Chemical Engineering Journal 156 (2010) 77–82

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Removal of *p*-nitrophenol using hydrodynamic cavitation and Fenton chemistry at pilot scale operation

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ARTICLE INFO

Article history:
Received 22 June 2009
Received in revised form 26 August 2009
Accepted 28 September 2009

Keywords:
Wastewater treatment
p-Nitrophenol
Hydrodynamic cavitation
Venturi
Orifice plate
Fenton's reagent

ABSTRACT

In the current work removal of *p*-nitrophenol has been investigated using hydrodynamic cavitation, either operated individually or in combination with H_2O_2 and conventional Fenton process. In hydrodynamic cavitation, two different cavitating devices viz. orifice plate and venturi have been used. Effect of different operating parameters such as initial concentration (5 g/l and 10 g/l), inlet pressure (over a range 5.7–42.6 psi) and pH (over a range 2–8) on the extent of removal has been investigated. In conventional Fenton process two loadings of $FeSO_4$, 0.5 g/l and 1 g/l were investigated and three ratios of $FeSO_4$:H_2O_2 viz. 1:5, 1:7.5 and 1:10 were used. Removal observed with venturi was higher than with orifice plate in combination with Fenton chemistry. For 5 g/l initial concentration of *p*-nitrophenol, maximum removal of 63.2% was observed whereas for 10 g/l solution it was 56.2%.

1. Introduction

The impact of industrial wastewaters discharges on the environment and human population can be tragic at times and instances of pollution with potentially adverse impacts in the longer term have continued to occur. New developments in the variety of fields to meet the ever-increasing requirements of human beings have also led to the presence of new compounds in the effluent streams of processing plants, which are not readily degraded by the conventional effluent treatment methods. It is of utmost importance to keep the concentration of chemicals in the effluent stream to a minimum level in order to comply with the environmental laws, which are becoming more stringent these days, hence there is a need to develop treatment schemes based on the use of advanced techniques such as advanced oxidation processes (AOPs), wet air oxidation, supercritical oxidation, etc. either individually or in combination with conventional methods, such that the stringent environmental regulations are met.

AOPs are defined as the processes that generate hydroxyl radicals in sufficient quantities to oxidize majority of the complex chemicals present in the effluent water [1]. Some of the AOPs, which have shown considerable promise for wastewater treatment applications, include cavitation, Fenton chemistry, and photocatalytic oxidation [2–4]. Usually a combination of different AOPs has been found to be more efficient for wastewater treatment [5–10] as compared to individual oxidation process. Cavitation based AOPs have been widely investigated for wastewater treatment applications but more focus has been on the use of ultrasonically induced cavitation [11–14]. However, it has been observed that use of sonochemical reactors, pose significant problems due to substantially lower energy efficiencies and higher costs of operation. Use of hydrodynamic cavitation reactors, which has been recently looked as an alternative to acoustic cavitation, is an emerging technology and there are not many instances where these reactors have been investigated for wastewater treatment applications. One of the possible reasons for this is that the intensity of cavitation generated in the reactors is lower as compared to the sonochemical reactors. There have been only a few reports where use of hydrodynamic cavitation has been applied for wastewater treatment and with much larger energy efficiency as compared to acoustic cavitation. Sivakumar et al. [15] have studied the applicability of hydrodynamic cavitation reactors for degradation of Rhodamine B samples (a pink coloured dye solution) and reported that hydrodynamic cavitation was indeed more energy efficient compared to acoustic cavitation. Chakinala et al. [16] have reported the use of a combination of hydrodynamic cavitation and advanced Fenton process for treatment of real industrial effluents and it has been observed that the novel combination results in about 60–80% removal of TOC under optimized conditions depending on the type of industrial effluent samples. In the current work, the use of the combination of hydrodynamic cavitation and Fenton chemistry for wastewater treatment applications has been investigated utilizing *p*-nitrophenol as a model pollutant. Nitrophenols pose significant health risks since they are carcinogenic and is also listed on the US

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doi:10.1016/j.cej.2009.09.042

