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Degradation of azo dyes using hydrodynamic cavitation and external oxidants

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ABSTRACT

Hydrodynamic cavitation (HC) with an orifice as the cavitating device was used to study the degradation of methyl orange dye. The operating parameters of the process, such as pH and inlet pressure, were optimized. The effect of hydroxyl radical promoters like Fenton oxidation and hydrogen peroxide (H₂O₂) on the extent of degradation was also investigated. It has been observed that acidic conditions (pH 2) favor the degradation of methyl orange. The combined effect of hydrodynamic cavitation with hydrogen peroxide was investigated at the solution's natural pH and an optimized solution pH 2. Maximum degradation of 99.2% was observed at natural pH, whereas complete degradation of methyl orange dye was observed at pH 2 with 8 ml/L of hydrogen peroxide addition. The hybrid process of HC/Fenton and HC/H₂O₂ showed the highest efficiency for the degradation of methyl orange with a minimum energy requirement (0.11 kWh) and operational cost (USD \$ 0.0062/L).

1. Introduction

A conventional treatment plant has enormous challenges when dealing with the complex and refractory compounds found in the wastewater flow from industrial units. Due to their colour and toxicity, unutilized dyes found in textile industry wastewater seriously harm the ecosystem. The manufacturing and dying processes result in the loss of 10–20% of the entire amount of dyestuff

produced [1,2]. In the textile and paper sectors, 50–70% of the colors used fall under the azo group [3,4]. Wastewater that contains dye poses a risk not just to humans but also to aquatic life and the environment. Human exposure to wastewater has several carcinogenic and mutagenic effects [5,6]. Therefore, before being released into the water body, dye-containing effluent must be treated. The completed dye degradation is not achieved using conventional physicochemical treatment methods.

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Another major issue associated with these methods is treating secondary solid waste [7,8]. Organic pollutants are degraded using advanced oxidation processes such as cavitation, Fenton oxidation, ozonation, and photocatalysis. Generated hydroxyl radicals ($\cdot\text{OH}$) from these processes have a very high oxidation potential of 2.80V [9]. Although AOPs are highly successful on a laboratory scale and have fewer industrial-scale applications [10,11]. Substantial efforts are required to develop the sustainable pathway of AOP for the industrial scale of operation. Cavitation is a unique technique extensively studied for the treatment of wastewater containing dyes. It has the ability to produce extremely reactive free radicals, which are crucial building blocks for speeding up dye oxidation. Cavitation refers to the nucleation, growth, and implosion of vapor-bubble cavities [12-14]. Hydrodynamic cavitation is a better approach for treating dye and is easily scalable for industrial operation [15-17]. Cavities in hydrodynamic cavitation are produced as a result of pressure changes and geometry changes brought on by constriction, such as orifice, venturi, etc. When liquid passes through constrictions, the kinetic head increases at the pressure head expense and results in formation of vaporous cavities [18,19]. Cavities develop when the pressure at the venturi throat reaches the liquid's vapor pressure or falls below it. This vaporous cavity continues to grow till it reaches the maximum size at very low pressure. The life span of the formed cavities is significantly less (~ 0.3 microseconds), and the temperature inside the cavities is about 10000 K and pressure of about 1000 atm [20]. Extreme temperatures within the compressing vaporous cavity split water molecules into $\cdot\text{H}$ and $\cdot\text{OH}$. The hydroxyl radicals ($\cdot\text{OH}$) have a very high oxidation potential for treating organic dye like methyl orange [11]. Methyl orange is widely used as an azo dye for various industrial applications and has shown different behavior in acidic (red color) and alkaline (yellow color) mediums. Dye decolorization is dependent on the breakdown of nitrogen double bonds ($-\text{N}=\text{N}-$). The structure of the methyl orange is chemically described as $(\text{C}_{14}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S})$ Sodium 4-[[4-(dimethylamino) phenyl] diazenyl] benzene-1-sulfonate. Very few studies have been conducted on the degradation of

methyl orange using the combined effect of hydrodynamic cavitation, Fenton reagent, and hydrogen peroxide. Literature is available on photolytic and ultrasonic degradation of methyl orange combined with various catalysts [21-23]. Still, no complete degradation of methyl orange has been found based on the literature survey [11,15,16]. Li et al., [17] studied methyl orange degradation using hydrodynamic cavitation combined with Cu nanoparticles and reported 6.8% and 83% degradation using HC alone, 40 mg/L of loading Cu nanoparticles, respectively. Yang et al., [24] studied methyl orange degradation using hydrodynamic cavitation with ClO_2 and reported 83% degradation achieved at optimum loading of 8 mg/L. Innocenzi et al., [21] also investigated the degradation of methyl orange dye (containing salts as a pollutant) using hydrodynamic cavitation combined Fenton process. However, the reported value of degradation is 90%. The degree of dye degradation is greatly dependent on the concentration of hydroxyl radicals. The dissociation of water molecules or the production of hydroxyl radicals are both favoured in an acidic environment. The too-low acidic medium does not favor the operation scale to avoid corrosion issues with the scale of operation. More investigations are required to scale up the process and optimize an operational parameter of hydrodynamic cavitated degradation of methyl orange. Thus, the present work's objective and novelty is to improve the treatment strategies for the total degradation of methyl orange in the presence of external oxidants such as Fenton and hydrogen peroxide at the optimized condition of process reaction parameters. Initially, the various operational process reaction parameters, such as effect of inlet pressure of cavitating device and solution pH has been investigated. Process intensification studies further have been investigated using a combined hydrodynamic cavitation process with hydrogen peroxide and Fenton process. The comparison of efficacy has been achieved in terms of higher degradation, energy consumption and operating cost.

