



UV/Fenton based treatment of paper recycling industry wastewater

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ABSTRACT

The efficiency of UV/Fenton process was investigated to treat the paper recycling industry wastewater. The effect of Fenton treatment on various water quality parameters namely, chemical oxygen demand (COD), biological oxygen demand (BOD), pH, electrical conductivity (EC), total suspended solids (TSS), total dissolved solids (TDS), total solids (TS), nitrates, phosphates, and heavy metals (Hg, Pb, Cd, Cr, Cu) was investigated. The COD, BOD, EC, TDS, TSS, TS, nitrates, phosphates, and heavy metals reduction percentages remained very high (>90%) for various wastewater samples treated. The strategy involving gradual increase in the mass loading (in terms of increasing the share of original wastewater in dilution showed that the UV/Fenton process was efficient to treat paper industry wastewater. The best results were obtained when paper industry wastewater was diluted with nine parts as real wastewater and one part dilution by freshwater. The findings of this study demonstrate that the UV/Fenton process can significantly break down a variety of pollutants from paper recycling industry wastewater.

Keywords: Advanced oxidation processes; Refractory organics; Paper industry; Wastewater treatment

1. Introduction

Recent rapid advancement in industrialization has resulted in increased release of toxic pollutants into the environment. Discharge of non-treated or partly treated wastewater from municipal and industries may result in water and soil pollution [1,2]. Paper industry is the third largest water consuming industry after metal and chemical industries and it is ranked at sixth in terms of primary environmental polluters after steel, leather, oil, cement, and textile industries [3,4]. The total freshwater consumption in paper mills is approximately 75–227 m³ to process one ton of the finished product, which ultimately leads to high volume of wastewater generation [5,6].

Pulping, pulp washing, bleaching and screening are among the most important sources of pollutant production [7,8]. Besides pollutants, these wastewaters encompass other characteristics, for example, high concentrations of biological oxygen demand (BOD), chemical oxygen demand (COD), acidic pH, low biodegradability, strong odor, color, fatty acids, suspended solids, tannins, metals, and more than 200–300 different types of organic compounds [9]. The discharge of toxins from the paper recycling industry imparts negative impacts in water, soil, air, humans and animals [10,11]. Thus, it is important to treat the wastewater before its final release in order to save natural ecosystems.

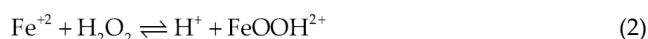
Advanced oxidation processes (AOPs) can be an effective mean to guarantee the removal of various lignocellulosic materials and color etc., from wastewater [12,13]. AOPs

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may effectively reduce the complex structured organic substances/molecules present in the wastewater which are non-degradable by microorganisms unless they are oxidized into less complex forms [14,15]. The mechanism of AOPs involves the generation of reactive oxidizing species, for example, hydroxyl radicals (OH[•]) [16,17]. AOP can accomplish partial mineralization, changing them into more biodegradable substances or even complete mineralization into inorganic substances (CO₂ and H₂O₂). AOP are effective techniques capable of converting toxic substances into safer compounds in a limited time and can be utilized to treat the petrochemical, chemical, and other industrial effluents, and also from the paper recycling industry [18,19]. AOP exploit the high energy of hydroxyl radicals, which attack most of organic molecules, polyphenols, unsaturated fatty acids, halogenated compounds, aromatic rings, and resin acids resulting from the decomposition process of mutagenic compounds or organic nitrogen [20,21]. Fenton process is a handy technique to produce hydroxyl radicals at an ambient temperature and pressure. Hydroxyl radicals are produced, when iron(II) reacts with hydrogen peroxide in the presence of Fe²⁺ [22,23] Eq. (1).



