

Synthesis and Dyeing Behavior of two Remazol Reactive Dyes with Sulfo Vinyl Sulfone Functionality on Cotton Fabric and their Degradation Study

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ABSTRACT

The article presents synthesis of two new remazol reactive dyes (Orange D and Black BE) having sulfo vinyl sulfone functionality for dyeing cotton fabric. The reaction yield was greater than 98 % for both the dyes and their purity (≥ 99 %) was determined by HPLC on BDS Hypersil C18 (250 \times 4.6 mm, 5 μ) analytical column. As the dyes contain more reactive sulfonic acid groups, they display superior performance on dyeing cotton fabric compared to their reference dyes (Reactive Orange 2R and Reactive Black B) used commercially. Due to enhanced water solubility, exhaustion method was used for dyeing cotton fabric. The degree of primary and secondary exhaustion from the dyeing bath was 62.8 % and 84.3 % for Orange D and 64.1 % and 84.4 % for Black BE respectively. Similarly, the fixation ratio and fixation yields were 85.3 % and 71.2 % for Orange D and 88.1 % and 74.3 % for Black BE respectively. The photo catalytic degradation of both the reactive dyes was studied in aqueous solution using TiO₂-P25 as a photo catalyst under solar and UV irradiation. Further, different operational parameters like pH of initial solution, contact time and initial dye concentration were optimized for dye degradation

Keywords: *Remazol Reactive dyes; Cotton fabric; Exhaustion method; Photo catalytic degradation*

INTRODUCTION

Reactive dyes contain one or more reactive groups through which they make covalent bonds with the substrate (fibre). This class of dyes was first introduced commercially by Imperial Chemical Industries (ICI) in 1956, as they achieve extremely high wash fastness properties by relatively simple dyeing methods. The distinct advantage of reactive dyes over direct dyes is their simpler chemical structures, brighter dyeing and show narrow absorption bands in their absorption spectra. The principal chemical classes of reactive dyes include azo (including metallized azo), triphenioxazine, phthalocyanine, formazan, and anthraquinones [1]. They find extensive use in textile industry due to variety of bright colour shades, superior wet fastness, simple application procedures and other economic considerations [2]. The characteristic features of a typical reactive dye includes, i) the reactive system which enables the dye to form covalent bonds with the -OH group in the cellulose, with -NH₂, -OH and -SH groups in proteins fibres and with -NH₂ group in polyamides, ii) the chromophoric group(s) which contributes to the colour and the substantivity for the fibre, iii) a bridging group that links the

reactive system with the chromophore and iv) the solubilising groups [2, 3]. It is well known that application of reactive dyes on cellulose fibres is carried out under highly alkaline conditions. As a consequence, a competing hydrolysis reaction takes place which increases with increase in pH and temperature for dyeing. Dye that does not diffuse into the fibre reacts with the hydroxyl group in water and gets hydrolyzed. This leads to loss of dye and poses an environmental threat as it passes into the waste water [4, 5].

The major contribution towards pollution is from the dyeing and finishing processes. While dye-substrate affinity is critical, synthetic dyes cannot be commercialized unless they pose little health risk under end-use conditions. Consequently, environmental safety is an essential consideration in molecular design. The bottleneck with these dyestuffs is their non bio-degradable nature due to highly complicated structures, and therefore it is difficult to eliminate their presence from the industrial effluents which are released into the water bodies [6]. Significant efforts have been made to reduce the resulting level of water pollution through reverse osmosis, coagulation/flocculation,

and activated carbon absorption [7]. Degradation of dyes by anaerobic microorganisms has been attempted but this approach results in the formation of toxic and carcinogenic compounds due to biological degradation [8]. One promising approach for complete destruction of dye molecules is that of photocatalysis. This is an advanced oxidation process, used to mineralize dye compounds [9]. TiO_2 as photo catalyst is being widely used to develop processes for decreasing water pollution caused by dyeing compounds, as it requires mild conditions and can oxidize most of the organic contaminants to CO_2 and H_2O [10, 11].

