Desalination and Water Treatment
www.deswater.com
doi: 10.5004/dwt.2021.26753

215 (2021) 160–166
March

UV-irradiated fly ash-catalyzed Fenton-type process for the removal of paracetamol in wastewater: nickel, copper, and manganese as active sites

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Received 15 May 2020; Accepted 1 November 2020

ABSTRACT

This study investigates the application of a photocatalytic process involving fly ash (FA) as a Fenton-like catalyst (without iron) for the removal of paracetamol (Para) in water. The active sites in Fenton-type reactions were found to be the trace metals in FA such as nickel, copper, and manganese. Furthermore, the removal efficiencies of the synergic process (UV-FA + H_2O_2) were compared with UV alone, FA, FA + UV, FA + H_2O_2, and H_2O_2 alone. Moreover, the effect of different pH, catalyst dose, a hydroxyl radical scavenger, and the effect of hydrogen peroxide dose was investigated. Para removal efficiency by different processes were found out to be Fly ash + UV + H_2O_2 > Fly ash + H_2O_2 > Fly ash > Fly ash + UV > UV. The adsorption of para on fly ash was found to have no significant effect on removal efficiencies. The optimum conditions were found out to be pH = 3, 8 mM concentration of H_2O_2, 0.2 g dose of fly ash for the synergic process. Although the synergic process was the best at pH 3, however, even near wastewater pH it was found to be effective. It is therefore concluded that fly ash + UV + H_2O_2 based synergic process is effective for the removal of pharmaceuticals even near wastewater pH.

Keywords: Paracetamol; Coal fly ash; Photo Fenton process; Wastewater

1. Introduction

The advanced oxidation (AOPs) processes are among the most suitable options used for water and wastewater treatment processes in combination with conventional methods [1,2]. Many homogeneous and heterogeneous AOPs were studied in the past, such as UV/O_3, catalytic ozonation, photocatalytic oxidation, Fenton and Fenton-like processes, etc. [3]. The classical homogeneous Fenton process involves the addition of ferrous salt along with hydrogen peroxide to produce hydroxyl radicals. Up till now, many modifications in the classical approach were investigated using various iron and non-iron homogeneous or heterogeneous processes involving hydrogen peroxide to promote the production of hydroxyl radicals, such processes are known as Fenton-like or Fenton-type processes [4,5].

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However, among them, heterogeneous Fenton-like processes were found to be highly efficient and economical [3,6].

In the last few decades, the pharmaceutical industries were greatly developed due to the high demand for life-saving drugs [7,8]. They are one of the major sources of wastewater containing refractory pollutants that are difficult to treat with conventional methods [7,8]. The presence of pharmaceuticals, surfactants, and pesticides in the hydrosphere is of great concern for the freshwater, coastal, and marine environment [9,10]. The non-biodegradability of a majority of the pharmaceuticals in wastewater is a serious problem due to their adverse effects on many aquatic, environmental, and health aspects [10,11]. Moreover, the release of pharmaceutically active compounds into the wastewaters may result in catastrophic events in the future, due to the generation of antimicrobial-resistant strains [12]. Therefore, it is indeed important to treat wastewater before being discharged.

In the past various methods were implied to treat pharmaceuticals in water and wastewater. Among them, biological methods were not highly successful due to the resistance of pharmaceuticals and their non-biodegradable nature. Therefore, AOPs were considered to be one of the most suitable options for the treatment of biologically resistant organic contaminants [3,6].

