DECOLORIZATION OF REACTIVE DYEING EFFLUENT BY SOLAR PHOTO-FENTON PROCESS COUPLED WITH LIME FLOCCULATION

Sajjala Sreedhar Reddy[1], Salam K. Al Dawery[2], Anwar Ahmed[1] and Velchuri Sairam[3]
[1]Department of Civil and Environmental Engineering, University of Nizwa, Oman
[2]Department of Chemical and Petrochemical Engineering, University of Nizwa, Oman
[3]School of Civil Engineering, VIT University, Vellore, India
E-Mail: sreedharreddy@unizwa.edu.om

ABSTRACT

The aim of this study is to decolorize reactive dyeing effluent by of solar based photo-Fenton process coupled with lime flocculation, with an objective of reducing reagent requirement of Fenton process and improve the pH of the treated effluent. Coupling with lime flocculation has reduced forty percent of H_2O_2 reagent requirements of photo-Fenton process for 98% decolorization of simulated reactive dyeing wastewater after a reaction period of 60 minutes and improved effluent pH. It is clear from the literature survey that greater reagent dose of Fenton process is required to decolorize reactive dye effluents containing auxiliary chemicals compared to aqueous solution containing reactive dye alone, due to the inhibitive effect of excess chloride ion present in wastewater. Burnt clay bricks made from chemical sludge, produced during lime flocculation, by replacing 20% of brick earth have the same properties as a standard burnt clay brick and are acceptable from environmental point of view.

Keywords: solar photo fenton, reactive dye, H_2O_2, TCLP, burnt clay brick.

INTRODUCTION

Reactive colorants were listed as the most problematic compounds in textile effluents [1-3]. These are distinguished by their water-friendly solubility and high stability and durability mainly due to their complex structure and synthetic origin [4]. As they are purposely designed to withstand degradation, they therefore offer a high resistance to chemical and photolytic degradation[5]. Under typical reactive dyeing conditions (pH≥10, temperature ≥ 60^0C, salt 60-100 g/l), as much as 50% of the initial mass of the dyes remains in the dye bath in the hydrolyzed form and has no affinity for the fiber [6-7]. Such a reactive dye concentration in the textile dyeing wastewater will seriously threaten the whole ecosystem of natural areas when it encounters them by causing aesthetic pollution, absorbing sunlight strongly responsible for the photosynthetic activity of aquatic plants and forming potentially carcinogenic and toxic aromatic amines under anaerobic conditions [8-12]. Therefore the management of spent reactive dye baths is the single most important environmental problem facing the textile industry. Achieving stringent regulations enforced by pollution control authorities worldwide for the discharge of textile effluents, becoming challenge for the existing treatment plants using conventional dye wastewater treatments methods [10,13,14]. To comply with the strict regulations, numerous researchers have suggested advanced oxidation processes as a viable alternative for efficient removal of reactive dye present in the textile effluents [5, 9, 13, 15-21, 22, 23]. Among the various advanced oxidation processes, the Fenton-type processes are considered most promising for the remediation of highly contaminated wastewater due to operational simplicity and are capable of carrying out deep mineralization of contaminants with effective oxidation [21, 22]. In addition, photo-assisted Fenton processes are better than other advanced oxidation technologies from an economic and environmental point of view, as they make use of photons with wavelengths similar to visible light, with the possibility of driven under solar radiation. Nonetheless, the high operating costs arising from the use of chemical reagents, effluent efficiency in terms of pH and iron sludge management are the key constraints of this technology [5, 23]. The combination of advanced oxidation technologies and other conventional wastewater treatment methods has proven to be more successful in economically treating contaminated drinking water supplies and industrial wastewater[24].

The present study aims to decolorize reactive dyeing effluent by solar photo-Fenton process coupled with lime flocculation, with the following objectives: (i) to study the effect of lime flocculation integration on (a) reagent requirement of solar photo Fenton process, and (b) treated effluent pH, and (ii) Feasibility of iron sludge usage as a substitute for brick earth in making burnt clay bricks.

