Degradation of p-nitrophenol using acoustic cavitation and Fenton chemistry

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ABSTRACT

Due to increasing human requirements, newer chemical species are being observed in the effluent streams with higher loadings such that efficacy of conventional treatment techniques is decreased and a combination of advanced oxidation processes is implemented for enhanced treatment ability and better energy efficiency. In the present work, the efficacy of combination of sonochemistry and Fenton chemistry has been investigated for wastewater treatment considering p-nitrophenol as model pollutant at pilot scale operation. Degradation of p-nitrophenol has been investigated under various operating conditions based on the use of ultrasound, Fenton process, ultrasound and H_2O_2, ultrasound and Fe, ultrasound and $FeSO_4$, ultrasound and conventional Fenton process and ultrasound and advanced Fenton process. Two different initial concentrations of 0.5 and 1% of p-nitrophenol have been used for the experiments. In conventional Fenton and advanced Fenton process, two loadings of $FeSO_4$ and Fe powder 0.5 and 1 g/l and three ratios of $FeSO_4$:H_2O_2 and Fe:H_2O_2 (1:5, 1:7.5 and 1:10) were investigated respectively. In all the systems investigated, maximum extent of degradation (66.4%) was observed for 0.5% p-nitrophenol concentration (w/v) using a combination of ultrasound and advanced Fenton process. The novelty of the work is in terms of investigating the efficacies of combined advanced oxidation processes based on the use of cavitation and Fenton chemistry at pilot scale operation and tries to establish the missing design related information for large scale operation of wastewater treatment.

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1. Introduction

Any wastewater treatment scheme, until recent years, was comprised of the conventional physical (screening, mixing, flocculation, sedimentation, floatation, filtration, etc.), chemical (precipitation, disinfection, oxidation using chemicals, etc.) and biological (removal of contaminants brought about by biological activity) methods; however each of these methods have some limitations. The physical methods only transform the pollutants into other forms; thus new waste disposal problems are generated. The treatment times with chemical/biological methods may be quite high and total mineralization may not be possible especially for a complex effluent stream containing a mixture of pollutants in high concentration. Also due to the increasing presence of molecules, refractory to the micro-organisms in the wastewater treatment streams, the conventional biological methods, also cannot be used for complete treatment of the effluent and thus, 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.

Advanced oxidation processes 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]. Hydroxyl radicals are powerful oxidizing reagents with an oxidation potential of 2.33 V, which is substantially higher than other oxidants such as hydrogen peroxide, permangnate ions, etc. Some of the AOPs, which have shown considerable promise for wastewater treatment applications, include cavitation, Fenton chemistry, and photocatalytic oxidation [2–4]. However, it has been observed that none of the methods can be used individually in wastewater treatment applications due to substantially lower energy efficiencies and higher costs of operation and usually a combination of different AOPs has been found to be more efficient for wastewater treatment [5–10]. It should be also noted here that almost all the earlier works are at a capacity less than 1 l. The required scale up ratio for translating the earlier literature information to large scale operation would be very large inducing a degree on uncertainty. In the present work, the use of the combination of acoustic cavitation and Fenton chemistry (conventional as well as advanced) for wastewater treatment applications has been investigated considering a model pollutant as p-nitrophenol at operating capacity of 7 l.

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Phenolic hydrocarbons including p-nitrophenol are widely used in pharmaceutical, petrochemical and other chemical manufacturing processes. It is widely used as synthetic intermediate or as a raw material in the manufacturing of pesticides, insecticides, wood preservatives and hence occurrence of p-nitrophenol in the effluent streams of these sources is quite common. However, nitrophenols pose significant health risks since they are carcinogenic and is also listed on the US Environmental Protection Agency's Priority Pollutants List. Thus, it is necessary to develop effective methods for their removal from water, or to reduce them to less harmful intermediates or achieve complete mineralization. With this background, p-nitrophenol has been selected as a model pollutant.