In our previous work [12, 13], we synthesized three reactive dyes (Red A, Red B and Red C) and investigated their colour fastness on 100% cotton fabric and studied their possible photo degradation. In continuation of that study, we herein describe the synthesis of two highly water soluble remazol reactive dyes having more reactive groups and studied their dyeing ability on cotton fabric by the exhaustion process. Further, investigations were done to efficiently degrade these dyes by photocatalysis using TiO_2 .

EXPERIMENTAL

Chemicals and materials

Reference reactive remazol dyes, Orange 2R and Black B of technical grade were obtained from JCL (Ahmedabad, India), H-acid (1-amino-8-hydroxynaphthalene-3,6-disulfonic acid, 98 %) and sulfo vinyl sulfone (4-(β -ethylsulphate)sulphonylaniline, 99.2 %) were procured from Mayur Chemicals (Ahmedabad, India) and Atul Ltd. (Valsad, India) respectively. Cyanuric chloride (2,4,6-trichloro-1,3,5-triazine, 98.4 %) and sodium naphthionate (99.0 %) was purchased from IDI Ltd. (Ahmedabad, India). Sodium chloride (NaCl), potassium chloride (KCl), sodium carbonate (Na_2CO_3), sodium hydroxide (NaOH), hydrochloric acid (HCl), sulphuric acid (H_2SO_4), sodium lauryl benzene sulfonate, tri-sodium phosphate, ammonium perchlorate were of commercial grade. Analytical grade ethyl acetate, *n*-propanol and *tetra*-butyl ammonium bromide were obtained from Merck Specialties Pvt. Ltd. (Mumbai, India). TiO_2 powder P-25 (mainly in anatase form, mean particle size of 30 nm, BET surface area, 50 m^2/g) was used as photo catalyst. Commercial cotton fabrics (plain weave, 36's warp and 30's weft, yarn density 72/inch warp and 69/inch weft) were dried at about 100 °C for over 6 h after de-sizing with ammonium perchlorate, NaOH and sodium lauryl benzene sulfonate [14].

Instrumentation and conditions

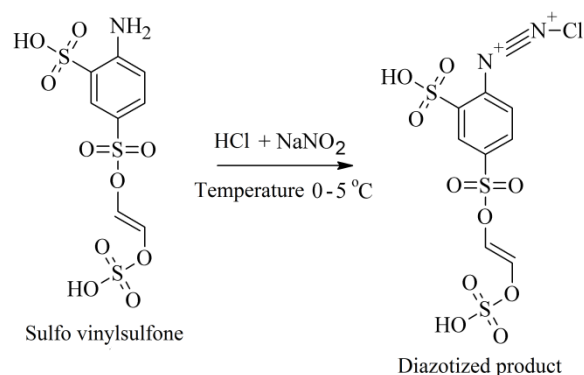
Sunlight intensity was measured using a digital lux meter from Sudershan Measuring & Engineering Pvt. Ltd., (New Delhi, India). A digital EH1000 pH meter from Line Seiki (Japan) was

used for pH measurements. A Varian Cary 400 UV-Visible Spectrophotometer (Palo Alto, CA, USA) with 10 mm matched quartz cells was used to carry out UV absorption studies. A Beckman DK-2A Double-beam Spectrophotometer (Maryland, USA) equipped with an Ulbricht sphere and powered by a hydrogen lamp was used to measure UV transmittance through the fabric samples. An SLI 440 dyeing machine (dye bath) manufactured by Sahas Laboratory Instruments (Ahmedabad, India) was used to dye the fabric samples. Thin layer chromatography (TLC) was performed on pre-coated silica gel 60 F254 plates from E. Merck (Darmstadt, Germany). Shimadzu Prominence UFLC with SPD-20A detector was used to determine purity using BDS Hypersil C18 (250 \times 4.6 mm, 5 μ particle size) analytical column (Tokyo, Japan). The mobile phase consisted of 1.5 mM *tetra*-butyl ammonium bromide in deionized water: acetonitrile (40: 60, v/v) and was delivered at a flow rate of 1.0 mL/min. The injection volume was kept at 20 μL .