Nowadays, several inexpensive materials have been suggested by researchers as catalysts in Fenton-like processes, such as natural materials, biosorbents, and waste produced from industries and agricultural lands [13–15]. These materials can be used for removing pollutants from wastewater. The fly ash (FA) is a waste material that is produced during the burning of powdered ignite coal into the fluidized bed combustion system for power generation. Large volumes of fly ash are produced each year as a waste product from these power plants. Almost 600 million tons per annum of FA is produced worldwide [16,17]. The disposal of this ash is a major environmental problem. Also, it could not be recycled as it is of no use in industries [18,19]. The coal fly ash has been successfully investigated (previously) as a Fenton-like catalyst for the removal of dyes and some organic pollutants [18–21]. In previous findings, iron was reported as an active site in Fenton-like processes using fly ash [18–21]. One of the major drawbacks of iron-containing Fenton like advanced oxidation processes is the requirement of strict acidic conditions to prevent the precipitation of iron that may still be considered to be the bottleneck for iron-based advanced oxidation processes [4]. Therefore, in current study fly, ash containing trace metals such as nickel, copper, and manganese was considered, keeping in view their complexes at various pH ranges [2], that may be more effective in Fenton-like process as compared with conventional active sites (iron-based) [23,24]. Hence, the photo-Fenton-like heterogeneous catalytic processes involving iron-free fly ash may operate even at basic pH and probably more effective in order to apply these processes on a larger scale.

The focus of the current investigation was to study a UV-assisted Fenton-like catalytic process using FA as a catalyst for the removal of pharmaceuticals, focusing on their charges in solution at studied pH values and their removal efficiency. Moreover, the effect of hydroxyl radical scavenger, reuse performance of the catalyst, and pH effects were investigated to further understand the process.

2. Experimental

2.1. Preparation of catalyst

FA was taken from a coal power plant in near Sahiwal city, Punjab, Pakistan and after washing with ultrapure deionized water thrice, it was dried in an oven for 6 h at 105°C. Then it was stored in a glass jar for use after complete drying.

2.2. Characterization

The structural studies of fly ash were investigated by using X-ray diffraction (XRD) technique, for that X-ray diffractometer (PANalytical X'Pert MPD, UK) having Cu K-α radiations (λ = 1.5405 Å) at 40 kV (at 40 mA). For elemental analysis, energy-dispersive spectroscopy (EDS) was used. The composition of fly ash was obtained by X-ray fluorescence (XRF) analysis. The fly ash surface morphological analysis was studied by scanning electron microscopy (SEM, Model JSM 6010L). The mass titration method was implied to quantify the isoelectric point (pH_{pzc}) [25]. Finally, surface area and pore size were quantified using Brunauer–Emmett–Teller (BET) method (ASAP 2020, Micrometrics , US).

2.3. Experimental procedure

The paracetamol (high performance liquid chromatography (HPLC) grade, purity 99%) was obtained from Signa Aldrich UK. The solutions of paracetamol (15 ppm) were prepared from its stock solution using the dilution equation ($C_1V_1 = C_2V_2$) in 100 mL flask. Although the pharmaceuticals may present in relatively low concentrations in real wastewater. However, to test the effectiveness of the studied process (FA/UV/H_2O_2) and to investigate the by-products formed if any, solution of relatively higher concentration of paracetamol (15 ppm) was used. The NaOH (1 N) and HCl (1 N) solutions were used to adjust the pH solutions, the pH of the solutions was measured by using a benchtop pH meter (HI-2211, Hanna instruments USA). The samples were drawn over fixed time intervals from a flask placed in UV-light (The lamp emits UV rays (20 W) with a maximum wavelength of 368 nm (Sylvania, Germany) containing a wooden box. All the reagents were of analytical grade and were used without further purifying them.

For paracetamol analysis, HPLC system (Hitachi Elite Lachrom L-2130) associated with C18 column (4.6 × 250 mm, Poroshell 120) was used. The mobile phase composition was 50:50 acetonitrile-phosphate buffer solution having composition, 0.2 M KH_2PO_4, and 0.2 M NaOH in ultra-pure deionized water. The injection volume was 10 μL and the flow rate was 1 mL/min and the wavelength were fixed to 254 nm. The percentage removal efficiencies of paracetamol were determined by using the following formula:

$$\text{Paracetamol Removal}\left(\%\right)=\frac{A_0-A_t}{A_0}\times 100 \qquad (1)$$

where A_0 is the peak area at $t = 0$ and A_t is the peak area time t.