Cavitation is described as the phenomena of formation, growth and subsequent collapse of microbubbles or cavities occurring in extremely small interval of time, releasing large magnitudes of energy [1]. The local effects of cavitation include generation of free radicals, hot spots and intense turbulence coupled with liquid circulation currents, the conditions being quite favorable for oxidation of pollutants. Indeed cavitation has been applied for treatment of p-nitrophenol. In one of the earliest works, Kotronarou et al. [11] have reported use of a simple ultrasonic horn operating at 20 kHz and investigated the mechanistic details of the degradation process. Sivakumar et al. [12] have investigated the degradation of p-nitrophenol using only ultrasonic irradiations at three different operating frequencies, i.e. 25, 40 kHz and combination of 25 + 40 kHz whereas use of high frequency ultrasound has also been investigated under argon-saturated aqueous solution [13].

Conventionally, Fenton chemistry is based on the use of Fe^{2+} ions in combination with hydrogen peroxide (optimized ratio of about 1:5 to 1:10) for generation of hydroxyl radicals and subsequent attack of the same on the pollutants [3]. There have been some reports related to the use of conventional Fenton chemistry for degradation of p-nitrophenol [14]. In recent years, there have been studies reporting the use of iron metal with H_2O_2 to give Fenton like mechanism [10,15,16] but at much lower costs of operation, mostly due to the fact that even waste iron can be used. It is hypothesized that initially iron metal is corroded in the presence of H_2O_2 under acidic conditions oxidizing Fe^0 to Fe^{2+}, which then further reacts with H_2O_2 in a Fenton-like process to generate hydroxyl radicals and Fe^{3+}. The Fe^0 then reduces the Fe^{3+} back to Fe^{2+} and hence comparatively higher quantum of hydroxyl radicals can be produced [10].

The present work reports the use of combination of sonochemistry and Fenton chemistry as well as advanced Fenton process based on the use of iron metal. There have been some reports depicting the use of Fenton like oxidation mechanisms for wastewater treatment [17,18] but not for p-nitrophenol. Effect of different operating combinations as well as loading of $FeSO_4$/Fe and hydrogen peroxide on the extent of degradation has been investigated.

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 for experimental studies. Hydrogen peroxide (30%, w/v), ferrous sulphate heptahydrate ($FeSO_4 \cdot 7H_2O$), iron powder (of 100 mesh size) all of AR grade were obtained from SD Fine Chem, India. All the chemicals were used as received.

2.2. Experimental setup

The experimental set up consists of an ultrasonic bath equipped with single large transducer having longitudinal vibrations and was
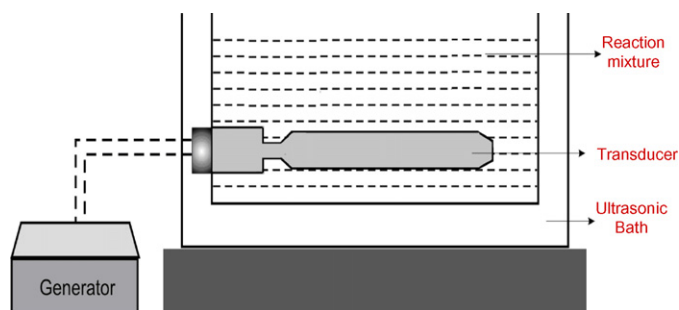

Fig. 1. Schematic representation of the experimental setup used for degradation of p-nitrophenol.

procured from Telsonic Ultrasonic, India. A schematic representation of the experimental assembly has been depicted in Fig. 1. The ultrasonic bath has an operating frequency of 25 kHz and rated power output of 1 kW. The bath is divided into two sections. The upper section is having dimensions of 35 cm length, 12 cm width and 17 cm height. The lower section is a V shaped channel of 3 cm height. The total body is made up of stainless steel. A drainage valve is also provided at the bottom of the bath. The internal of the bath consists of a transducer fitted at the bottom of the bath horizontally along the length of bath. The energy to this transducer is provided by a generator which is a separate unit. It has been generally observed that sonochemical reactors with higher irradiating surface results in better energy efficiency for degradation of wastewater [19]. Stirring was provided for experimental runs with solid particles so that the particles are well suspended in the reactor. Experiments with only stirring were performed in a stirred glass reactor with 500 ml capacity. Pitched blade turbine type of impeller was used at a speed of 1000 rpm, so that no mass transfer resistance prevailed in the reactor.

