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Sequential photo-Fenton and sono-photo-Fenton degradation studies of Reactive Black 5 (RB5)

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ABSTRACT

Sequential treatment studies have been done using photo-Fenton and sono-photo-Fenton, and compared with individual processes to analyze possible favorable effects of combined systems for the degradation of Reactive Black 5 (RB5). The coupled system enhances the degradation as compared to the ultrasound or UV–light irradiation processes along with the reduction in treatment time. Batch runs were carried out to investigate the process operational conditions: pH, H_2O_2 dosage, Fe^{2+} dosage, and RB5 concentration; to obtain the results at best possible operating conditions render advanced oxidation processe (AOP) competitive with other processes; and to ensure the rapid and complete transformation of the toxic organic compounds to benign chemicals. Degradation observed was 69% under Fenton and 93% under photo-Fenton with experimentally optimized conditions i.e. pH 4, Fe^{2+} concentration $0.050\,gL^{-1}$ and H_2O_2 $0.150\,gL^{-1}$ after 20 min. Sono-photo-Fenton treatment enhances the degradation up to 98% with experimentally optimized parameters within 12 min of reaction.

Keywords: Reactive Black 5; Dye; Photo-Fenton; Sono-photo-Fenton

1. Introduction

Synthetic dyes are useful in many industries such as textile [1,2], paper printing, food, pharmaceutical, leather, and cosmetics [3], and classified into acid, reactive, direct, basic, vat, disperse, metal complex, mordant, and sulphur dyes. There are more than 10,000 dyes used in textile industry, out of them 70% are azo dyes, which is complex in structure and synthetic in nature [4]. Azo dyes are synthetic organic dyes with basic structure Ar–N = N–Ar, where Ar is aromatic compound [5].

Reactive Black 5 (RB5), an azo-based chromophore, is widely used in many industries due to its bright color, excellent colorfastness, and ease of application [6]. It binds easily to textile fibre, such as cotton, through covalent bonds [7,8] and it is highly stable, soluble, highly toxic, and cheap. Conventional [9] and biological [10] methods alone are not enough for the effective removal of azo dyes, so finding an effective method for the degradation of azo dyes is required [11].

Advanced oxidation processes (AOPs) are advanced treatment processes, which are used to oxidize organic compounds that are resistant to the conventional methods of treatment. AOP's provide

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nonspecific oxidant [12] i.e. hydroxyl radical (OH*) which is highly reactive and have high electrochemical oxidation potential. Hydroxyl radicals attack organic compounds and cause chemical decomposition of given compounds by H-abstraction [13–17]. These radicals are produced by means of oxidants such as H_2O_2 and O_3, ultraviolet irradiation, ultrasound, and homogeneous or heterogeneous catalysts [18].

The technique of ultrasound when combined with other AOPs [19,20] would lead to faster degradation rates. The use of ultrasound has been recognized, for many years, in a wide variety of processes such as cleaning, drying, degassing, plastic welding filtration, biological cell disruption, and extraction for chemical reactions. So, the process of Sonication (i.e. the act of applying sound, usually ultrasound, energy to agitate particles in a sample, for various purposes) can also be an attractive treatment option [21].

The main objective of this study is to perform sequential studies of photo-Fenton and sono-photo-Fenton for visualizing best suitable treatment option for the degradation of RB5. A lot of studies have been reported on the degradation of RB5 in literature using various advanced techniques. To the best of our knowledge limited work has been done in the area of coupling both processes. This modification can be applied for existing advanced oxidation technologies so that drawbacks of individual technique can be overcome by sequencing so as to reduce the treatment time. In sequential studies, selecting AOP as pretreatment option can be justified by treating stable intermediates in the subsequent treatment i.e. sono-photo-Fenton and vice versa. Various literature studies depict the use of combined systems like physico-chemical [22], ozone-biological [23], photochemical-biological [24], photocatalysis [25], etc. for the treatment of dyes, but little attention has been paid on coupling ultrasound with other AOPs with sequential studies. Experiments were performed to analyze the feasibility of decolorization and degradation of RB5 by Fenton, photo-Fenton, Sono-Fenton, and Sono-photo-Fenton processes. The effects of solution pH, contact time, H_2O_2 dose, and Fe^{+2} dose and initial dye concentration on RB5 removal efficiency were studied.