The method of creating HO[•] radicals is incredibly straightforward, requiring neither sophisticated equipment nor special reactants. Considering iron as common and non-toxic element, and hydrogen peroxide as simple to use and safe for the environment, this reaction is a desirable oxidative procedure for wastewater treatment. It must be emphasized that the system behavior cannot be fully explained based on the reaction alone [Eq. (1)]. In fact, adopting an appropriate pH value (2.7–2.8) can cause the decrease of Fe³⁺ to Fe²⁺, as has been noted in numerous recent researches (Fenton-like) proceeding at an appreciable rate.



In these conditions, iron can be considered as a real catalyst as illustrated in Eqs. (2) and (3).

Advanced oxidation pretreatment based on Fenton's reagent is exceptionally successful to treat wastewater from different industries. Toxic compounds can be degraded by this technique [24]. According to Pérez et al. [25] the Fenton process was very successful in treating paper mill effluent. Sevimli [26] investigated the primary reactive species produced by the process and concluded that Fenton's reagent has been used to remove refractory organics and color from pulp and paper effluents due to the great oxidative strength of the OH radical. Xu et al. [27] also reported total organic carbon and COD removal using the Fenton process from wastepaper pulp effluent [27]. Previous studies used UV/Fenton process to treat wastewater from paper industry and removed the individual contaminants like dyes and other organics. The objective of this study was to investigate the performance of UV/Fenton to treat real wastewater to reduce COD, BOD, pH, electrical conductivity (EC), total suspended solids (TSS), total dissolved solids (TDS), total

solids (TS), nitrates, phosphates, and heavy metals (Hg, Pb, Cd, Cr, Cu) from paper recycling industry wastewater.

2. Materials and methods

2.1. Source and characterization of wastewater

The effluent wastewater was collected as grab sample from a paper and paperboard mill. The samples were collected directly from the pipe's end and received no pretreatment. The collection, handling and wastewater storage was done according to the procedures described in Standard Methods for the Examination of Water and Wastewater [28]. The effluents were characterized and analyzed for pH, COD, BOD, EC, TSS, TDS, TS, nitrate, phosphates, and heavy metals (Hg, Pb, Cd, Cr, Cu) analyses as shown in Table 1.

2.2. Experimental set-up

The experiments on UV irradiation were operated using a UV reactor of 750 mL supplied with UV lamps emitting light at 254 nm wavelength as shown in Fig. 1. Sample of wastewater was added into the chamber which contained wastewater, 20 mM FeSO₄ and 35% H₂O₂. The reactor was wrapped in aluminum foil to the secure getaway of UV light. The sample pH was maintained at 3 by using 0.1 N sulfuric acid (H₂SO₄). At each interval of 24 h, the sample was taken and further analyzed for parameters, that is, pH, COD, BOD, EC, TDS, TSS, TS, nitrates, phosphates and heavy metals (Hg, Pb, Cd, Cr, Cu). All the experiments were carried out in triplicates [29].

This experiment was based on dilution ratios of collected wastewater. The wastewater was diluted as shown in Table 2.

2.3. Preparation of Fenton reagent

To prepare 20 mM solution of FeSO₄·7H₂O, 5.5 g of FeSO₄ were dissolved in 1 L of distilled water. The experiment was

Table 1
Influent characterization of paper recycling industry wastewater

Parameters	Paper recycling industry wastewater
pH	7.93 ± 0.05
EC, μS/cm	4,493.66 ± 0.57
COD, mg/L	1,020.33 ± 2.08
TDS, mg/L	560.66 ± 1.52
TSS, mg/L	311 ± 1.73
TS, mg/L	871.66 ± 1.15
BOD, mg/L	427.33 ± 1.52
Nitrate, mg/L	72.33 ± 3.51
Phosphate, mg/L	127.33 ± 0.57
Hg, mg/L	11.13 ± 0.005
Pb, mg/L	3.64 ± 0.003
Cd, mg/L	6.01 ± 0.005
Cr, mg/L	11.44 ± 0.002
Cu, mg/L	11.76 ± 0.002