2.3. Experimental procedure

The sonochemical reactor was filled with 7 l of aqueous solution of p-nitrophenol. For experiments involving Fenton chemistry three different ratios of $FeSO_4$:H_2O_2 viz. 1:5, 1:7.5 and 1:10 (by weight) were investigated at two different loadings of $FeSO_4$ [20]. Similar ratios were used for the experiments with advanced Fenton process based on the use of Fe powder. Sonication of mixture was started simultaneously with stirring. The reaction was carried out for 90 min and sample was collected after every 15 min.

The operating initial pH for the system was selected as 3.7 on the basis of preliminary experiments performed over the range 2–4 which is usually the optimum range for Fenton chemistry. It was observed that there was a marginal change (less than 2%) in the extent of degradation for the combined operation and hence 3.7 was selected as the initial pH for all the treatment schemes. pH was adjusted using 2 M sulphuric acid.

The reaction mixture was cooled using external cooling agent such as ice and chilled water to counter the heat released into the system due to the action of ultrasound. The operating temperature of the solution was maintained at 28 ± 2 °C.

Analysis of the samples was done using Chemito-2500 UV-Vis spectrophotometer at λ = 401 nm [11,12]. Before analysis of samples small amount of NaOH was added so that sample becomes alkaline ensuring correct determination of p-nitrophenol using spectrophotometer [12]. Concentration of p-nitrophenol was calculated by measuring the absorbance of p-nitrophenol with the help of the calibration chart prepared earlier with known concentration. The main aim of the present work was to maximize the degradation of p-nitrophenol using combination of advanced oxidation processes at pilot scale operation and hence colori-
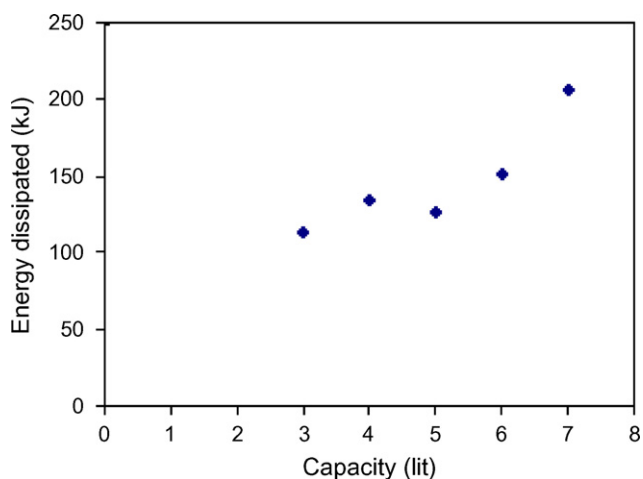
Fig. 2. Calorimetric plot of sonochemical reactor giving the extent of energy dissipation into the reactor.

Fig. 3. Degradation of p-nitrophenol using only ultrasound.

metric analysis has been used to monitor the removal of the parent compound. Sivasankar and Moholkar [21] have also recently reported similar approach for sonochemical degradation of p-nitrophenol where monitoring of p-nitrophenol removal was done spectrophotometrically. The detailed discussion on the degradation mechanism and the various primary and secondary products is readily available in the open literature [11,13,22].

All the experiments were repeated two times and the reported values are average of the two runs. The experimental errors were within 2% of the value of extent of degradation.

