

Re-using the steel and bleach waste in advance oxidation processes (AOPs) for dye-bath effluent treatment

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ABSTRACT

Two types of wastes, that is, blast furnace flue dust (BFD) from steel manufacturing industry and bleach waste (BW) from textile industry have been utilized for the Fenton-like and photo-Fenton-like processes to assess their efficacy in terms of dye-bath effluents treatment for color and chemical oxygen demand (COD) removal. BFD was used as a catalyst while BW was utilized as H_2O_2 source. The results of this study illustrated that BFD catalyst can be effectively utilized for the treatment of dye-bath effluents when used along with ultraviolet (UV) irradiation. The BFD/H₂O₂/UV process resulted in almost more than 90% of the color removal and more than 60% COD removal within 5 min. However, the process without irradiation of UV, that is, BFD/H₂O₂ was a slow process giving a color removal of about 77% and COD of 67% in 2 h. The process conditions such as pH, concentration of H₂O₂ and concentration of catalyst affects all the processes similarly. However, the time for complete reaction was less for BFD/H₂O₂/UV than BFD/BW. These investigations showed that these processes can be used at higher industrial level to achieve the maximum efficiency in waste treatment processes.

Keywords: Blast furnace flue dust; Fenton-like process; Photo-Fenton-like process

1. Introduction

Metal industries contribute considerably to the generation of waste. Therefore, these industries demonstrate increasing support for studies that investigate the use of these substances for applications in other methods consequently adding value to materials that are treated as waste matter [1]. A steel industry utilizes diverse type of mechanisms for its production process. Therefore, it is quite usual to get numerous materials which are apparently useless along with the end products or finished products. But managing all those leftovers or refuse requires a lot of attention in terms of ecological saving to convert the worthless refuse into useful resource. Therefore, it is essential to categorize the leftovers according to their properties for their appropriate application [2,3]. A kind of refuse of steel industry is the blast furnace flue dust (BFD) which is amongst the most problematic operational element. BFD is produced in huge amounts and may contain some alkalis and heavy metals which make it likely to harm the environment. Therefore, it is needed to carry out the appropriate endpoint options for this waste [4]. The testing of waste toxicity through various techniques has progressively advanced during current decades as a competent technique for environmental risk assessment [5].

BFD is a gas loaded with fine particulates of dust [3]. The particles of BFD vary in size from 500 microns to less than 75 microns. The Mossbauer analysis of BFD gives a mineralogical composition of iron genre having around 51% hematite,

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42% magnetite, and 7% maghemite [4] and X-ray diffraction configuration depicts that BFD is made up of hematite, quartz, magnetite and wustite. There are also small amounts of SiO₂ (4.1%), Al₂O₃ (1.2%) and CaO (2.3%) in it. Trace quantities of Na, Ca, K, Mn, Ti, V and Cl may also be present [2,3].

Another waste "bleach wastewater" generated from textile industry has significant importance to replace analytical grade hydrogen peroxide as a reagent in the Fenton process to obtain additional benefit of rendering it disposable without any added treatment [6].

Textile industry is the major factor of severe environmental pollution that causes risk to the ecosystems [7]. Textile effluents contain heavy pollutants which pollute water, soil and land if discharged without treatment [6,8]. Currently, there are about 14 million chemicals present in the environment and are highly toxic beyond the acceptable level [9]. Globally, the main environmental problem is heavy metal pollution [10]. These effluents contain different types of dyes when they entered the water streams these dyes lead towards the death of aquatic life due to reduced sunlight penetration and dissolved oxygen [11]. They affect human and environmental health by contaminating food chain and drinking water [12]. Complete mineralization along with color removal is required to safeguard environment [13].

Azo dyes are the main extensively used dyestuffs in textile industries, food factories, tannery industries, plastics and cosmetics manufacturing industries [14]. The global dye production comprises of 70% of azo compounds which have an intricate chemical configuration [15]. These dyes exist in the environment for longer time span due to stability and are considered highly toxic [16,17]. Azo dyes are challenging for degradation techniques as they possess complex nature representing double nitrogen bond and additional chromophores imparting color [13]. Several health problems occurred due to the exposure of these chemical agents to living organisms [18]. The efficient decolorization processes are immediately required [19].