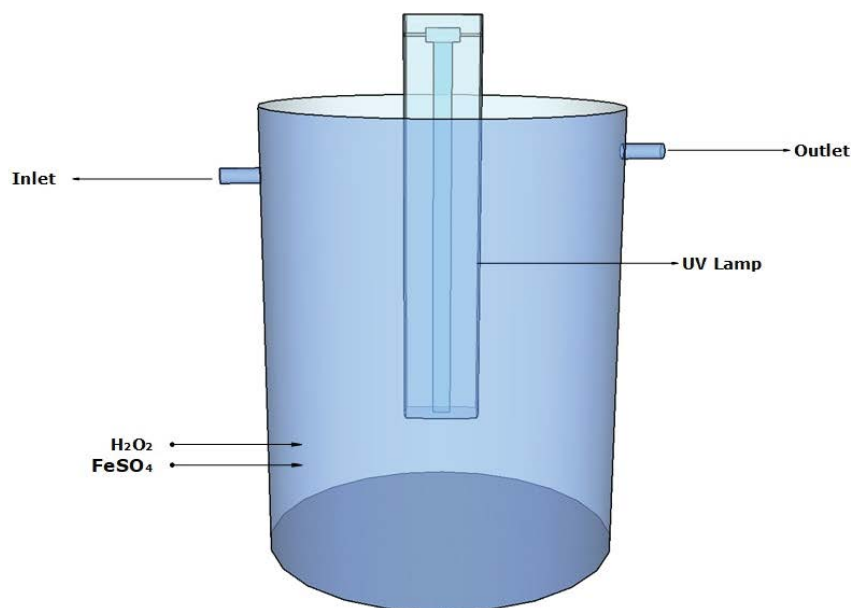


Fig. 1. Schematic diagram of UV/Fenton reactor for treatment of paper recycling industry wastewater.

Table 2
Dilutions for wastewater in UV/Fenton reactor

S. No.	Code	Wastewater (mL)	Distilled water (mL)
1.	1:9 WW	100	900
2.	2:8 WW	200	800
3.	3:7 WW	300	700
4.	4:6 WW	400	600
5.	5:5 WW	500	500
6.	6:4 WW	600	400
7.	7:3 WW	700	300
8.	8:2 WW	800	200
9.	9:1 WW	900	100
10.	10:0 WW	1,000	0

then performed by using this solution. After performing a Jar test with various ratios of FeSO_4 and H_2O_2 , the COD was measured. The ratio that reduced COD more effectively was chosen.

2.4. Analytical procedures

The parameters that were analyzed at laboratory scale were COD, BOD, TDS, TSS, TS, nitrate, phosphate, pH, EC, and heavy metals (Hg, Pb, Cd, Cr, and Cu). All analysis were carried out according to "Standard Methods for the Examination of Water and Wastewater" [28]. The COD of the samples were measured through closed reflux colorimetric method using COD digester (HACH - LTG 082.99.40001, Dubai, United Arab Emirates). In COD vial sample 2.5 mL was digested in digester with 1.5 mL of the digestion solution and 3.5 mL of sulfuric acid reagents. The COD digestion takes place for 2 h at 150°C and later COD reading was

measured by COD spectrophotometer. The pH of the samples was checked by means of digital pH meter (Jenway Model 520, Stone Staffordshire, ST15 0SA United Kingdom). Electrical conductivity values of the wastewater samples were analyzed by using conductivity meter.

The concentration of phosphates was analyzed by using UV-VIS Spectrophotometer (IRMECO UV-Vis, U2020, Lütjensee, Germany). Water sample (approximately 20 mL) was taken in beaker, then stannous chloride (0.4 mL) was added. Then 1 mL of aluminum molybdate was added. Afterwards blue color was developed. When blue color was formed the sample was examined on spectrophotometer at 680 nm and the readings were noticed [28].