2. Materials and methods

2.1. Materials

Methyl Orange ($C_{14}H_{14}N_3NaO_3S$) is also called Acid Orange 52, Helian thin, and Orange III procured from S. D. Fine Chemicals, Mumbai, India and the molecular weight is 327 g/mole. The aqueous solution of methyl orange was prepared using demineralized water. Methanol (CH_3OH), H_2SO_4 (98% w/w), sodium hydroxide (NaOH), H_2O_2 (30% w/v industrial grade), and Ferrous Sulfate Heptahydrate ($FeSO_4 \cdot 7H_2O$) were purchased from M/s Loba Chemicals Pvt. Ltd., Mumbai, India

2.2. Equipment and procedure

The hydrodynamic cavitation reactor setup used in experimental work has been depicted in Figure 1 which consists of a treatment tank and regenerative pump (1.1 kW). The output from the pump is divided into two lines: the mainline and the bypass line. A cavitating device, consisting of a 2 mm aperture, is part of the mainline. The upstream

(P1) and downstream pressures (P2) of the cavitation device is measured using pressure gauge. According to several literatures [25,26], the initial concentration of methyl orange solution was 20 mg/L and kept constant for all studies. In all of the studies, 10 liters were considered the treatment volume. Experiments were carried out by varying the inlet pressure over the range of 2 to 5, while studies of pH were carried out over the range of 2 to 12, with the goals of determining the optimal values for pressure and pH, respectively. After a standard period of time, the 5 mL sample was removed from the solution. The samples were filtered using Whatman's filter paper (1 μ m), and the degradation of methyl orange was then examined using a Chemito SpectraScan UV 2600 double-beam UV-VIS spectrophotometer at a wavelength of 464 nm. The present work's experimental error is within 2% of the average value presented in the figures and discussion.

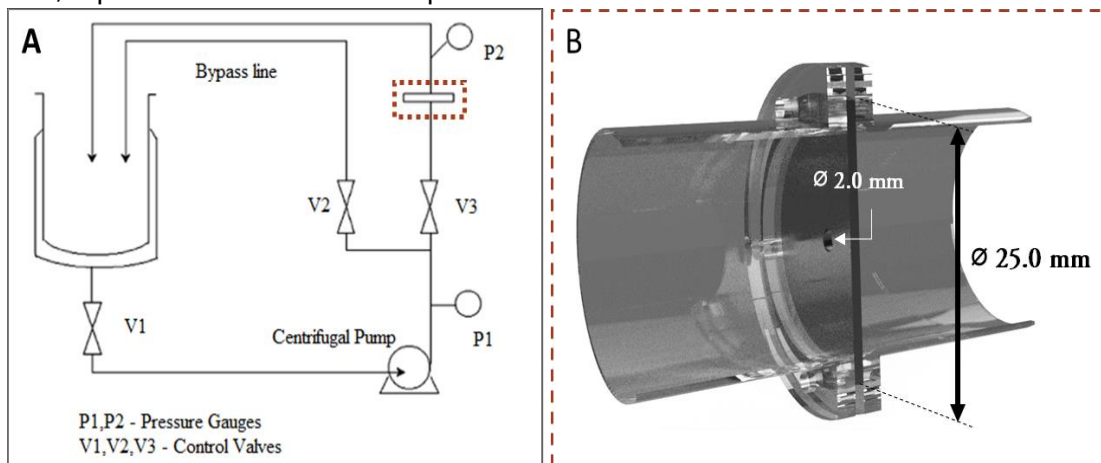


Fig. 1. Schematic representation of the experimental setup for (A) hydrodynamic cavitation and (B) the orifice plate.

3. Results and discussion

3.1. Effect of inlet pressure

The cavitation intensity of any cavitating device is dependent on inlet pressure and cavitation number. Hydrodynamic cavitation was used in experiments to examine the impact of inlet pressure of cavitating device on methyl orange degradation over a range of 2 bar to 5 bar. In order to create the cavitation effect, a 2-mm orifice was used. The tendencies obtained are depicted in Figure 2. Methyl orange decomposition was shown to be more extensive at higher inlet pressures (from

2 to 4 bar) and to decrease further at higher inlet pressures (5 bar). The maximum degradation of methyl orange as 6.41% was obtained at an optimum inlet pressure of 4 bar in 60 min. It may be because with increasing inlet pressure of orifice, local energy dissipation rate and intensity of turbulence increased. Due to high turbulence in the mixture, a more significant number of cavities collapse, which increased the generation of cavitation intensity. The increase in cavitation collapse leads to a higher temperature and pressure condition, which breaks the water molecule trapped in the cavity, thereby increasing

hydroxyl radicals. Hence, the degradation of methyl orange is higher with increasing inlet pressure up to the optimal value of pressure. Also, with an increase in the inlet pressure, velocity through the orifice increased, and the cavitation number decreased and resulted in hydroxyl radicals [27,28].