Advanced oxidation processes (AOPs) have proven to be more successful in pollutant removal from textile wastewater [20]. The AOPs are chemical oxidation processes based on the reactive radicals generation, that is, •OH radical and consists of various processes such as Fenton and Fenton-like reactions and photo-Fenton and photo-Fenton-like reactions [21]. Their implementation is simple and easy along with production of less residual products [22,23]. Among AOPs, Fenton and photo-Fenton processes are the most encouraging techniques [24]. The catalysts used during oxidation processes are hydrogen peroxide and iron ions [25]. Oxidation of organic or inorganic substrates by active oxygen species generated by the reaction of peroxides (typically H₂O₂) and Fe ions include Fenton and other reactions of this kind, for example, Fenton-like process [26,27]. In Fenton-like process, the ferrous ions are regenerated by reacting with remaining hydrogen peroxide producing additional hydroxyl radicals [26,28].

The photo Fenton process combines ultraviolet radiation and hydrogen peroxide with ferrous ion complexes generating additional •OH and thus breaking down the pollutants at a faster rate than that of the process without ultraviolet (UV) radiation [26]. The treatment of effluents through radiation utilization has many advantages such as degradation of non-biodegradable compounds, reduction of secondary pollution, environment friendly and cost-effective [29]. In contrast to the Fenton, photo Fenton process regenerates ferrous ions in a cyclic manner by photochemical reduction of ferric ions and process proceeds with the availability of hydrogen peroxide [30]. Heterogeneous catalysts reinforced by iron have been used to develop Fenton-like systems [31,32]. These catalysts (e.g., zeolite, goethite, clays laden with iron, wastes containing iron, etc.) replace the conventional homogeneous catalysts breaking down H_2O_2 at a faster rate and degrading the substrate rapidly [13,33]. As a result, to evaluate the treatment efficiency, the evaluation of toxicity is very significant because sometimes end products may be more toxic than parent compounds [7].

This study is aimed to evaluate the efficiency of BFD as a catalyst, in terms of color and chemical oxygen demand (COD) removal in photo-Fenton and Fenton-like systems to investigates the process conditions (time, pH, concentration of H_2O_2 and bleach waste [BW], concentration of iron catalyst) and the utilization of blast furnace dust with bleach wastewater of textile industry as an alternative source of hydrogen peroxide.

2. Materials and methods

2.1. Sampling

The BFD samples were taken from Pakistan Steel Mills, Karachi. The approximate composition of BFD sample in terms of physiochemical configuration was obtained from the Pakistan Steel Mills material testing laboratory. X-ray powder diffraction analysis (by: X-ray powder diffraction Lab, Department of Physics, Government College University, Lahore) was used to characterize the sample. The textile wastewater samples were collected from a textile industry named Indus Home Ltd. using grab sampling technique from dyeing unit outlet and washing unit outlet according to APHA procedures [34].

Dye-bath effluents contained reactive dyes along with other chemicals which were yellow FN-2R (0.10% v/v), turquoise GN (1.12% v/v), blue-FNR (0.41% v/v), tanalene KDC (0.50 g/L), common salt (60 g/L) and soda ash (20 g/L). Bleach wastewater contained chemicals which were caustic soda liquid 50% v/v, hydrogen peroxide 50% v/v (bleaching agent), oxidative desizer (sodium per sulfite $Na_2S_2O_8$), stabilizers and sequestrants. Samples were stored at 4°C after collection.

2.2. Analytical methods

Hydrogen peroxide (H_2O_2 ; 35% w/w) of analytical grade from Merck, Germany, was used. The pH of the reaction solutions and samples was adjusted using 1 M H_2SO_4 and 0.1 M NaOH solutions. Chemicals used for titrimetric procedure of H_2O_2 content determination include sulfuric acid, potassium permanganate and sodium oxalate. pH, electrical conductivity, salinity, total dissolved solids (TDS), temperature, COD and color were measured according to standard methods of water and wastewater [34]. The chemicals and reagents used were listed in Table 1.