The nitrate concentration was analyzed by UV-VIS Spectrophotometer (IRMECO UV-Vis, U2020). 20 mL of sample was taken and 1 mL of 0.1 N HCl was added. The solution was mixed properly. Solution was placed to cuvette. The absorbance of the sample was determined at 220 nm. The absorbance of the sample obtained at 220 nm was changed to concentration, using calibration curve [28].

Heavy metals concentration was analyzed using Atomic Absorption Spectrophotometer (PerkinElmer Analyst 700, Beatrixlaan 301 7312 DG Apeldoorn, The Netherlands) at definite wavelength. Wastewater samples were filter and analyzed on Atomic Absorption Spectrophotometer [28].

3. Results and discussion

3.1. Effect of pH

The pH was measured in order to assess the chemical condition of the solution at the start and completion of the procedure. The pH of wastewater analyzed for different dilutions of experiment is presented in Fig. 2. The pH of the wastewater sample was consistent around 3. As the efficiency of Fenton process was maximum at pH of 3, pH of

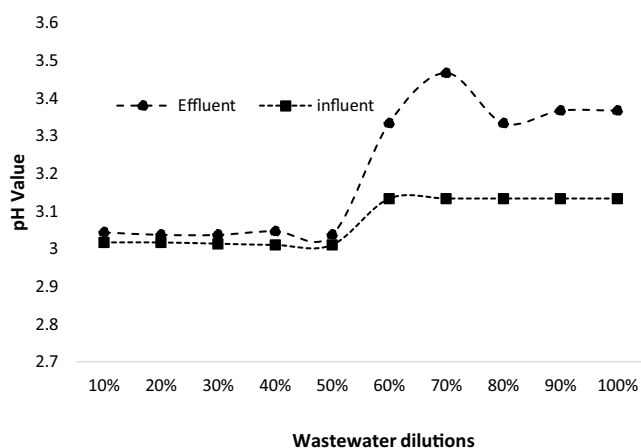


Fig. 2. Effect of pH on paper recycling industry wastewater.

the solution was maintained as mentioned in methodology section. The effluent the pH values were also in the range of 3.04, to 3.15 for various dilutions as shown in Fig. 2. It has already been demonstrated that the pH of the medium and the applied Fe^{2+} to H_2O_2 molar ratio were the two main factors affecting the efficiency of UV-Fenton process [30]. Acidic pH ranges up to 4 was optimum for the reaction. The degradation of organic matter in the wastewater samples took place in acidic pH range. The results were in accordance with the earlier finding regarding the use of UV-Fenton reagent [28–31]. The generation of hydroxyl radicals takes place in acidic environment [32]. On the other hand, a decrease in pH caused increased H^+ ions to enter the reaction. Tri-valent iron in acidic medium is reduced to bivalent iron when it accepts an electron from the hydrogen ion. This generates hydroxyl radicals, and the cycle repeats itself if hydrogen ions are still present [33]. According to a study's, lowering pH can enhance the removal effectiveness. The maximum removal performance occurred at pH = 3, contact duration of 120 min, and 10 mL/L H_2O_2 and 30 g/L iron powder, which was 98.8% iron [34].

3.2. Effect on COD and BOD removal

COD of all wastewater samples was determined before and after treatment of wastewater with different ratios of wastewater by Fenton process. For 1:9 dilution, COD of the wastewater was 91.63 mg/L and the highest COD of wastewater was observed at full strength wastewater (10:0) which was 149.66 mg/L. The obtained results show a decrease in effluent COD. Overall, the range of COD reduction was 85.33% to 91.63% for various dilutions of wastewater at reaction time of 24 h as shown in Fig. 3. A study observed 51% COD removal when the Fenton process was applied [35]. According to Samarghandi and Khojasteh [36], the effectiveness of Fenton process in COD removal was 43%–55% after 24 h of treatment. The effectiveness of COD removal was similar at pH ranges of 2.5 to 4.0. Another study observed COD reduction in range of 68%–70% for UV/Fenton process [37]. Moreover, the highest treatment efficacy was achieved at an optimum pH of 3. The COD removal efficiencies were 82.6% for pH 3; an increase in pH