Synthesis of Remazol Reactive dyes

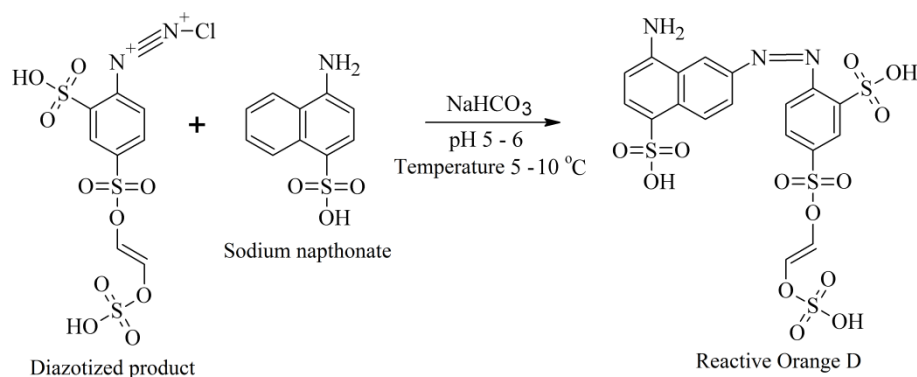
Reactive Orange D

Step I: A diazonium salt of sulfo vinyl sulfone was prepared by reacting with HCl and NaNO_2 at 0-5 °C based on standard method as illustrated in Scheme I [15].



Scheme 1 Synthesis of diazonium salt of sulfo vinyl sulfone

Step II: The diazonium salt of sulfo vinyl sulfone was added drop wise with stirring to a solution of 1.0 M sodium naphthionate, by maintaining pH 5-6 at a temperature of 5-10 °C. The process was monitored by TLC using a mixture of ethyl acetate, *n*-propanol and water (1:6:2, v/v/v) as the eluent. The dye was separated from the solution by salting out with 7.0 % NaCl and 9.0 % KCl (by volume), filtered, buffered to pH 7.0 \pm 0.2, and finally dried to obtain Reactive Orange D in 98.4 % yield (Scheme II).



Scheme II Synthesis of Remazol Reactive Orange D.

Reactive Black BE

Step I: This step was identical to the one described for Reactive Orange D.

Step II: To a 0.5 M alkaline solution of H-acid, prepared by dissolving 160.5 g in 250 mL water and adding 26.5 g sodium carbonate. 1.0 M solution of diazotised sulfo vinyl sulfone (at 5-10 °C, pH = 5-6 from trisodium phosphate) was then coupled with 0.5 M H-acid by maintaining pH 2-3 at 0-5 °C. The process was controlled by TLC using a mixture of ethyl acetate, *n*-propanol and water (1:6:2, v/v/v) as the eluent. The dye was separated from the solution by salting out with 7.0 % NaCl and 9.0 % KCl (by volume), filtered and then buffered to pH 7.0±0.2, and finally dried to fine powder. The yield of Reactive Black BE was 99.2 % (Scheme III).

Dye application on cotton fabric

Dyeing of cotton fabric was carried out using the method suggested previously [5, 12]. For the dyeing experiment, bleached cotton fabrics (with 10 × 4 cm dimension) were used. After washing in solution containing 2 mL/dm³ non-ionic detergent, the fabric samples were washed repeatedly in water and dried at room temperature. Prior to dyeing all fabric samples were wet in cold water. The dye bath contained 1.0 % dye together with 80 gm NaCl and 20 gm Na₂CO₃ per litre of solution. Due to high water solubility, it was possible to carry out application processes with a liquor ratio of 1:5 of NaCl per L of dye bath as compared to 1:8/10 ratio used commercially. The dyed samples were dried at room temperature before measurement.