2.3. Experimental layout

A lab-scale rector was used for the treatment of effluents by photo-Fenton-like process to evaluate the effect of

Table 1 Chemicals with their purity

Chemical	Purity, %
Hydrogen peroxide	35
Sodium persulfite	99
Sulfuric acid	95–98
Sodium hydroxide	≥98
Potassium permanganate	95–99
Sodium oxalate	99

ultraviolet radiation on the treatment process. The efficiency of BFD and BW using the Fenton-like and photo-Fenton-like processes was assessed by COD and color removal of tested wastewater samples. All the tests were performed at ambient temperature. Tests were performed using different doses of BFD (Fig. 1).

2.4. Experimental procedures

Preliminary tests for Fenton-like reactions were performed for H₂O₂ dose optimization and identifying the effects of various doses of BFD, pH, UV and BW on samples. The ultraviolet radiations 254 nm of wavelength were utilized. The processes without BFD utilization were compared for color removal of the effluents. Effluents were exposed to UV light (alone) in the reactor for 60 min at pH 7 and were analyzed for color removal after regular intervals. 100 mL sample was exposed to various H₂O₂ doses (i.e., 2.94, 5.88, 8.82, 11.77 and 14.71 mmol/L) and analyzed for color removal after time interval of 1, 2, 4 and 24 h. Similar procedure was repeated with bleach waste as hydrogen peroxide source. Optimal doses of hydrogen peroxide and bleach waste were used with 80 min of UV radiation exposure at pH 7. Effluents were analyzed for color removal after regular time intervals. The approximate experiment repetition was five runs while the final average removal efficiency was calculated and mentioned. The results were analyzed through SPSS for windows, Version 16.

3. Results and discussion

The results showed that the concentration of COD and pH in bleach wastewater was high as compared with dyebath effluents. The concentration of electrical conductivity (EC), salinity and TDS was high in dye-bath effluents as compared with bleach water as shown in Table 2.

The significant technology which is generally utilized for the characterization of crystal particles and micro materials was powder X-ray diffraction [35]. The powder X-ray diffraction analysis of BFD indicated the presence of oxides of iron mainly magnetite and hematite (Fig. 2). The studies showed that the amount of ferrous (Fe²⁺) produced by this catalyst (BFD) was about 10% (w/w) of the total soluble iron and almost 90% of the soluble iron consists of Fe³⁺ which is most probably owing to magnetite, maghemite and hematite existence in BFD which gives rise to ferric planes in the particle of catalyst in bulk [4].



Fig. 1. Schematic of lab-scale reactor used for photo-fenton-like process.

Table 2

Physiochemical properties of bleach waste and dye-bath effluents

Parameters	Bleach wastewater	Dye-bath effluents
COD	6,580 mg/L	4,260 mg/L
pН	10.85	10.83
EC	2,305 µS	3,530 µS
Salinity	2.3 ppt	52.9 ppt
TDS	1,406 mg/L	48,500 mg/L
Absorbance	-	3.4
Lambda max	-	462 nm
H_2O_2 content	13 mg/mL	-



Fig. 2. X-ray diffraction analysis of blast furnace flue dust.

3.1. Effect of H₂O₂ doses (analytical grade)

The results depicted in Fig. 3 shows that 11.77 mmol/L dose of H_2O_2 resulted in 59.4% color removal with 1 g/L of BFD dose. The color removal efficiency decreased from 59.4% to 55.7% on further increasing the H_2O_2 dose. A gradual decrease in the removal efficiency was noticed because the increased concentration of H_2O_2 inhibits the process [36].

*3.2. Effect of H*₂*O*₂*doses (bleach waste source)*

It also appears in Fig. 4 that 8.82 mmol/L of H_2O_2 with 1 g/L catalyst (BFD) gives the highest color removal efficiency of 43.3% which goes on decreasing by increasing the dose. BW was used as H_2O_2 source. Although BW produced lesser efficiency and reaction rates due to impurities effect (solids of BW may cause decrease in contact of catalytic active sites surface to H_2O_2) but H_2O_2 (BW) dose used with the same concentration of catalyst was lesser than the analytical grade H_2O_2 dose. This can be due to the presence of another oxidant present in the bleach waste, that is, sodium persulfate which produced hydroxyl ions in the acidic



Fig. 3. Effect of various H_2O_2 doses (mmol/L) (analytical grade) on color removal of dye-bath effluent by Fenton-like process (BFD content 1 g/L, pH: 3 ± 0.2).



