

Visible Light Photocatalytic Decolourization of Methyl Orange Using N-Doped TiO₂ Nanoparticles

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Abstract

In the present study, nitrogen doped TiO₂ (N-TiO₂) has been synthesized by simple sol-gel technique. The light absorption property and morphology of the N-TiO₂ were characterized by Diffused Reflectance UV-VIS spectrometer and scanning electron microscopy (SEM). The results showed a clear shift in the light absorption from UV region (380) to visible region (410 nm). SEM results showed the morphology was changed after doping process compared with the pure anatase TiO₂. The photocatalytic activity was examined by the photo decolorization of the Methyl Orange which was used as a pollutant model in this study under visible light irradiation. The main operational parameters, namely, initial pollutant concentration, doped photocatalyst loading, pH of photocatalysis solution and visible light intensity on the rate of photocatalytic decolorization. The results show that N doping induced the enhancement of photocatalytic decolorization. Methyl orange removal efficiency results at optimum experimental conditions have revealed more than 98% degradation after 60 minutes of illumination.

Keywords: photocatalysis, N-TiO₂, methyl orange, visible light, bleaching.

1. Introduction

Textile dyes (like, azo dyes) and other industrial dyestuffs constitute one of the largest groups of organic compounds that represent an increasing environmental danger. About 1–20% of the total world production of dyes is lost during the dyeing process and is released in the textile effluents [1]. Such colored dye effluents can threaten the surrounding ecosystems, apart from the environmental pollution; some of the dyes have health hazards. The toxicity and potentially carcinogenic nature of these substances and their manufacturing precursors represent an increasing danger in aquatic life [2]. Traditional physical treatment methods, like adsorption on activated carbon or ultrafiltration, can be applied to remove this kind of organic compounds from wastewaters but, as they are non-destructive, they just transfer the pollutants from water to another phase and originate the problem of secondary pollution. Besides, biological methods result ineffective to degrade azo dyes due to their resistant to biodegradation. Therefore, advanced oxidation processes (AOPs) appear as effective alternative methods to treat textile effluents. The main advantage of these processes is that they can destroy organic molecules, leading to their complete mineralization or to biodegradable intermediate compounds. Among AOPs, heterogeneous photocatalysis with titanium dioxide (TiO₂)

has been successfully applied to degrade organic dyes [3]. TiO₂ is used mainly due to its nontoxicity, high photocatalytic activity, capability to adsorb reactants under efficient photonic activation ($h\nu \geq E_g$), cheap, availability, and stability against photocorrosion. Furthermore, TiO₂ can be supported on various substrates such as glass, fibers, stainless steel, inorganic materials, sand, and activated carbon which allow its continuous reuse [4-6]. In addition, anatase is reported to give the best combination of photoactivity and photostability because anatase is the most active allotropic form among the various ones available, either natural (rutile and brookite) or artificial (TiO₂-B, TiO₂-H) [7]. The most of the restrictions are concerned with the need for an UV excitation source because TiO₂ is photoactive only under excitation wavelength < 385 nm due to its large band gap ($E_{bg}=3.2$ eV) [8]. Thus, the photocatalytic efficiency of TiO₂ is expected to be low under visible light irradiation as UV light is about 10% of the overall solar intensity [8-10]. To overcome this drawback, metal or non metal doping strategy has been adopted [11]. Doping is an effective method to enhance visible light absorption; moreover, recent reports revealed that nitrogen is an optimal candidate among the doping species such as the transition metals and nonmetals [5].

2. Experimental Details

2-1 Materials

Methyl Orange, (C₁₄H₁₄N₃SO₃Na) was purchased from Fisher Chemical. This compound was chosen as a simple model of a series of common azo-dyes largely used in the industry. Water in this experiment was purified by nanofiltered-deionization water supply unit (NFDW) and was used throughout. For the preparation of doped TiO₂ nanoparticles, the materials used were titanium tetra-isopropoxide [Ti(OC₃H₇)₄] (97%) from Aldrich, Iso-propopanol (99.8% Riedel-Dehaen Hannover) and Urea (99.5%) purchased from Fluka. All other reagents, (NaOH, H₂O₂ and HNO₃) used were high purity and purchased from Fluka, Scharlau and Medex products, respectively.

2-2 Preparation of N-Doped TiO₂

For the preparation of TiO₂ nanoparticles, Ti (IV)-isopropoxide is used as a starting material. TiO₂ sol was prepared by gradual addition of 10 milliliter of titanium tetra-isopropoxide [Ti(OC₃H₇)₄] to 0.83M of urea solution which was already prepared by dissolving 10 g urea in 200 milliliter of nano filtered deionized water at room temperature with at a rate of addition of 2 ml/ min. and vigorous stirring. Then capping agent, 5 milliliter isopropanol was added to prevent agglomeration of particles. Afterwards, the solution was stirred further for 2 hrs to achieve complete hydrolysis. The resulting transparent colloidal solution was aged for 24 hours. Subsequently, filtered by Buchner and dried at 60°C for 2 hrs and finally calcined at 400°C for 4 hrs under air [12].

2-3 Characterization of N- Doped TiO₂

UV-Vis spectroscopy measurement was carried out on a Shimadzu UV-3600 spectrophotometer coupled with a spherical integration unit to obtain the optical absorption threshold of the sample, using BaSO₄ as the reference sample, within the wavelength range of 200–800 nm. Scanning Electron Microscopy (SEM, Phenom™) was used to explore the morphological and structural properties of the N-TiO₂ powder.