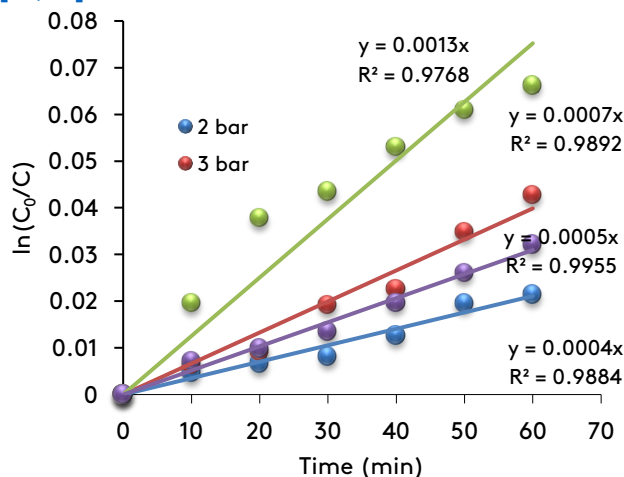


Fig. 2. Effect of inlet pressure on the extent of degradation of methyl orange using hydrodynamic cavitation (initial concentration of methyl orange: 20 mg/L, natural pH: 6.8).

A study on the degradation of methyl parathion using an orifice plate with a diameter of 2 mm reported a maximum of 22.4% degradation of methyl parathion at 4 bar inlet pressure in 120 mins for 20 mg/L methyl parathion solution [22]. Dhanke and Wagh [29] also observed similar results during the investigation of Acid-Red-18 degradation reported, wherein a maximum degradation of 60% was attained at 6 bar of intake pressure with the same size as the orifice plate. As a result, the remaining experiments in this investigation were all conducted with an optimal inlet pressure of 4 bars. The kinetics of methyl orange degradation were studied to determine kinetic parameters such as reaction order and reaction rate constant. A first-order kinetic model was found to match the data well, and the model equation (1) can be given as follows:

$$\ln\left(\frac{C_0}{C}\right) = kt \quad (1)$$

where C_0 initial concentration at time zero C instantaneous concentration at time t as

illustrated in Figure 2, the first-order kinetic study ($\ln(C_0/C)$ vs. time) yielded a line with a slope equal to the pseudo-first-order kinetic rate constant. It can be seen from the figures that a kinetic rate constant of 0.0013 min^{-1} was obtained for at a 4 bar of input pressure, and a regression value of $R^2 = 0.98$ indicates that the experimental data were well fit.

3.2. Effect of solution pH

In the current study, the impact of solution pH was examined using a range of solutions from 2 to 12 at dye concentration of 20 mg/L and the optimal inlet pressure of 4 bar. To modify the pH of the solution, a molar solution of 0.5 mol/L H_2SO_4 or 1 mol/L KOH was used. The obtained results of impact of solution pH have been depicted in Figure 3. According to the Figure 3, the extent of degradation increases as the pH of the solution decreases, with acidic pH favoring degradation over alkaline pH. Nearly 10% of degradation was obtained in an alkaline medium at pH 12. The maximum removal of 81.6 % was obtained at a pH of 2. The higher degradation efficiency of dye solution at low pH may be due to H^+ ions directly involved in the reduction reaction [30]. Also, dye molecules present in the molecular state at low pH easily enter the cavities gas-water interface due to their hydrophobic nature and get readily attacked by hydroxyl radicals. The dye molecule gets ionized in an alkaline medium and remains in a bulk solution [31,32]. Saharan et al., [23] reported the maximum degradation of 72.72% of orange-G in an acidic medium at a pH of 2. It was also reported that the degradation of orange-G decreased at solution pH of 9 while no degradation was reported at solution pH of 11 and 13. Sun et al., [33] studied acid black 1 degradation with the help of acoustic cavitation where it was reported that the degradation efficiency increases from 5.13% to 98.83% while a solution of pH decreases towards acidic from 6 to 3. It is important to note here that the hydroxyl radical formation controls dye degradation. Therefore, the information provided would be very helpful for determining the optimum solution pH for more decomposition of dye.

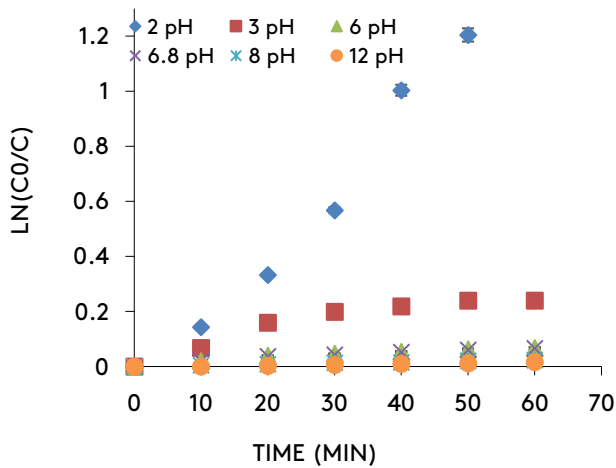


Fig. 3. Effect of pH on the extent of methyl orange degradation (20 mg/L initial concentration, 4 bar inlet pressure).

