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Solar driven decolourisation of Reactive Yellow 14 by advanced oxidation processes in heterogeneous and homogeneous media

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Abstract

This paper evaluates the decolourisation of an azo dye Reactive Yellow 14 (RY14) by three advanced oxidation processes viz., solar/TiO_2, solar/H_2O_2 and solar/H_2O_2/Fe^{2+} (photo-Fenton). The results showed that all the three processes could be effectively used for the decolourisation. The study on the effects of various experimental parameters such as pH, dye concentration, light intensity on the solar decolourisation revealed that these parameters influenced the removal rate. The photodecolourisation efficiencies with solar irradiation are comparable to UV irradiation. It is found that these three solar processes are viable techniques for the decolourisation of RY14.

Keywords: Reactive Yellow 14; Advanced oxidation processes; Solar light; Decolourisation

1. Introduction

Industrial wastewater contains rich source of non-biodegradable contaminants [1]. In recent years advanced oxidation processes (AOPs) are effectively used to detoxify noxious and recalcitrant pollutants in industrial wastewater. The potentialities offered by AOPs can be utilized to integrate the biological treatments by an oxidative degradation of toxic or refractory substances entering or leaving the biological stage. AOPs generate powerful reactive species hydroxyl radical (·OH). These ·OH radicals attack rapidly and non-selectively the most organic molecules. The versatility of AOPs is also enhanced by the fact that they offer different possible ways for hydroxyl radical production and thus allowing a better compliance with the specific treatment requirements.

The advanced oxidation processes can make use of solar energy instead of artificial light sources. The artificial light sources need high electrical power, which is costly and hazardous. Furthermore, solar energy is an abundant natural energy source in tropical countries like India.

Among the advanced oxidation processes the homogeneous AOPs employing H_2O_2 [2–5] and H_2O_2/Fe^{2+} [6,7] and heterogeneous AOPs employing semiconductor materials as photocatalysts [8–12] have been found to be very effective in the degradation of pollutants. The reactive azo dyes are most important class of synthetic organic dyes used in the textile industries and are therefore common industrial pollutants. These dyes are found to be non-biodegradable.

In the present work we report the photodegradation of a reactive class mono azo dye Reactive Yellow 14 (RY14) by three AOPs using TiO_2, H_2O_2 and H_2O_2/Fe^{2+} with solar light. We have analysed the influence of various parameters on photodecolourisation to find out the optimum conditions for efficient colour removal.

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The structure and absorption maximum of Reactive Yellow 14 are shown in Table 1.

2. Experimental

2.1. Materials

The commercial azo dye Reactive Yellow 14 obtained from Colour Chem Pondicherry was used as such. A gift sample of TiO_2-P25 was obtained from Degussa (Germany). It has the particle size of 30 nm and BET specific surface area of 50 m^2/g. AnalaR grade reagents H_2O_2 (30% w/w) and $FeSO_4 \cdot 7H_2O$ (Merck) were used as received. The double distilled water was used to prepare experimental solutions. The natural pH of the aqueous dye solution is 5.5. The experimental solutions were adjusted to the decided pH by the addition of H_2SO_4 or NaOH.

2.2. Irradiation experiments

All photocatalytic and photochemical experiments were carried out under similar conditions on sunny days of 2002–2003 between 11 AM and 2 PM. An open borosilicate glass tube of 50 ml capacity 40 cm height and 20 mm diameter was used as the reaction vessel. The suspension was magnetically stirred in the dark for 30 min to attain adsorption–desorption equilibrium between dye and TiO_2. Irradiation was carried out in the open-air condition. Fifty milliliters of dye solution with TiO_2 was continuously aerated by a pump to provide oxygen and for the complete mixing of reaction solution. During the illumination time no volatility of the solvent was observed.