Fig. 4. Effect of various H_2O_2 doses (mmol/L) (bleach waste) on color removal of dye-bath effluents by Fenton-like process (BFD content 1 g/L, pH: 3 ± 0.2).

medium [37]. This could also be due to the decomposition of H_2O_2 and subsequently degradation rate of pollutant was increased at higher doses of catalyst, though excessive dose may also cause side reactions to occur and hence reduction in decomposition of H_2O_2 [26].

It is suggested that increasing the dose of hydrogen peroxide above a certain concentration do not increase the decomposition rate of H_2O_2 because additional concentration of H_2O_2 may cause initiation of competing reactions causing hydroxyl radicals to recombine by the scavenging activity of H_2O_2 [38] and due to this concentration of iron in the main phase and total catalytic active sites which cause the H_2O_2 to decompose remain constant for all the H_2O_2 concentrations [4].

3.3. Effect of various BFD doses

In Fig. 5, the rate of reaction and decolorization increased with the increase in the catalyst dose from 0.5 to 3 g/L from 58.4% to 77.7% respective efficiencies. Above 3 g/L, the color removal efficiency was marginal. The results depicted that in the non-irradiated process (Fenton-like), the rise in catalytic dose steered enhanced reaction rate and improved efficiency in terms of discoloration. This owes to an augmentation in number of catalytic active sites and increased amount of soluble iron leached to solution which led to an increased •OHion generation. The results illustrated that the color removal rate was enhanced with the rise in catalytic dose up to 3 g/L and after that there was no appreciable amount of decolorization observed.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + \bullet OH$$
(1)

3.4. Effect of various BFD doses/UV radiations

The results in Fig. 6 indicated that UV radiations increase the rate of decolorization of wastewater significantly. In the



Fig. 5. Effect of various BFD doses (g/L) on color removal of dye-bath effluent by Fenton-like process (BFD/H₂O₂) (H₂O₂ content 11.77 mmol/L, pH: 3 ± 0.2).



Fig. 6. Effect of various BFD doses (g/L) on color removal of dye-bath effluent by photo Fenton-like process (BFD/H₂O₂/UV) (H₂O₂ content 11.77 mmol/L, pH: 3 ± 0.2).

irradiated system, the efficiency of the process in terms of color removal increased slightly with the increasing amount of BFD from 0.5 to 1.5 g/L with color removal of 74.3% and 78.1%, respectively, that is, 3.8% increase in decolorization. At 2 g/L about 5.3% increase in color removal was observed as compared with the removal with 1.5 g/L. The decolorization was further improved up to 3 g/L achieving 97.2% color removal. On further increasing the catalyst amount, the color removal was nominal. The results obtained by the photo Fenton-like process illustrated that ultraviolet radiations increase the rate of reaction attaining a color removal efficiency of over 90% within 4 min. The reaction completed attaining decolorization above 97% within 12 min with 3 g/L dose.

The results demonstrated that there was slow increase in decolorization with increasing dose of catalyst from 0.5 to 1.5 g/L but as the catalyst concentration increases faster reaction rates were observed up to 3 g/L. It may be attributed to the fact that catalyst amount above 1.5 g/L caused greater amount of soluble iron to be leached into the medium (due to acidic conditions, i.e., at pH 3) and active sites on the surface of the catalyst were also increased due to distribution of ferrous (Fe²⁺) on the surface of the catalyst (BFD). The removal rate of pollutants by Fenton oxidations processes could be strongly enhanced through ultraviolet radiations (visible light) of higher than 300 nm wavelength [39]. The irradiation by UV causes rise in hydroxyl radical generation hence efficiency of the process was also increased due to reduction (photolytic) of ferric to ferrous and by reactions (photochemical) of ferric complexes [27].

$$\operatorname{Fe}^{2+} + \operatorname{H}_2O_2 \to \operatorname{Fe}(OH)^{2+} + \bullet OH$$
 (2)

Consequently hydroxyl radical generation occurred on the surface of BFD as well as in aqueous medium (due to cycling of Fe²⁺ and Fe³⁺ by UV radiation) proposing homogeneous and heterogeneous reactions to take place [40]. Hence the reaction rate and decolorization efficiency was enhanced considerably by UV irradiation. Aguilar et al. [15] described that the percentage reduction in color during the Fenton-like treatment processes was enhanced as compared with other electro oxidation processes due to the lack of Fe²⁺.