3.3. Effect of H_2O_2 loading at natural solution pH

Hydrogen peroxide (a potent oxidizing agent) provides additional hydroxyl radicals for the degradation of methyl orange. The rate formation of hydroxyl radical from the interaction of hydrodynamic cavitation is not sufficient for the complete degradation of the targeted compound. Combining the synergy of hydrogen peroxide with hydrodynamic cavitation is highly beneficial for providing a higher concentration of hydroxyl radical. Thus, the effect of H_2O_2 loading with a range of 2 ml/L to 10 ml/L on degradation of methyl orange has been investigated at natural pH. All experiments were carried out at an already optimized 4 bar inlet pressure with a fixed 20 mg/L dye concentration and natural pH for a fixed time of 60 min. The results of effect of H_2O_2 loading on degradation of methyl orange were shown in Figure 4. It is clearly observed from the figure that the addition of hydrogen peroxide strongly affects the degradation efficiency of methyl orange at natural pH. The obtained results indicates that the dye degradation increase from 62.50% to 99.21% with an increase in H_2O_2 concentration from 2 to 8 ml/L with further increase in hydrogen peroxide concentration up to 10 ml/L, decreasing the extent of degradation (85.80%) of methyl orange observed. The increase in the extent of degradation with an increase in hydrogen peroxide concentration is mainly due to the enhanced generation of hydroxyl radical due to the splitting of hydrogen peroxide and an increase in micro-

mixing. Furthermore, the extent of degradation of methyl orange has been decreased at higher hydrogen peroxide loading due to hydroxyl radicals' scavenging. Zupac et al. (2013) [34] reported the pharmaceutical degradation containing wastewater using 30% w/v of H_2O_2 loading of 20 ml/L was an optimum with a degradation in the order of 19, 19.9, 23, 29, 89 and 99.9% for ibuprofen, naproxen, clofibric acid, ketoprofen, carbamazepine, and diclofenac, respectively, in 30 min at a 6 bar of inlet pressure.

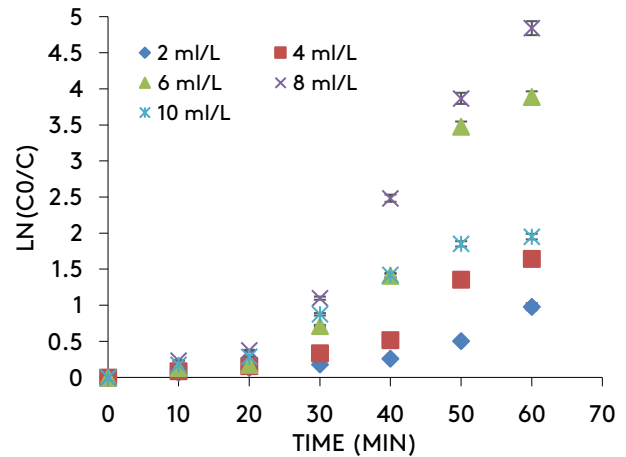


Fig. 4. Effect of H_2O_2 loading on the degradation of methyl orange at natural pH of solution 6.8 (initial concentration: 20 mg/L, inlet pressure: 4 bar).

3.4. Combination of HC and H_2O_2 at optimum pH

It is interesting to analyze the effect of varying pH at constant/optimize the concentration of H_2O_2 as the pH of the solution plays a crucial role in dissociating water molecules and helps to enhance the concentration of hydroxyl radicals. Experiments were conducted to combine the effect of HC with varying concentrations of H_2O_2 (0.025 to 0.2 mL/L) at pH 2 optimization. The initial inlet pressure (4 bar) and initial dye concentration of 20 mg/L were kept constant. The obtained results have been given in Figure 5. The figure shows that complete degradation of methyl orange was obtained using the combined HC/ H_2O_2 at optimal pH with higher loadings of hydrogen peroxide, i.e., 0.2 ml/L in 50 min. It is essential to emphasize an important point here that the lower loadings of 0.025 ml/L to 0.2 ml/L of hydrogen peroxide (were sufficient to degrade methyl orange at optimal pH compared to that required loading of hydrogen peroxide at natural pH using the HC/ H_2O_2 process

(2 ml/L to 10 ml/L loading of hydrogen peroxide). It has been also observed that above 90% of degradation was obtained for loading hydrogen peroxide of 0.025 ml/L at a pH of 2. In contrast, a 6 ml/L to 8 ml/L concentration of hydrogen peroxide was required to achieve the same degradation of methyl orange at natural solution pH. It has been shown that the degradation of methyl orange increases rapidly in an initial period of 10 min using optimal pH compared to natural pH using a combined HC/H₂O₂ process with varying hydrogen peroxide loadings. Gogate and Bhosale, [35] discussed the effect of H₂O₂ concentration on dyes (Orange acid II and Malachite or brilliant green) using an orifice (2 mm) as a device for hydrodynamic cavitation. It was reported that the extent of degradation of orange acid II increased from 60% to 96% with the H₂O₂ concentration varying from 95.2 to 571.2 mg/L at a 5 kg/cm inlet pressure. However, the extent of degradation varied from 77% to 86% with a similar range of H₂O₂ concentrations for Malachite or brilliant green. It should be mentioned that raising the concentration of hydroxyl radical by adding hydrogen peroxide over the recommended level is difficult to do since the pollutant availability controls the regulatory mechanisms [36–38].