2.3. Procedure

At specific time intervals 2 ml of the sample was withdrawn and centrifuged to separate the catalyst. One milliliter of the centrifugate was diluted to 10 ml and its absorbance at 410 nm was measured. The absorbance at 410 nm ($n \rightarrow \pi^*$ transition of $-N{=}N-$ group) is due to the colour of the dye solution and it was used to monitor the decolourisation. For solar/H_2O_2 process a desired molar ratio of dye/H_2O_2 at pH 3 was freshly prepared before the experiments. For photo-Fenton process a desired molar ratio of dye/Fe^{2+}/H_2O_2 solution was freshly prepared from $FeSO_4 \cdot 7H_2O$ and the dye stock solution. The pH of the solutions was adjusted to 3.0 for solar/H_2O_2 and photo-Fenton processes. After irradiation the absorbance was measured immediately to avoid further reaction. The pH of the solutions after irradiation was adjusted to 5.5 before taking the concentration measurements.

2.4. Solar light intensity measurements

Solar light intensity was measured for every 30 min and the average light intensity over the duration of each experiment was calculated. The sensor was always set in the position of maximum intensity. The intensity of solar light was measured using LT Lutron LX-10/A Digital Lux meter. The solar intensity was 1100 × 100 Lux and it was nearly constant during the experiments. UV spectral analysis was done using Hitachi U-2001 spectrophotometer. The pH of the solution was measured by using HANNA phep (model H 198107) digital pH meter.

3. Results and discussion

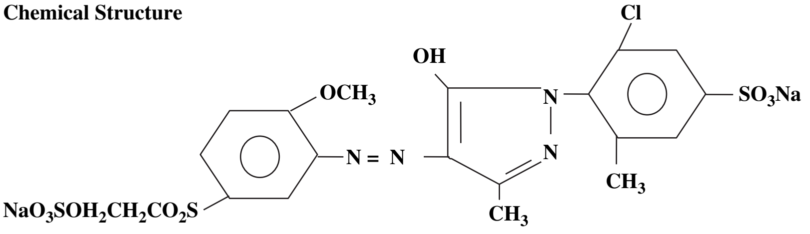
3.1. Solar/H_2O_2 process

The photodecolourisation of RY14 under different conditions is shown in Fig. 1. The dye does not decolourise on irradiation in the absence of H_2O_2. The dye solution with H_2O_2 in dark undergoes 12.7% decolourisation in 120 min. At the same time, irradiation with solar light and H_2O_2 gives 71.2% decolourisation. The decolourisation of dye is due to oxidation by hydroxyl radical (Eqs. (2) and (3)) formed in the solution

Table 1
The characteristic of Reactive Yellow 14 dye

Azo dye	Formula	Abs	M_w
Reactive Yellow 14	$C_{20}H_{19}N_4O_{11}S_3Na_2Cl$	410,254 nm	669.0

Chemical Structure



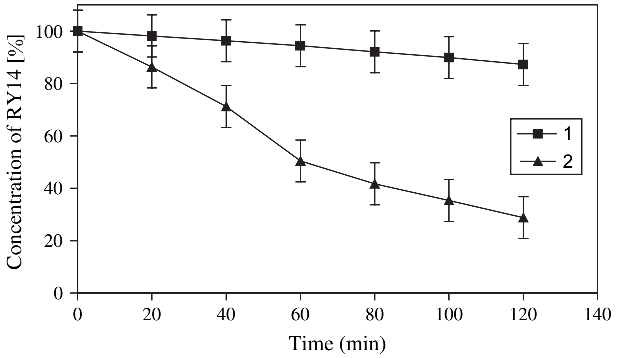


Fig. 1. Photochemical degradability of RY14 by solar/H_2O_2. $[H_2O_2] = 10$ mM, $[RY14] = 5 \times 10^{-4}$ mol/l, pH = 3.0 ± 0.1. 1. Dye solution with H_2O_2 in dark; 2. dye solution with solar light and H_2O_2.

according to Eq. (1). Xu [13] reported that direct photolysis of H_2O_2 by photons produces $^{\bullet}OH$ radical.