3.5. Effect of BFD/BW process

As shown in Fig. 7, the rate of reaction and decolorization efficiency was less with the dose range up to 2 g/L and was marginally increased at 2.5 and 3 g/L doses with color removal efficiency of 53.2% and 57.5%, respectively.

The BFD/BW process, as illustrated by the results, was a slow process in terms of color removal of the wastewater. The efficiency and reaction rate of the process might be reduced by the effect of turbidity of the solution due to excessive solids of bleach waste. The turbidity of the medium (of catalyst and BW) caused agglomeration of the solid causing decrease in suitable contact of catalyst with H_2O_2 [26]. Besides this, almost 90% of the soluble iron leached by BFD was in the form of Fe³⁺ and only 10% of it is in the form of Fe²⁺ which was distributed on the surface of the catalyst. The reduction of Fe³⁺ by H_2O_2 without the involvement of UV was slow and therefore ineffective [40]. Therefore, in non-irradiated system, the reduction in the contact with the catalyst surface due to turbidity further reduced reaction rate due to decrease in hydroxyl radical generation.

3.6. Effect of BFD/BW/UV process

The addition of irradiation to the BFD/BW process has almost negligible effect in terms of increase in color removal efficiency but the rate of reaction was improved due to some extent improved cycling of Fe³⁺ to Fe²⁺ by irradiated process as compared with non-irradiated one. But it was speculated that turbidity of the medium (due to bleach waste) reduces the direct photolysis of hydrogen peroxide as well as cycling of Fe³⁺ to Fe²⁺ by UV irradiation, for which rate of reaction of BFD/BW/UV process was not comparable with that of BFD/ H₂O₂/UV.

Hence it was inferred that turbidity of the medium interferes with the process efficiency in terms of color removal of the effluents. Maximum decolorization achieved within 10 min was 37% with 3 g/L dose of catalyst. 80 min exposure with 3 g/L of catalyst produced 56% color removal as shown in Fig. 8.



Fig. 7. Effect of various BFD doses (g/L) on color removal of dye-bath effluent by Fenton-like process (BFD/BW) using bleach wastewater as H_2O_2 source (H_2O_2 content 8.82 mmol/L, pH: 3 ± 0.2).

3.7. COD removal by Fenton-like process

COD removal was slower with Fenton-like process of this study as compared with photo-Fenton-like process. Process with bleach waste being used as H_2O_2 source produced maximum COD removal of about 45% while with that of analytical grade H_2O_2 produced around 68% as shown in Fig. 9. When the two processes were compared in terms of rate of COD removal, their difference was comparable with that of decolorization. So it was assumed that the factors that affect the rate of decolorization in both the processes also affect the rate of COD removal [24].

3.8. COD removal by photo Fenton-like process

COD removal rate was increased significantly by the UV irradiation of the process utilizing analytical grade hydrogen peroxide. COD removal was achieved up to 89% with this process. Around 88% of COD was removed within 40 min of UV irradiation. About 62% of COD was removed within 5 min while 77% was removed within 20 min of the reaction time.

The process utilizing BW as source of H_2O_2 produced around 47% of COD reduction of the wastewater. The results indicated that rate of COD removal by BFD/BW/UV was almost half than that of BFD/H₂O₂/UV. It may be speculated that the factors that affect the decolorization efficiency also affect rate of COD removal by BFD/BW/UV process as shown in Fig. 10.

3.9. Effect of pH

The aqueous pH has a major effect on the efficiency of Fenton-like process. The results demonstrated that the percentage color removal decreased with the increase in pH.



Fig. 8. Effect of various BFD doses (g/L) on color removal of dye-bath effluent by photo Fenton-like process (BFD/BW/UV) (H_2O_2 content 8.82 mmol/L, pH: 3 ± 0.2).