2. Reagents and chemical analysis

RB5 (Fig. 1) was purchased from Sigma and used without further purification. Hydrogen peroxide and $FeSO_4{\cdot}7H_2O$ used in Fenton experiments, as well as all other chemicals were purchased from Merck. Distilled water was used throughout the investigations. The initial pH of the dye solution was 8.2; 0.1 N solutions of H_2SO_4 and NaOH were used for further adjustments in the initial pH. $Na_2S_2O_3$ (Sigma–Aldrich) was added into the sample to quench the oxidation before spectrophotometer analysis.

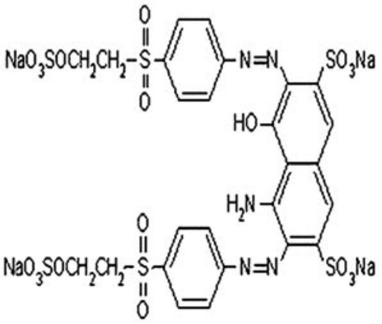

Fig. 1. Structure of Reactive Black 5.

COD of initial dye solution (160 mgL^{-1}) was determined by using Standard APHA method [26] (APHA 2220). UV Intensity was measured hourly during experimental days with Eppley (model No. 33013) radiometer. The degradation studies were performed with UV–visible Spectrophotometer (Hitachi V-500 UV/VIS (Japan)) double-beam spectrophotometer with RB5 having $\lambda_{\max}$ at 310 and 595 nm. The samples were also analyzed using HPLC [Shimadzu, SED-20A] for the confirmation of degradation.

3. Experimental conditions and equipment detail

Batch experiments were performed for Fenton (in the absence of both UV–visible irradiation and ultrasonicator), photo-Fenton (in the presence of UV–visible irradiation), sono-Fenton (ultrasonicator alone), and sono-photo-Fenton in both simultaneous and sequential manner to compare all the oxidation processes.

An immersion well photochemical reactor made of Pyrex glass equipped with a 125 W medium pressure mercury lamp was used in the study. Immersion well reactor comprised of three concentric cylinders, with water circulated jacket to prevent short wavelength UV radiation and IR radiations. For homogenous catalytic experiments, the pH of the 200 mL RB5 solution was adjusted to 4 to avoid iron precipitation. Appropriate amount of hydrogen peroxide was added to the solution followed by the addition of ferrous ions and the simultaneous start of ultraviolet and/or ultrasound irradiation. Sample was aerated continuously with flow rate of 4.5 Lh^{-1}.

4. Results and discussion

The oxidation processes are usually described in terms of pseudo-first order kinetics and half life time $t_{1/2}$. Initially rate constants were calculated by the slope of $-\ln(C/C_0)$ vs. t (min), where C and C_0 are the concentrations at time t and zero, respectively. Sono-Fenton and photo-Fenton processes showed better degradation rates than Fenton due to sonication and UV irradiation. Sono-photo-Fenton was found to be most efficient due to the presence of both sonication and UV radiation, simultaneously (Table 1).

Rate constants and $t_{1/2}$ were calculated for 2 min data only because degradation and decolorization capacity of RB5 increases invariably in the presence of both UV and US processes, simultaneously and individually [27]. This happens because sonication and UV both are important parameters that can directly influence the formation of OH* for the oxidation of RB5 solution.