$$H_2O_2 + h\nu \rightarrow {}^{\bullet}OH + {}^{\bullet}OH \tag{1}$$

$${}^{\bullet}OH + \text{dye} \rightarrow \text{dye intermediate} \tag{2}$$

$${}^{\bullet}OH + \text{dye intermediate} \rightarrow CO_2 + H_2O + \text{Mineralisation products} \tag{3}$$

The effect of initial pH of dye solution on the decolourisation of RY14 was carried out in the pH range between 1 and 8. The results are shown in Fig. 2. It is found that the dye decolourisation is maximum at pH 3. Increase in the pH of dye solution from 1 to 3 increases the decolourisation from 13.5 to 51.0% in 60 min. Further increase in pH from 3 to 8 decreases the decolourisation rate. The difference in the decolourisation efficiencies is due to the variation in the hydrogen peroxide decomposition to $^{\bullet}OH$ radical. In the pH range 3–8 the factors which heavily influenced the decomposition are [15,16]:

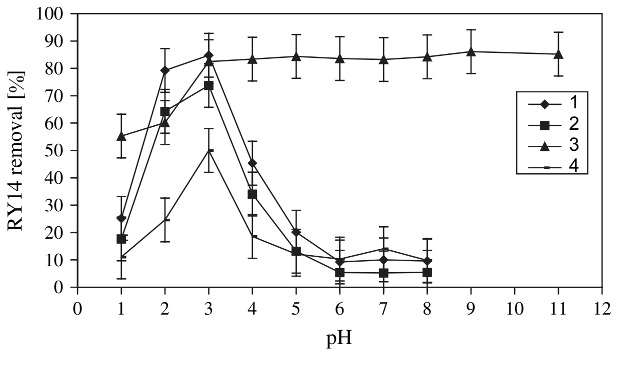


Fig. 2. Effect of pH on the decolourisation of RY14 under different conditions. 1. Photo-Fenton process: $[RY14] = 5 \times 10^{-4}$ mol/l, $[H_2O_2] = 10$ mM, $[Fe^{2+}] = 0.05$ mM, irradiation time = 30 min; 2. Fenton process: $[RY14] = 5 \times 10^{-4}$ mol/l, $[H_2O_2] = 10$ mM, $[Fe^{2+}] = 0.05$ mM, time = 30 min; 3. solar/TiO_2-P25: TiO_2-P25 = 4 g/l, $[RY14] = 5 \times 10^{-4}$ mol/l, irradiation time = 40 min; 4. solar/H_2O_2: $[H_2O_2] = 10$ mM, $[RY14] = 5 \times 10^{-4}$ mol/l, irradiation time = 60 min.

1. At this pH range H_2O_2 spontaneously decomposed into water and oxygen rather than producing hydroxyl radical (Eq. (4)).

$$2H_2O_2 \xrightarrow{h\nu} 2H_2O + O_2 \tag{4}$$

2. At pH above 3 the concentration of hydroperoxy anions (HO_2^- conjugate base of H_2O_2) increases. The hydroperoxy anions formed reduced the concentration of H_2O_2 and $^{\bullet}OH$ radical (Eqs. (5) and (6)).

$$H_2O_2 + HO_2^- \rightarrow H_2O + O_2 + {}^{\bullet}OH \tag{5}$$

$${}^{\bullet}OH + HO_2^- \rightarrow H_2O + O_2^{\bullet -} \tag{6}$$

The effect of the pollutant concentration is also an important parameter in photochemical degradation. The effect of various initial dye concentrations on the colour removal (Fig. 3) reveals that the increase in initial dye concentration decreases the removal rate. This decrease in decolourisation is due to the following reasons.