MATERIALS AND METHODS

Materials

Commercial grade Hydrogen peroxide (30% w/w), ferrous sulphate, Sulfuric acid and lime procured from Muscat were used in the photo Fenton's and lime flocculation process. Sigma Aldrich made LR grade chemicals (Na_2CO_3, NaCl, NaOH, HCl) were used in the preparation of simulated reactive dyeing wastewater. Reactive dye (Procion brilliant blue M.R) used in the preparation of simulated dye wastewater was obtained from ATIC industries, Atul, Gujarat, India.

Preparation Simulated Reactive Dyeing Effluent

Simulated reactive dyeing wastewater was prepared according to the reactive dyeing protocol practiced in the dyeing industries at Nagari, Andhra Pradesh, India.

Reactive dyeing wastewater was prepared by transferring 0.6 g dye, 5g of NaCl, 2g of NaOH and 5 g of Na_2CO_3 into a beaker and to that 8.4 ml of 20% HCl and one liter tap water added. The solution was heated on a stirring plate at 65^0C for one hour. It is important to choose the correct concentration of dye in the wastewater before the treatment tests are performed. By assuming that 20 percent of the dye remains in the expended reactive wastewater, the concentration of 120 mg / l of dye was maintained in all the decolorization tests.

Decolorization Procedure

As shown in Figure-1, simulated reactive dyeing wastewater was decolorized by coupling photo-Fenton process with lime flocculation. Both Photo-Fenton process and lime flocculation experiments were carried out using laboratory jar test apparatus in the open atmosphere during sunny days of May-2019 at University of Nizwa, Nizwa, Oman. There is an evidence in the literature that OH radical mediated (generated from the reactions of Fe (11)/Fe (111) and H_2O_2) decolorization of aqueous solutions of different reactive, direct, acid and basic dyes is highly effective at pH: 1-3[2, 3, 14, 15, 25, 26]. Hence some trial decolorization experiments were carried out at pH: 1, 2 and 3 for different $FeSO_4$/ H_2O_2 dosages. The preferable operating pH was found to be 2. The pH of the reactive dye wastewater was adjusted to 2, with the help of 0.1N H_2SO_4 solution. In photo Fenton's process, after the addition of $FeSO_4$ and H_2O_2 to the reactive dye wastewater, the reaction mixture was stirred at 100 rpm till the end of reaction period.

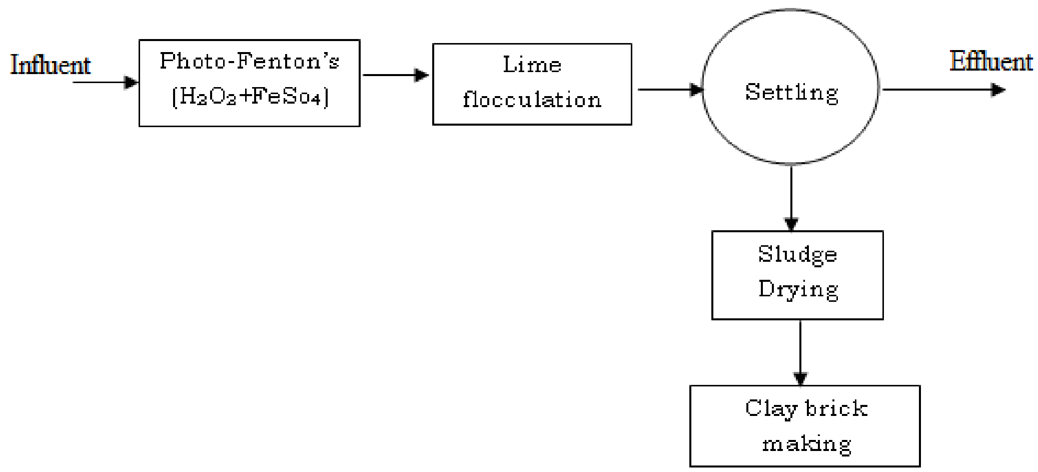

Figure-1. Schematic of decolorization of simulated reactive dyeing wastewater by Photo-Fenton process coupled with lime flocculation.

