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# Solar Photocatalytic Decolorization of Four Commercial Dyes in Aqueous Solution of Two Forms of Titanium Dioxide $TiO_2$

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## ABSTRACT

The photocatalytic decolorization of four commercial dyes with different structures and different substitute groups has been investigated using pure $TiO_2$ and $TiO_2$ sol-gel as photocatalysts in aqueous solution under solar radiation. Influence of different kinds of dye and photocatalyst on photo-catalytic process was studied. Solution temperature and daily photon flux during experiment was calculated. The result decolorization percentage of four dyes was in the following order: reactive yellow > methylene blue > tubantin blue > tubantin red; this is due to molecular structural difference among these four molecules of dyes. High decolorization when used $TiO_2$ sol-gel due to small particle size, small surface area and wide energy gab. The daily photon flux average was nearly constant during the experiments, owing to its large surface area pure $TiO_2$ caused higher average solution temperature than caused by $TiO_2$ sol-gel when they are used as potocatalytic decolorizers.

المستخلص
تتم التحقيق من التحفيز الضوئي لإزالة لون أربعه أصباغ تجاريه ذات تراكيب مختلفة تحتوي علي مستبدلات مختلفة باستخدام محلول مائي لثاني اوكسيد التيتانيوم النقي وثاني اوكسيد التيتانيوم محضر عن طريق سول-جل كمحفزات ضوئية تحت أشعه الشمس . تمت دراسة تأثير اختلاف نوع الصبغة ونوع المحفز الضوئي على عمليه التحفيز الضوئي. ايضا تم حساب درجه حراره المحلول وتدفق الفوتونات أثناء التجربة. ترتيب النسبة المئوية لإزالة لون الاربعه أصباغ كالأتي: الصبغة الصفراء النشطة اكبر من الميثيل الأزرق اكبر من صبغة التيوبيتان الزرقاء اكبر من صبغة التيوبيتان الحمراء يرجع ذلك للاختلاف في التركيبة الجزيئية بين الأصباغ الاربعه . وضحت النتائج أن اعلى نسبه ازالة الون عند استخدام ثاني اوكسيد التيتانيوم سول-جل وذالك لصغر حجم جزئياته وصغر مساحة سطحه ومستوي طاقة واسع. ومتوسط التدفق الفوتونات اليومي تقريبا ثابت أثناء التجربة, زيادة متوسط درجة حرارة المحلول اعلى عند استخدام ثاني اوكسيد التيتانيوم النقي مقارنه بثاني اوكسيد التيتانيوم سو-جل وذلك لكبر مساحه سطح ثاني اوكسيد التيتانيوم النقي عند استخدامها كمحفزات لإزالة اللون .

***KEYWORD:*** *solar photo; commercial; dyes ; decolrization; pure titanium dioxide, titanium dioxide sol-gel; Photon flux.*



## INTRODUCTION

Synthetic dyes are chemical compounds having complex aromatic structures (Yang *et al*., 2011) which were used in textile, paper, rubber, plastic, leather, cosmetic, pharmaceutical, and food industries (Yao *et al*., 2009; Rafatullah *et al*., 2010; Hu et al., 2010; Sen *et al*., 2011; Charumathi and Das, 2012; Alvarenga *et al*., 2015). Their disposal into the environment and contamination of water is a major problem (Alvarenga *et al*., 2015). Biological, physical, chemical and physico-chemical methods such as coagulation and flocculation (Aleboyeh *et al*., 2008; Harrelkas et al., 2009; Zonoozi *et al*., 2009), membrane separation (Sachdeva and Kumar, 2009; Amini *et al*., 2011) sonochemical degradation (Abbasi and Asl, 2008), electrochemical (Gupta et al., 2007; Fan *et al*., 2008), activated carbon adsorption(Xu et al., 2008; Tan et al., 2008; Foo and Hameed, 2010), and adsorption (Crini, 2005; Crini and Badot, 2008) were used for removal of dyes. However, these methods have their drawbacks, amongst which, the generation of hazardous waste or secondary intermediates, slow degradation rates and high costs are limitations that stand out.