4.1. Photo-Fenton studies

4.1.1. Effect of H_2O_2

H_2O_2 concentrations were varied from 0.088 to 0.4 gL^{-1} at constant Fe^{2+} concentration and the experiment was conducted for 30 min. Percentage removal of dye increased with the increasing dosage of H_2O_2 till 0.150 gL^{-1} after that removal of dye decreased with the increasing dosage of H_2O_2. This decrease is due to the fact of scavenging of $OH^{\cdot}$ radicals by H_2O_2 [28,29], equation presenting the condition:

$$OH^{\cdot} + H_2O_2 \rightarrow H_2O + HO_2^{\cdot}$$

$$HO_2^{\cdot} + OH^{\cdot} \rightarrow H_2O + O_2$$

Degradation and decolorization of dye solution increased with increasing time in the presence of H_2O_2, UV and Fe^{2+}. For RB5 concentration of 0.1 gL^{-1}, optimum dose of H_2O_2 came out to be 0.150 gL^{-1} (Fig. 2(a)).

Table 1
Value of k and $t_{1/2}$ for first 2 min at 310 and 595 nm

Experiments	Value of k (min^{-1}) at 310 and 595 nm		Value of $t_{1/2}$ (min) at 310 and 595 nm	
US	0.1501	0.0604	4.6218	11.5515
UV	0.1621	0.0723	4.2784	9.6254
US + Fe^{2+}	0.1264	0.5415	5.5005	1.2836
UV + Fe^{2+}	0.1382	0.0722	5.0224	9.6254
US + H_2O_2	0.1743	0.0727	3.9836	9.8252
UV + H_2O_2	0.1921	0.1501	3.6094	4.6205
Fenton	0.4225	2.0644	1.6513	0.3362
Sono-Fenton	0.4924	2.2201	1.4094	0.3133
Photo-Fenton	0.7561	2.3583	0.9173	0.2942
Sono-photo-Fenton	0.8765	2.5985	0.7917	0.2674

4.1.2. Effect of ferrous ions concentration

Ferrous ions dosage was varied in the range between 0.030 and 0.250 gL^{-1} at fixed H_2O_2 and pH. The amount of iron in dye solution is an important parameter influencing the oxidation processes. Iron acts as a catalyst and enhances the oxidation process. Hence with increasing iron dosage, free radical production increased, so dye removal efficiency also increased. Increasing iron dosage from 0.030 to 0.084 gL^{-1}, degradation and decolorization of RB5 dye increased and further addition of iron decreased the removal efficiency. It may be explained by redox reaction that $OH^{\cdot}$ scavenged either by the reaction with hydrogen peroxide or by the reaction with Fe^{2+} as mentioned in the equations [30]:

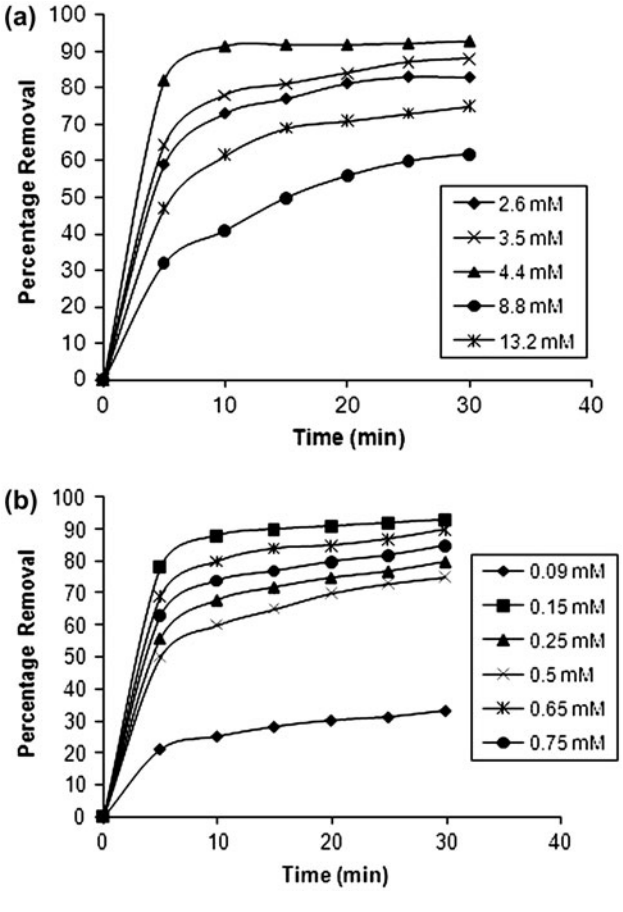

Fig. 2. % degradation of RB5 by photo-Fenton process. (a) Effect of variation of H_2O_2 dose, and (b) Effect of variation of Fe^{2+} (RB5 = 0.1 gL^{-1}; pH 4).