3. Results

3.1. FA characterization

The point of zero charge of fly ash was found to be in the basic range that is pH_{pzc} = 9.1 (Table 1). The previous findings indicate that point of zero charge of fly ash depends on silica, alumina, and calcium content. Moreover, the results of current study agree with previous findings (indicating alkaline nature of fly ash) [26]. Hence, the significant alumina and calcium content (Table 2 and Fig. 2), as indicated by the fly ash composition (Table 2) and the elemental analysis as shown in Fig. 2, which may lead to alkaline pH_{pzc} [25–27]. The XRD patterns (Fig. 1) indicate that there were no obvious crystalline phases in studied coal fly ash samples. The SEM results shown in Fig. 2 indicate a highly dense, irregular, hollow, and porous morphology of fly ash. The specific surface area and pore size of the coal fly ash were found to be 11.35 m^2/g and 1.35 nm, respectively (Table 1). Fig. 2 shows the EDX scan of FA, the results indicate that apart from silica and alumina content, the FA contain significant amounts of trance transitions metals such as Cu, Mn, and Ni. These transition metals may be quite useful in the catalytic activity of FA by interacting with H_2O_2 to produce hydroxyl radicals. It is important to mention here that although the basic components of the FA (silica, alumina, calcium, potassium, and trace metals) were the same, however, their composition may alter due to production processing in different power plants. This may alter its chemical composition and properties [19]. The above-mentioned basic components of studied FA are similar to FA components reported in some recent studies on FA [16,19,28,29]. However, the elemental composition of basic components and trace metals may slightly vary [16,19,28,29]. The specific surface area of studied FA was found to be slightly higher than some earlier reports [16,19,28,29].

3.2. pH effect

The paracetamol removal efficiency was compared by varying the pH for the photo-Fenton like process (Fig. 3). Results are shown in Fig. 3 indicates that the highest removal efficiency was achieved at acidic pH. This is may be due to poising of active sites of catalyst at high pH values [30,31]. Previous investigations also reported that such processes were more effective near acidic pH and that the removal efficiency may decrease by increasing pH [13,30–31]. It may be due to the formation of $M(OH)^+$ complexes at low pH (2–4) [13,30–31]. On the other hand, Fe^{2+} ions are unstable at a pH > 4.0, which may lead to the production of ferric hydroxy complexes [13,30–31]. In addition, the formed complexes interact with hydroxides in water and form $[M(OH)_4]^-$ complexes at basic pH values. Moreover, in alkaline pH, the hydrogen peroxide may also be unstable and decompose to give oxygen and water [32,33]. It is important to mention here that in current investigation the FA used contains trace amounts of metals such as Mn, Ni, Cu, etc. (Fig. 2). Therefore, unlike conventional Fenton-like processes involving iron species, it provides more flexibility in terms of pH range [22]. The results clearly indicate that even at pH 6 and 8 (near wastewater, pH = 6–9) significant removal efficiency of paracetamol obtained. For example, the removal efficiency of para was 60% (in 120 min) and 28.5% for pH = 6 and 8, respectively (Fig. 3). This may be due to the workability of active sites (Mn, Cu, and Ni) in broader pH range [22] as compared to conventional ferrous based fly ash catalyst and UV-irradiation, that may decompose H_2O_2 leading to produce of $^{\bullet}OH$ radicals in the solution [34].

3.3. Comparison of processes for paracetamol removal

The results presented in Fig. 4 show the comparative study of various processes with photocatalytic