At the end of reaction period, reaction mixture was aided with lime flocculation, in the same jar test apparatus. In the lime flocculation, after adding stock lime solution to the reaction mixture, contents were rapidly mixed at 100 rpm for two-minutes followed by slow mixing (10 rpm) for 20 minutes to accomplish coagulation and flocculation.

The formed flocks were left to settle quiescently for about one hour, after which supernatant was tested for the residual reactive dye concentration and pH. The residual reactive dye concentration was determined colorimetrically using spectronic made spectro photometer. The absorbance of the dye was measured at 605 nm.

Later settled sludge was dried in a hot air oven at 105^0C for 24hours and utilized in burnt clay brick making as a substitute to clay. Clay is replaced with sludge starting from 0% (100% clay and 0% sludge) up to 30% (70% Clay and 30% sludge). For casting of bricks, 70 mm x 70 mm x 70 mm moulds are used. A total of 12 burnt clay brick samples (4 bricks for each proportion) were prepared at Civil engineering materials laboratory of University of Nizwa. Water content of 25% was maintained for casting of bricks based on previous studies conducted in textile sludge usage in clay brick making [27]. The prepared bricks were then tested for water absorption and compressive strength according to IS: 3495 (part I and II): 1992 [28]. To know the acceptability of sludge based burnt bricks from environmental point of view, toxicity characteristic leaching procedure (TCLP) was conducted in accordance with USEPA Method-1311 [29]. Concentration metals present in the sludge was analyzed at using atomic absorption spectrophotometer as per the procedure described in the standard methods for the examination of water and wastewater[30].

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RESULTS AND DISCUSSIONS

Optimization of Solar Photo-Fenton's Reagent

The effect of concentrations of H_2O_2 and Fe^{2+} on the removal of color from simulated reactive dye wastewater having a dye concentration of 120 mg/l was investigated in detail. The concentrations of H_2O_2 and $FeSO_4$ were varied in the ranges between 150 mg/l – 5000 mg/l and 80 mg/l - 200 mg/l respectively.

Optimum H_2O_2 Dosage

To optimize H_2O_2 dosage, experiments were carried out at different dosages of H_2O_2, keeping dye concentration and ferrous sulphate dosage at 120 mg/l and 140 mg/l respectively. The H_2O_2 concentration was varied from 150 mg/l to 5000 mg/l, and the obtained results are presented in Figure-2. At a fixed ferrous sulphate concentration, the H_2O_2 concentration of 1000 mg/l was effective in decolorizing 98% of color from simulated dyeing plant effluent in one hour of reaction period.

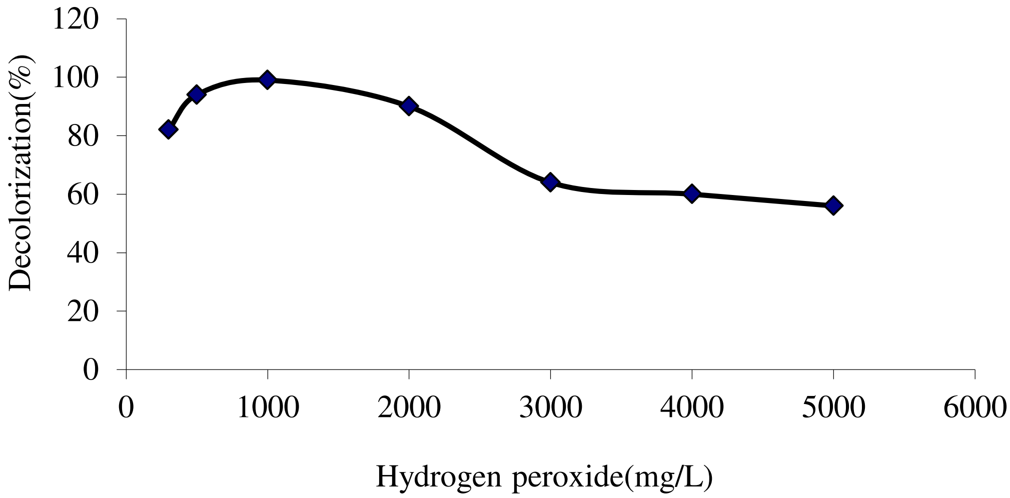

Figure-2. Effect of H_2O_2 dosage on decolorization (Dye=120 mg/l; Ferrous Sulphate: 140 mg/l; pH=2).