1. Increase in the initial dye concentration induces a rise in the internal optical density and the solution becomes more and more impermeable to UV radiation. This reduces the rate of photolysis of H_2O_2 by solar light radiation.
2. At constant H_2O_2 concentration and light intensity the hydroxyl radical concentration becomes

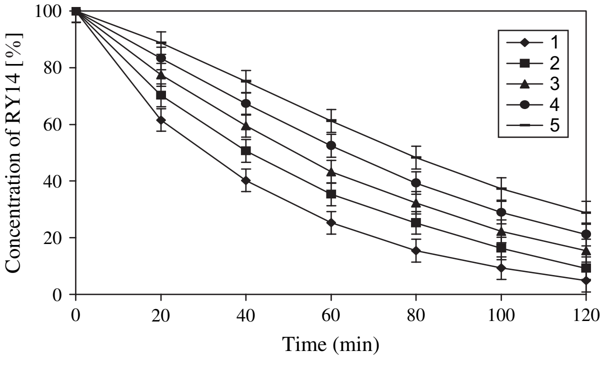


Fig. 3. Effect of various initial dye concentrations on the decolourisation of RY14 by solar/H_2O_2. $[H_2O_2] = 10$ mM, pH = 3.0 ± 0.1. 1, 1×10^{-4} mol/l; 2, 2×10^{-4} mol/l; 3, 3×10^{-4} mol/l; 4, 4×10^{-4} mol/l; 5, 5×10^{-4} mol/l.

Table 2
Pseudo first order rate constants of RY14 decolourisation by solar/H_2O_2

Concentration of dye ($\times 10^{-4}$ mol/l)	Decolourisation k (min^{-1})
1	0.024
2	0.017
3	0.012
4	0.009
5	0.006

constant for all dye concentrations. This limits the degradation reaction.

The kinetics of decolourisation of RY14 in solar/H_2O_2 process follows pseudo first order kinetics. Many authors had reported pseudo first order kinetics for UV/H_2O_2 process [5,14]. The calculated pseudo first order rate constant is given in Table 2. Increase in the initial dye concentration decreases the pseudo first order kinetics. At low H_2O_2 concentration formation of hydroxyl radical through photolysis of H_2O_2 is the rate determining step. When H_2O_2 concentration is in excess the decolourisation obeys the pseudo first order with respect to dye concentration (Eq. (7))

$$-\frac{\mathrm{d}c}{\mathrm{d}t} = K_1 C_{\mathrm{A}} C_{\cdot\mathrm{OH}} \tag{7}$$

At constant hydroxyl radical concentration $C_{\cdot\mathrm{OH}}$ should be a constant hence Eq. (7) is given as

$$-\frac{\mathrm{d}c}{\mathrm{d}t} = K_1 C_{\mathrm{A}} \tag{8}$$

K_1 is the pseudo first order rate constant at given time.

By integrating Eq. (8) with restriction $C = 0$, $t = 0$

$$\ln\left(\frac{C_0}{C}\right) = -kt \tag{9}$$

3.2. Fenton and photo-Fenton processes

The decolourisation of RY14 by Fenton and Photo-Fenton processes is shown in Fig. 4. At the time of 30 min colour removal was 73.7 and 80.3% in Fenton and photo-Fenton processes, respectively. The decolourisation of dye in Fenton process is due to the hydroxyl radical generated by reaction (10). The generated ferric ion in solution reacts with residual H_2O_2 (Eq. (11)) to produce hydroperoxy radical and ferrous ion. But hydroperoxy radical did not play important role in the decolourisation, hence Fe^{2+} ion is continuously generated from the reaction mixture (Eq. (12)). The decolourisation continues until hydrogen peroxide is completely consumed in the reaction.