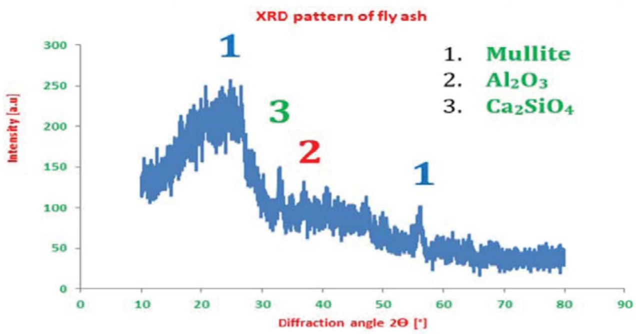


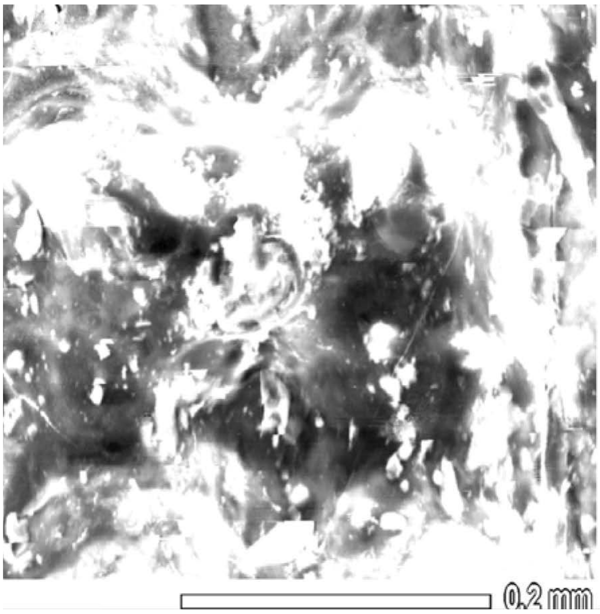
Fig. 1. XRD pattern of FA.

Table 1
Properties of coal fly ash and paracetamol

Properties of coal fly ash	Pore diameter (nm)	Specific surface area (m^2/g)	Point of zero charge (pH_{pzc})
	1.35	11.35	9.1 ± 0.3
Properties of paracetamol	Formula	Molar mass (g/mol)	pka
	$C_8H_9NO_2$	151.2	9.38

Table 2
Chemical composition of coal fly ash

Components	SiO_2	Al_2O_3	CaO	K_2O	NiO	MnO_2	CuO	Carbon residual
Amount (wt.%)	31.54	26.25	5.71	2.42	2.31	1.3	2.12	27.54



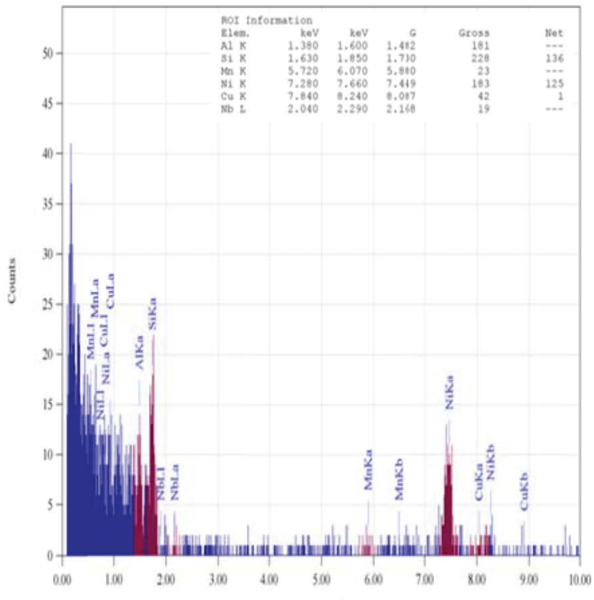


Fig. 2. SEM and EDX of fly ash.