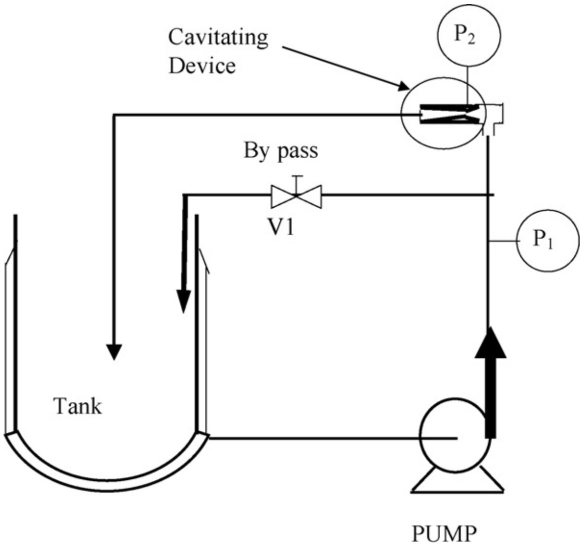

Fig. 1. Schematic representation of the hydrodynamic cavitation set up.

environmental protection agency's priority pollutants list. Due to this fact, *p*-nitrophenol has been selected as a model pollutant.

2. Materials and methods

2.1. Materials

p-Nitrophenol was obtained from Loba chemie, India, and was 98% pure. It was diluted to required concentrations using distilled water. Hydrogen peroxide (30%, w/v) and ferrous sulphate heptahydrate ($FeSO_4 \cdot 7H_2O$), all of AR grade were obtained from SD Fine chem, India. Solutions of sulphuric acid (2 M) and sodium hydroxide (2 M) were used for adjustment of pH. All procured chemicals were used as received.

2.2. Experimental setup

Schematic of the experimental setup used for hydrodynamic cavitation has been shown in Fig. 1. The setup essentially consists of a closed loop circuit including a holding tank of capacity 15 l, a centrifugal pump (2900 rpm) of power rating 370 W and a valve. Calorimetric studies were performed and it was observed that the conversion efficiency is 63.7% indicating that actual power dissipation into the liquid is 235.7 W. All experiments were performed at 7 l of operating capacity based on the pump capacity so as to maintain desired flow rate in the circulation loop and ensure generation of cavitating conditions. The suction side of the pump is connected to the bottom of the tank. The discharge from the pump branches into two lines. The main line consists of a venturi or orifice plate which acts as a cavitating device, located just after the pressure gauge. The cavitation device is made of acrylic. A by-pass line and control valve (V1) is provided to control the liquid flow through the main line. Pressure gauges are provided to measure the inlet pressure and the fully recovered downstream pressure which in most of the cases was equal to 1 atm. During the experiment, the by-pass valve was kept open till the pump reached its maximum speed and is then totally or partially closed to achieve desired pressure in the cavitation chamber.

2.3. Experimental procedure

The holding tank was filled with 7 l of aqueous solution of *p*-nitrophenol of desired concentration. Pump was started and flow in the by-pass line was controlled to set the required inlet pressure. The reaction was carried out for 90 min and sample was collected after every 15 min. Aim was to maximize the extent of degradation using different combinations of hydrodynamic cavitation and Fenton chemistry and hence a base was considered as 90 min of treatment for comparison purpose. Analysis of the samples was done using chemito-2500 UV–vis spectrophotometer at $\lambda = 401$ nm [17,18]. Concentration of *p*-nitrophenol was calculated by measuring the absorbance of *p*-nitrophenol with the help of the calibration chart prepared earlier with known concentrations.

The experiments with hydrodynamic cavitation operating individually were performed with venturi and orifice as the cavitating device. Fig. 2 gives the geometrical details of the venturi used in the work. Orifice plate of diameter 25 mm with a single hole of 2 mm diameter was the second cavitating device considered in the work. To examine the effect of inlet pressure on the extent of removal, the inlet pressure was varied from 5.7 psi to 42.6 psi. The resultant flow rate through the system and cavitation number has been given in Table 1 for each operating pressure. The operating temperature was maintained at $30 \pm 4\,^{\circ}C$ (due to unavailability of cooling system, strategy was applied to operate hydrodynamic cavitation setup for a period of 10 min followed a cooling period of 20 min).