3. Results and discussions

3.1. Energy efficiency studies

Energy efficiency, i.e. fraction of the total supplied electrical energy actually dissipated into the system has been estimated as a function of the volume of the reaction mixture with an aim of quantifying the zone of influence of the ultrasonic irradiations [19]. It is important always to quantify the actual energy being dissipated in the system as it is always different from the supplied electric energy or the rated power of the equipment. The basics about the calorimetric estimation of acoustic power dissipation have been reported earlier [23–25]. It should be noted that in the present work, the heat absorbed by the immersed transducer section has not been considered in the estimation of energy efficiency (compared to the liquid, the transducer has much lower mass as well as specific heat and hence the possible error introduced is always less than 0.5%). Energy efficiency studies were undertaken using water and with varying capacity in the range of 3–7 l (7 l has been chosen as maximum operating capacity due to safety issues as the total capacity of the reactor is 7.5 l) with an aim of obtaining the optimum volume for maximum energy transfer into the system (for all the volumes considered in the work, the transducer surface was entirely submerged in the liquid). The obtained results have been depicted in Fig. 2 and it can be seen that the extent of energy dissipation increases with an increase in the volume of reaction mixture (the extent of energy dissipation per unit volume remains more or less constant) indicating that the zone of influence of ultrasonic waves propagation is throughout the maximum capacity of the reactor. The sound wave emission is from the transducer located at the bottom of the reactor in the longitudinal direction, i.e. in a direction perpendicular to the base of the reactor. With an operating capacity in the range of 2–4 l, the liquid height achieved in the reactor with the given geometry is not sufficient to absorb all the sup-
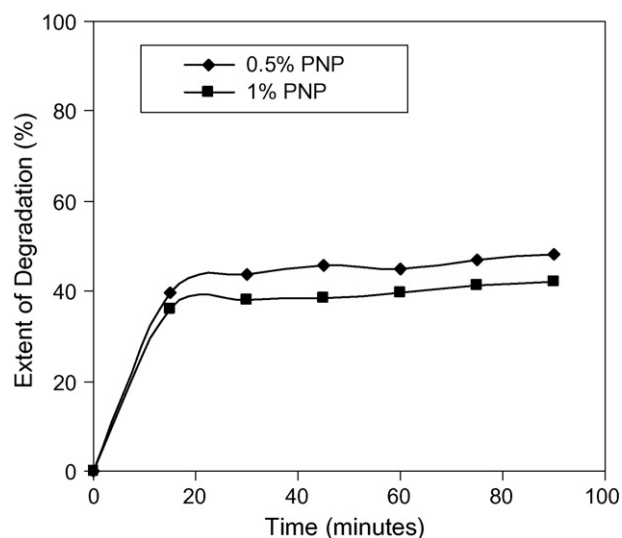
plied energy from the vibrating transducer. The quantum of energy absorbed into the system increases with an increase in the height of the solution in the reactor due to an increase in the capacity and hence there is a corresponding increase in the energy dissipation. Experiments with actual pollutant were then performed using this optimum volume as 7 l.

3.2. Degradation using only ultrasound

Initially, the extent of degradation was investigated using ultrasound induced acoustic cavitation phenomena at two initial concentrations of the pollutant and the obtained results have been shown in Fig. 3. It can be seen from the figure that initial rate of degradation is quite high after which the extent of degradation approaches a constant value. The initial high rates can be possibly attributed to the degassing effect of ultrasound. In the initial stages the presence of dissolved air in the solution, decreases the threshold pressure at the inception of cavitation, which results in substantial rise in the number of cavities generated, leading to higher reaction rates, initially. As observed, when time progresses, the dissolved air is liberated by degassing action and its role in generating cavitation becomes insignificant in subsequent time period. The extent of degradation of p-nitrophenol for 0.5% solution (w/v) was 48.3% whereas for 1% solution (w/v) the degradation was lower at 42%. The obtained results are consistent with the literature [12] which shows that sonochemical degradation is usually faster at lower concentrations of the pollutant species.

3.3. Degradation using combination of ultrasound and hydrogen peroxide

The controlling mechanism of sonochemical degradation of p-nitrophenol is the production of free radicals and their subsequent attack on the pollutant species. Considering this mechanism and knowing that hydrogen peroxide also dissociates in the presence of ultrasound giving hydroxyl radicals, a combination of ultrasound and hydrogen peroxide was investigated as a treatment strategy. Five different concentrations of hydrogen peroxide were used for this purpose in the range of 0.5–5 g/l. The obtained results have been shown in Fig. 4 for 0.5% initial concentration of p-nitrophenol whereas Table 1 gives the results for both the concentrations of p-nitrophenol and for different combinations of ultrasound, stirring and hydrogen peroxide (the trends of variation with time was
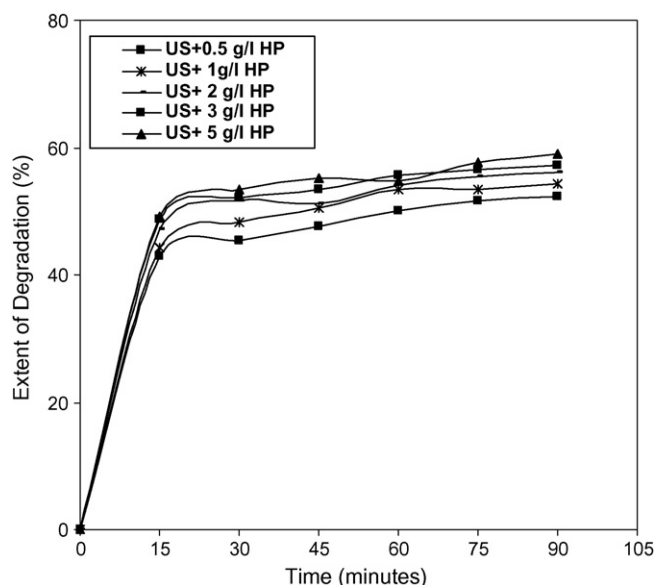
Fig. 4. Degradation of p-nitrophenol (0.5% initial concentration) using combination of ultrasound and hydrogen peroxide.