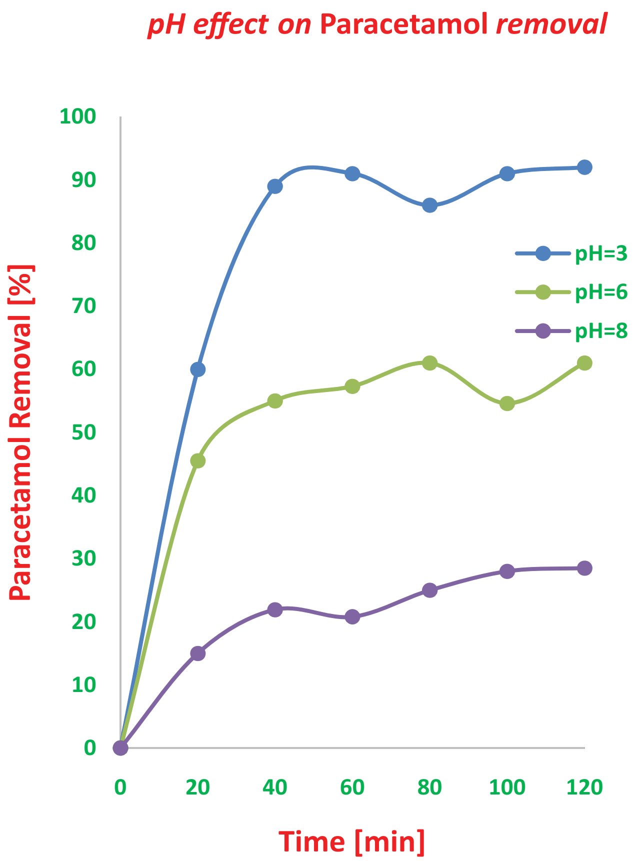


Fig. 3. Effect of pH on the removal efficiencies of paracetamol in fly ash-based photo Fenton-type process (C_0 = 15 mg/L; pH = 3, 6, 8; H_2O_2 = 8 mM; volume = 100 mL; catalyst dose = 0.2 g/100 mL; t = 120 min).

Fenton-like process using FA. The results were found to be in the order of FA + para + H_2O_2 + UV > FA + para + H_2O_2 > FA + para > para + H_2O_2 > para + UV. The removal efficiencies were 60.4% > 43.5% > 28% > 8.1% > 5.1%, respectively, in the first 20 min (Fig. 4). This indicated that the best degradation was obtained by using photocatalytic Fenton-like process. This may be due that fact that the photolytic exposure of M(III) complexes results in the regeneration to M(II), hence it further reacts with more H_2O_2 [13,30–31].

In addition, the catalyst may provide its surface for the reactions of pollutants [13]. The results further reveal that although para may not significantly adsorb on the surface of FA (11.8% in 120 min). The low adsorption of para on FA may be due to the similar charge (positive charge) on para (pH < pKa value of the para) and CFA (pH < pH_{pzc} of FA) (Table 1). The least efficient process was the para + UV (direct photolysis) where the removal was only 9% (in 120 min) in which UV light directly reacted with acetaminophen. The results in Fig. 4 reveal that even in the absence of UV-irritation significant amount of the para was removed for FA + H_2O_2 process, which suggests that FA do catalyze the removal of para in water. Therefore, it is suggested that in the synergic process (FA + para + H_2O_2 + UV) adsorption of para, Direct UV-irritation, UV-H_2O_2 interaction, and role of catalyst (FA) all are significant for the degradation of pharmaceuticals.

3.4. Fly ash dose

The optimum dose of fly ash was determined by performing different experiments. The selected doses were 0.1 g/100 mL (1 g/L), 0.2 g/100 mL (2 g/L), and 0.5 g/100 mL (5 g/L). After a contact time of 120 min, the total removal of paracetamol for different doses of catalyst was in order 0.5 > 0.2 > 0.1 g and removal efficiency was 94% > 90% > 41%, respectively (Fig. 5). The results

