollutant

197 molecules since local turbulence created by cavitation can increase the mass transfer of ozone

198 from gas phase to the bulk liquid phase [25]. In addition to this, ozone dissociates in presence

199 of cavitation and generates atomic oxygen ($O^{\bullet}$) which further reacts with water molecule to

200 generate highly reactive $OH^{\bullet}$ radicals [13]. Hence the combination of hydrodynamic

201 cavitation and ozone can be a promising option for the treatment of wastewater.

202 With this background, the effect of loading of ozone on the extent of mineralisation of

9

203 industrial pesticide effluent has been evaluated by applying HC + Ozone process and by

204 varying the loading of ozone from 0.5 to 3 g/h. All the experiments have been carried at an

205 inlet pressure of 6 bar and pH of 7 and the samples withdrawn at regular interval of time were

206 subjected to COD and TOC analysis. It can be seen from the Fig. 4 and Fig. 5 that, with an

207 increase in the loading of ozone, the rate of COD as well as TOC reduction increases. The

208 rate constant (k), extent of COD and TOC reduction and the moles of pollutant degraded at

209 various loadings of O_3 have been summarised in Table 3. The rate and the extent of COD

210 reduction of 1.38×10^{-3} min^{-1} and 14.77 % obtained by using individual hydrodynamic

211 cavitation have enhanced significantly (nearly by 80%) to 2.47×10^{-3} min^{-1} and 25.69 % by

212 using HC in combination with 0.75 g/h of ozone. Similar trend has also obtained in case of

213 effect of ozone on the rate and extent of TOC reduction.

214 Although, the efficacy of HC has increased noticeably in the presence of ozone,

215 increasing the loading of ozone beyond 0.75 g/h (Ozone to liquid flow rate ratio as 0.000787)

216 has shown only marginal increase in the rate of COD and TOC reduction. Similarly, the

217 moles of effluent degraded have also not increased very significantly by increasing the

218 loading of ozone beyond 0.75 g/h. Hence, an optimal loading of 0.75 g/h ozone should be

219 used in the combined process of HC and ozone, for the effective treatment of industrial

220 pesticide effluent.

221 Similar results have also been observed in the literature reports for the treatment of

222 synthetic wastewater containing single organic pollutant. Gore et al. [13] have observed that

223 efficiency of HC for the degradation of reactive orange 4 dye (RO4) enhances significantly

224 by combining it with ozone. TOC reduction of 14.67% obtained in case of HC has increased

225 to 76.25% when HC was coupled with ozone. Similarly, Wu et al. [25] have also

226 demonstrated the enhanced effect of suction-cavitation on the ozonation of phenol using

227 orifice as a cavitator.

228   *3.3   Effect of HC and HC + Ozone processes on the biodegradability index*

229   Biodegradability index (BI), a ratio of BOD_5: COD is a measure of the extent to which

230   a wastewater is amenable to biodegradation [3]. The wastewater is fairly biodegradable if BI

231   is greater than 0.4. However, wastewater with BI in the range of 0.3-0.4 is also amenable to

232   biological treatment with a BI value ≥ 0.3 necessary for aerobic treatment and ≥ 0.4 for

233   anaerobic treatment. The wastewater cannot be treated biologically if BI is less than 0.3 [9,

234   26]. Wastewater effluent pre-treatment using hydrodynamic cavitation or its combination with

235   ozone can reduce the toxicity of wastewater and thus can enhance its biodegradability.

236   The biodegradability index of the industrial pesticide effluent has been evaluated after

237   the pre-treatment of HC or HC + Ozone processes and the results obtained have been

238   summarised in Table 4. It has been observed that pre-treatment of HC for either diluted or

239   non-diluted effluent could lead to only marginal enhancement in the BI even after 120 min of

240   treatment time. However, the pre-treatment of combination of HC and ozone process has

241   shown significant impact on the biodegradability of the wastewater effluent. The BI of the

242   effluent has substantially increased from 0.121 to 0.324 after pre-treatment of HC + Ozone (3

243   g/h) over the treatment duration of 120 min. In order to obtain higher BOD/COD ratio, the

244   degradation process can be continued for longer period of time or the cavitation conditions

245   can be made more severe.

246   Similar observation has been reported by Padoley et al. [9] while studying the effect

247   of HC pre-treatment for distillery wastewater. It was observed that, HC treatment of distillery

248   wastewater (with 25 % dilution) at inlet pressure of 13 bar results into an increase in the BI

249   from 0.14 to 0.32 after 50 min. It was reported that, HC pre-treatment results into an increase

250   in the efficiency of the conventional biological process by almost 6 times in terms of COD

251   removal and biogas formation. Bis et al. [27] have also observed that the application of HC

252   can enhance the biodegradability index of the mature landfill leachate. The orifice plate

253   having three holes of 10 mm diameter and cavitation number of 0.033 was observed to be the

254 most suitable one for enhancing the biodegradability index of mature leachate. By using this

255 configuration and maintaining 30 recirculation passes through the cavitation zone at inlet

256 pressure of 7 bar, the highest biodegradability index (BI) was observed. The results obtained

257 were attributed to the physical and chemical effects of cavitation.

258   Overall, it has been observed that HC in combination with ozone can effectively be

259 utilized for reducing the toxicity and thereby enhancing the biodegradability index of the

260 pesticide wastewater effluent.

261 *3.4 Effect of loading of H_2O_2 on the TOC reduction using HC + H_2O_2 process*

262   Hydrogen peroxide being a potential oxidant can be used for enhancing the efficacy

263 of hydrodynamic cavitation since it dissociates easily in the presence of cavitation providing

264 additional highly reactive hydroxyl radicals [28]. The effect of loading of H_2O_2 on the TOC

265 reduction has been evaluated by using the loadings of H_2O_2 such as 2, 5 and 10 g/L for the

266 treatment of industrial pesticide effluent (5 times diluted) with pH adjusted to 7. The results

267 obtained have been depicted in Fig. 6, which indicated that the effect of H_2O_2 on the TOC

268 values of the effluent during combined process of HC and H_2O_2 followed first order kinetics.