Determination of dye exhaustion, dye fixation and fixation yield

A detailed description on primary and secondary dye exhaustion, dye fixation ratio and fixation yield was reported in our previous work [12]. These parameters were evaluated and compared with reference reactive dyes under identical conditions. Remazol Reactive Orange 2R and Remazol Reactive Black B were used as comparison standards for Reactive Orange D and

Reactive Black BE respectively. The percentage of dye bath exhaustion was calculated by the equation,

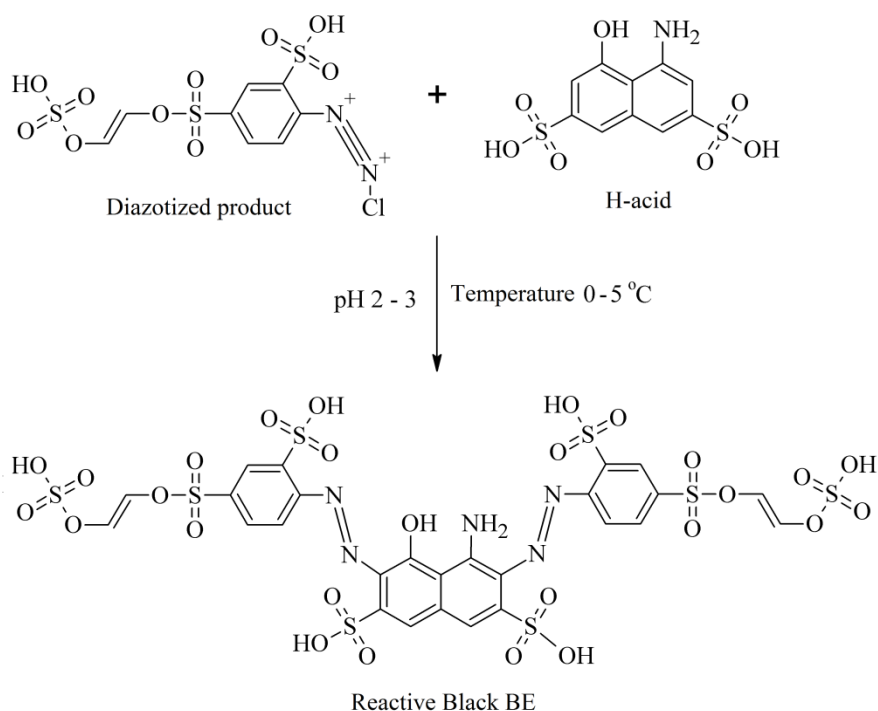
% E = (C₁-C₂/C₁) × 100, where, 'E' is the degree of dye exhaustion from the dye bath, in percentage; C₁ and C₂ are the concentration of the dye before and after dyeing, respectively.

Fixation ratio is defined as the percentage of the dye chemically bound to cellulose at the end of the dyeing process, relative to secondary exhaustion. Fixation yield is the percentage of dye chemically bound to cellulose at the end of the dyeing process, relative to the amount of dye used for the experiment. The following equation was used to calculate the dye fixation ratio for both the dyes,

T = (C₁/C₂) × 100, where, T is the fixation ratio in percentage; C₁ and C₂ are the concentration of the dye before and after extraction, respectively. The fixation yield for dyeing was calculated using the formula, F = T × E/100, where F is the fixation yield in percentage.

Dye degradation study

Degradation studies were carried out by exposing 500 mL of 10 mg/L dye solution in presence of 0.25 g of TiO₂ under direct sunlight for 4 h. No provision for additional aeration was made as stirring was enough to provide sufficient air (oxygen) in the reaction mixture. Suitable aliquots of solution were withdrawn and analyzed for its dye concentration at pre-determined intervals by spectrophotometry to evaluate the effect of contact time. The effect of pH on dye degradation was also evaluated by equilibrating the solution at different initial pH of 5.0, 7.0 and 9.0 respectively. This was achieved by addition of 0.1M HCl or 0.1M NaOH solutions. Two different light sources were used to study the effect of light source viz. sunlight and UV irradiation (using a high pressure mercury lamp). Regeneration of TiO₂ was attained by chemical itching (rinsing with conc. H₂SO₄). The photo degradation experiments were repeated four times consecutively to check the reusability of regenerated TiO₂.



Scheme III Synthesis of Remazol Reactive Black BE.