$$H_2O_2 + OH^{\cdot} \rightarrow H_2O + OH_2^{\cdot}$$

$$Fe^{+2} + OH^{\cdot} \rightarrow OH^{-} + Fe^{+3}$$

The maximum degradation and decolorization was observed at 0.050 gL^{-1} iron dosage in reaction time 30 min. The less degradation and decolorization was observed at small iron dosage which may be because of less production of OH´ for the oxidation process.

4.1.3. Effect of pH

The effect of pH was investigated on RB5 degradation and decolorization between 1 and 8 (Fig. 3(a)). The experiment was carried out for 30 min under controlled pH and suitable dose of Fe^{2+} (0.050 gL^{-1}) and H_2O_2 (0.150 gL^{-1}). At low pH (1–3), percentage removal of dye solution was not significant because reaction between hydrogen peroxide and ferrous ion was affected, which leads to reduction in the $OH^{\cdot}$ radical production [31]. Also percentage removal decreased with increasing pH i.e. 5–8 due to scavenging of H^+ ions [32]:

$$OH^{\cdot} + H^{+} + e^{-} \rightarrow H_2O$$

The degradation and decolorization of dye solution was similar at pH 3 and 4 i.e. 90–94% (which is operating pH for Fenton).

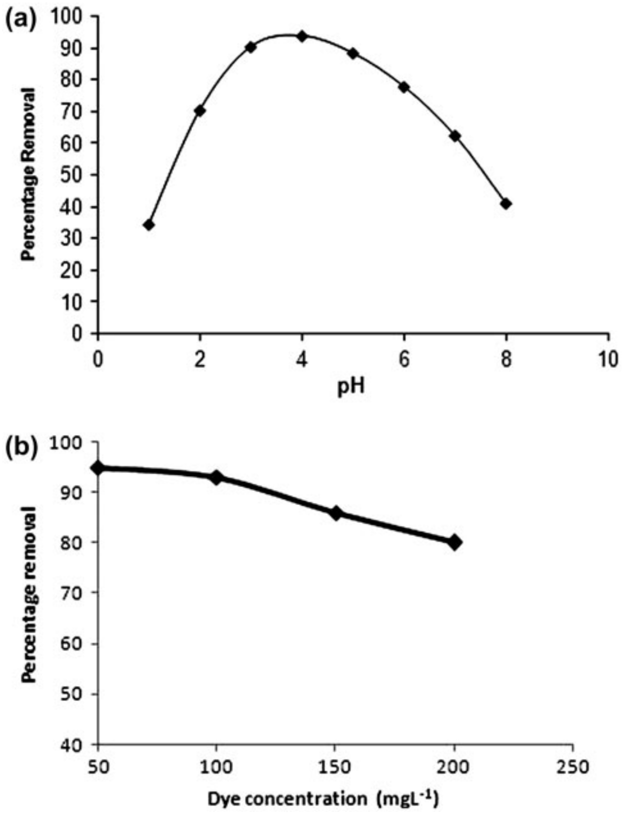

Fig. 3. Effect of (a) variation in pH, and (b) initial dye concentration on the degradation of RB5 by photo-Fenton processes. [RB5] = 0.1 gL^{-1}; [H_2O_2] = 0.150 gL^{-1} 4.4 mM; [$FeSO_4 \cdot 7H_2O$] = 0.050 gL^{-1}.