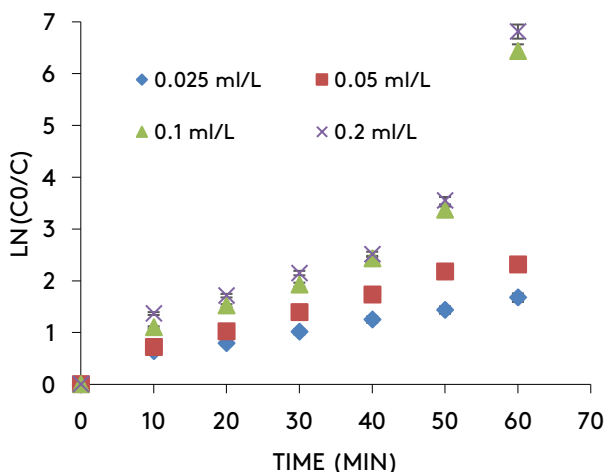
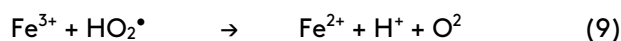
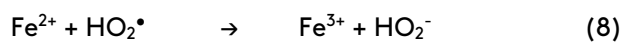
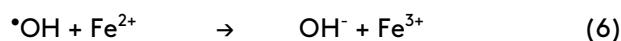
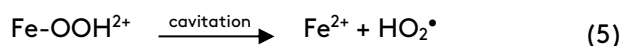
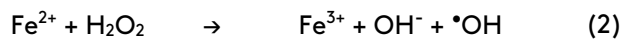


Fig. 5. Effect of H₂O₂ loading on the degradation of methyl orange at optimum pH of solution 2 (initial concentration: 20 mg/L, inlet pressure: 4bar).

3.5. Effect of Fenton/H₂O₂ loading at optimum pH

The Fenton is a very effective process for treating industrial effluents. It results in producing many hydroxyl radicals by dissociating H₂O₂ under acidic pH conditions. Initially, experiments were carried

out at already optimized operating parameters such as pH of 2, the inlet pressure of 4 bar, and fixed 20 mg/L initial concentration. The FeSO₄·7H₂O loading was varied over the range from 0.00625 gm/L to 0.2 gm/L using constant loading of 0.2 ml/L H₂O₂. Figure 6 illustrates the decomposition of methyl orange during the HC/Fenton process. The degradation of methyl orange increased with a decrease in Fenton loading from 0.2 gm/L to 0.00625 gm/L. The maximum 98.94% degradation was obtained at 0.00625 gm/L of FeSO₄·7H₂O in 60 min of treatment time. It is crucial to optimize the quantity of Ferrous catalyst and H₂O₂ dosage as a significant increase in Fe ions leads to an increase in the concentration of unutilized iron salts and increases the total dissolved solids (TDS). The effect of FeSO₄·7H₂O and H₂O₂ dosage was investigated at optimum pH (2) and inlet pressure of 4 bar. The free radical mechanism of the Fenton reaction is as shown in Eqs (2) – (9) [39–42].



Similar results of removal of pollutants from wastewater with the help of combined hydrodynamic cavitation and advanced Fenton's process have been reported in the literature. Mishra and Gogate, (Mishra and Gogate, 2010) studied degradation of Rhodamine B using hydrodynamic cavitation (venturi) in conjunction with Fenton's reagent at a loading of FeSO₄:H₂O₂ in the ratio of 1:5 at optimum pH (2.5) and inlet pressure (4.8 atm) and reported maximum extent of degradation 99.9% and 57% total organic carbon (TOC) removal. Chakinala et al., [43] reported about 85% chemical oxygen demand (COD) removal for real industrial effluent by a liquid whistle reactor (hydrodynamic cavitation) and advanced Fenton process and higher pressure (2000 psi) with the subsequent addition of H₂O₂

(1900 mg/L) results in rapid TOC removal (60-80%). It's should be noted here that the significant degradation of methyl orange is dependent on the amount of dosage or concentration of ferrous catalyst and loading of hydrogen peroxide and hence, the importance of the present work is clearly established.

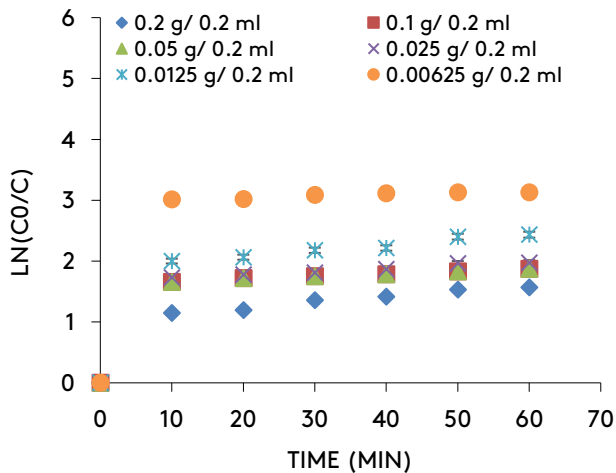


Fig.6. Effect of Fenton loading on degradation at optimum pH of solution 2 (initial concentration: 20 mg/L, inlet pressure: 4bar, constant loading: 0.2 ml/L H₂O₂).