269 Table 5 summarizes the values of rate constant (k) and extent of TOC reduction and moles of

270 the effluent degraded (based on TOC) after 120 min of operation. The results obtained have

271 clearly shown that the rate and extent of TOC reduction increases with an increase in the

272 loading of H_2O_2. Combined process of HC and H_2O_2 has observed to be more efficient than

273 HC operated individually, since the rate constant of 0.56×10^{-3} min^{-1} (TOC reduction-

274 6.58%) obtained using only HC has significantly enhanced to 2.42×10^{-3} min^{-1} (TOC

275 reduction- 22.85%) by using HC in combination with 2 g/L of H_2O_2. Similarly, moles of the

276 pollutant from industrial pesticide effluent degraded (based on TOC) in case of individual HC

277 (4.614×10^{-3} moles) has also increased appreciably by using HC in combination with 2 g/L of

278 H_2O_2 (1.749×10^{-2} moles). However, further increase in the loading of H_2O_2 has resulted in

12

279 monotonic increase in the TOC reduction, even though loading of H_2O_2 has increased by 5

280 times i.e. from 2 g/L to 10 g/L. Hence, optimized loading of H_2O_2 should be used for

281 effective mineralization of pesticide effluent. The results obtained are attributed to the fact

282 that at very high loading of H_2O_2 the detrimental effects are observed due to recombination/

283 scavenging of $OH^\bullet$ radicals by H_2O_2 present.

284   In a similar study of treatment of actual industrial wastewater effluent, Chakinala et

285 al. [16] have also observed that the efficacy of hydrodynamic cavitation enhances appreciably

286 by using it in combination with H_2O_2. It was found that the extent of TOC reduction increases

287 with an increase in the loading of H_2O_2 although increase in the TOC reduction was marginal

288 at higher loading of H_2O_2.

289 *3.5 Influence of H_2O_2 on standard COD estimation*

290   While studying the effect of addition of H_2O_2 on the COD values of the industrial

291 pesticide effluent, it has been observed that the COD values of the effluent samples are

292 increasing after the pre-treatment using combined process of HC and H_2O_2. The unusual

293 trend of the results has indicated the possibility of H_2O_2 interference on the COD estimation.

294   To confirm the existence and extent of H_2O_2 interference on the COD estimation,

295 COD analysis of industrial pesticide effluent has been evaluated after adding different

296 loadings of H_2O_2 such as 2, 5, 7 and 10 g/L. The samples have been prepared by using five

297 different dilutions of the effluent and COD analysis has been carried out immediately after

298 the addition of fixed quantity of H_2O_2. The COD values have also also been evaluated without

299 addition of H_2O_2 and keeping identical extent of dilutions of effluent. Later, the COD

300 overestimation was obtained by determining the increase in the COD values due to the

301 addition H_2O_2. It has been observed that the presence of hydrogen peroxide leads to an

302 overestimation of the COD values and the extent of COD overestimation has found to be

303 proportional to the concentration of H_2O_2. Fig. 7 demonstrates the linear relationship between

13

304 COD overestimation and concentration of H_2O_2. The slope of the straight line has indicated

305 that the equivalence of 1 mg/l of H_2O_2 is 0.441 mg/l of COD. The other details of regression

306 analysis including the relative standard deviation and confidence limit of 95% have been

307 depicted in Table 6. Each ΔCOD value reported in the table is arithmetic mean of COD

308 overestimation values of five different concentrations of effluent loaded with fixed

309 concentration of H_2O_2.

310   Many other previous reports have also confirmed the interference of H_2O_2 on the

311 COD estimation [17, 18]. Hydrogen peroxide leads to the COD overestimation since it gets

312 consumed during COD analysis as per the oxidation reaction given in Eq. 1 [19] leading to

313 more utilization of potassium dichromate and higher COD values.

314 $K_2Cr_2O_7 + 3H_2O_2 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O + 3O_2$ (1)

315   Lee et al. [29] have reported that theoretical COD value of 1 mg/L of H_2O_2 is 0.470

316 mg/L. The COD overestimation obtained in the present work is marginally lower than the

317 theoretical one, since some fraction of H_2O_2 might have got consumed for the oxidation of

318 effluent even though COD analysis was carried out immediately after the addition of H_2O_2.

319 Although, it has been observed that extent of overestimation of COD is proportional to the

320 concentration of H_2O_2, the exact COD overestimation correlation is dependent on type of the

321 wastewater under question.

322 *3.6 Effect of loading of H_2O_2 on the rate of COD reduction using HC + H_2O_2 process*

323 *(based on corrected COD values)*

324   For studying the effect of loading of H_2O_2 on the rate of COD reduction, the

325 determination of residual concentration of H_2O_2 and corrected COD values is very essential.

326 Fig. 8 shows the change in the residual concentration of H_2O_2 during combined process of

327 HC and H_2O_2. It has been observed that the residual concentration of H_2O_2 decreases with

328 time for all the loadings of H_2O_2 during HC + H_2O_2 process. The results obtained have clearly

14

329   indicated that very high loading of H_2O_2 is not beneficial, since the rate of decomposition of

330   H_2O_2 has decreased with an increase in the initial loading of H_2O_2. Substantial decrease in the

331   residual concentration of hydrogen peroxide can be obtained if the effluent will be treated for

332   longer period of time using HC + H_2O_2 process. Various researchers have reported the

333   degradation rate of H_2O_2 under cavitation [1, 30]. The values of residual concentration of

334   H_2O_2 have been used further to obtain the extent of COD overestimation, which was further

335   used for estimating the true/corrected values of COD as below.