Hydrogen peroxide was also used as an additive (over a concentration range of 0.5–5 g/l) as an additional source of hydroxyl radicals to intensify the removal using hydrodynamic cavitation.

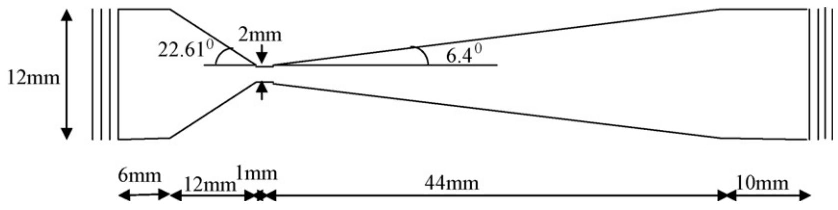

Fig. 2. Geometric specifications of a venturi used as a cavitating device.

Table 1
Cavitation number calculations.

Venturi				Orifice			
Pressure (psi)	Flow rate (l/min)	Velocity (m/s)	Cavitation no.	Pressure (psi)	Flow rate (l/min)	Velocity (m/s)	Cavitation no.
5.7	2.1	10.15	1.57	5.7	1.42	7.54	3.43
19.9	4.77	25.31	0.3	19.9	2.61	13.85	1.01
34.1	6.16	32.69	0.18	34.1	3.67	17.36	0.51
42.6	6.69	35.5	0.15	42.6	4.01	21.28	0.43

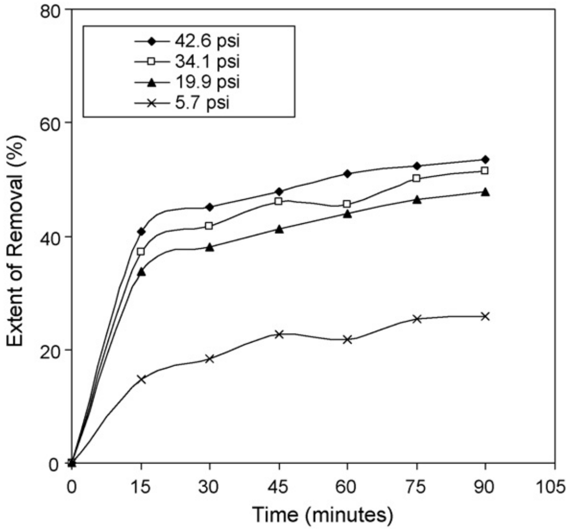

Fig. 3. Removal of *p*-nitrophenol (0.5% solution) using venturi at various inlet pressures.

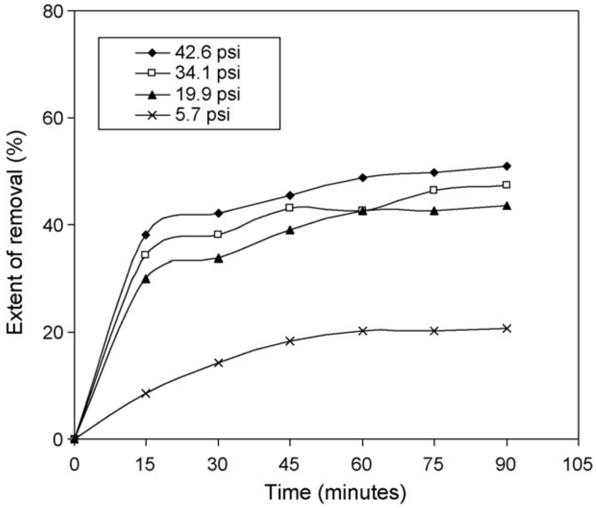

Fig. 4. Removal of *p*-nitrophenol (0.5% solution) using orifice plate at various inlet pressures.