3.6. Effect of pH on Fenton/H₂O₂ loading

The pH of a solution with Fenton loading plays a crucial role in enhancing hydroxyl ions concentration. To investigate the effect of solution pH on Fenton/H₂O₂ loading on the degradation of methyl orange, the optimal inlet pressure of 4 bar and an initial dye concentration of 20 mg/L were used. The range of methyl orange solution pH was used from 2 to 6 in the present study while Fenton reagent of 0.00625 gm/L FeSO₄·7H₂O and 0.2 mL/L H₂O₂ constants. The obtained trends of effect of solution pH on Fenton/H₂O₂ loading on degradation of methyl orange have been given in Figure 7. It has been observed from the figure that the methyl orange is completely degraded at a pH of 3 within 60 min of treatment time. This may be due to the fact that as the iron power easily dissolves under acidic solution pH, it produces more Fe ion which further react with H₂O₂ and generates hydroxyl radical which results in higher degradation at lower pH. At lower pH (< 4), the iron metal is oxidized into Fe⁰ to Fe²⁺ in presence of hydrogen peroxide which further reacts with H₂O₂ and produces a number of hydroxyl radicals [44,45]. However, with an

increase in pH from 4 to 6, percentage degradation decreased significantly from 93.11% to 22.16%. Results were consistent with the literature findings and reported that the Fenton process's optimum pH is in the range of 3-4 [46]. Due to the synthesis of Fe (II) and the buffer's ability to prevent free radicals from forming, the rate of decomposition reduces for pH values greater than 4, which precipitates ferric oxy-hydroxide and prevents the regeneration of ferrous ions [47]. In addition, the hydroxyl radical's oxidation potential was lower in basic solutions than in acidic solutions, so higher pH values result in lower degradation. The investigation on a combination of hydrodynamic cavitation with Fenton's chemistry for degradation of dichlorvos reported 91.5% degradation at a loading of FeSO₄:H₂O₂ in the ratio of 3:1 using an orifice with a 2 mm hole diameter, which was remarkably higher than that, obtained using hydrodynamic cavitation alone [48]. Also, Bagal and Gogate, [49] investigated the degradation of 2, 4 di-nitro phenols using HC/Fenton process and reported 83.42% degradation at H₂O₂: FeSO₄ loading ratio of 1:6 at optimized pH of 4, inlet pressure 4 bar, and 20 mg/L initial concentration. Overall, it can be said that acidic conditions are more favorable for the degradation of methyl orange dye using a combined HC/Fenton process.

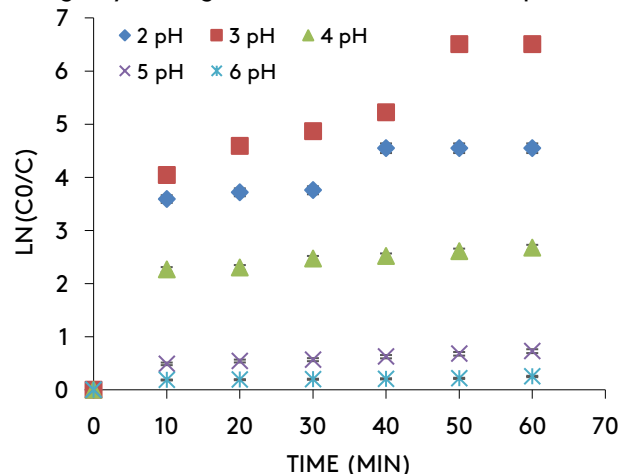
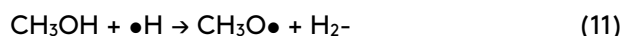
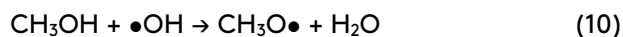


Fig. 7. Effect of varying pH of solution on the degradation of methyl orange at constant Fenton loading (initial concentration: 20 mg/L, inlet pressure: 4bar, constant loading: 0.00625 g/L FeSO₄·7H₂O 0.2 ml/L H₂O₂).

3.7. Effect of methanol

The experiment was conducted in the presence of methanol to examine the impact of produced

hydroxyl radicals on the decomposition of methyl orange. Methanol acts as a scavenger for hydroxyl radicals. Dosage of methanol was varied over the range of 0.5 to 1 ml/L with optimal conditions (inlet pressure 4 bar, pH 2 and 20 mg/L initial concentration). One can observe that there is a significant decrease in degradation with an increase in methanol dosage from 0.25 to 0.5 ml/L beyond which there is no degradation observed. The maximum degradation of 1.54% has been observed at 0.25 ml/L of methanol. Zheng et al. [50] found that the introducing of methanol as a radical scavenger showed a decrease in the rate of phenol's degradation, validating free radical attack as a regulating mechanism. According to research conducted by Devi and Kumar [51], who investigated the photodegradation of methyl orange dye, methanol has a higher ability to react with hydroxyl radicals with a second-order rate constants of $9.7 \times 10^8 \text{ mol}^{-1} \text{ s}^{-1}$ and $2.6 \times 10^6 \text{ mol}^{-1} \text{ s}^{-1}$, respectively, demonstrated in equations (10) and (11).



Overall, it is possible to say that methanol performs the function of a radical scavenger throughout the degradation process; as a result, the level of degradation that was obtained was significantly lower when compared to other sets of investigations that were explored in the present study. Additionally, it is important to highlight in this context that even with increased amounts of methanol loading, no degradation of methyl orange was shown to occur.