336   Extent of COD overestimation, mg/L = residual concentration of H_2O_2, mg/L $\times$ 0.441

337   True/ Corrected COD in mg/L= measured COD value - extent of COD overestimation

338   The corrected COD values obtained have been used to further study the effect of

339   loading of H_2O_2 on the rate of COD reduction at various loadings of H_2O_2 such as 2, 5 and 10

340   g/L. The results obtained have been illustrated in Fig. 9. The results obtained while studying

341   the effect of loading of H_2O_2 on the rate of TOC and COD have indicated that the combined

342   process of HC and H_2O_2 is more efficient than HC alone for reducing the toxicity of

343   wastewater effluent. The details of rate constant (k), extent of COD reduction and moles of

344   effluent degraded at various loadings of H_2O_2 have been summarized in Table 7. It has been

345   observed that rate and extent of COD reduction increases monotonically with an increase in

346   the loading of H_2O_2, since the rate of COD reduction obtained in case of 2 g/L of H_2O_2 has

347   not increased significantly even after increasing the loading of H_2O_2 by 5 times i.e. to 10 g/L.

348   Similarly, moles of the pollutant degraded based on COD values have not enhanced

349   proportionately with an increase in the loading of H_2O_2. Hence, based on the rate of

350   decomposition of residual H_2O_2 and rate of COD reduction it has been concluded that even

351   though efficacy of hydrodynamic cavitation greatly enhances in presence of H_2O_2, the use of

352   very high loading of H_2O_2 should be avoided for the sake of any apparent increased rate of

353   COD reduction. In the multivariate analysis of mineralization of phenol using hydrodynamic

354 cavitation, Chakinala et al. [31] have also indicated that an optimum loading of H_2O_2 must be

355 used for enhancing the efficacy of HC since an excess amount acts as a scavenger for

356 hydroxyl radicals and also contribute to excess COD in the pollutant stream.

357 *3.7 Comparison of energy efficiency and cost effectiveness*

358 The energy efficiency of cavitation based processes can be expressed in terms of

359 cavitational yield, which is defined as the ratio of quantifiable effects of cavitation per unit

360 energy supplied to the system [32]. In the present study, the cavitational effects have been

361 measured on the basis of number of moles of pollutant degraded and the power consumption

362 (KWh/m^3) have been evaluated on the basis of treatment time required for obtaining the 60 %

363 COD reduction. Table 8 provides the comparison of energy efficiency and cost effectiveness

364 of various processes such as HC and its combination with various loadings of H_2O_2 or Ozone.

365 The major fraction of the operating cost of these processes is typically utilized for supplying

366 the electrical energy to the system, since the cost of reagents such as H_2O_2 is very nominal as

367 compared to the cost of electrical energy [33]. Due to this, the cost of reagents is neglected in

368 the present study, while determining the cost effectiveness of various processes.

369 It has been observed that energy efficiency of individual hydrodynamic cavitation

370 notably enhances by combining it with H_2O_2. The cavitational yield of 1.589×10^{-9} moles/J

371 obtained in the case HC has approximately enhanced by 2, 3 and 4 times as a result of

372 combining it with 2, 5 and 10 g/L of H_2O_2 respectively. However, marginal enhancement in

373 the energy efficiency and cost effectiveness was observed by combining HC with ozone. This

374 is because additional energy (ozonator- 180 W and oxygen concentrator – 420 W) supplied to

375 generate the ozone has not resulted in significant enhancement in the rate of COD reduction.

376 Due to this, the combined application HC and ozone is not very economically attractive if

377 applied for the treatment of pesticide industry wastewater effluent under question. However,

378 it can be used as a pre-treatment tool for increasing the BI, which can be further treated with

16

379   simple biological means. It has also been observed that power consumption of hydrodynamic

380   cavitation alone is very high due to the low rate of COD reduction leading to high treatment

381   time. This makes the application of only HC a non-feasible and expensive option for the

382   treatment of industrial wastewater effluent. However, the operating cost of the HC can be

383   reduced if the wastewater effluent is available at considerable hydrostatic heads or pressures.

384   In that case treatment cost of HC can be reduced by designing the pressure reduction devices

385   in such a way that they will work in a hydrodynamic cavitation mode, without supply of any

386   additional energy [34].

387   Another option for reducing the treatment cost of HC is to use it in combination with

388   optimum loading of H_2O_2. It was observed that moles of effluent degraded in case of HC has

389   increased substantially by using HC in combination with H_2O_2, even though energy supplied

390   to both processes is identical. The combination of HC with H_2O_2 has found to be a relatively

391   cost-effective option; since it has significantly reduced the treatment time required for

392   obtaining desired COD reduction leading to less power consumption. The electrical cost of

393   10250.99 Rs. /m^3 incurred during HC has drastically reduced to 1951.63 Rs./m^3 (i.e. almost

394   by 4 times) when HC was combined with 10 g/L of H_2O_2. The results obtained are attributed

395   to the fact that hydrogen peroxide can easily dissociate in presence of hydrodynamic

396   cavitation leading to increased generation of hydroxyl radicals resulting into higher rate of

397   COD reduction [35]. Although, cost of electricity has appreciably reduced due to the

398   combination of HC and H_2O_2, even the reduced cost are excessive and cannot be observed by

399   a pesticide manufacturing unit and hence additional optimization is needed. However this

400   work has indicated the direction in which future effort needed.

401   **4. Conclusions**

402   The treatment of industrial pesticide effluent using HC and its combination with H_2O_2

403   and ozone has established the following important conclusions:

17

404 • The efficacy of hydrodynamic cavitation does not get enhanced appreciably by

405   increasing the extent of dilution of the effluent.

406 • The treatment of effluent using HC + Ozone (3 g/h) for the duration of 120 min

407   enhanced the biodegradability index from 0.121 to 0.324.

408 • The rate of TOC reduction obtained in case of individual hydrodynamic cavitation

409   enhanced approximately by 5 and 11 times by using HC in combination with 3 g/h of

410   ozone and 10 g/L of H_2O_2 respectively.

411 • H_2O_2 interferes with the COD analysis leading to the COD overestimation of 0.441

412   mg/L for 1 mg/L of H_2O_2.

413 • The rate of COD reduction obtained in case of individual hydrodynamic cavitation

414   has also enhanced roughly by 2.5 and 5 times by using HC in combination with 3 g/h

415   of ozone and 10 g/L of H_2O_2 respectively.