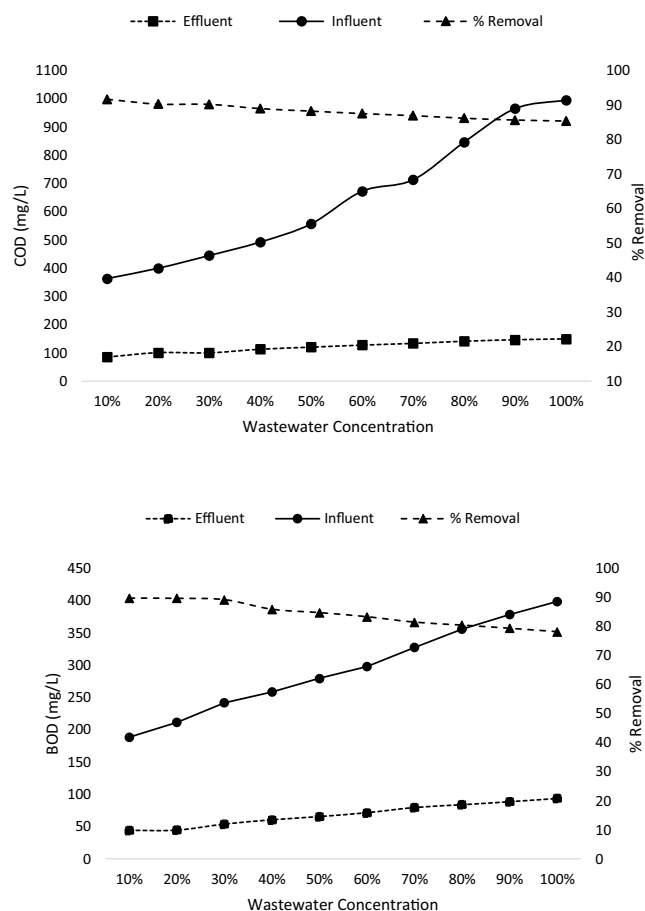


Fig. 3. Effect of UV-Fenton process on chemical oxygen demand (A) and biological oxygen demand (B) removal from paper recycling industry wastewater.

resulted in decreased COD removal efficiencies. At initial pH 11, COD removal efficiency was 54.03% [38]. The Fenton method's ability to degrade COD has also been found to be enhanced by UV radiation in several other studies [39,40]. Due to the continuous generation of reactive oxygen species, particularly hydroxyl radicals, the photo-Fenton technique is considered to be more efficient.

The highest BOD removal efficiency was noted during the experiment with 1:9 dilution. The influent BOD concentration at this point was 188.33 mg/L and the effluent concentration was 44 mg/L with removal efficiency of 89.70%. The lowest removal of BOD was observed at 10:0 wastewater when the influent concentration was 398.33 mg/L and BOD effluent concentration lowered to only 93.33 mg/L with 78.15% removal. The BOD removal efficiencies during treatment were 89.70%, 89.62%, 89.16%, 85.88%, 84.71%, 83.30%, 81.43%, 80.42%, 79.32% and 78.15% for 1:9, 2:8, 3:7, 4:6, 5:5, 6:4, 7:3, 8:2, 9:1, and 10:0 wastewater as shown in Fig. 3 and Table 3. According to a study, the highest BOD removal efficacy of 68% was recorded during the UV-Fenton treatment [41]. A difference between COD and BOD reveals how much organic matter in wastewater is difficult to break down [42]. Radicals generated by the Fenton mechanism enhanced the oxidation of organic matter [43].