Advanced oxidation processes have been suggested as potential alternative method for decolorizing dyes (Neamtu *et al*., 2004; Domínguez *et al*., 2005; Hsing et al., 2007; Guimaraes *et al*., 2012) including ozonation (Muthukumar and Selvakumar, 2004), $H_2O_2$/UV(Muthukumar *et al*., 2005; Kasiri and Khataee, 2011), photo-fenton system (Parıltı and Akten, 2010; Karthikeyan *et al*., 2011; Weng *et al*., 2013) and photo-catalytic oxidation ($TiO_2$ /UV). These methods are based on the formation of $HO^{\cdot}$ radical and its destructive behavior on organic matter.

In Photo-catalysis systems when a semiconductor such as $TiO_2$ absorbs energy of photon equal to or greater than its band gap width which generated electron / hole pair ($e^-$/ $h^+$) owing to promotion of an electron ($e^-$) from the valence band to the conduction band. The photo-generated electrons can reduce the dye or react with electron acceptors such as $O_2$ adsorbed on the Ti (III)-surface or dissolved in water, reducing it to superoxide $O_2^{\bullet -}$ radical anion. The photo-generated holes can oxidize the organic molecule to form $R^+$, or react with $OH^-$ or $H_2O$, oxidizing them into $OH^\bullet$ radicals. The resulting $OH^\bullet$ radical, being a very strong oxidizing agent (standard redox potential +2.8 V) can oxidize most dyes to mineral acids, carbon dioxide and water (Qamar *et al*., 2005), these processes are presented in Fig 1.

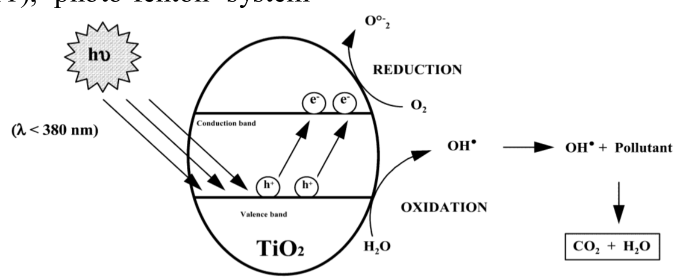
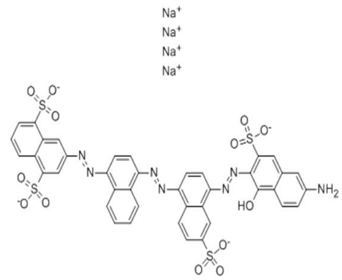
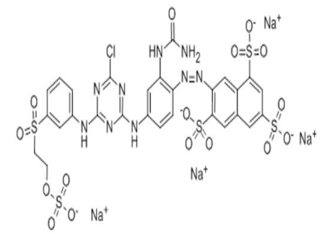



***Fig.1. General Mechanism of the photo-catalysis***

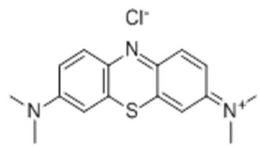
Since the real textile dyeing industry is using different kinds of dyestuffs, so four dyes (tubantin blue, tubantin red, methylene blue and reactive yellow) are selected study of their photodecolorization under solar light using two photo-catalysts, pure $TiO_2$ . and $TiO_2$ sol-gel which is prepared by sol-gel method. Also photon flux and temperature solution will be determined. Fig 2 shows the chemical structure of four dyes