For experiments involving Fenton chemistry, three different ratios of $FeSO_4$:H_2O_2 viz. 1:5, 1:7.5 and 1:10 were investigated at two different loadings of $FeSO_4$ [19].

3. Results and discussions

3.1. Removal using hydrodynamic cavitation alone

Removal of *p*-nitrophenol was investigated using two different cavitating devices viz. venturi and orifice plate. Four different inlet pressures over the range 5.7–42.6 psi were employed to investigate the effect of pressure on removal of *p*-nitrophenol. The obtained results have been depicted in Fig. 3 for venturi as the cavitating device and in Fig. 4 for orifice plate as a cavitating device. As seen from the figure, the extent of removal increases with an increase in the pressure for both the cavitating devices. Also the extent of increase in the extent of removal is significant for an increase in the pressure from 5.7 psi to 19.9 psi. The trends shown in Figs. 3 and 4 are only for 5 g/l (0.5% by weight) concentration of *p*-nitrophenol and exactly same trends in variation with operating pressure were observed for 10 g/l (1% by weight) solution (actual values were different). The maximum removal for 5 g/l initial concentration of *p*-nitrophenol was 53.4% whereas for 10 g/l initial concentration it was 44.8% at an operating inlet pressure of 42.6 psi. Thus, it is clear that the extent of removal is higher for lower initial concentration of pollutant as is commonly observed with the cavitationally induced degradation of pollutants [10,16]. It should be also noted here that the net moles of *p*-nitrophenol removed will be higher in the case of initial loading of 10 g/l as compared to 5 g/l initial loading.

The observed increase in the extent of removal with an increase in the inlet pressure can be attributed to enhanced cavitational activity at higher pressures. Bubble dynamics studies [20] have indicated that the cavitational intensity generated at the collapse of the cavity increases as a result of an increase in the inlet pressure of the system. Due to an increase in the cavitational collapse intensity, higher temperature and pressure pulse is generated resulting in an enhanced dissociation of the water molecules trapped in the cavity thereby leading to higher concentration of hydroxyl radicals existing in the reactor. Also there exists a critical operating pressure for onset of cavitating conditions [21,22] which explains the significant increase observed in removal of *p*-nitrophenol for an initial increase in the operating pressure from 5.7 psi to 19.9 psi. Similar results have been reported in the literature. Chakinala et al. [23] have reported that the iodine yield in a Weissler reaction increases with an increase in the operating pressure from 500 psi to 1500 psi. Vichare et al. [24] have also reported similar results using a model reaction of oxidation of potassium iodide.

In the present work, apart from investigating the effect of inlet pressure, effect of type of cavitating device has also been investigated. The obtained results indicated that the extent of removal is marginally higher for the case of venturi (53.4% removal) as compared to that obtained with orifice (51%). This can be attributed to the fact that at same operating pressure, the liquid velocity in the case of venturi is higher as compared to the orifice plate (Table 1) and hence the number of passes through the cavitating zone for the same time of operation will be higher in the case of venturi. Due to a higher number of passes through the cavitating device, the liquid experiences cavitating conditions for more duration resulting into higher removal rates [15]. Also the operating cavitation number is lower in the case of venturi as compared to orifice (Table 1) which also indicates that the cavitational intensity is higher in the case of venturi leading to higher extent of removal.

3.2. Removal using combination of hydrodynamic cavitation and hydrogen peroxide

To introduce more $OH^\bullet$ radicals in the system, hydrogen peroxide was added to the holding tank. Six different concentrations of hydrogen peroxide were investigated ranging from 0.5 g/l to 10 g/l with venturi. The obtained results have been depicted in Fig. 5. It can be seen from the figure that removal of *p*-nitrophenol was enhanced from 53.4% for operation using only venturi to 59.9% for 5 g/l concentration of *p*-nitrophenol but beyond that an increase to 10 g/l concentration of *p*-nitrophenol resulted in lower extent of removal due to the scavenging effects of unutilized hydrogen peroxide. The observed trends were exactly similar to higher loading of *p*-nitrophenol as well (10 g/l; results shown in Table 2) though the final extents of removal were lower as compared to that obtained for 5 g/l concentration similar to operation with hydrodynamic cavitation alone. It can be also seen from Table 2 that the extent of intensification is more for higher loading of pollutant indicating that generation of higher amount of free radicals by way of using hydrogen peroxide is indeed more beneficial for the case of highly loaded effluents. Credence to the obtained results can also be obtained from similar literature illustrations. Wang et al. [25] have reported that the extent of degradation of rhodamine B in a hydrodynamic cavitation reactor based on swirling jet increases with an