Table 3
Influent and effluent dynamics of chemical oxygen demand of paper recycling industry wastewater during treatment

Chemical oxygen demand	Influent	Effluent	% Removal
10%	363.33	85.33	91.66
20%	399.66	101	90.21
30%	444.667	100.333	90.17
40%	492.33	113	88.92
50%	556.66	120.33	88.21
60%	672.33	127.67	87.46
70%	712.66	133.67	86.89
80%	845.66	141.33	86.18
90%	964.66	146.67	85.65
100%	995	149.66	85.33

3.3. Removal of TDS, TSS and TS

Analysis of TDS for various wastewater samples before and after treatment by Fenton process is presented in Fig. 4. The results show a decrease in the effluent TDS concentration as compared to the influent and it shows a removal efficiency of 90.96% for 1:9 dilution, 88.46% for 2:8 dilution, 87.21% for 3:7 dilution, 84.48% for 4:6 dilution, 83.35% for 5:5 dilution, 81.86% for 6:4 dilution, 80.61% for 7:3 dilution, 79.60% for 8:2 dilution, 78.47% for 9:1 dilution, 77.05% for 10:0 wastewater at 24 h hydraulic retention time (HRT) as shown in Fig. 4. The Fenton reagent generates OH^- radicals, which may interact with dissolved solids which are seemingly organics in paper recycling industrial wastewater, thus accomplishing their degradation. In this manner, it ends up in the reduction of TDS.

The highest TSS removal efficiency was noted during the experiment with 1:9 dilution. The influent TSS concentration at this point was 102.66 mg/L and the effluent concentration came out to be 23 mg/L with removal efficiency of 92.60%. The lowest TSS removal was observed at 10:0 wastewater when the influent concentration was 286.33 mg/L and TSS effluent concentration lowered to only 101.66 mg/L with 67.30% removal. The TSS removal efficiencies were in the range of 35 to 86.13%, for various dilutions (Fig. 4). Almost a similar trend was observed regarding TS removal from the wastewater under investigation.

3.4. Effect of nitrate and phosphate

The dynamics of nitrate concentrations before and after treatment are presented in Fig. 5. The highest nitrate removal was noted during the experiment with 1:9 dilution. The influent nitrate concentration at 1:9 was 29.54 mg/L and the effluent concentration was 4.29 mg/L with nitrate removal efficiency of 94.077%. The lowest removal of nitrate was observed at 10:0 and the nitrate concentration of influent was 69.17 mg/L and the effluent nitrate concentration lowered to only 11.19 mg/L with 84.49% removal. The nitrate removal efficiencies during treatment were 94.07%, 93.41%, 91.83%, 89.51%, 88.00%, 87.32%, 86.32%, 86.24%, 86.06% and 84.49% from 1:9, 2:8, 3:7, 4:6, 5:5, 6:4, 7:3, 8:2, 9:1, and

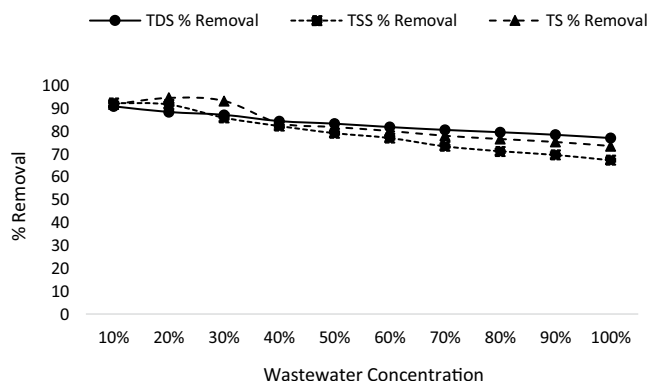


Fig. 4. Effect of UV-Fenton treatment on total dissolved solids, total suspended solids and total solids on paper recycling industry wastewater.