416 • The combined process of HC and H_2O_2 has proved to be the most energy efficient

417   and cost effective since electrical cost of 10250.99 Rs./m^3 incurred during HC has

418   appreciably reduced to 1951.63 Rs./m^3 (i.e. almost by 4 times) when HC was

419   combined with 10 g/L of H_2O_2. Although, the cost incurred during HC has

420   successfully reduced by combining it with process intensifying agents, the reduced

421   treatment costs are still prohibitive. However, this work has clearly indicated the

422   direction in which more efforts are needed.

18

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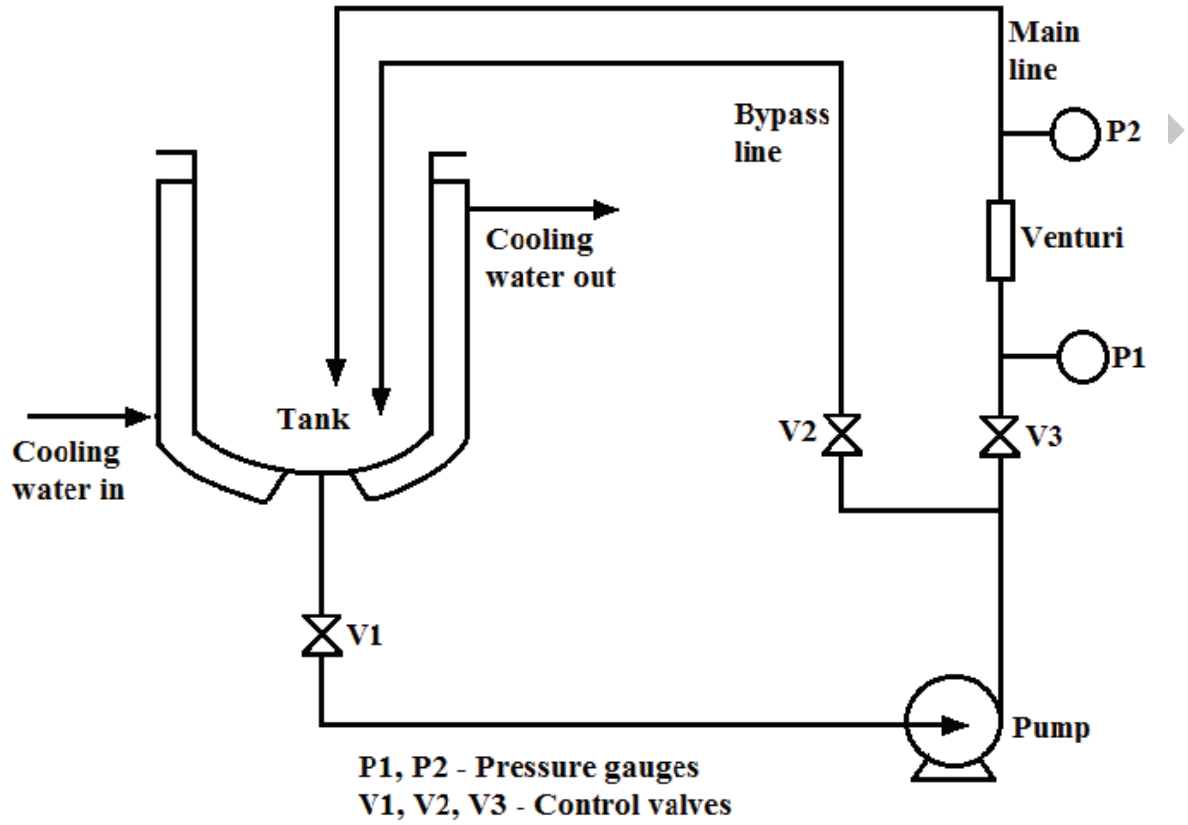**List of Figures:**

List of Tables:

Fig. 1. Schematic representation of hydrodynamic cavitation set-up.
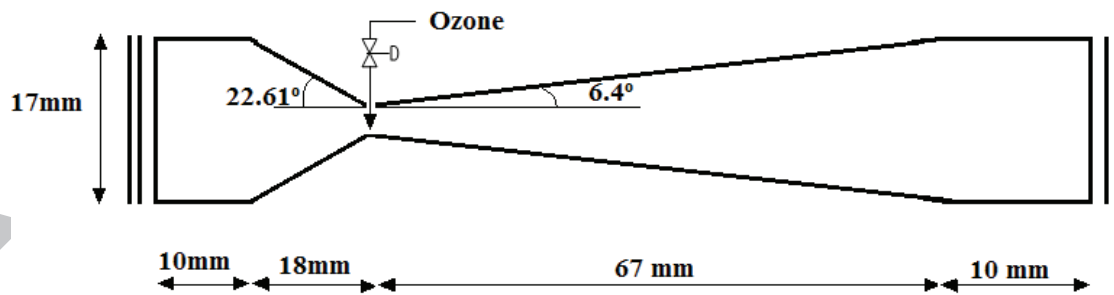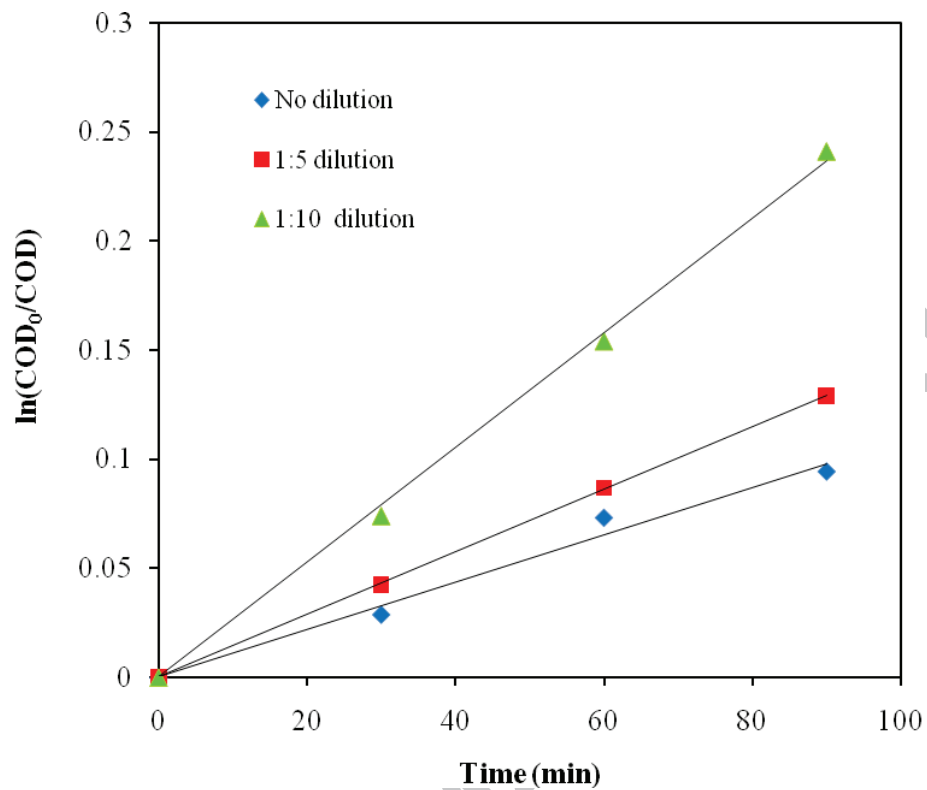