4.1.4. Effect of dye concentration

The degradation and decolorization of RB5 were studied by varying the dye concentration from 0.050 to 0.200 gL^{-1} at constant pH of 4, 0.150 gL^{-1} H_2O_2 and 0.050 gL^{-1} Fe^{2+}. It can be estimated from the Fig. 3(b) that with the increasing concentration of dye, degradation and decolorization decreases. It occurs due to the fact that; increase in dye concentration increases the number of dye molecule but not OH radicals. And this increasing number of dye molecule obstructs the penetration of photons entering into the solution, therefore less OH radical were produced [33]. UV–vis spectra of photo-Fenton process for RB5 solution with all optimized parameters (Fig. 4) clearly shows the vanishing of peaks in both UV and visible region.

4.2. Comparison between US/dye, US/dye/Fe^{2+}, and US/dye/H_2O_2

Some experiments were performed to study the effect of individual parameters along with the combinations i.e. US/dye, US/dye/Fe^{2+}, and US/dye/H_2O_2 with all optimized parameters (Fig. 5). Sonication is used to speed dissolution by breaking intermolecular interaction in RB5 solution. This occurs because

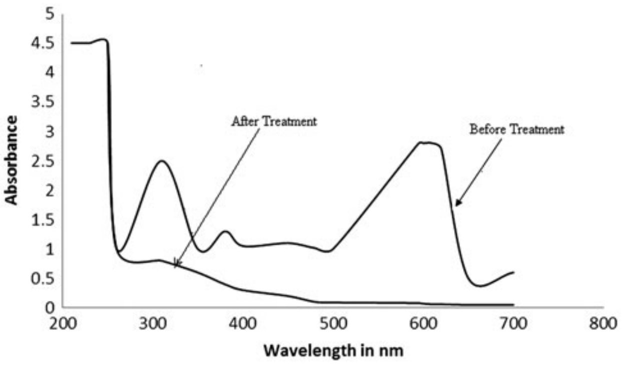

Fig. 4. UV-visible absorption spectra of RB5 solution during degradation with photo-Fenton process. [RB5] = 0.1 gL^{-1}; [H_2O_2] = 0.150 gL^{-1}; [$FeSO_4 \cdot 7H_2O$] = 0.050 gL^{-1}; pH 4.

sonication induces acoustic cavitation [34] the formation, growth, and collapse of bubbles in a RB5 solution. Distinct experiments were performed with reaction time of 240 min and maximum degradation was observed to be 14, 17, and 20%, respectively at pH 4.

4.3. Comparison between sono-Fenton, Fenton, photo-Fenton, and sono-photo-Fenton

Actually the percentage degradation and treatment time for any compound decides the economy of the process during the commercial applications. The ultrasonic wave influences the photo-Fenton and Fenton process by increasing the efficiency of degradation by inducing acoustic cavitation which further produces hydroxyl radicals [35,36]. Fig. 6 shows the comparison between sono-Fenton, Fenton, and sono-photo-Fenton. The percentage degradation increases up to 21% in case of photo-Fenton and 7% in case of photo-Fenton when combined with sonication.

Moreover, the treatment time was also reduced in conjunction with ultrasound i.e. sono-Fenton took 10 min less than dark Fenton for the same percentage of degradation and it was 12 min in case of sono-photo-Fenton. Thus from results, it can be concluded that sonication increases the degradation efficiency of photo Fenton and dark Fenton.

4.4. Sequential sono-Fenton and photo-Fenton processes

The effect of sequential sono-Fenton and photo-Fenton process on degradation and decolorization of RB5 solution was studied. The coupling system of sono-photo-Fenton was combined sequentially, applying first US than UV and vice versa at all optimized parameters (Fig. 7). Degradation and decolorization appeared to be more in UV–US process than US–UV process. This is due to the fact that photo-Fenton alone provides better result than sono-Fenton [37] and in sequential reaction; sonication provides the beneficial effects to photo-Fenton process to increase the degradation and decolorization efficiency. Moreover, the degradation time is drastically reduced in case of sequential treatment (UV–US) thus improves the economy of the process for field scale applications.