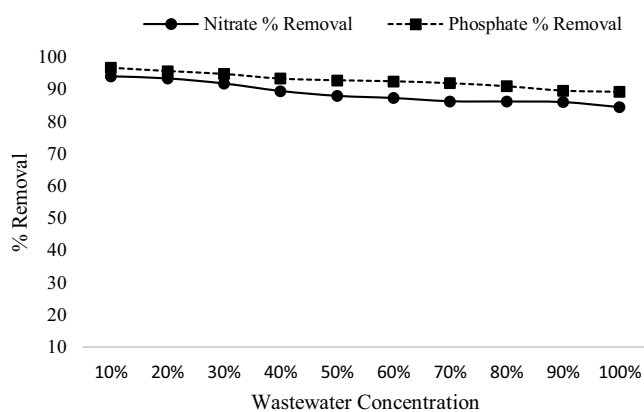


Fig. 5. Dynamics of nitrates and phosphates during paper recycling industry wastewater.

10:0 wastewater. A study showed that Fenton process could effectively remove nitrates. Due to the large concentrations of HO^\bullet produced by the oxidation process, these chemicals are removed during this procedure [44].

Fig. 5, also showed patterns of phosphate removal during treatment. The maximum phosphate removal was reported at 1:9 dilution during which the influent phosphate concentration was 37.99 mg/L and effluent had a phosphate concentration of 4.16 mg/L and a removal efficiency of 96.73%. The lowest phosphate removal efficiency of was at 10:0 wastewater. A study was conducted by [45] to investigate the phosphate removal from the landfill leachates. The best phosphate removal (98%) was observed at the end of treatment time (45 min). This phenomenon can be described by the increment of both pH and dissolved Fe ions in the reactor which formed $\text{Fe}(\text{OH})_n$ flocks that removed the pollutant by complexation [46].

3.5. Effect of heavy metals (Hg, Pb, Cd, Cr, Cu)

The removal or oxidation of various metal ions were also studied during the research and were presented in Fig. 6. The highest Hg removal efficiency was noted during the experiment with 1:9 dilution. The influent Hg concentration at this point was 3.52 mg/L and the effluent

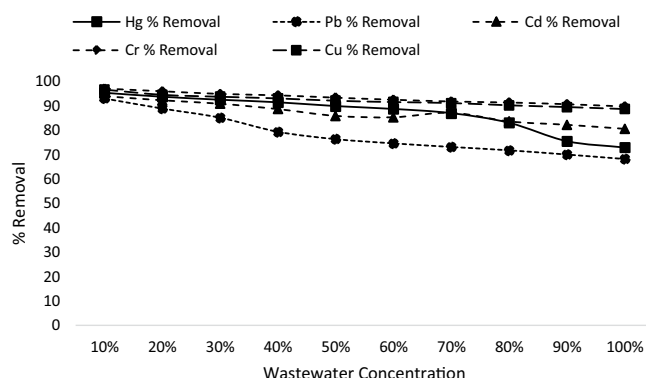


Fig. 6. Effect of treatment on heavy metals (Hg, Pb, Cd, Cr, Cu).

concentration came out to be 0.51 mg/L with Hg removal efficiency of 95.38%. The highest Pb removal was also noted during the experiment with 1:9 dilution. The influent Pb concentration at this point was 1.193 mg/L and the effluent concentration was 0.25 mg/L with Pb removal of 93.04%. The lowest removal of Pb was observed at 10:0 wastewater when the influent concentration was 3.29 mg/L and Pb effluent concentration lowered to only 1.156 mg/L with 68.22% removal.

The highest Cd removal efficiency was noted during the experiment with 1:9 dilution. The influent Cd concentration at this point was 1.726 mg/L and the effluent concentration was 0.36 mg/L with Pb removal efficiency of 94.01%. The lowest removal of Pb was observed at 10:0 wastewater when the influent concentration was 5.73 mg/L and Pb effluent concentration lowered to only 1.166 mg/L with 80.59% removal. The maximum Cr concentration removed was 1.923 mg/L and the effluent concentration was 0.33 mg/L with a removal of 97.11%. The highest Cu removal efficiency was 2.69 mg/L and the effluent concentration was 0.49 mg/L with Cu removal efficiency of 96.68%. The lowest removal of Cu was observed at 10:0 wastewater when the influent concentration was 10.71 mg/L and Cu effluent concentration lowered to only 1.326 mg/L with 88.718% removal. A study found that 93%, 100%, and 73% reductions of heavy metals such Cr, Cd, Cu, and Pb were reported at pH of 2.5 during the photo-Fenton treatment [47].