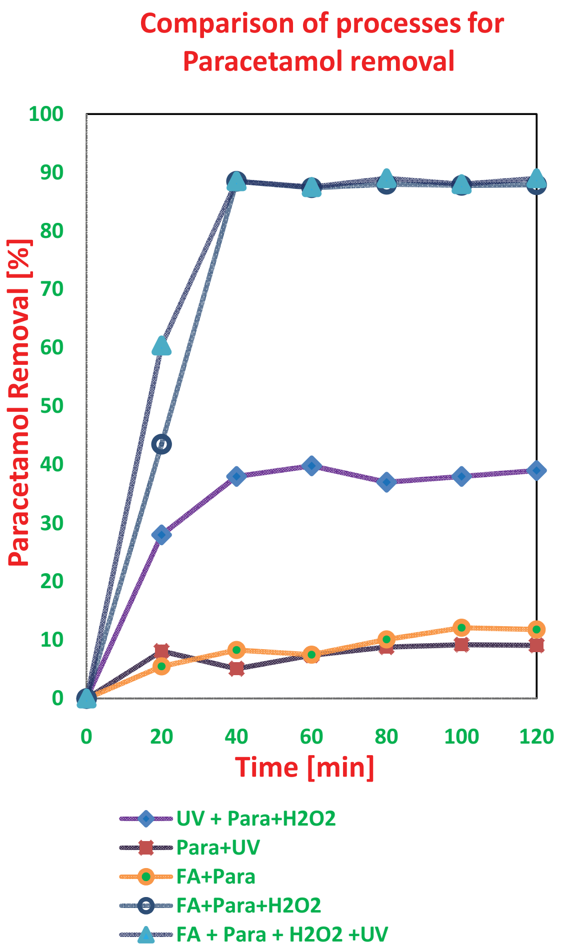


Fig. 4. Comparison of various processes for the removal efficiencies of paracetamol (C_0 = 15 mg/L; pH = 3; H_2O_2 = 8 mM; volume = 100 mL; catalyst dose = 0.2 g/100 mL; t = 120 min).

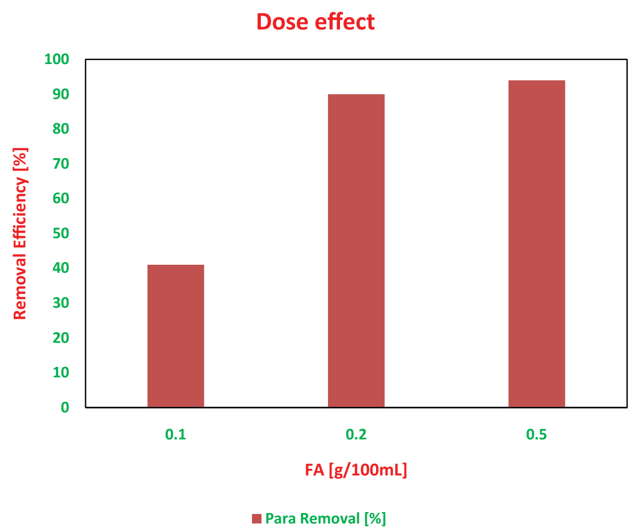


Fig. 5. Effect of catalyst dose on the removal of paracetamol in fly ash-based photo Fenton-type process (C_0 = 15 mg/L; pH = 3; H_2O_2 = 8 mM; volume = 100 mL; catalyst dose = 0.1 g/100 mL, 0.2 g/100 mL, 0.5 g/100 mL; t = 60 min).

indicate that by the increase in catalyst dose, the reaction rates were increased, and higher degree of performance was achieved as when the amount of fly ash was increased more metal ions were reacting with hydrogen peroxide resulting in the formation of more •OH radicals [13,30–31]. Considering the economic aspects and overall removal efficiency 0.2 g of fly ash was considered as the optimum dose.

3.5. Effect of H_2O_2 concentration

The results show that (Fig. 6) after a contact time of 120 min the total removal of paracetamol was in order of 32 > 8 > 16 > 4 mM and the removal efficiency was 92% > 91% > 90% > 78%, respectively. The hydrogen peroxide concentration in solution was directly related to the production of •OH radicals and according to this the more the number of hydroxyl radicals more will be the removal [13,30–31]. In case of higher H_2O_2 doses as (Fig. 7), the removal efficiency was not a high as an increase in dose. The reason might be that at higher doses scavenging of hydroxyl radicals occurs (two radicals combine to form stable H_2O_2). Similar findings were reported by another researcher's [13,30–31]. Therefore, 8 mM was selected as optimum dose of H_2O_2, moreover higher concentrations of hydrogen peroxide are not preferred due to its significant importance on the environment [30].