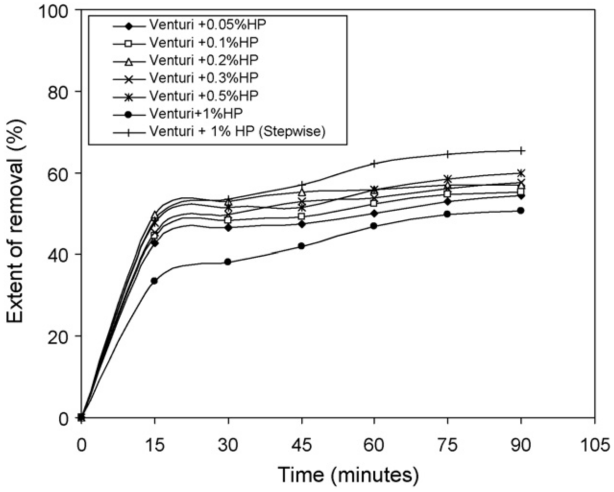

Fig. 5. Removal of *p*-nitrophenol (0.5% solution) using combination of hydrodynamic cavitation (venturi) and hydrogen peroxide.

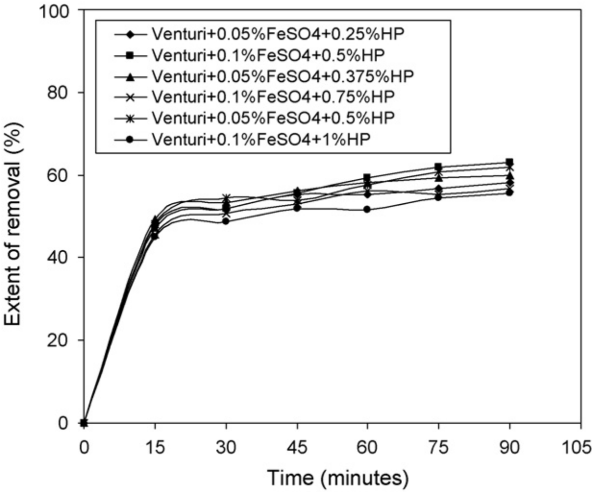

Fig. 6. Removal of *p*-nitrophenol (0.5% solution) using hydrodynamic cavitation (venturi) + $FeSO_4$ + H_2O_2.

increase in the hydrogen peroxide concentration over the range 0–150 mg/l though the observed increase at 150 mg/l was quite marginal as compared to 100 mg/l and hence loading of 100 mg/l was considered to optimum loading. Guo et al. [3] have reported similar existence of optimum concentration for the degradation of 2,4-dinitrophenol where H_2O_2 concentration was varied from 100 mg/l to 800 mg/l.

At the observed optimum concentration of H_2O_2, the operation was investigated with orifice plate under otherwise similar operating conditions. It has been observed that the extent of removal was only 52.4% with orifice as compared to 59.9% with venturi. The observed intensification using venturi is higher as compared to orifice attributed to higher intensity cavitation resulting in higher extent of decomposition of hydrogen peroxide generating additional hydroxyl radicals.

Table 2
Removal of *p*-nitrophenol under various operating parameters.