In the Fenton's system, hydrogen peroxide (H_2O_2) and Fe^{2+}/Fe^{3+} (functioning as semi-conductors) are coupled in an acidic environment. Electron-hole pairs (e^-/h^+) are generated in the semiconductor's conduction band and valance band, respectively, when the semiconductor-electrolyte interface is contacted to radiation. These charge carriers can reduce or oxidize the heavy metal ions present in wastewater that has a sufficient redox potential after migrating to the semiconductor surface. The generated electron-hole pairs should be trapped, and the hydroxyl ions (OH^-) are the most likely places for holes to be trapped. As a result, powerful hydroxyl radicals ($\cdot OH$), superoxide radicals ($O_2^{\cdot -}$), and hydroperoxyl radical anions ($HO_2^{\cdot -}$) are generated [48].

The elimination of heavy metal complexes involves the use of OH^\cdot to break the bond between organic ligands and metal ions, which then allows the organic matter that is released to be destroyed. Hydroxide precipitation is usually employed to remove the released heavy metal ions.

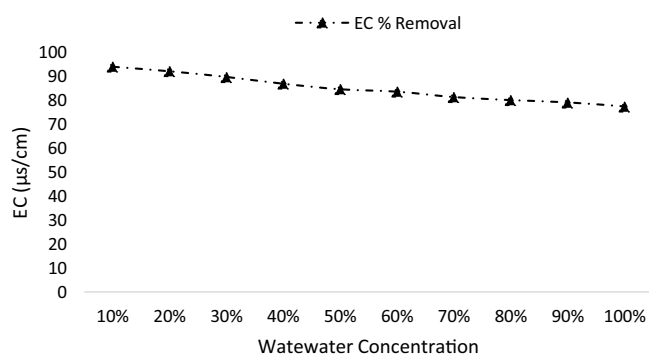


Fig. 7. Dynamics of electrical conductivity in response to UV-Fenton treatment of paper industry wastewater.

Strong oxidants (OH^\cdot) produced by the $Fe-H_2O_2$ system accelerate the oxidation of heavy metals in wastewater [49].

3.6. Effect of electrical conductivity

Analysis of EC was done for all wastewater samples before and after treatment by Fenton process and presented in Fig. 7. The results showed a decrease in effluent EC compared to the influent, in which the EC reduction efficiency was 93.92% for 1:9 dilution, 92.03% for 2:8 dilution, 89.66% for 3:7 dilution, 86.83% for 4:6 dilution, 84.53% for 5:5 dilution, 83.52% for 6:4 dilution, 81.29% for 7:3 dilution, 80.01% for 8:2 dilution, 79.06% for 9:1 dilution, 77.38% for 10:0 wastewater at 24 h HRT as shown in Fig. 7.

4. Conclusion

The UV-Fenton method proved to be very efficient for treating wastewater from the paper recycling industry. The UV/Fenton technique resulted in COD removal range of 85.33% to 91.63% for various wastewater samples within 24 h. BOD reduction range was 78.15% to 89.70% for 10:0 wastewater within 24 h at pH 3 throughout the experiment. The UV/Fenton process showed a significant result for other water quality parameters like nitrates, phosphates, TDS, TSS, TS, EC and heavy metals removal in the paper industry wastewater. The strategy involving gradual increase in the mass loading is recommended for the efficient UV/Fenton process to treat paper industry wastewater. The UV/Fenton process appears to be a promising solution for the treatment of wastewater from the paper recycling industry.

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