3.8. Comparative analysis of process

Table 1 displays the results of the analysis of the data using the three parameters of process duration, degree of degradation, and pseudo-first-order kinetic rate constant. It has been found that the pH of the solution is a key factor in accelerating degradation. When the pH of the solution was adjusted from its natural state to 2, the amount of decomposition of methyl orange increased from 6% to 81%. Additional research was conducted with the addition of hydrogen peroxide and Fenton/hydrogen peroxide hydroxyl radical promoters. In order to examine the influence of

H_2O_2 , the concentration of H_2O_2 was varied while the solution's natural and optimal pH levels were maintained throughout the experiment. For H_2O_2 loading at natural and optimum pH solutions, the rate of degradation and the total period of time required to complete it were almost identical. The rate of the reaction was accelerated to 20%, while the amount of H_2O_2 loading was decreased from 8 to 0.1 mL/L. The combined process has a synergistic effect when compared to the individual operations. As shown below, the synergistic index was calculated as the ratio of the rate constant obtained at optimal loading in the combined process to the sum of the rate constants obtained in the individual process:

$$f = \frac{k_{\text{HC}/\text{H}_2\text{O}_2}}{k_{\text{HC}} + k_{\text{H}_2\text{O}_2}}$$

The combined HC/ H_2O_2 process was carried out at a natural pH of 6.8, at a pressure of 4 bar, and with an optimal loading of 6ml/L of H_2O_2 . The studies are also carried out in the absence of HC to compute the synergistic index at optimal loading of 6ml/L of H_2O_2 . The results demonstrate a 38.8% degradation extent with first order kinetics fitting well in the data and a rate constant k of 0.0076 min^{-1} . The rate constant values for alone HC, only H_2O_2 (6ml/L), and combined HC/ H_2O_2 are 0.0013 min^{-1} , 0.0076 min^{-1} , and 0.0538 min^{-1} , respectively. The calculated synergistic index using above equation is 6.04 which clearly demonstrate the efficacy of the combined process at natural pH.

$$f = \frac{0.0538}{0.0013 + 0.0076} = 6.04$$

A synergistic index has also been calculated at an ideal pH of 2 and a loading of 0.1 ml H_2O_2 . The obtained data demonstrate that the extent of degradation is 54.21% with a first order rate constant of 0.0127 min^{-1} utilizing an ideal loading of 0.1 ml H_2O_2 in the absence of HC. The rate constant values for alone HC, only H_2O_2 (0.1 ml/L), and combined HC/ H_2O_2 are 0.025 min^{-1} , 0.0127 min^{-1} , and 0.0825 min^{-1} , respectively, yielding a synergistic index of 2.18.

$$f = \frac{0.0825}{0.0013 + 0.025} = 2.18$$

Further research was conducted using an optimized pH of the solution to evaluate the effect of Fenton/ H_2O_2 loading as well as at lower pH levels (2-6). To optimize the pH of the solution, the Fenton requirement was reduced by 50%, with a

marginal increase in the extent of degradation and a significant increase in reaction rate. The synergy of Fenton, H₂O₂, and optimize pH of solution results in a greater extent of degradation and many reductions in process time required for the extent of degradation. Thus, synergy helps reduce the processing time, amount of energy requirement, and catalyst loadings. The results were further analyzed based on the amount of moles of methyl orange degradation per kWh energy and the cost required to degrade per liter of methyl orange. Results of the same analysis with cavitation yield have shown in Table 2. It indicates that the amount of energy requirement for synergy of Fenton/H₂O₂ at optimizing pH of a solution is much lesser than other methods. The treatment cost of USD \$ 0.0062/L is required to complete removal of methyl orange using hydrodynamic cavitation. It was also observed that the similar treatment cost is required to above 90% extent of degradation for various treatment scheme investigated in this study. Similar results in removing pollutants from wastewater using a combination of hydrodynamic

cavitation and Fenton's chemistry/ advanced Fenton's process have been reported in the literature. A study on the degradation of methomyl using hydrodynamic cavitation (venturi) in conjunction with Fenton's reagent at a loading of FeSO₄:H₂O₂ in the ratio of 1:20 at optimum pH (2.5) and inlet pressure (5 bar) observed complete degradation with a specific degradation rate of $161.41 \times 10^{-3} \text{ min}^{-1}$ in 30 min [52]. Patil and Gogate, [22] also investigated the effect of the Fenton reagent with range of ratios from 1:0.5 to 1:4 (H₂O₂:FeSO₄) at a fixed concentration of H₂O₂ as 100 mg/L (20 mg/L initial concentration of methyl parathion, 4 bar of inlet pressure, 39 °C) using an orifice plate. The degradation rate increased with an increase in the H₂O₂:FeSO₄ ratio, while the maximum TOC removal (76.6%) was obtained for H₂O₂:FeSO₄=1:4, with a rate constant of $6.36 \times 10^{-3} \text{ min}^{-1}$. It is essential to understand that the significant degradation of methyl orange depends on the amount of dosage or concentration of iron catalyst and H₂O₂.

Table 1. Optimized parameters for the degradation of methyl orange.

Parameter	Optimized parameters	Process time (min)	Extent of degradation (%)	Rate constant	
				$k \times 10^{-2} \text{ min}^{-1}$	R ²
Initial pressure	4 bar, pH natural (6.8)	60	6.4	0.13	0.99
pH	pH: 2	60	81.6	2.5	0.98
8 ml/L H ₂ O ₂ at pH 6.8	8 mL/L	60	99.2	6.9	0.94
0.2 ml/L H ₂ O ₂ at pH 2	0.1 mL/L	60	99.8	8.3	0.94
FeSO ₄ .7H ₂ O: 0.00625 g/L and H ₂ O ₂ : 0.2 mL/L	pH: 2	60	95.1	7.2	0.97
FeSO ₄ .7H ₂ O: 0.00625 g/L and H ₂ O ₂ : 0.2 mL/L	pH: 3	60	98.9	9.9	0.96

Initial concentration of methyl orange: 20 mg/L.