Beyond 1000 mg/l of H_2O_2 dosage, the percentage decolorization was decreased, due to the scavenging of hydroxyl radical by H_2O_2 at high concentrations[3] as shown in the eq (1).

$$*OH + H_2O_2 \longrightarrow *HO_2 + H_2O \quad (1)$$

A research by Haber and Weiss (1934) [31] on catalytic decomposition of H_2O_2 in the presence of iron have showed that the decomposition occurred through both OH and HO_2 radicals which initiated chain reactions. Each transformation has its own reaction rate and requites sufficient H_2O_2 to be added to push the reaction beyond that point. This could be the reason for low decolorization at a H_2O_2 concentration less than 1000 mg/l.

Optimum Ferrous Sulphate Dosage

Experiments were carried out to decolorize reactive dye wastewater at five different ferrous sulphate concentrations with hydrogen peroxide concentration remaining unchanged at 1000 mg/l. As shown in Figure-3, with 120 mg/l of dye concentration, percentage decolorization has showed a remarkable dependence on the dosage of ferrous sulphate. Results indicate that the percentage decolorization increased as the ferrous sulphate dosage was increased from 80 mg/l to 140 mg/l. Decolorization is low at small concentration of ferrous sulphate due to lowest hydroxyl radical production available for oxidation [3]. It is also noticed from the Figure-3, that the percentage of decolorization was decreased with the increase of ferrous sulphate in the range of 140 mg/l to 180 mg/l. This reduction in percentage decolorization could be due to the recombination of hydroxyl radical with the over dosage of ferrous sulphate [32] as shown in the eq (2).

$$^{*}OH + Fe^{2+} \longrightarrow OH^{-} + Fe^{3+} \quad (2)$$

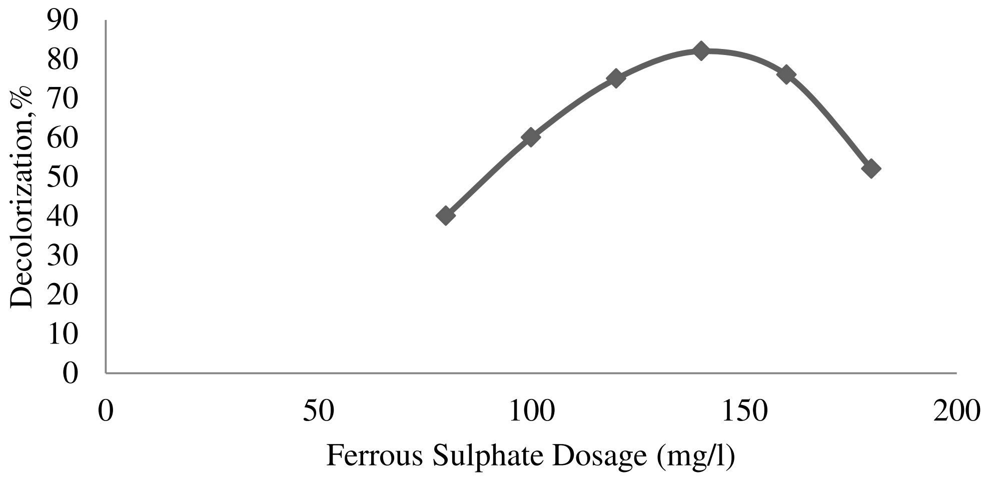

Figure-3. Effect of ferrous sulphate dosage on decolorization (Dye=120 mg/l; H_2O_2: 1000 mg/l; pH=2).