3.6. Hydroxyl radical scavenging effect

The NaCl has been previously used as a radical scavenger due to the scavenging effect of chloride ions [35–37]. The results given in Fig. 7, clearly indicate that in the presence of NaCl the removal efficiency of synergic process was significantly decreased as compared with out NaCl. For example, the removal efficiency of paracetamol was 87.5% without NaCl and with the addition of NaCl, it was reduced to 55%. Therefore, the results clearly suggested that the studied synergic process operates through radical mechanisms. Hence, NaCl plays a significant role as a hydroxyl scavenger in the degradation of paracetamol. The decrease in removal efficiency in the presence of NaCl may be due to the reaction of chloride ions with •OH radicals [37]. Following are the reactions of the chlorides as scavengers of hydroxyl radicals were reported in previous findings [37].

$$Cl^- + {}^{\bullet}OH \leftrightarrow ClOH^{\bullet -} \quad (2)$$

$$ClOH^{\bullet -} + H^+ \leftrightarrow Cl^{\bullet} + H_2O \quad (3)$$

$$Cl^{\bullet} + Cl^- \leftrightarrow Cl^{2\bullet -} \quad (4)$$

$$Cl^{2\bullet -} + H_2O \rightarrow ClOH^{\bullet -} + H^+ + Cl^- \quad (5)$$

3.7. Mechanism

Fig. 8 indicate the mechanism of photo-Fenton process on FA. The hydroxyl radical's generation was confirmed

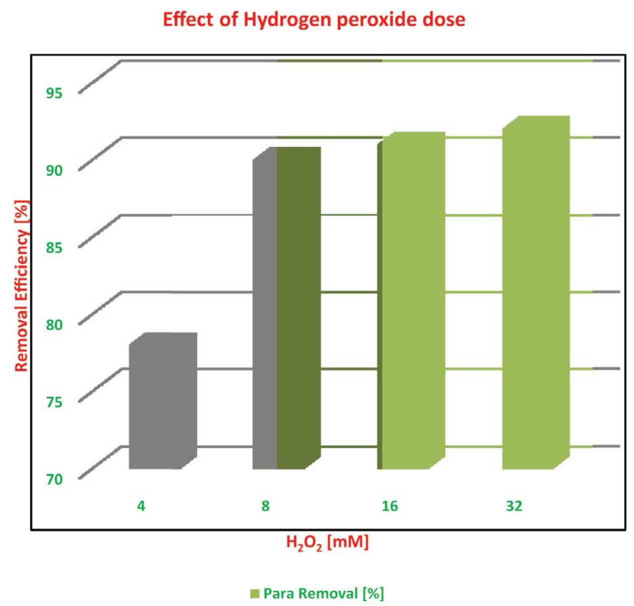


Fig. 6. Effect of hydrogen peroxide dose on the removal of paracetamol in fly ash-based photo Fenton-type process (C_0 = 15 mg/L; H_2O_2 = 4, 8, 16, 32 mM; pH = 3; volume = 100 mL; catalyst dose = 0.2 g/100 mL; t = 60 min).