exactly identical as depicted in Fig. 4 and hence only final values after 90 min of treatment have been given). It can be seen from Fig. 4 and Table 1 that extent of degradation increases with an increase in the concentration of H_2O_2. For 0.5% solution of p-nitrophenol the degradation was in the range of 52% for 0.5 g/l to 59% for 5 g/l. For 1% solution of p-nitrophenol the degradation was between 45.1% for 0.5 g/l and 53% for 5 g/l. Thus the maximum degradation was with 5 g/l of hydrogen peroxide in both the cases. Similar trends have been observed in the literature for degradation of 2,4-dinitrophenol where H_2O_2 concentration was varied from 100 to 800 mg/l [3] and for phenol where H_2O_2 concentration was varied from 1 to 5 g/l [10]. It should be noted here that use of hydrogen peroxide in combination with ultrasonic irradiation resulted in an enhanced extent of degradation but additional stirring did not result any further improvement (as shown in Table 1), possibly indicating that liquid circulation generated due to the cavitational effects is capable of overcoming the mass transfer limitations.

The results obtained with combination of ultrasound and hydrogen peroxide were compared with combination of stirring and hydrogen peroxide. The degradation was lower as compared to the combination of ultrasound and hydrogen peroxide. Quantitatively, for 0.5% solution of p-nitrophenol, extent of degradation was 24.2% for hydrogen peroxide loading of 0.5 g/l, where as for 1% p-nitrophenol solution the extent of degradation was 17.6% at

Table 1
Degradation of p-nitrophenol using different combinations of ultrasound, hydrogen peroxide and stirring.

Operating parameters	Extent of degradation for 0.5% solution (%)	Extent of degradation for 1% solution (%)
Only US	48.3	42.0
US + 0.5 g/l H_2O_2	52.3	45.1
US + 1 g/l H_2O_2	54.4	46.3
US + 2 g/l H_2O_2	56.2	48.1
US + 3 g/l H_2O_2	57.1	51.3
US + 5 g/l H_2O_2	59.0	53.2
US + stirring + 0.5 g/l H_2O_2	52.5	45.5
US + stirring + 1 g/l H_2O_2	55.3	46.2
US + stirring + 2 g/l H_2O_2	55.7	49.2
Stirring + 0.5 g/l H_2O_2	24.3	17.6
Stirring + 1 g/l H_2O_2	32.5	24.2
Stirring + 2 g/l H_2O_2	36.8	29.6

Table 2
Degradation of p-nitrophenol using different combinations of ultrasound and Fe/FeSO$_4$.

Operating parameters	Extent of degradation for 0.5% solution (%)	Extent of degradation for 1% solution (%)
US + 0.5 g/l FeSO$_4$	50.1	43.8
US + 1 g/l FeSO$_4$	52.0	44.5
US + 0.5 g/l Fe	53.8	45.2
US + 1 g/l Fe	54.8	46.6

same loading of H_2O_2. Thus it is quite evident that stirring with hydrogen peroxide cannot generate OH• radicals and the obtained degradation is only due to the oxidizing action of H_2O_2.