Table-1. Fenton process operating parameters from literature for greater than 90 % of colour removal.

Name of the Dye	**Dye concentration (mg/L)**	**Optimum H_2O_2 Dosage (mg/L)**	**Optimum Ferrous Sulphate Dosage (mg/L)**	**Optimum pH**	**Reference**
Reactive Black 5 (RB5)	100	400	100	3	[3]
Reactive Black 5 (RB5)	200	1000	225	3	[3]
Composite Reactive Dye (Remazol Black 5(RB5)+Remazol Red RB(RR)+Remazol Yellow 84(RY)+Remazol Brilliant Blue(RB))	300	1000	500	4	[40]

It is evident from Table-1, that operating parameters of Fenton process for removing reactive dye is not uniform. Based on the recent literature survey it was found that many successful tests with Fenton reaction were performed on aqueous solutions containing reactive dye alone. However, effluents from reactive dyeing industry contain dyes, detergents and salts NaCl, or Na_2SO_4.

In the presence of excess chloride ion, chloride ion radicals are produced due to the reaction of HO• radicals with Cl^- ions. Chloride ion radicals possess much lower oxidative potential than HO• radicals [23]. For the decolorization of industrial reactive dye effluents, therefore, greater dose of reagents is needed compared to aqueous solutions containing reactive dye only.

Lime Flocculation for Pre-Oxidized Reactive Dye Wastewater

According to the above optimized pre-oxidation conditions (H_2O_2: 1000 mg/l, $FeSO_4$: 140 mg/l and reaction period: one hour), lime flocculation for pre-oxidized dyeing effluents were designed to compare the process removal efficiency under different conditions. As shown in Table-2, twenty eight different process conditions were employed to reduce the H_2O_2 requirement for 98% decolorization, and to improve the effluent quality in terms of pH.

Table-2. Description of different process conditions.

Process description	H_2O_2 (mg/l)	$FeSO_4$ (mg/l)	Pre-oxidation period after which lime added (min.)	Lime (mg/l)	Decolorization (%)	Effluent pH
1	1000	140	60	0	98	2.4
2	1000	140	30	50	99	7.6
3	1000	140	30	100	99.2	8.9
4	1000	140	30	150	92	10.4
5	600	140	60	0	94	2.7
6	600	140	60	50	99	7.2
7	600	140	60	100	99	8.4
8	600	140	60	150	94	10.2
9	600	140	30	0	80	2.2
10	600	140	30	50	96	7.8
11	600	140	30	100	97	8.6
12	600	140	30	150	92	10.4
13	300	140	60	0	80	2.3
14	300	140	60	50	90	7.2
15	300	140	60	100	94	8.6
16	300	140	60	150	90	10.6
17	300	140	30	0	64	2.3
18	300	140	30	50	80	7.5
19	300	140	30	100	85	8.7
20	300	140	30	150	83	10.6
21	150	140	60	0	20	2.4
22	150	140	60	50	40	7.6
23	150	140	60	100	50	8.3
24	150	140	60	150	42	10.2
25	150	140	30	0	12	2.5
26	150	140	30	50	32	7.5
27	150	140	30	100	46	8.7
28	150	140	30	150	38	10.6

It is observed from Table-2, that lime requirements for decolorization very much depend on H_2O_2 dosage and pre-oxidation reaction period. With the process condition '6'(H_2O_2 = 600 mg/l; $FeSo_4$ = 120 mg/l; pre-oxidation reaction period = 60 min and lime = 50 mg/l) 99% of decolorization was achieved by saving 40% of the hydrogen peroxide requirement and effluent quality in terms of pH was improved from 2.7 to 7.2.

Plasticity Index of Clay-Sludge Mixture

The effect of moisture on the plastic behavior of the clay and sludge mixture was evaluated using the plasticity index. Non-plastic soil usually has a plasticity index (PI) value of 0-7, 7-17 for low plastic soil and more than 17 for high plastic soil [33]. The results from Atterbeg's studies on sludge and clay mixtures shown in Table-3, indicates that the PI value is increasing as the proportion of sludge in the mix increases [34]. The PI values shown in Table 3 indicate that up to 30 per cent of sludge can be used as a substitute for clay in brick making without losing plastic behavior.