RESULTS AND DISCUSSION

Reactive Orange D and Reactive Black BE, which are the derivatives of 1-amine-8-hydroxynaphthalene-3,6-disulphonic acid were synthesized having more reactive groups compared to commercial Orange 2R and Black B dyes used as reference. Both the dyes were synthesized by reacting diazotized sulfo vinyl sulfone with either sodium naphthionate or H-acid to give Reactive Orange D or Black BE respectively. Due to the presence of an additional sulphonic acid group better color yield was displayed and both the dyes were highly water soluble. The absorption spectra for Orange D and Black BE in deionized water (0.004 %) gave wavelength of maximum absorption (λ_{\max}) at 520 nm and 630 nm respectively as reported previously [16]. Figure 1 shows the chromatograms of the dyes taken on BDS Hypersil C18 column using 1.5 mM *tetra*-butyl ammonium bromide in deionized water: acetonitrile (40: 60, v/v) as the mobile phase. The

purity was $\geq 99\%$ for both the dyes as ascertained from HPLC measurements.

Dyeing efficiency

Maintaining a maximum temperature of 80 °C for 90 min during time course for dyeing was found most suitable for maximum efficiency. On account of low liquor ratio in the dye bath, 1:5 of NaCl per L the salinity of the effluent decreased considerably. Both the dyes had strong affinity towards the cellulose fibers possibly due to the linear structure of the dyes. The degree of dye exhaustion from the dye bath depends mainly on type of reactive groups. The results obtained substantiate that the dyes are quantitatively bound with the cellulose fibers. The synthesized dyes gave superior fixation ratio and yield compared to the reference standards (Orange 2R and Black B) used commercially (Table 1).

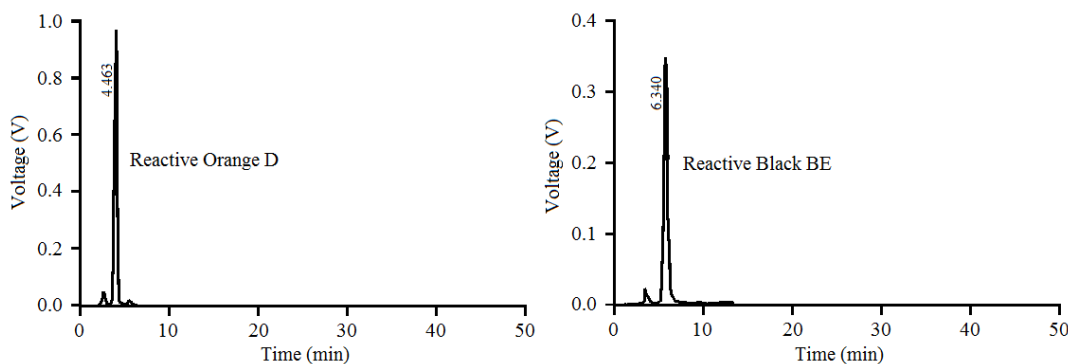


Figure 1 HPLC chromatograms of synthesized Reactive Orange D and Reactive Black BE.

Table 1 Degree of dye exhaustion from the dye bath, dye fixation ratio and fixation yield

Dye	Degree of exhaustion (%)		Fixation ratio (%)	Fixation yield (%)
	Primary exhaustion	Secondary exhaustion		
Orange 2R	54.6	75.1	76.1	57.1
Orange D	62.8	84.3	85.3	71.2
Black B	57.1	78.2	79.3	62.0
Black BE	64.1	84.4	88.1	74.3

Comparison of colour quality and strength for dyeing cotton fabric

To evaluate the colour difference and strength, a comparative study with the synthesized dyes and their commercial counterparts (Orange 2R and Black B) was carried out. Figure 2 shows a representative sample of cotton fabric dyed with both the reactive dyes. The synthesized dyes had brighter shade and colour depth. The results show 8 % and 43 % higher colour value and strength respectively for Orange D and Black BE compared to Orange 2R and Black B. This outcome can significantly reduce the consumption of dyes and hence the cost for dyeing process.