Fig. 9. COD removal by Fenton-like processes using H_2O_2 (analytical grade) and bleach wastewater as source of hydrogen peroxide. (BFD content 3 g/L, H_2O_2 content 11.77 mmol/L and 8.82 mmol/L, respectively).



Fig. 10. COD removal by photo-Fenton-like processes using H_2O_2 (analytical grade) and bleach wastewater as source of hydrogen peroxide (BFD content 3 g/L, H_2O_2 content 11.77 mmol/L and 8.82 mmol/L, respectively).



Fig. 11. Effect of pH on Fenton-like process using bleach waste as $\rm H_{2}O_{2}$ source.



Fig. 12. Effect of pH on Fenton-like process using $\rm H_2O_2$ (analytical grade).



Fig. 13. Color removal by treatment of dye-bath effluents using H_2O_2 (analytical grade)/UV and bleach wastewater/UV (H_2O_2 content 11.77 and 8.82 mmol/L, respectively, pH: 7).

At pH 3 color removal achieved with BW (as hydrogen peroxide source) was 56.3% and at pH 2 it was nearly 53%. At pH 4, 5 and 6 color removal achieved was 48%, 37% and 25% shown in Fig. 11, respectively. Decolorization achieved at pH 2 and 3 differ with slight effect but according to the results, pH 3 was considered optimum.

Decolorization achieved with H_2O_2 (analytical grade) depicted pH 3 as optimum giving 77% color removal. Color removal decreased on increasing the pH. At pH 5 and 6, color removal achieved was least, that is, 49% and 47% as shown in Fig. 12, respectively, with min or difference between the results of both. The results achieved by both the sources of hydrogen peroxide demonstrated that pH range 2–3 give better results with BFD as catalyst of Fentonlike process. This is because under acidic conditions (at pH 3 ± 0.2) more soluble iron was leached from BFD [4]. The Fenton reagent showed decline in its action when the pH is higher because it causes ferric hydroxides to precipitate and form iron oxohydroxides which are comparatively less reactive species. So hydroxyl radical generation was reduced since free iron ions concentration is decreased [27]. Hydroxyl radicals drop their oxidation potential as the pH increases [26]. It also suggested that optimum pH for the Fenton reaction was found to be around 3, irrespective of the pollutant to be degraded.

Rate of reaction was almost half in case of BW/UV process as compared with H2O2/UV process. Bleach waste requires longer period of irradiation for color removal treatment and color removal achieved was 45% after 80 min as shown in Fig. 13. This extension in UV exposure time was probably by the limited penetrations of photons due to the presence of impurities (total dissolved solids, total suspended solids) in bleach wastewater [41]. Studies showed that hydrogen peroxide being a strong oxidant shows its effectiveness when used to oxidize some exemplary compounds or utilized for the treatment of such wastewaters which require flexible oxidation conditions. However, the degradation rate attained was low when H₂O₂ is applied alone to the wastewater. The dose, concentration of effluents and exposure time appear to be important factors for treatment with H₂O₂. The dose is the most important factor because of its scavenging activity when used in excessive concentrations [42].

4. Conclusion

This study shows that the waste produced by the blast furnace of steel manufacturing industry can treat real textile dye-bath effluents effectively through photo Fenton-like process (using analytical grade H₂O₂). The processes using bleach waste as source of hydrogen peroxide along with BFD catalyst also showed promising performance in terms of color removal, COD removal and reaction rate. The color and COD removal by the Fenton-like process (BFD/H₂O₂) was satisfactory but the reaction rate was slower which can be improved by the application of ultraviolet radiation. The ultraviolet radiations greatly enhanced the reaction rate as well as color removal and COD removal efficiency, that is, almost complete color removal and more than 80% COD removal can be achieved. The performance of other processes (not utilizing BFD) was not effective other than UV/H₂O₂ which gives almost 70% color removal with 80 min reaction time. BFD can be used efficiently in photo-assisted Fenton-like (BFD/H₂O₂/ UV) process achieving color and COD removal more than 90% and more than 60%, respectively, within 5 min. These investigations showed that these processes can be used at higher industrial level to achieve the maximum efficiency in waste treatment processes.

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