Operating Parameters	Removal of 0.5% solution (in %)	Removal of 1% solution (in %)
HC (Venturi) along at 42.6 psi	53.4	44.8
HC (Venturi) + 0.05% H_2O_2	54.3	45.4
HC (Venturi) + 0.1% H_2O_2	55.2	46.8
HC (Venturi) + 0.2% H_2O_2	57.1	48.9
HC (Venturi) + 0.3% H_2O_2	57.6	52.4
HC (Venturi) + 0.5% H_2O_2	59.9	54.1
HC (Venturi) + 1% H_2O_2	50.6	43.6
HC (Venturi) + 1% H_2O_2 (Stepwise)	65.5	58.3
HC (Venturi) + 0.05% $FeSO_4$ + 0.25% H_2O_2	58.1	51.1
HC (Venturi) + 0.1% $FeSO_4$ + 0.5% H_2O_2	63.2	55
HC (Venturi) + 0.05% $FeSO_4$ + 0.375% H_2O_2	59.9	51.7
HC (Venturi) + 0.1% $FeSO_4$ + 0.75% H_2O_2	56.6	56.2
HC (Venturi) + 0.05% $FeSO_4$ + 0.5% H_2O_2	61.8	53.1
HC (Venturi) + 0.1% $FeSO_4$ + 1% H_2O_2	55.7	49.9

To avoid scavenging action of the excess hydrogen peroxide, it was thought desirable to add hydrogen peroxide in stepwise manner keeping the total concentration of hydrogen peroxide similar. With this background, additional set of experiment was performed where H_2O_2 was added in stepwise manner, i.e. 2 g/l after every 15 min (total loading remains as 10 g/l). It has been observed that the extent of removal after 90 min treatment was 65.5% as against 50.6% observed when all the hydrogen peroxide was added initially. The stepwise addition ensures that at any moment in the reactor the concentration of hydrogen peroxide is not in excess and hence results in favorable results for removal process. Though the reports of stepwise addition of hydrogen peroxide are not available for the case of hydrodynamic cavitation, credence to the obtained results can be obtained based on the work of Chakinala et al. [16] for a combination of hydrodynamic cavitation and advanced Fenton process and Kosaka et al. [26] for a combination of ozone and hydrogen peroxide where mechanism for intensification is also based on generation of additional free radicals in the system. Chakinala et al. [16] observed that addition of the oxidant in three stages (at time t = 0 min, 60 min and 120 min) seems to be the most optimized option for treatment of real industrial effluent as this approach reduces the TOC to 40% of the initial value in only 150 min as against residual TOC being 60% for the case of entire oxidant being added initially. Kosaka et al. [26] reported that the residual TOC at the end of 40 min treatment time was lowest for the mode in which the hydrogen peroxide concentration was increased in steps as compared to other two modes where the concentration was either kept constant or decreased in steps with the total loading of hydrogen peroxide remaining the same in all the cases.

3.3. Removal using combination of hydrodynamic cavitation and Fenton chemistry

Efficacy of combination of hydrodynamic cavitation and Fenton chemistry for removal of *p*-nitrophenol has been investigated for three different ratios of $FeSO_4$:H_2O_2 viz. 1:5, 1:7.5 and 1:10 with venturi as the cavitating device. The obtained results have been depicted in Fig. 6 for 5 g/l of *p*-nitrophenol solution whereas the final extents of removal for 5 g/l and 10 g/l concentration of the pollutant at the end of 90 min have been given in Table 2. It has been observed that at same concentration of H_2O_2, the extent of removal increased with an increase in the $FeSO_4$ loading. This can be attributed to the generation of higher quantum of free radicals due to presence of higher quantum of Fe^{2+} ions. The maximum