Table-3. Effect of sludge proportion on Atterberg limits of clay – sludge mixture.

Sludge Proportion (%)	Liquid limit (%)	Plastic limit (%)	Plasticity index (PI)
0	48.20	28.40	19.80
10	50.24	32.00	18.24
20	53.36	37.00	16.36
30	56.34	41.00	15.34

Water Absorption of Bricks

Water absorption is a crucial factor influencing burnt clay brick longevity. The less infiltration of water into the burnt clay brick, greater resilience of the brick is expected and the resistance to the natural environment. As shown in Figure-4, the value of water absorption is increased from 6 % (0 % sludge) to 18 % (30 % sludge) with the increase of sludge proportion in the clay - sludge mixture. As seen in the previous section addition of sludge lowers the plastic nature of the mixture and also decreases the bonding ability of the mixture. Higher the amount of sludge, adhesiveness of the mixture decreases and the internal pore size of the brick increases. Similar trend was observed in a study conducted by Weng *et al*. [34], Mageed *et al*. [35] and Jahagirdar *et al*. [27]. According to IS: 1077-2007 [37], the water absorption of burnt clay bricks should not be more than 20% by weight up to class12.5 and 15 % by weight for higher classes.

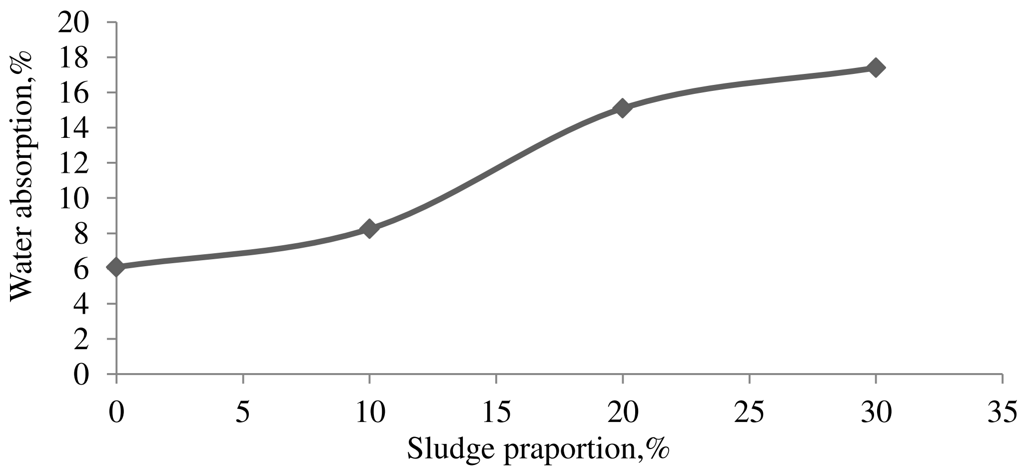

Figure-4. Effect of sludge proportion on water absorption of burnt clay bricks.

Compressive Strength of Bricks

The compressive strength test measures the functional performance of a building material. Figure-5 shows the results of the compressive strength test on the burnt clay bricks made from mixtures of clay and sludge. With the rise of the sludge proportion in the brick making mixture, compressive strength was decreased. These finding are closely matching to the research findings of Weng *et al*. [34], Mageed *et al*. [35] and Jahagirdar *et al*. [27] on sludge based burnt clay bricks. Jahagirdar *et al*. [27] reported that less silica content in sludge compare to base material is responsible for decrease in compressive strength of sludge based bricks. Addition of up to 20 wt. % sludge conformed to minimum compressive strength of 3.5 N/mm^2 according to IS 1077-1992 [36].