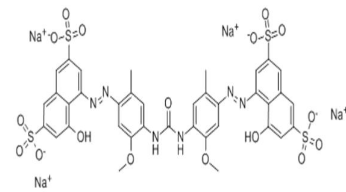
Tubantin blue BRR HC



Tubantin red 6BLL C



Methylene blue



Reactive Yellow 145

***Fig (2): chemical structure of dyes under study*** [38]

## Materials and Methods

### Materials

### Chemicals

Pure titanium dioxide (particle size 49 nm, specific surface area 46.962 m²/g and energy gab $E_g$) from Aldrich, $TiO_2$ sol-gel (particle size 49nm, specific surface area 46.962 m²/g and energy gab $E_g$)was prepared by sol-gel method. Tubantin blue BRR-HC, tubantin red, methylene blue and reactive and yellow 145 from Bezema, Ferrioxalate actinometry (0.006M) was prepared , distilled water, sodium hydroxide (1%), sulphuric acid (0.5M), 1-10 phenanthroline, sodium acetate All other chemicals were of analytical grade

### Apparatus

Sensitive balance ,Magnetic stirrer , Aqunova spectrophotometer (Jenway), Compound parabolic collector (CPC) solar collector was built by Dr. Saleh Hamdo at Solar Energy Equipment Co), borosilicate tube (length 50 cm,d 5cm) (built by technician Ahmed at physics Department – faulty of science – University of Khartoum) with slot on end to outlet the product.

## Methods

### Photo-reactor setup

All experiments were performed under natural solar radiation. The solar CPC was constituted by one CPC unit (0.2 $m^2$) tilted 15° N local latitude. The cylindrical photochemical reactor (800 $cm^3$ capacity) was made-up of borosilicate glass (length 50 cm, d 5cm). The slot was opened during experiments described in fig 3.

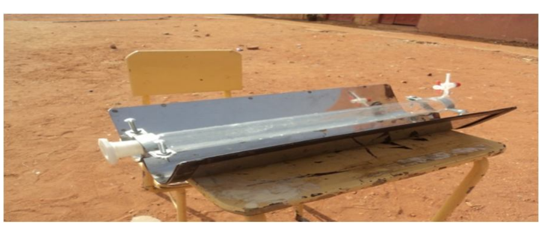


***Fig (3):1 Photo-reactor setup***

### Determination of photon flux

2.947g of ferrioxalate actinometry (0.006M) solution was prepared in 100 $cm^3$ of $H_2SO_4$(0.5 M) and diluted with distilled water to 1 $dm^3$. 450 $cm^3$ ($V_1$) of solution irradiated under solar light using photo-reactor in Fig 3 for 30 min. 10 $cm^3$ ($V_2$) of irradiated solution was given into 50$cm^3$ ($V_3$) volumetric flask containing a mixture of 5 $cm^3$ of 0.1% 1.10-phenanthroline solution and 2.5 $cm^3$ buffer (82g sodium acetate and 10 $cm^3$ $H_2SO_4$ diluted to $1dm^3$ with distilled water), then diluted to the mark with distilled water. A reference solution was prepared in the same way except that it had not been irradiated . Both solutions were kept in the dark for (about 30 min) until full colour development was achieved, and the absorbance difference between the two samples are measured at 510 nm [optical path length l = 2.5cm, ε(510nm) = 11100 $dm^3mol^{-1}cm^{-1}$]. The photon flux amount $q_{n,p}$ einstein $s^{-1}$was calculated by equation (1) (Kuhn et al., 2004)

$$q_{n,p} = \frac{\Delta A V_1 V_3}{\Phi(\lambda)\, \varepsilon(510nm)\, V_2 l t} \quad -(1)$$

**Photo-decolorization of dye**

In all experiments the reactor was washed with distilled water. 50ppm of each dyes (tubantin blue, tubantin red, methylene blue or reactive yellow) solution was taken. The pH 9 was adjusted using $H_2SO_4$ (1%) or $NH_4OH$ (1%). 1g/$dm^3$ of catalyst (pure $TiO_2$ or $TiO_2$ sol-gel) was added then the mixture was stirred for 30min and was put in the photoreactor containing 20 $cm^3$ of $H_2O_2$ (30%) and exposed to solar radiation for five hours. The sample was taken every hour and separated by centrifuge then absorbance was read at 436, 525 and 620 nm, and the average was taken, and the percentage of colour removal was determined by equation (2):

$$decolorizatin\,\% = \frac{A_i}{A_f} \times 100 \quad -(2)$$

Where $A_i$ is initial absorbance, $A_f$ is final absorbance. Then plot of the decolorization percentage versus irradiation time was obtained by using origin 8.6 computer programmer