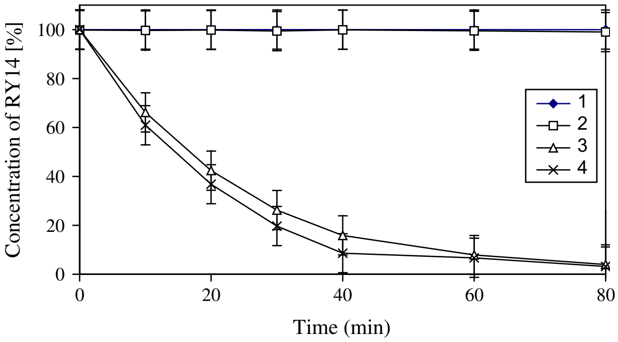


Fig. 4. Photochemical degradability of RY14 under different conditions. [RY14] = 5×10^{-4} mol/l, [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3 ± 0.1. 1. Solar/dye; 2. UV/dye/Fe^{2+}; 3. dye/H_2O_2/Fe^{2+}/dark (Fenton process); 4. Solar/dye/H_2O_2/Fe^{2+} (photo-Fenton process).

$$\mathrm{Fe}^{2+} + \mathrm{H_2O_2} \rightarrow {}^{\bullet}\mathrm{OH} + \mathrm{Fe}^{3+} + \mathrm{OH}^- \tag{10}$$

$$\mathrm{Fe}^{3+} + \mathrm{H_2O_2} \rightarrow \left[\mathrm{Fe}^{3+}\cdots\mathrm{OH_2}\right]^{2+} + \mathrm{H}^+ \tag{11}$$

$$\left[\mathrm{Fe}^{3+}\cdots\mathrm{OH_2}\right]^{2+} + \mathrm{H}^+ \rightarrow \mathrm{H\dot{O}_2} + \mathrm{Fe}^{2+} + \mathrm{H}^+ \tag{12}$$

The changes in the UV–visible spectrum of RY14 at different irradiation time by photo-Fenton process are displayed in Fig. 5. The enhancement in the photo-Fenton decolourisation is due to additional hydroxyl radical produced by the photoreduction of hydroxylate ferric ion to ferrous ion (Eq. (13)) and the direct photolysis of H_2O_2 (Eq. (1)).

$$\mathrm{Fe(OH)}^{3+} + \mathrm{UV} \rightarrow {}^{\bullet}\mathrm{OH} + \mathrm{Fe}^{2+} \tag{13}$$

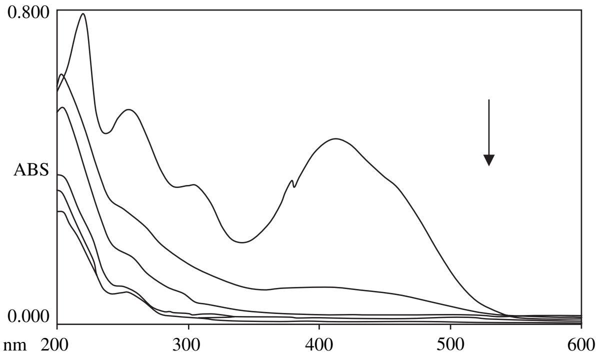


Fig. 5. The changes in UV–visible spectrum on irradiation by means of solar light of an aqueous Fenton's reagent containing RY14. [RY14] = 5×10^{-4} mol/l, [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3 ± 0.1.

However, the difference in the decolourisation between these two processes is only 6.6% at 30 min at constant Fe^{2+}, H_2O_2 and dye concentrations. This indicates that even in photo-Fenton process the decolourisation occurs mainly due to Fenton reaction (Eq. (10)). The efficiency of these processes was strongly influenced by the solution pH. The results are shown in Fig. 2. Both processes are efficient at pH 3. The increase in pH decreases the decolourisation efficiencies. This decrease is due to the precipitation of Fe^{3+} as hydroxide, which affects the Fe^{2+} generation and also reduces the transmission of the solar light in photo-Fenton process. The effect of various initial dye concentrations on the decolourisation of RY14 by photo-Fenton process is shown in Fig. 6. Increase in the initial dye concentration decreases the decolourisation. At the time of 20 min $1-3 \times 10^{-4}$ mol/l dye concentration the maximum decolourisation was reached. In photo-Fenton process hydroxyl radical is mainly responsible for dye decolourisation and its concentration remains constant for all dye concentrations. The increase in dye concentration increases the number of dye molecule and not the hydroxyl radical concentration, hence the removal rate decreases.