Fig. 2. Geometric specifications of a circular venturi (with throat diameter- 2mm)

Fig. 3. Effect of dilution on the mineralization of industrial pesticide effluent (Subjected to: treatment volume- 6 L, inlet pressure- 6 bar, pH-7)
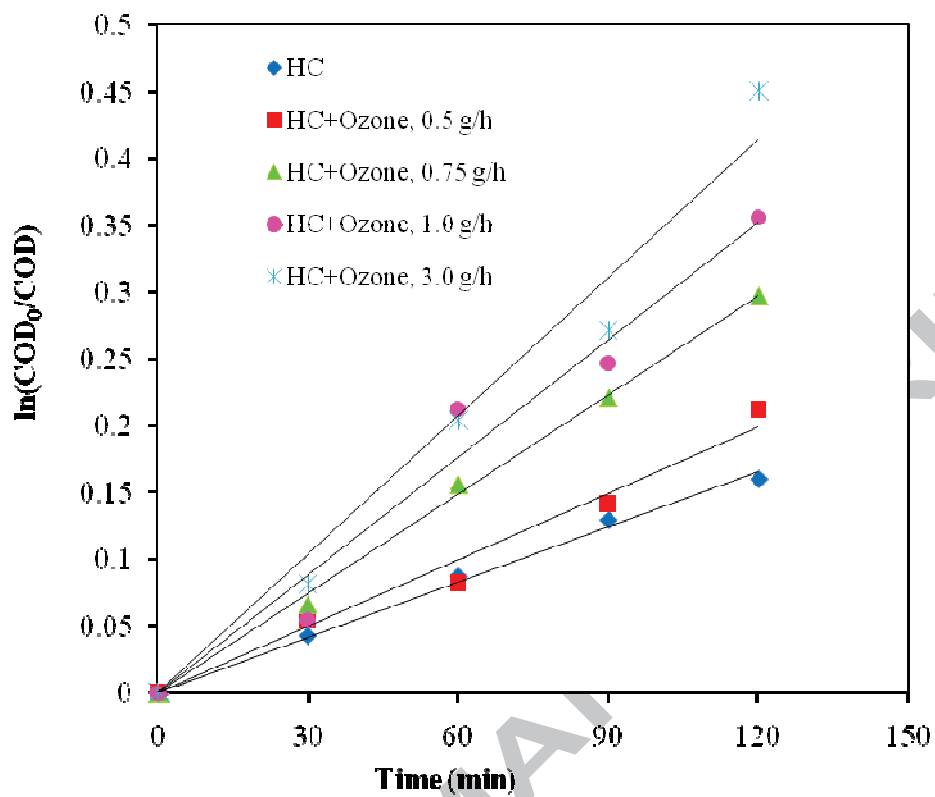