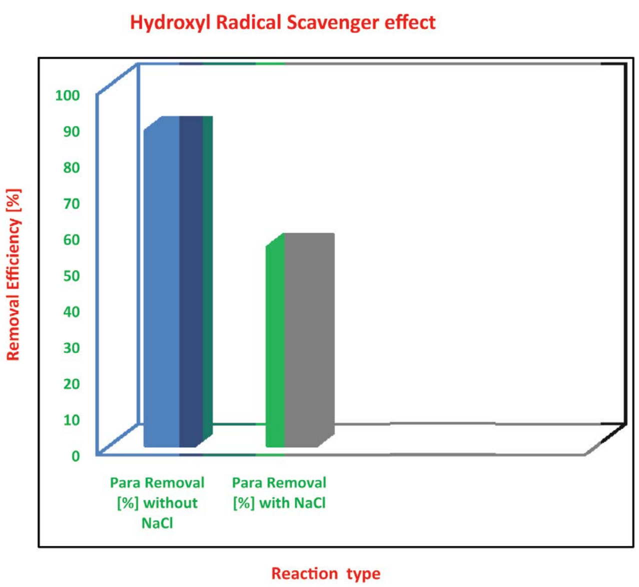


Fig. 7. Effect of hydroxyl radical scavenger (NaCl) effect on the removal of paracetamol in fly ash-based photo Fenton-type process ($C_{0(para)}$ = 15 mg/L; NaCl = 100 mg/L; H_2O_2 = 8 mM; pH = 3; volume = 100 mL; catalyst dose = 0.2 g/100 mL; t = 60 min).

in the current investigation (Fig. 7), the results presented in Fig. 8 clearly suggested that paracetamol degradation involves a radical mechanism. The production of hydroxyl radicals in the synergic process may be due to either the direct UV-irradiation on the metal complexes formed on the surface of FA with the interactions of water molecules [13,30–31] or by Fenton-type interaction of hydrogen

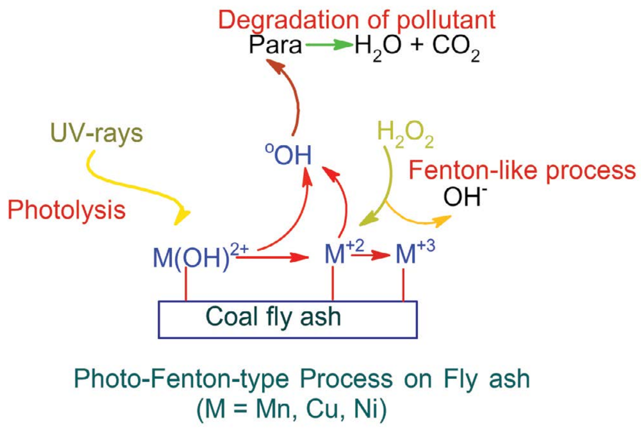


Fig. 8. Proposed mechanism of photo-Fenton type process on FA.

peroxide with the transition metals (Ni, Mn, and Cu) in FA (Fig. 8). The catalyst dose effect and hydrogen peroxide concentration effect indicate the importance of both species in paracetamol removal (Figs. 5 and 6). Therefore, unlike conventional Fenton-like process, the photo-Fenton-like processes are more efficient.

3.8. Cost evaluation

The operating cost of the studied photocatalytic Fenton like process was evaluated based on main factors by following equation:

$$\text{Operating Cost}(\$) = \text{Electrical Energy Cost} + \text{Cost of Chemicals} \quad (6)$$

At optimum conditions, for 60 min treatment, the electrical energy cost for 20 W UV lamp was determined to be \$0.014 based on local electrical energy charges. Based on 8 mM concentration of H_2O_2, the cost of chemical was determined to be \$0.03. Hence, the operating cost of the studied photocatalytic process was determined to be \$0.044 at optimum conditions for the treatment of 15 mg/L of paracetamol, respectively.

4. Conclusions

A comparative study of removal of paracetamol was done by using fly ash, UV light, and H_2O_2 in different combinations. Following conclusions were drawn from study.

- The removal efficiency was found out to be maximum for the Photocatalytic Fenton-like process (FA + H_2O_2 + UV) as compared to others that was found to be in the order of FA + Para + H_2O_2 + UV > FA + Para + H_2O_2 > FA + Para > ACE + H_2O_2 > Para + UV 60.4% > 43.5% > 28% > 8.1% > 5.1%, respectively).
- Although the photocatalytic Fenton-like process was effective at pH 3, still significant removal efficiency of ACE was achieved at pH = 6 and 8 (60% and 28.5% for pH = 6 and 8, respectively, that is near wastewater pH).

- The studied synergic process operates through radical mechanism as indicated by radical scavenger effect.

Acknowledgments

The IEER, UET Lahore (Pakistan) contribution is greatly acknowledged.

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