3.3. Solar/TiO_2 process

The photocatalytic decolourisation of RY14 with solar light and TiO_2 is shown in Fig. 7. The dye is completely decolourised in 80 min. The changes in the UV–visible spectrum of RY14 at different irradiation time are displayed in Fig. 8. The decolourisation of the dye is mainly due to the oxidation of dye by hydroxyl radical and photogenerated hole. In solar/TiO_2 process TiO_2 utilizes UV part of the solar spectrum (wavelength short than 380 nm) to produce e^- and hole. The highly oxidative h^+ ($E^\circ = 2.7$ eV) directly reacts with the surface adsorbed dye molecule or indirectly oxidises the organic compounds via formation of ·OH radical.

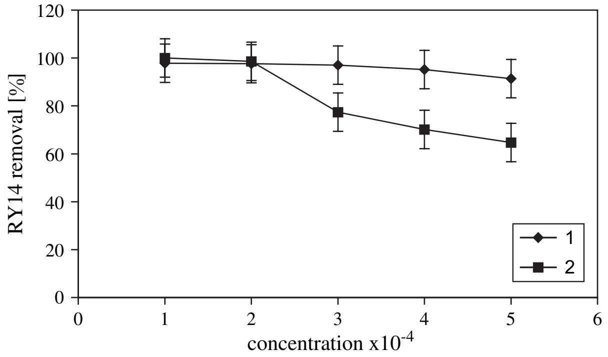


Fig. 6. Effect of various initial dye concentrations on the decolourisation of RY14 under different conditions. 1. Photo-Fenton process: $[H_2O_2] = 10$ mM, $[Fe^{2+}] = 0.05$ mM, pH = 3.0 ± 0, irradiation time = 30 min; 2. solar/TiO_2-P25: TiO_2-P25 = 4 g/l, pH = 5.5 ± 0.1, irradiation time = 40 min.

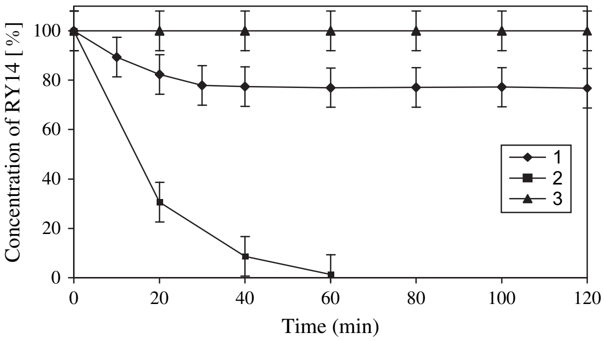


Fig. 7. Photodegradability of RY14 by UV/TiO_2-P25. TiO_2-P25 = 4 g/l, $[RY14] = 5 \times 10^{-4}$ mol/l, pH = 5.5 ± 0.1. 1. Dye solution treated with TiO_2-P25 in dark. 2. Dye solution irradiated with UV light in the absence of TiO_2-P25. 3. Dye solution irradiated with UV light in the presence of TiO_2-P25.

The effect of pH on the photodecolourisation by solar/TiO_2 is shown in Fig. 2. The results indicate that the increase in pH of the solution from 1 to 9 increases the decolourisation from 20.2 to 82.1% at the time of 40 min. This can be reasonably explained by the presence of sulphonic and ethyl sulphonic groups in the structure of RY14. The dye when hydrolysed gives these groups and hence the dye behaves as a negatively charged molecule. The surface charge property of TiO_2 changes with change in pH of the solution. The point of zero charge (P_{ZC}) for TiO_2-P25 is 6.8 [18]. In acidic medium the TiO_2 surface is positively charged. At acidic pH the electrostatic attraction between positively charged TiO_2 surface and negatively charged dye leads to strong adsorption. This makes the photocatalyst to change from white to yellow colour. The strong adsorption of dye in the TiO_2 surface blocks the active site of the catalyst and decreases the catalytic activity. Similar observation was reported by Poulios and Tsachpinis [19]. In alkaline medium the adsorption is less.