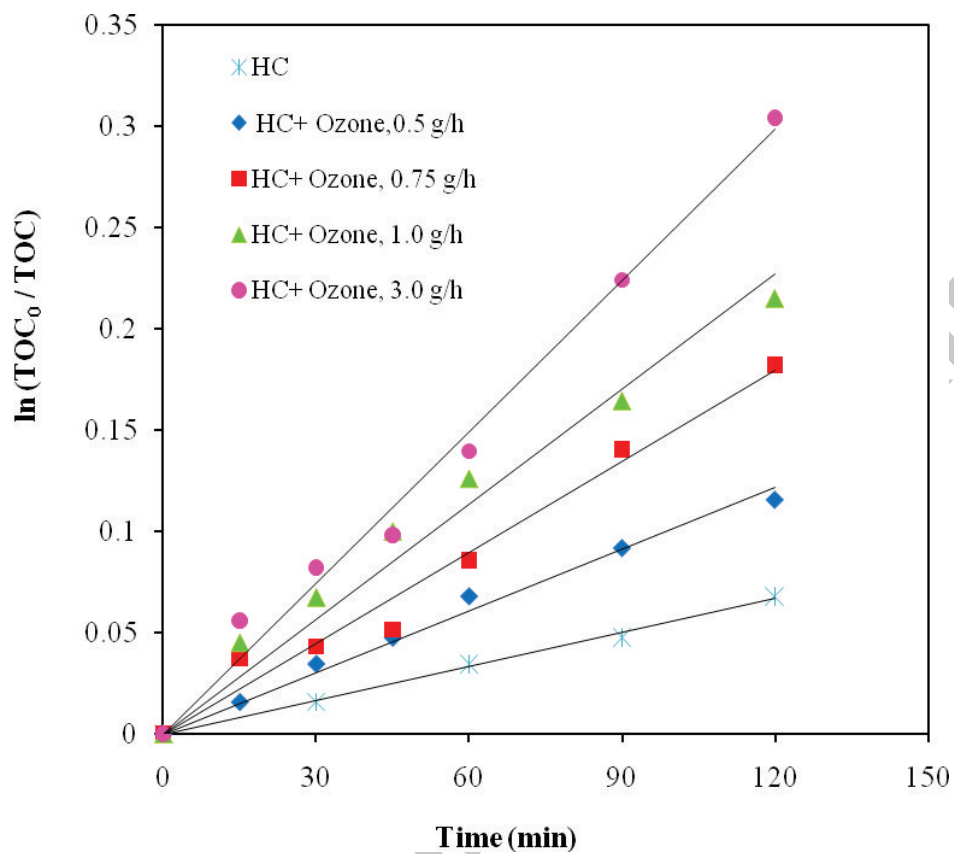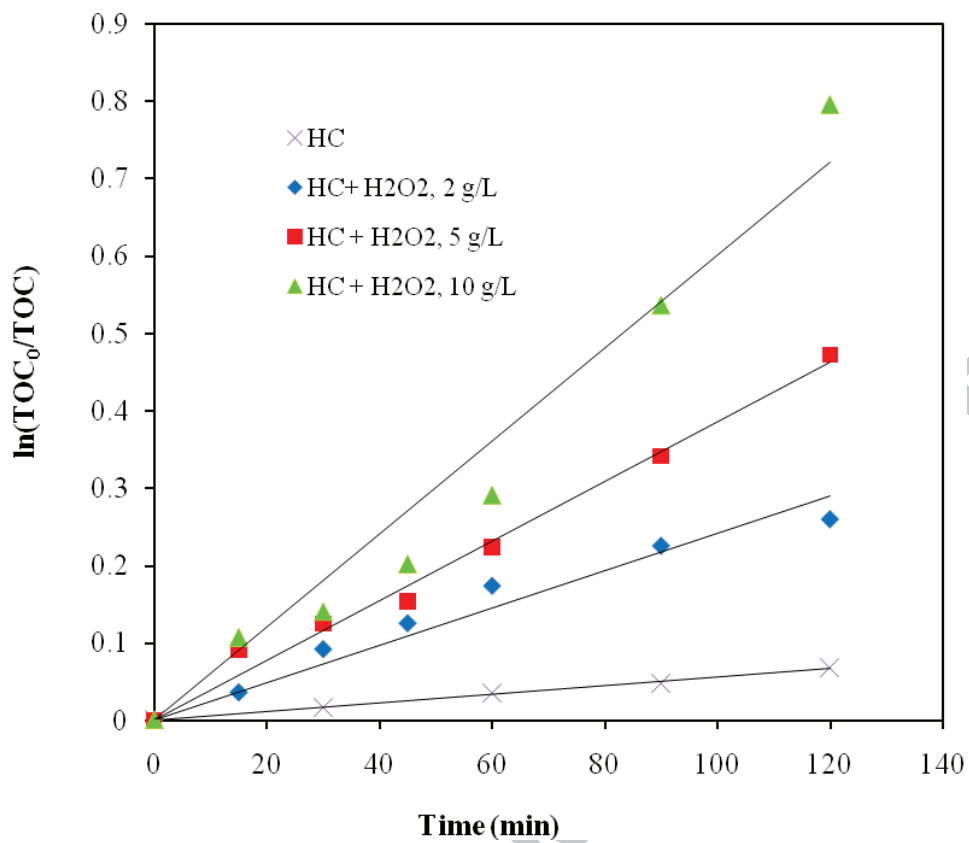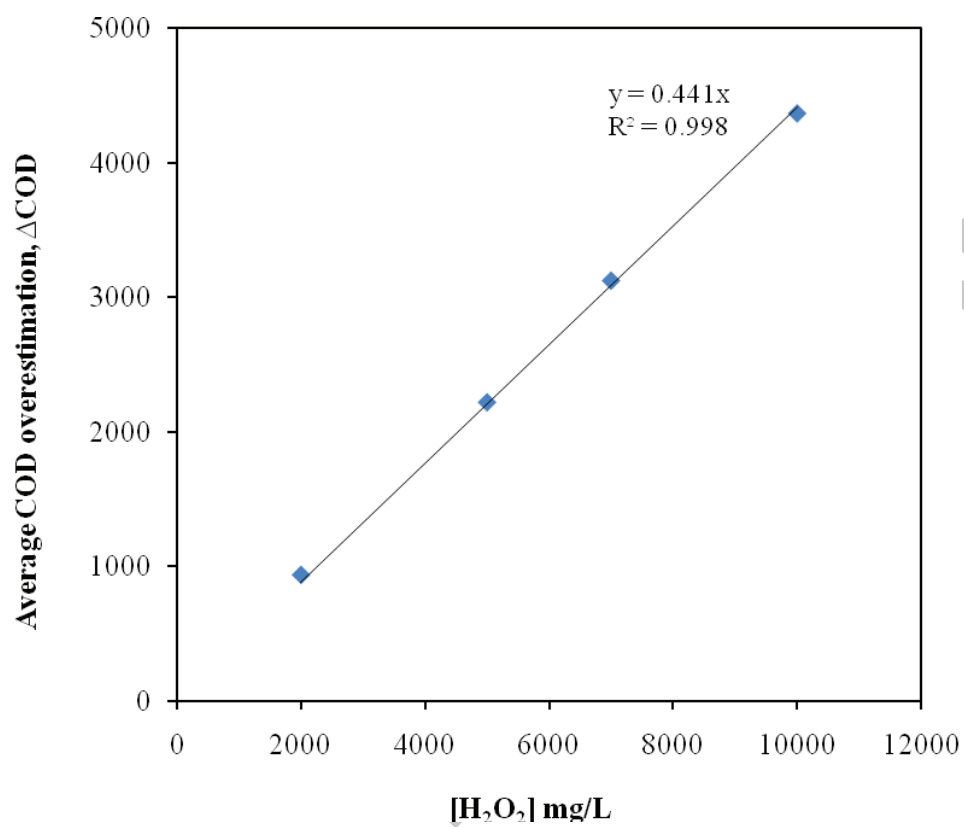
Fig. 4. Effect of loading of ozone on the COD reduction of industrial pesticide effluent (Subjected to: treatment volume- 6L, inlet pressure- 6 bar, pH-7)