Table 2. Energy and Cost analysis,

Parameter	Cavitation Yield	Energy Required (kWh)	Operational cost (USD \$/L)
Initial pressure at 4 bar	3.23×10^{-6}	1.72	0.098
pH at 2	4.12×10^{-5}	0.13	0.0077
8 ml/L H ₂ O ₂ at pH 6.8	5.01×10^{-5}	0.11	0.0089
0.2 ml/L H ₂ O ₂ at pH 2	5.04×10^{-5}	0.11	0.0062
FeSO ₄ .7H ₂ O: 0.00625 g/L and H ₂ O ₂ : 0.2 mL/L solution pH 2	4.99×10^{-5}	0.11	0.0064
FeSO ₄ .7H ₂ O: 0.00625 g/L and H ₂ O ₂ : 0.2 mL/L solution pH 3	5.05×10^{-5}	0.11	0.0062

Initial concentration of methyl orange: 20 mg/L.

3.9. Degradation mechanism

Advanced oxidation processes (AOPs) have been accepted as an efficient technique for degrading dyes. These methods require the generation of highly reactive OH^- by chemical, solar, or other energy sources. The most important feature of AOPs is their extraordinarily strong generation of oxidizing radicals, which allows them to detoxify a wide range of persistent organic pollutants without selectivity. Several investigations have demonstrated the production of OH^- utilizing AOPs for dye degradation [14,53–55]. AOPs not only produce OH^- but also hydroperoxyl (HO_2^-) radicals, which aid in the degradation of organic and inorganic substances present in industrial effluent. In general, when cavitating conditions (like high energy densities) are met, methyl orange dye is broken down by an oxidation process that starts with free radicals attacking the dye molecule and then reacts with intermediates. According to the literature, methyl orange dye degraded into seven primary byproducts during the cavitation phenomena [56,57]. Additionally, it was said that the solution still included a large number of intermediate species. It is observed that seven byproducts primarily show both hydrophilic and hydrophobic species. The hydrophobic compounds are more easily broken down by the cavitation process because they are often surface active at the solution-bubble interface [56]. Methyl orange dye is demethylated and hydroxylated into a variety of intermediates, including m/z of 290 and 320, during the cavitation phenomenon. This process entails the homolytic rupture of the amine group's nitrogen-carbon bond, which results in the replacement of a hydrogen atom for a methyl group. Although sequential demethylation is the most likely mechanism for the creation of intermediates such as m/z of 306, m/z of 276, m/z of 322, m/z of 292 and m/z of 308 [16,56–58], hydroxyl radical attack is the primary cause of methyl orange dye degradation. Evidently, it is reasonable to anticipate that demethylation and hydroxylation will either couple with one another or occur independently of one another due to simultaneous and independent events. As a result, the specific demethylation and hydroxylation processes, as well as their combinations, govern the by-products of MO breakdown, which are

subsequently mineralized into carbon dioxide and water [59].

4. Conclusions

The current study established that hydrodynamic cavitation (HC) can be used to degrade methyl orange dye. It has been observed that process intensification is achieved effectively using HC/Fenton process and HC/ H_2O_2 . The optimized parameters were observed to be inlet pressure of 4bar, 2 pH, and a 20-mg/L dye concentration. Degradation of methyl orange was found to be maximum in acidic conditions. The combination of hydrodynamic cavitation and the oxidizing agent H_2O_2 accelerates the degradation of methyl orange in comparison to HC alone. Complete degradation of methyl orange was obtained at Fenton reagent (0.00625 g/L and 0.2 ml/L) with treatment cost of USD \$ 0.0062/L. Based on the literature, it clearly indicating that hydroxyl radical attack is the primary cause of methyl orange dye degradation. Overall, the hybrid HC /Fenton process provides the intensified degradation approach and promises to be better and more economical than HC compared with process intensified additives (hydrogen peroxide) and HC when operated alone.

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Appendix-I Sample calculations

I) Calculations of cavitation yield (0.2 ml/L of H_2O_2 , 4 bar, 2 pH, 20 mg/L)

Degradation of methyl orange obtained = 99.89%

Time of treatment = 60 min

Volume = 10 L

Power rating of pump = 1.1 kW (1100W)

Concentration removed after treatment (mg/L) = $\frac{99.89}{100} \times 20(\text{mg/L}) = 19.978 \text{ mg/L}$

Power density in J/L = $\frac{1100 \times 60 \times 60}{10} = 3.96 \times 10^5 \text{ J/L}$

Cavitation yield = $\frac{19.978}{3.96 \times 10^5} = 5.04 \times 10^{-5} \text{ mg/J}$

II) Cost of treatment

Energy efficiency = 5.04×10^{-5} mg/J

Energy required for removal of methyl orange = $\frac{20}{5.04 \times 10^{-5}} = 3.96 \times 10^5$ J/L

Energy required for removal of phenol = $\frac{3.96 \times 10^5}{3,600,000} = 0.11$ kWh

In Pune, MH, considering 1 kWh = Rs. 4.67

Cost of treatment = $0.11 \times 4.67 = 0.5137$ Rs/L

Cost of additive (1 L of H₂O₂) = Rs. 26.24

Cost of FeSO₄.7H₂O (1 kg) = Rs. 9

For 0.2 ml of H₂O₂ = Rs. 0.0052

Hence, total cost of treatment of 99.89% removal of methyl orange/L = Rs. 0.515/L

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