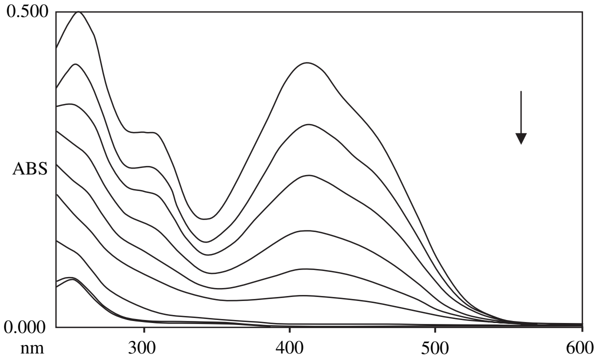


Fig. 8. The changes in UV–visible spectrum on irradiation by means of solar light of an aqueous suspension of TiO_2 containing RY14. TiO_2 = 4 g/l, $[RY14] = 5 \times 10^{-4}$ mol/l, pH = 5.5.

Furthermore the alkaline medium is also favorable for the formation of hydroxyl radical. Hence in alkaline medium more decolourisation was observed.

The effect of initial dye concentration on the decolourisation is shown in Fig. 6. Increase in the dye concentration from 1 to 5×10^{-4} mol/l decreases the decolourisation from 100 to 89.2% in 40 min. With the increase in initial dye concentration, more and more dye is adsorbed on the TiO_2 surface. This affects the light absorption capacity of the catalyst. The increase in dye concentration also decreases the path length of photon entering into the solution.

3.4. *Effect of solar light intensity*

Solar light intensity is an important parameter for the photocatalytic and photochemical processes. The efficiencies of these processes depend on the solar light intensity. The effect of solar light intensity on the decolourisation of RY14 was investigated at different times of the year 2002–2003. The results are shown in Fig. 9. Increase in solar light intensity from 700 to 1250 Lux increases the decolourisation from 73.2 to 91.2%, 85.3 to 93.4% (40 min) and 28.7 to 42.1% (60 min) for solar/TiO_2, solar/H_2O_2/Fe^{2+} and solar/H_2O_2 processes, respectively. The enhancement of removal rate is due to increase in hydroxyl radical production. Light intensity determines the amount of photons absorbed by the catalyst. With the increase in the solar power the catalyst absorbs more photons and this produces more hydroxyl radicals. In solar/H_2O_2 and solar/H_2O_2/Fe^{2+} processes the photolysis of H_2O_2 and photoreduction of Fe^{3+} depend directly on the incident light intensity. At low intensity the photolysis of H_2O_2 and photoreduction are limited. It appears that the solar power tested in our study lies in the linear range and hence all photons produced were effectively used.

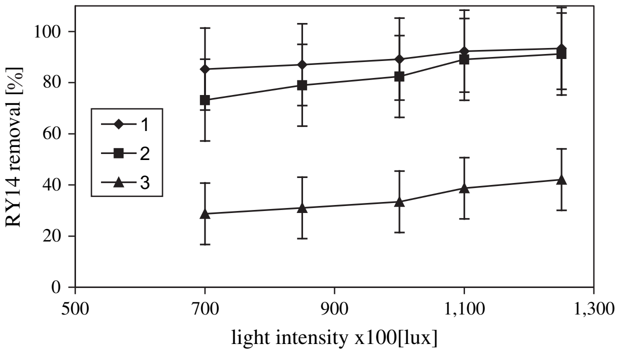


Fig. 9. Effect of various solar light intensity on the decolourisation of RY14 under different conditions. 1. Photo-Fenton process: [RY14] = 5×10^{-4} mol/l, [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3.0 ± 0, irradiation time = 40 min; 2. solar/TiO_2-P25: [RY14] = 5×10^{-4} mol/l, TiO_2-P25 = 4 g/l, pH = 5.5 ± 0.1, irradiation time = 40 min; 3. solar/H_2O_2: [RY14] = 5×10^{-4} mol/l, [H_2O_2] = 10 mM, pH = 3.0 ± 0, irradiation time = 40 min.