Fig. 5. Effect of loading of ozone on the TOC mineralization of industrial pesticide effluent (Subjected to: treatment volume- 6L, inlet pressure- 6 bar, pH-7)

Fig. 6. Effect of loading of H_2O_2 on the TOC mineralization of industrial pesticide effluent (Subjected to: treatment volume- 6L, inlet pressure- 6 bar, pH-7)

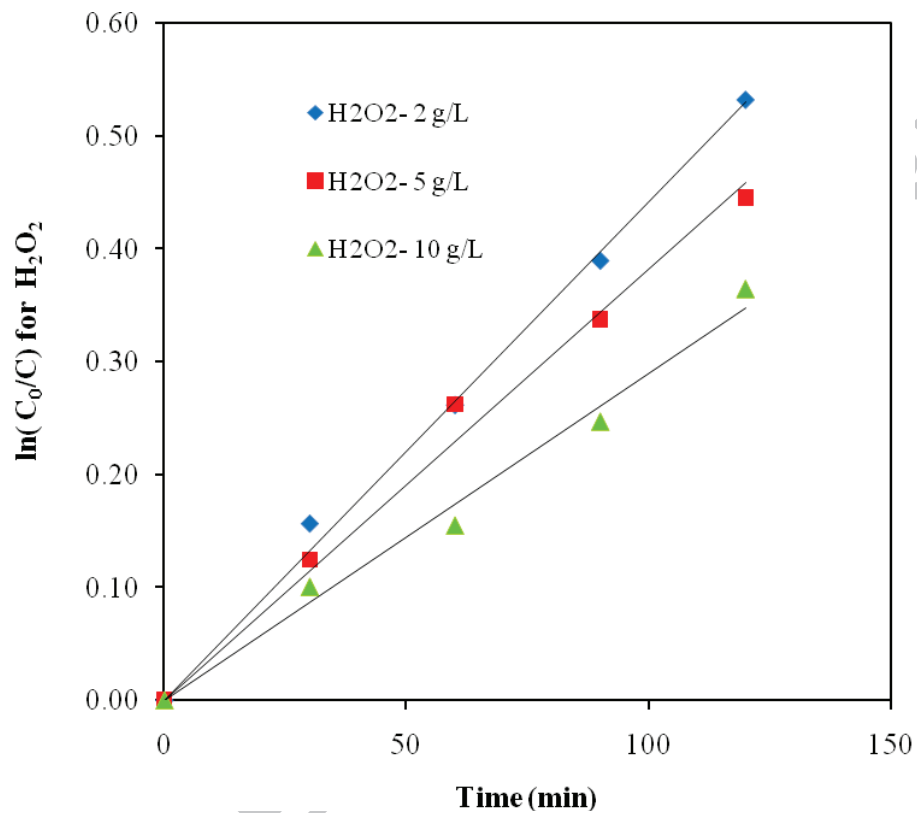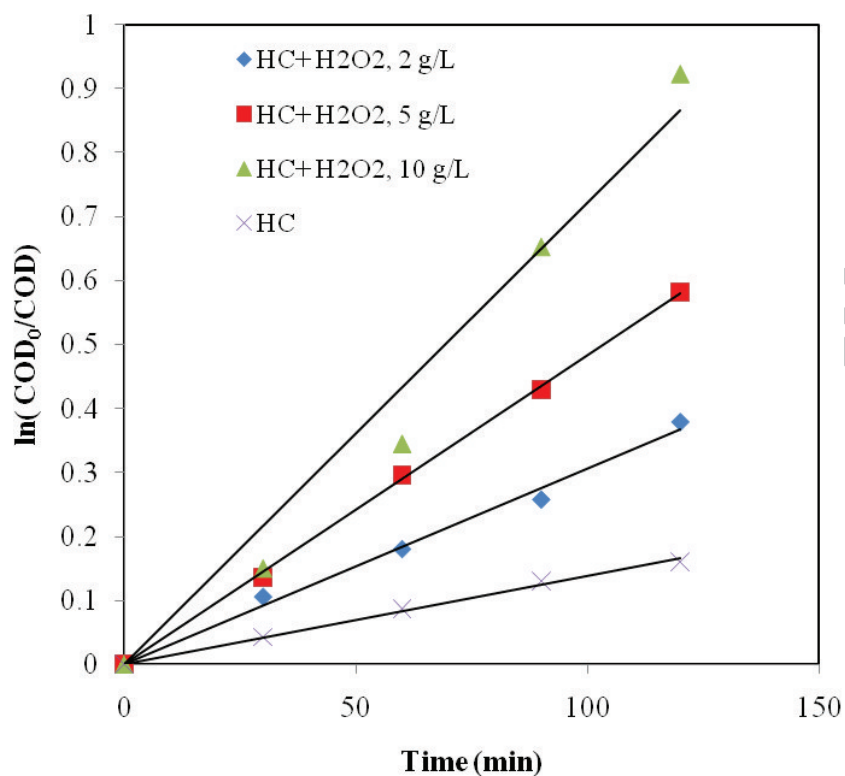**Fig.7 Effect of concentration of H_2O_2 on the average COD overestimation**

Fig. 8. First order rate of decomposition of H_2O_2 during HC + H_2O_2 Process

Fig. 9. Effect of loading of H_2O_2 on the COD reduction of industrial pesticide effluent (Based on corrected COD values and subjected to: treatment volume-6L, inlet pressure- 6 bar, pH-7)

Table 1

Characteristics of industrial pesticide effluent

Parameters	Values
pH	0.3-0.5
Total suspended solids	106 mg/L
Total dissolved solids	1,08,500 mg/L
Biological oxygen demand	2100-2200 mg/L
Chemical oxygen demand	17000-18000 mg/L
Ratio of BOD_5/COD	0.123

Table 2

Rate constant of COD removal (k), extent of COD reduction and moles of pollutant degraded at various dilutions of effluent.