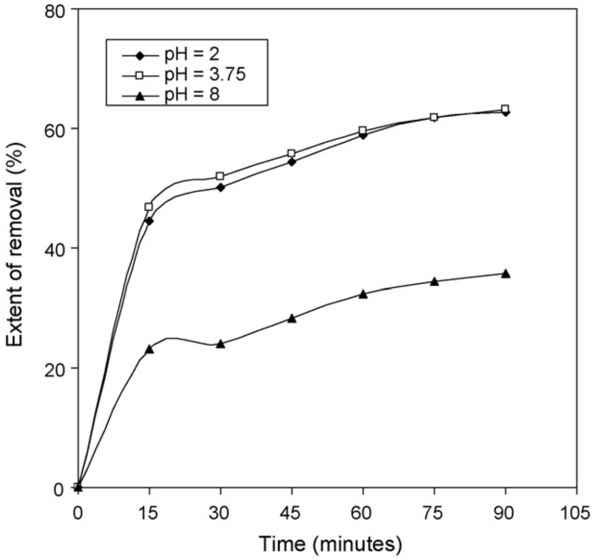

Fig. 7. Removal of *p*-nitrophenol (0.5% solution) using combination of hydrodynamic cavitation (venturi) and Fenton chemistry ($FeSO_4$ and H_2O_2) at various pH.

extent of removal using this approach for 5 g/l initial concentration was found to be 63.2% at 1 g/l loading of $FeSO_4$ and 5 g/l loading of H_2O_2. For 10 g/l initial concentration of *p*-nitrophenol the maximum extent of removal (56.2%) was observed at 1 g/l of $FeSO_4$ and 7.5 g/l of H_2O_2 loading. Due to presence of higher loadings of the pollutant, higher loading of hydrogen peroxide is required to yield maximum extents of removal. Not much information is available on removal of pollutants using combination of hydrodynamic cavitation and Fenton chemistry but some studies have been reported for the combination of ultrasound induced acoustic cavitation and Fenton chemistry. Song et al. [4] have reported that the degradation of Acid Red 88 increased with an increase in the loading of Fe^{+2} from 0.036 mM to 0.108 mM at constant H_2O_2 loading of 1.96 mmol/l. Sun et al. [9] also reported similar results for the case of acid black 1 at constant H_2O_2 loading of 8.0 mM and Fe^{+2} loading varied from 0.01 mM to 0.05 mM.

3.4. *Effect of operating pH*

Removal of *p*-nitrophenol was investigated at three different pH values 2.0, 3.75 and 8.0 using venturi and optimum $FeSO_4$ and H_2O_2 concentration obtained earlier. The observed results have been depicted in Fig. 7. It can be seen from the figure that maximum removal of 63.2% was observed at pH value of 3.75, although there was very little difference in removal value at pH 2.0 and at pH 3.75. In terms of the ease of maintaining operating pH, pH of 3.75 seems to be the optimum choice. At pH 8.0 (alkaline conditions), the extent of removal was 35.7% which is considerably lower than that under acidic conditions. The observed results can be attributed to the fact that under acidic conditions generation of hydroxyl radicals due to decomposition of hydrogen peroxide is favored and also the oxidation capacity of hydroxyl radicals is higher under acidic conditions. The trends in the removal of pollutant with various pH values are in close agreement with the reported literature [9,27]. Sun et al. [9] reported that the decolourisation efficiency of acid black 1 increased from 5.13% to 98.83% as a consequence of the pH value decreasing from 6 to 3. Similarly, Zhang et al. [27] have observed that the optimum pH value was in the range of 2–4 for the investigation related to the decolourisation of C. I. Acid Orange 7.

4. Conclusions

The present work has clearly established the utility of combination of hydrodynamic cavitation and Fenton process. Amongst the different cavitating devices investigated in the work, venturi results in more intense cavitation as compared to single hole orifice and higher inlet pressures are recommended for maximizing the extent of removal. Stepwise addition of oxidant was found to be more beneficial for intensification of the removal of *p*-nitrophenol for a combination of hydrodynamic cavitation and hydrogen peroxide at higher loadings. For the combined use of hydrodynamic cavitation and Fenton chemistry, an optimum loading of $FeSO_4$ as 1 g/l and H_2O_2 concentration as 5 g/l was observed for 5 g/l as initial *p*-nitrophenol concentration and the maximum extent of removal under these conditions was 63.2%. For 10 g/l initial *p*-nitrophenol solution, the maximum extent of removal was 56.2% under optimized conditions. Efficacy of removal using the combined approach was found to be strongly dependent on the operating pH and pH of 3.75 was found to be optimum.

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