## RESULTS and DISCUSSION

### Influence of different kinds of dyes

**Table (1): The photo-decolorization of four kinds of dyes by using pure $TiO_2$ & $TiO_2$ sol-gel**

| Decolorization % | | Methylene blue | Tubantin red | Tubantin blue | Reactive yellow |
|---|---|---|---|---|---|
| 0 hour | Pure $TiO_2$ | 0 | 0 | 0 | 0 |
| | $TiO_2$sol-gel | 0 | 0 | 0 | 0 |
| 1 hour | Pure $TiO_2$ | 20.06770 | 23.40590 | 18.79460 | 20.26516 |
| | $TiO_2$ sol-gel | 66.08000 | 59.77000 | 44.11230 | 56.37130 |
| 2 hours | Pure $TiO_2$ | 54.34048 | 40.43520 | 34.50630 | 55.70741 |
| | $TiO_2$sol-gel | 81.66340 | 68.03000 | 58.56510 | 48.94510 |
| 3 hours | Pure $TiO_2$ | 84.91110 | 38.82220 | 36.60660 | 77.59770 |
| | $TiO_2$ sol-gel | 88.53240 | 87.02000 | 95.65900 | 77.59770 |
| 4 hours | Pure $TiO_2$ | 98.75890 | 65.71640 | 66.58490 | 96.24171 |
| | $TiO_2$ sol-gel | 95.08930 | 97.27000 | 100.2597 | 101.1823 |
| 5 hours | Pure $TiO_2$ | 99.23900 | 96.10656 | 79.56900 | 101.9904 |
| | TiO2 sol-gel | 101.5900 | 67.85440 | 101.8973 | 114.6835 |

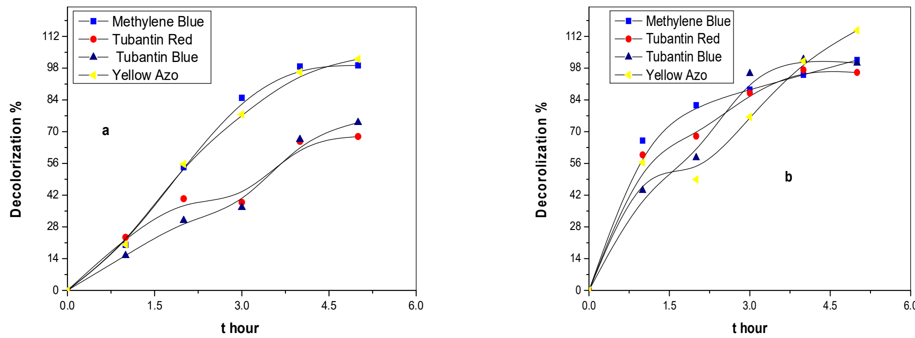



***Fig (4): The photo-decolorization of four kind of dyes by using pure $TiO_2$ and $TiO_2$ sol-gel ■ methylene blue ● tubantin red ▲ubantin blue ◄reactive yellow***

Table (1) summarizes the photo-decolorization methylene blue, tubantin blue , tubantin red and reactive yellow for each catalysts. Fig (4) indicates that decolorization percentage of the four dyes were in the following order: reactive yellow > methylene blue > tubantin blue > tubantin red, due to the molecular structural differences among these four molecules of dyes. The functional groups (nitrite groups, alkyl side chain, chloro, carboxylic, sulfonic substituent, and hydroxyl groups) of the molecular structure of dye tend to decrease the solubility of molecules in water (Khataee and Kasiri, 2010), and vary their adsorption characteristics and susceptibility to photo-degradation system (Zhang *et al*., 2011). Decolorization of reactive yellow azo dye is high due to presence of two hydroxyl substituents next to the azo bond. Moon et al. (2003), Buitron et al., (2004) maintained that the electronic properties of a hydroxyl group are −I and +M effects; the number of hydroxyl groups in the dye molecule can intensify this resonance and consequently the decolorization of the dye. Tubantin red was low owing to presence of chloro, ethyl side chain and five sulfonic substituent. The presence of chloro groups in the dye molecule decreases the process, alkyl side chain decreases the solubility of molecule in water, and more sulfonic substituents are less reactive in the photocatalytic process (Khataee and Kasiri, 2010).