Process	Rate constant of COD removal, $k \times 10^3$ min^{-1}	Regression coefficient	R^2	% COD reduction after 2 h	C_{A0}, based on COD, moles/L	C_{A0}-C_A, based on COD, moles/L
HC, No dilution	1.09	0.00109	0.99	8.98	0.386	0.0473
HC, 1:5 dilution	1.38	0.00144	0.99	14.77	0.0825	0.0126
HC, 1:10 dilution	2.63	0.00268	0.99	21.42	0.0350	0.0095

Table 3

Rate constant (k), extent of COD and TOC reduction and moles of pollutant degraded at various loadings of O_3

Process	Rate constant for COD removal in $k \times 10^3$ min^{-1}	Regression coefficient for COD removal	R^2	Rate constant for TOC removal in $k \times 10^3$ min^{-1}	Regression coefficient for TOC removal	R^2	% COD reduction after 2 h	% TOC reduction after 2 h	$C_{A0}-C_A$ based on COD, moles/ L
HC	1.38	0.001354	0.99	0.56	0.00056	0.99	14.77	6.58	0.0126
HC+ Ozone, 0.5 g/h	1.66	0.001702	0.98	1.01	0.000972	0.98	19.08	10.92	0.0146
HC+ Ozone, 0.75 g/h	2.47	0.002493	0.98	1.5	0.001491	0.99	25.69	16.63	0.0203
HC+ Ozone, 1.0 g/h	2.94	0.003033	0.97	1.89	0.001718	0.97	29.98	19.36	0.0238
HC+ Ozone, 3.0 g/h	3.45	0.003635	0.99	2.48	0.002443	0.97	36.26	26.204	0.0272

Table 4
Effect of HC and HC+ Ozone processes on biodegradability index

Process	Time in Minutes	Number of passes	Mean COD mg/L	Confidence limit of 95%	Mean BOD mg/L	Confidence limit of 95%	BOD/COD
HC, No dilution	0	0	12360	12360±48	1490	1490±3	0.121
	60	108	11490	11490±41	1740	1740±5	0.151
	120	216	11250	11250±40	1805	1805±6	0.160
HC, 1:5 dilution	0	0	2640	2640±8	325	325±4	0.123
	60	108	2420	2420±8	375	375±3	0.155
	120	216	2250	2250±7	400	400±5	0.178
HC+ Ozone, Ozone-1.0 g/h 1:5 dilution	0	0	2560	2560±9	320	320±4	0.125
	60	108	2070	2070±7	460	460±5	0.222
	120	216	1794	1794±5	510	510±5	0.284
HC+ Ozone, Ozone-3.0 g/h 1:5 dilution	0	0	2570	2570±8	322	322±4	0.125
	60	108	2097	2097±7	470	470±5	0.224
	120	216	1638	1638±6	530	530±5	0.324

35

Table 5

Rate constant (k) and extent of TOC reduction and moles of pollutant degraded at various loadings of H_2O_2

Process	Rate constant for TOC removal, $k \times 10^3$ min^{-1}	Regression coefficient	R^2	% TOC reduction (120 min)	C_{A0} moles/L	C_{A0}-C_A moles/L
HC	0.56	0.00056	0.99	6.58	0.0711	4.614E-03
HC+ H_2O_2, 2 g/L	2.42	0.00223	0.95	22.85	0.0694	1.749E-02
HC+ H_2O_2, 5 g/L	3.86	0.00378	0.98	37.66	0.0718	2.661E-02
HC+ H_2O_2, 10 g/L	6.02	0.00648	0.96	54.87	0.0755	3.884E-02

Table 6

COD overestimation (ΔCOD) values for various concentrations of H_2O_2

[H_2O_2], g/L	Average ΔCOD	Relative standard deviation, %	Confidence limit of 95%
2	940	2.29	940 ± 18.83
5	2221	4.23	2221 ± 82.40
7	3124	1.68	3124 ± 45.99
10	4366	3.36	4366 ± 128.47

Table 7

Rate constant (k) and extent of COD reduction and moles of pollutant degraded at various loadings of H_2O_2

Process	Rate constant for COD removal, $k \times 10^{-3}$ min^{-1}	Regression coefficient	R^2	% COD reduction (120 min)	C_{A0} moles/L	C_{A0}-C_A moles/L
HC	1.382	0.001354	0.995	14.77	0.0825	0.0126
HC + H_2O_2, 2 g/L	3.064	0.003036	0.992	31.57	0.0806	0.0248
HC + H_2O_2, 5 g/L	4.835	0.004870	0.999	44.18	0.0801	0.0352
HC + H_2O_2, 10 g/L	7.215	0.007834	0.972	60.29	0.0818	0.0474

Table 8
Comparison of cavitational yield and cost effectiveness of various processes

Process	Cavitational yield, moles effluent degraded/J	Rate constant, k (based on COD) min^{-1}	Treatment time in h to achieve the 60% COD reduction	Power consumption in KWh/m^3	Cost of Electricity Rs./m^3
HC	1.589E-09	1.38E-03	11.050	2025.89	10250.99
HC + H_2O_2, 2 g/L	2.796E-09	2.68E-03	5.707	1046.26	5294.05
HC + H_2O_2, 5 g/L	4.248E-09	4.55E-03	3.359	615.74	3115.65
HC + H_2O_2, 10 g/L	6.011E-09	7.26E-03	2.104	385.70	1951.63
HC + Ozone, 0.75 g/h	1.658E-09	2.47E-03	6.183	1751.79	8864.07
HC + Ozone, 1.0 g/h	1.943E-09	2.94E-03	5.194	1471.74	7447.03
HC + Ozone, 3.0 g/h	2.224E-09	3.45E-03	4.427	1254.18	6346.16

37

Highlights:

- ✓ Treatment of industrial effluent using hydrodynamic cavitation (HC) based processes

- ✓ Biodegradability index of the effluent increased from 0.123 to 0.324 using HC+Ozone

- ✓ Higher rate of COD and TOC reduction obtained using HC+H_2O_2 as compared to HC alone

- ✓ Cost estimation is done for individual HC and HC based hybrid processes

- ✓ Combined HC and H_2O_2 process is the most effective and economical one