Effect of light source on photo degradation of dyes

To find the effect of light source for the degradation/removal of dyes, two light sources were used. The results indicate that the decolorization efficiency of dye with solar light irradiation was around 83-85 %, and 50-60 % with artificial UV light irradiation (Figure 3). The higher degradation under solar light can be attributed to higher temperature of the dye solution attained under

solar irradiation [17]. A similar observation has been reported in the process of removal of methylene blue whereby the colour removal rate with solar light irradiation was almost twice that of artificial UV light irradiation [18]. Ong and co-workers [17] carried out a similar study for the degradation of Congo Red and Reactive Yellow 2 and observed higher degradation under sunlight irradiation.

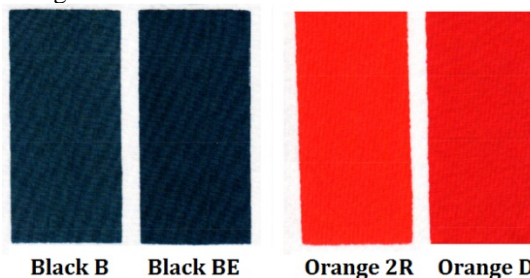


Figure 2 Comparative assessments of colour difference and strength for dyed cellulose fabric.

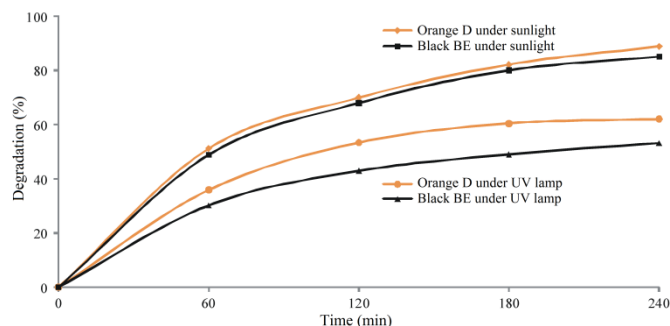


Figure 3 Effect of light source on the photo degradation of Orange D (5.0 mg/L) and Black BE (5.0 mg/L).

Effect of pH on photo degradation

For the photo degradation of dye under sunlight/TiO₂ system it is essential to study the effect of pH on dye removal. The impact of pH on the efficiency of photo degradation process can be understood from the ionization state of the surface of TiO₂. The point of zero charge (PZC) of TiO₂ is reported as 6.9 and as a result, the TiO₂ surface will be positively charged when pH < 6.9 and negatively charged when pH > 6.9 [19]. Since both the dyes are ionic in nature, under acidic conditions, when pH is less than pH_{PZC}, the surface of TiO₂ will be positively charged and dye molecules will be readily absorbed by the surface. Under alkaline conditions, the number of negatively charged surface sites on the TiO₂ would be more and therefore the photo degradation efficiency will be less due to electrostatic repulsion. The results obtained show 80-85 % dye degradation at pH 5.0 compared to ~ 45 % at pH 9.0 under sunlight after 4h (Figure 4).

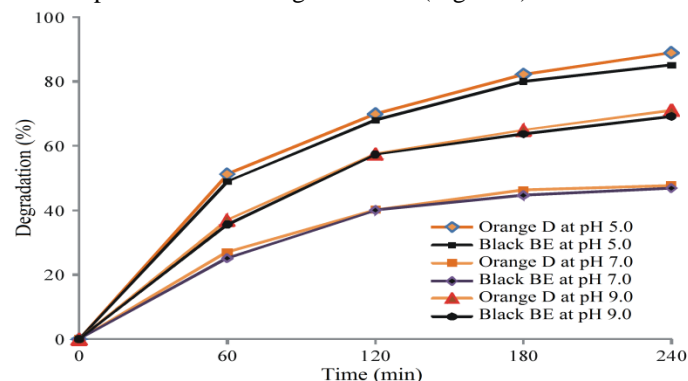


Figure 4 Effect of pH on degradation of Orange D (5 mg/L) and Black BE (5 mg/L) under sunlight.