**Influence of photo-catalyst**

***Table (2): properties for both pure titanium dioxide and titanium dioxide sol-gel***

| **Photo-catalyst** | **Surface area** | **Particles size** | **Energy gab** |
|---|---|---|---|
| **Pure titanium dioxide** | 46.962 $m^2/g$ | 49.11070 nm | 3.03eV |
| **Titanium dioxide sol-gel** | 38.264 $m^2/g$ | 22.0198 nm | 3.23eV |

Table (1) and Fig (5) showed that higher decolorization caused by $TiO_2$ sol-gel due to its smaller particle size, smaller surface area and wider energy gab than that caused by pure $TiO_2$ which was appeared in table (2). Carp et al., (2004) reviewed that large surface area are usually associated with large amounts of crystalline defects, which favour the recombination of electrons and holes leading to a poor photoactivity. Particle size is an important parameter for photocatalytic efficiency; since the predominant way of electron–hole recombination may be different depending on the particle size; also increase in the band gap and consequently, a blue shift in the absorption edge.

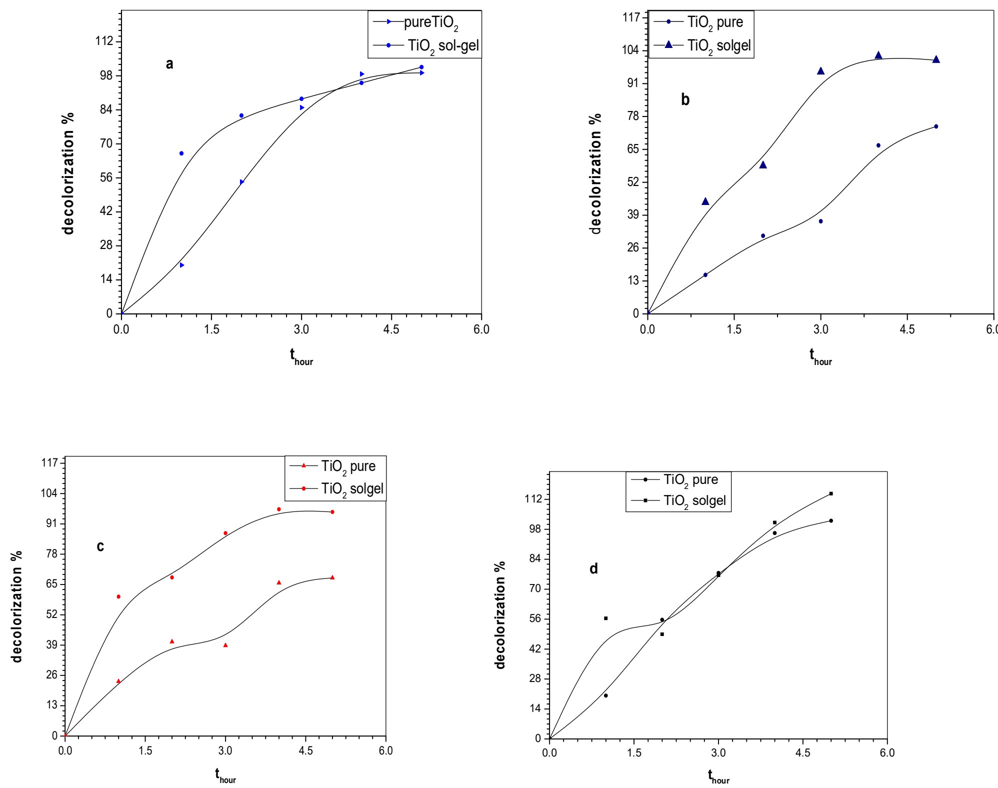



*Fig (5): Influence of type of titanium dioxide (pure $TiO_2$ and $TiO_2$ sol-gel) on photo-decolorizaton of dyes (a) methylene blue(b) tubantin blue (c) tubantin red (d) reactive yellow.*