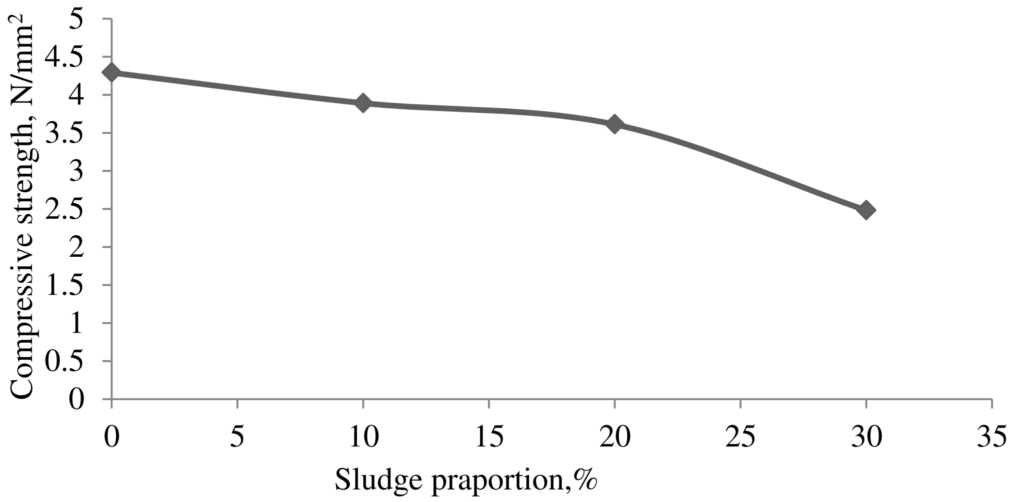

Figure-5. Effect of sludge proportion on compressive strength of burnt clay bricks.

TCLP Test of Bricks

Heavy metals such as lead (Pb), chromium (Cr), cadmium (Cd) and copper (Cu) are used extensively in the manufacture of textile dyes [37, 38]. Such heavy metals are highly toxic and can bio-accumulate when are released into water bodies [39].

Leaching tests were carried out using the 1311 method developed by the United States Environmental Protection Agency (USEPA) [29] to determine the degree of immobilization of heavy metals in the fired clay bricks made with sludge. As shown in Table-4, the concentrations of heavy metals were below the limits set by USEPA [29]. It can therefore be concluded that sludge produced from photo Fenton's coupled with the lime flocculation is suitable for the manufacture of fired clay bricks.

Table-4. TCLP test results of sludge based burnt clay bricks.

Elements (PPM)	Sludge proportion			US EPA Regulatory limits (PPM)[29]
	10%	20%	30%	
Chromium	0.430	0.478	0.556	5.0
Lead	0.060	0.185	0.189	5.0
Cadmium	0.002	0.004	0.005	1.0

CONCLUSIONS

The following conclusions are drawn based on this study:

a) Solar based photo Fenton process proved to be very successful in decolorizing simulated reactive dyeing wastewater.

b) The optimal conditions for solar based photo-Fenton process for achieving 98% of color removal from simulated reactive dye wastewater are pH=2, Ferrous Sulphate: 140 mg/L, H_2O_2=1000 mg/l and reaction period=60 min.

c) Bigger Fenton's reagent dose is needed for decolorizing reactive dye effluents containing auxiliary chemicals compare to aqueous solutions containing reactive dye alone.

d) Through coupling with lime flocculation, 40% of the H_2O_2 requirement of solar-based photo-Fenton process can be reduced to achieve 98% color removal from simulated reactive dyeing wastewater.

e) Lime requirement for pre-oxidized reactive dye wastewater depends very much on the dosage of H_2O_2 and the reaction time of pre-oxidation.

f) The pH of the effluent increased from 2.7 to 7.2 by combining photo-Fenton oxidation process with lime flocculation.

g) Making of burnt clay bricks with the chemical sludge produced from solar photo Fenton process coupled with lime flocculation is feasible, promising and environmentally appropriate.

h) Increasing the proportion of chemical sludge resulted in a decrease in compressive strength and an increase in water absorption for the burnt clay brocks.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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