Effect of contact time and initial dye concentration

The result of contact time and initial dye concentration for the removal of dye are represented by Figure 5. The percentage removal of dyes was concentration dependent, a decrease in the percent of removal in both the dyes was observed on increasing the concentration of dye solution from 5.0 to 9.0 %. This is because more dye molecules are adsorbed on the surface of TiO₂ which inhibits photo degradation as it stops photons from activating TiO₂, thereby reducing the generation of hydroxyl radicals on the surface of TiO₂. Further, with a decrease in the formation of hydroxyl radicals on the surface of TiO₂, there is a corresponding decrease in the relative number of hydroxyl radicals attacking the compound, resulting in reduced degradation. The adverse effect of higher initial concentration towards photo degradation has been reported previously [20, 21]. Additionally, there was a gradual increase in dye degradation with increase in contact time from 1 to 4 h.

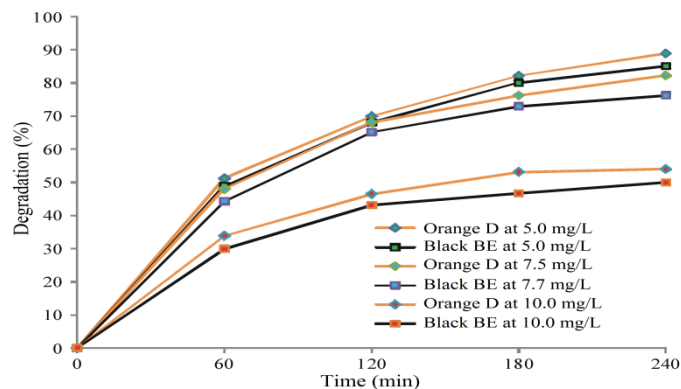


Figure 5 Effect of contact time and initial concentration of Orange D and Black BE dyes under sunlight at pH 5.

Effect on reusability of TiO₂

For repetitive use of TiO₂ catalyst, it is essential to verify the regeneration efficiency for practical purposes. A study was conducted to check the efficiency of TiO₂ on multiple usages. The results for dye degradation after first and fourth use with the same catalyst and same initial dye concentration are illustrated in Figure 6. The extent of dye degradation decreased from 88.9 % to 48.4 % for Orange D and from 85.02% to 42.9 % for Black BE after fourth use at 4.0 h due to the decrease in photo-catalytic activity of TiO₂ after repetitive use. This can be due to the presence of adsorbed dye molecules on the TiO₂ surface, which was overcome by regenerating the TiO₂ surface with aggressive chemical itching using conc. H₂SO₄. Such regeneration process recovered ~ 85 % of the initial activity.

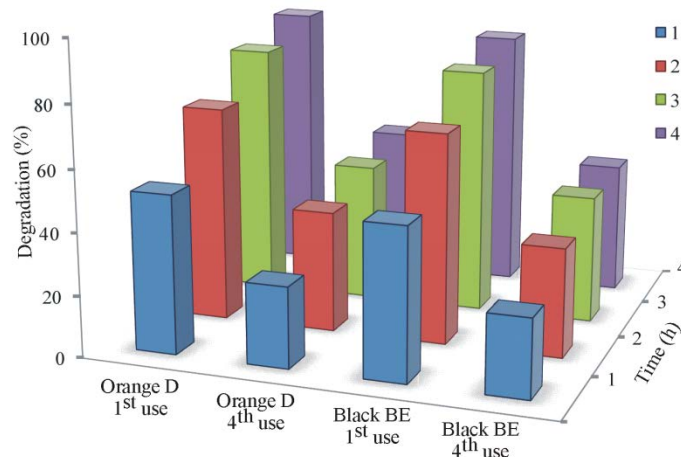


Figure 6 Efficiency of TiO₂ on repetitive use for dye degradation.

CONCLUSION

In summary, the synthesized reactive dyes display superior dyeing properties on cellulose fiber compared to their respective commercial dyes. This was possible due more reactive groups and enhanced water solubility. Other merits of these dyes include higher primary and secondary dye exhaustion, greater fixation yields and superior colour strength. Further, the possibility of dye degradation using the inexhaustible and economical solar light and TiO₂ as photo catalyst in an aqueous medium is also investigated. From the results it is apparent that TiO₂ displays superior performance in degrading anionic dyes under acidic medium.

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