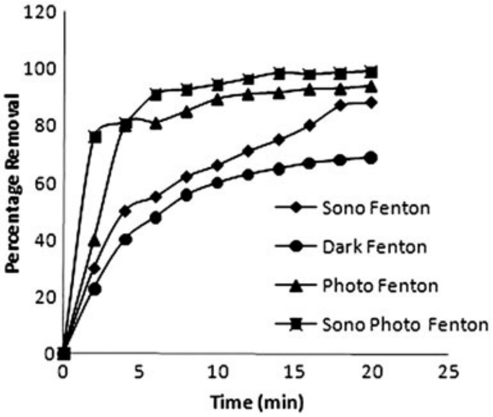

Fig. 6. Comparison of Fenton, sono-Fenton, photo-Fenton and sono-photo-Fenton. $[RB5] = 0.1\ gL^{-1}$; $[H_2O_2] = 0.150\ gL^{-1}$; $[FeSO_4{\cdot}7H_2O] = 0.050\ gL^{-1}$; pH 4.

4.5. Mineralization studies

Reduction in COD during the Fenton treatment was used to monitor the degree of mineralization with optimized conditions pH 4, $Fe^{2+} = 0.050\ gL^{-1}$, $H_2O_2 = 0.150\ gL^{-1}$, and $C_0 = 0.100\ gL^{-1}$. The RB5 dye under experimental conditions shows almost complete

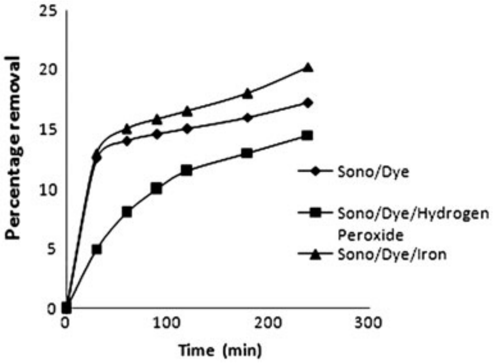

Fig. 5. % degradation of RB5 due to sonolytic [US], US + H_2O_2, US + Fe^{2+} $[RB5] = 0.1\ gL^{-1}$; $[H_2O_2] = 0.150\ gL^{-1}$; $[FeSO_4{\cdot}7H_2O] = 0.050\ gL^{-1}$; pH 4.

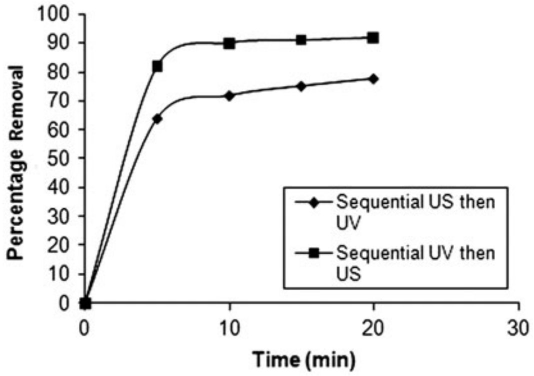

Fig. 7. Comparison of sequential US → UV process and UV → US process. $[RB5] = 0.1\ gL^{-1}$; $[H_2O_2] = 0.150\ gL^{-1}$; $[FeSO_4{\cdot}7H_2O] = 0.050\ gL^{-1}$; pH 4.

decolorization by the end of 20 min, whereas complete mineralization took 30 min as confirmed by 98% reduction in COD [38].

5. Conclusion

- The efficiency of degradation varied in the following order; sono-photo-Fenton > photo-Fenton > UV to US process > US to UV process > sono-Fenton > Fenton, while dye was completely decolorized in all processes. Therefore, sono-photo-Fenton process is much more efficient than other processes to degrade the RB5 solution.
- The effective degradation of RB5 seems to be related to the benefits of ultrasound for the complete degradation of organic pollutants in textile wastewater with reduced degradation time. All the oxidation processes occurs at pH 4, $Fe^{2+} = 0.050\,gL^{-1}0$, $H_2O_2 = 0.150\,gL^{-1}$, and dye concentration = $0.1\,gL^{-1}$.
- The results of sono-photo-Fenton degradation of dye showed that it could be used as efficient and environmental friendly technique for the complete degradation of recalcitrant organic pollutants, which will increase the chances for the reuse of wastewater.

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