3.5. *Comparison of solar irradiation with UV irradiation*

The photocatalyic and photochemical decolourisation of RY14 have also been carried out in the presence of UV light (λmax = 365 nm). The results are compared with solar light irradiation in Fig. 10. The decolourisation efficiencies of RY14 in the presence of UV irradiation are comparable to solar irradiation. At the time of 40 min 91.3 and 89.2% of decolourisation were observed in UV/TiO_2 and solar/TiO_2 processes, respectively. This small difference in the rate of decolourisation is due to difference in the input energy. In solar process TiO_2 utilizes the near-UV part of the solar spectrum (wavelength shorter than 380 nm), whereas in UV process 365 nm is used. The irradiation wavelengths are close and so the removal efficiencies are not much different. In UV/H_2O_2 and solar/H_2O_2 processes 34.2 and 24.9% of decolourisation were observed, respectively. The H_2O_2 photolysis by UV irradiation is more. In solar light only 5% of UV irradiation is used in the photolysis hence UV process is more efficient than solar process. In photo-Fenton process the efficiencies of UV and solar radiations are close to each other. In both processes the decolourisation is mainly due to Fenton reaction hence the removal efficiencies are close to each other. In all the processes except H_2O_2 process the decolourisation is found to be nearly equal in both UV and solar irradiations. If the processes are carried out with concentrated sunlight using reflectors then solar processes will be more efficient. In tropical countries like India

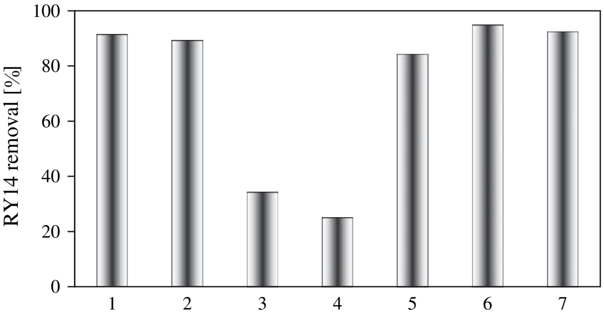


Fig. 10. Comparison of various processes on the decolourisation of RY14 under UV and solar light. 1. UV/TiO_2-P25: TiO_2-P25 = 4 g/l, pH = 5.5 ± 0.1, irradiation time = 40 min; 2. solar/TiO_2-P25: TiO_2-P25 = 4 g/l, pH = 5.5 ± 0.1, irradiation time = 40 min; 3. UV/H_2O_2: [H_2O_2] = 10 mM, pH = 3.0 ± 0, irradiation time = 60 min; 4. solar/H_2O_2: [H_2O_2] = 10 mM, pH = 3.0 ± 0, irradiation time = 60 min; 5. Fenton process: [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3.0 ± 0, irradiation time = 30 min; 6. UV/photo-Fenton process: [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3.0 ± 0, irradiation time = 30 min; 7. solar/photo-Fenton process: [H_2O_2] = 10 mM, [Fe^{2+}] = 0.05 mM, pH = 3.0 ± 0, irradiation time = 30 min.

intense sunlight is available throughout the year and hence it could be used instead of costly and hazardous UV light.

4. Conclusions

RY14 dye could be effectively decolourised by solar/TiO_2, solar/H_2O_2 and solar/H_2O_2/Fe^{2+} processes. Solar/H_2O_2 and solar/H_2O_2/Fe^{2+} processes are efficient at pH 3, whereas solar/TiO_2 process is efficient at pH 9. Increase in initial dye concentration decreases the removal rate in all the three processes. The kinetics of decolourisation follows pseudo first order in these processes. Increase in light intensity increases the removal efficiencies. In all the processes except H_2O_2 process the decolourisation efficiencies by UV and solar light are close to each other.

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