*Table (3): Solution temperature (average) and daily photon flux average during photo-decolorization of the four dyes*

| Dye | Solution temperature (average) °C | | Daily photon flux (average) Einstein/s | |
|---|---|---|---|---|
| | Pure $TiO_2$ | $TiO_2$ sol-gel | Pure $TiO_2$ | $TiO_2$ sol-gel |
| **Methylene blue** | 46.2 | 41.2 | $3.1264E^{-5}$ | $3.1167E^{-5}$ |
| **Tubantin blue** | 40.8 | 38.8 | $3.1125E^{-5}$ | $3.1164E^{-5}$ |
| **Tubantin red** | 41.2 | 41.6 | $3.1174E^{-5}$ | $3.1101E^{-5}$ |
| **Reactiv yellow** | 40.8 | 39.4 | $3.1207E^{-5}$ | $3.1264E^{-5}$ |

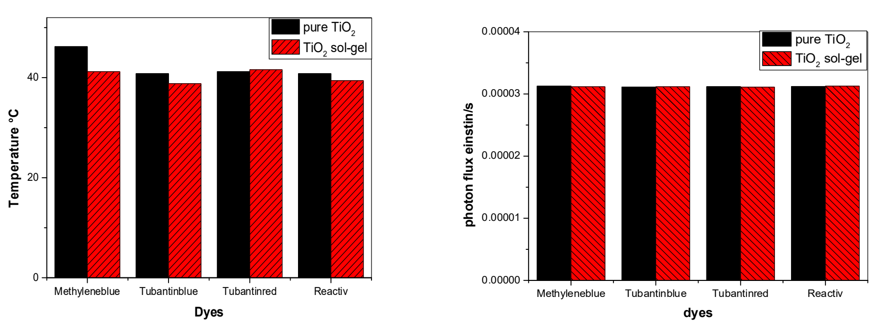



*Fig (6): Average of solution temperature and average of daily photon flux during decolorization of four commerical dyes*

Decolorization experiments of four dyes were conducted at 10 pm – 3 am on 2d/11 – 29th/11, Solar light intensity (photon flux) was measured for every hour by using ferrioxalate actinometry, and the average of photon flux over the duration of each experiments was calculated. Table (3) summarizes the average photon flux and average temperature solution during decolorization of the four kinds of dyes under study by using two kinds of photo-catalysts (pure $TiO_2$ and $TiO_2$ sol-gel) .Fig (6) indicates that The photon flux was nearly constant during the experiments. Fig (6) indicated that average temperature solution increased over the duration of each experiment. Using pure $TiO_2$ caused higher solution temperature that that caused by using $TiO_2$ because it has large surface area; thus, it has large number of crystalline defects which facilitate the recombination of electrons and holes and generation of heat( Carp et al., 2004).

## CONCLUSION

This study demonstrates that certain commercial dyes with different structure and different substitute groups can be decolorized by solar photocatalysis. The experimental results indicate that decolorization. Percentage of the four dyes were in the following order: reactive yellow > methylene blue > tubantin blue > tubantin red, due to the chemical structural difference among these four molecules of dyes. High decolorization observed on using $TiO_2$ sol-gel due to small particle size, small surface area and wide energy gab. The average of the daily photon flux was nearly constant during experimentation, when using pure $TiO_2$ the increase of solution temperature was higher than when using $TiO_2$ sol-gel due to the fact that pure $TiO_2$ has large surface area.

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