3.4. Degradation using ultrasound and Fe powder/FeSO$_4$

The obtained results using combination of ultrasound and FeSO$_4$ or Fe powder have been shown in Table 2 (only final values are given and the trends for variation with time were similar to that represented in Fig. 4). It can be seen from the table that use of combination of ultrasound with FeSO$_4$ results in only marginal improvement in the extent of degradation of p-nitrophenol as compared with the use of Fe powder at similar loadings. The obtained results can be attributed to the fact that, in the presence of ultrasonic irradiations, the Fe^0 surface area is increased and the reactive surface is cleaned continuously [26–29] contributing to enhanced degradation. Also the presence of Fe particles provide additional nuclei for enhanced cavitational effects. In both the cases, the degradation was enhanced with an increase in Fe powder and FeSO$_4$ dosages. Zhang et al. [26] have reported similar results for degradation of C. I. Acid Orange 7 for a range of Fe loading as 0.5–2 g/l and for C. I. Reactive Black 8 [27].

3.5. Degradation using ultrasound and Fenton chemistry

Cavitational reactors can be used in combination with Fenton chemistry and due to similarity in the mechanism of degradation, in terms of generation of free radicals and its subsequent attack on the pollutants, it is expected to get synergistic results. Three ratios of FeSO$_4$:H_2O_2 viz. 1:5, 1:7.5 and 1:10 (by weight) were incorporated to study the effects of FeSO$_4$ loading as well as H_2O_2 concentration and the obtained results have been shown in Table 3. Comparing the data with that obtained using combination of ultrasound and H_2O_2 as well as combination of ultrasound and FeSO$_4$, it can be said that the extent of degradation of p-nitrophenol is enhanced using combination of ultrasound and conventional Fenton process. In the case of ultrasound coupled with Fe^{2+}/H_2O_2, hydrogen peroxide reacts with ferrous ions to generate active hydroxyl radicals, which may accelerate the degradation of model pollutant via the usual Fenton chemistry [30]. The resulting Fe^{3+} can react with H_2O_2 to generate an intermediate complex (Fe–O$_2$H^{2+}) which can be effectively dissociated into Fe^{2+} and HO$_2$• under ultrasonic irradiation. The isolated Fe^{2+} further reacts with H_2O_2 and generates a higher concentration of hydroxyl radicals compared to that in the absence of
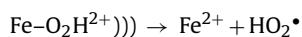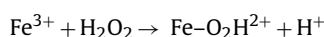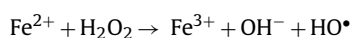
Table 3
Degradation of p-nitrophenol using combination of ultrasound and conventional Fenton chemistry.

Operating parameters	Extent of degradation for 0.5% solution (%)	Extent of degradation for 1% solution (%)
US + 0.5 g/l FeSO$_4$ + 2.5 g/l H_2O_2	52.9	47.1
US + 1 g/l FeSO$_4$ + 5 g/l H_2O_2	57.6	50.1
US + 0.5 g/l FeSO$_4$ + 3.75 g/l H_2O_2	54.3	48.5
US + 1 g/l FeSO$_4$ + 7.5 g/l H_2O_2	52.0	52.2
US + 0.5 g/l FeSO$_4$ + 5 g/l H_2O_2	56.2	49.9
US + 1 g/l FeSO$_4$ + 10 g/l H_2O_2	49.2	47.5

Table 4
Degradation of p-nitrophenol using combination of ultrasound and advanced Fenton process.

Operating parameters	Extent of degradation for 0.5% solution (%)	Extent of degradation for 1% solution (%)
US + 0.5 g/l Fe + 2.5 g/l H_2O_2	62.7	55.7
US + 1 g/l Fe + 5 g/l H_2O_2	66.4	58.0
US + 0.5 g/l Fe + 3.75 g/l H_2O_2	63.6	56.6
US + 1 g/l Fe + 7.5 g/l H_2O_2	63.2	59.2
US + 0.5 g/l Fe + 5 g/l H_2O_2	65.0	57.8
US + 1 g/l Fe + 10 g/l H_2O_2	62.7	55.2

ultrasonic irradiation, which is ascribed to the enhanced degradation efficiency of model pollutant in the coupled ultrasonic process ($US/Fe^{2+}/H_2O_2$) compared to US and Fe^{2+}/H_2O_2 systems operated individually [3,31]. The various radical reactions taking part in the combined operation can be given as follows:

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

$$Fe^{3+} + H_2O_2 \rightarrow Fe-O_2H^{2+} + H^+$$

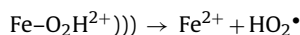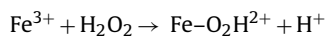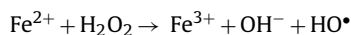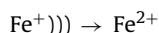$$Fe-O_2H^{2+}))) \rightarrow Fe^{2+} + HO_2^\bullet$$

A careful analysis of the results given in Table 3 indicates that the extent of degradation is marginally higher at higher loading of $FeSO_4$ at a constant loading of hydrogen peroxide, which is consistent with earlier reported results for pollutants like Acid Red 88 [4] and Acid black 1 [9]. It has also been observed that H_2O_2 concentration was more controlling in deciding the extent of degradation of p-nitrophenol. Maximum extent of degradation of 57.6% was observed for 0.5% p-nitrophenol solution at 1 g/l $FeSO_4$ loading and 5 g/l of H_2O_2 concentration. Beyond 5 g/l concentration of H_2O_2, extent of degradation was lowered due to scavenging effect of the excess concentration of H_2O_2 [4,9]. The optimum loading of hydrogen peroxide was found to be also dependent on the initial loading of the pollutant. For 1% p-nitrophenol solution, the optimum H_2O_2 concentration was 7.5 g/l where maximum degradation of 52.2% has been observed.

3.6. Ultrasound + Fe + H_2O_2 (US + advanced Fenton process)

Similar to conventional Fenton process, use of advanced Fenton process was investigated for quantifying the extent of degradation under different operating conditions. In this case, iron powder of 100 mesh size was used in place of $FeSO_4 \cdot 7H_2O$. Unlike $FeSO_4$, iron powder is insoluble in water and hence stirring was provided so that iron powder is well suspended in the reactor and it gets evenly distributed in the reactor. The obtained results have been shown in Table 4 (90 min of treatment time) for all the cases including two different concentrations of p-nitrophenol solution. It has been observed that maximum degradation of 66.4% was obtained at 1 g/l of Fe dosage and 5 g/l H_2O_2. Beyond 5 g/l of H_2O_2 concentration the degradation was lowered as in the case of conventional Fenton process. For 1% p-nitrophenol solution, maximum degradation of 59.2% was observed at 1 g/l Fe dosage and 7.5 g/l of H_2O_2 concentration. Beyond that there was no increase in the degradation even with higher concentration of H_2O_2 due to scavenging effect of $OH^\bullet$ radicals which was evident in degradation of C. I. Reactive Black 8 [31]. Zhang et al. [31] have reported that the degradation of C. I. Reactive Black 8 is not enhanced above H_2O_2 concentration of 5.88 mM. The extent of degradation was higher in the case of advanced Fenton process as compared to the conventional Fenton process at equivalent loadings. Ultrasound in combination with AFP yielded about 10% more degradation than ultrasound combined with conventional Fenton process. In the mechanism of AFP, Fe is corroded and gets converted to Fe^{2+} after that the mechanism is similar to conventional Fenton process. The different radical reactions taking part in the process are as follows:

$$Fe^+))) \rightarrow Fe^{2+}$$

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

$$Fe^{3+} + H_2O_2 \rightarrow Fe-O_2H^{2+} + H^+$$

$$Fe-O_2H^{2+}))) \rightarrow Fe^{2+} + HO_2^\bullet$$

4. Conclusions

The present work has clearly established the utility of using a combination of ultrasound induced cavitational reactors and conventional or advanced Fenton process for degradation of p-nitrophenol at pilot scale operation. The results also suggest that an optimum loading of hydrogen peroxide exists and it is critical to establish this optimum concentration as any unutilized hydrogen peroxide can add to effluent COD. Advanced Fenton process (AFP) is more efficient in degradation of pollutants than conventional Fenton process, attributed to the fact that presence of solid particles enhances the cavitational activity. These results are quite important considering the fact that use of waste iron particles can indeed be thought off which can lead to overall lower treatment costs. The data presented here adds useful information in terms of scale up strategies for large scale operation of the hybrid treatment schemes for industrial wastewater.

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