2-4 Photocatalysis Procedure

For the visible photocatalytic process, irradiation was performed in a batch photoreactor of 100 milliliter in volume with a Philips 500 W halogen lamp. The lamp cooled continuously by circulating cold water by means of a thermostatic water bath. The experimental procedure was as follows; 100 milliliter of methyl orange solution (10 mg/l), 50 mg of doped TiO₂ powder and 2 milliliter of 50% wt/wt H₂O₂ were delivered into the glass cell. The suspension was first bubbled with O₂ gas (flow rate of 30 ml/min) with continuous stirring in the dark for 20 min to acquire the equilibrium adsorption. Then after, the suspension was irradiated by means of visible light until complete mineralization of the dye is achieved. During illumination, samples of 5 ml were collected at regular times, centrifuged and filtered. The discoloration was monitored by measuring the absorbance at λ max = 465 nm. The M.O. degradation was calculated as follows:

$$\text{Degradation\%} = (A_0 - A) / A_0 \times 100$$

where, A₀ is the initial absorbance of M.O. and A is the absorbance at sampling time.

3. Results and Discussion

3-1 UV-VIS spectroscopic feature of methyl orange

Methyl orange, which is a well known acid-base indicator, is orange in basic medium and red in acidic medium. Its structure, shown in Fig.(1) is characterized by a sulfonate group, which is responsible for the high solubility of these dyes in water. When dissolved in distilled water, the UV-visible spectrum of methyl orange showed two absorption maxima. The first band observed at 271 nm and the second band, more intense, at 465 nm as presented in Fig.(2). The latter band was used to monitor the photocatalytic degradation of methyl orange.

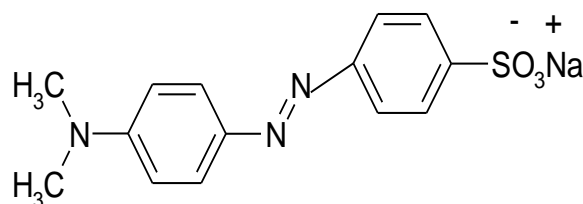


Fig. (1) Structure of Methyl Orange (MO).

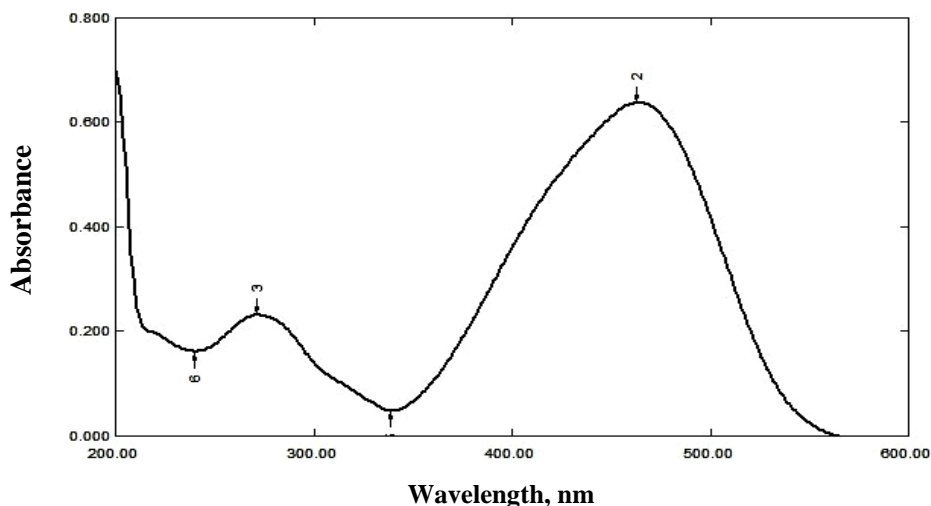


Fig. (2) UV-VIS spectrum for methyl orange.

3-2 Characterization of N-TiO₂

Fig.(3) shows UV-Vis diffused reflectance spectra for the pure and doped-TiO₂. As expected, pure TiO₂ showed absorption only in the UV region (less than 400 nm), due to the intrinsic band gap absorption of TiO₂. In comparison with undoped TiO₂, N-doped TiO₂ sample presents a similar light absorption band and have apparent absorption in the visible region. That is to say, with the preparing method of this paper, N doping do have effect on the light absorption property. This narrowed band gap will facilitate excitation of electrons from the valence band to the conduction band in the doped oxide semiconductor under visible light illumination, which can result in higher photocatalytic activities [13,14]. Scanning Electron Microscopy (SEM) images of undoped TiO₂ particles (anatase) revealed very smooth and homogeneous surfaces formed by very fine aggregated particles (Figure 4), whereas the N-TiO₂ particles exhibited slightly more compacted surface structures. The difference in morphology could be ascribed to nitrogen dopant. Similar conclusion has also made by others researchers [15].

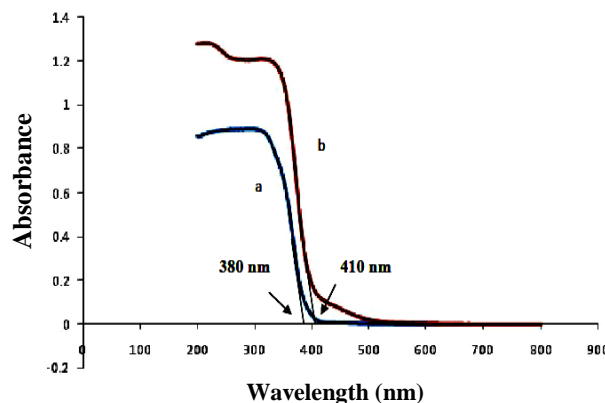
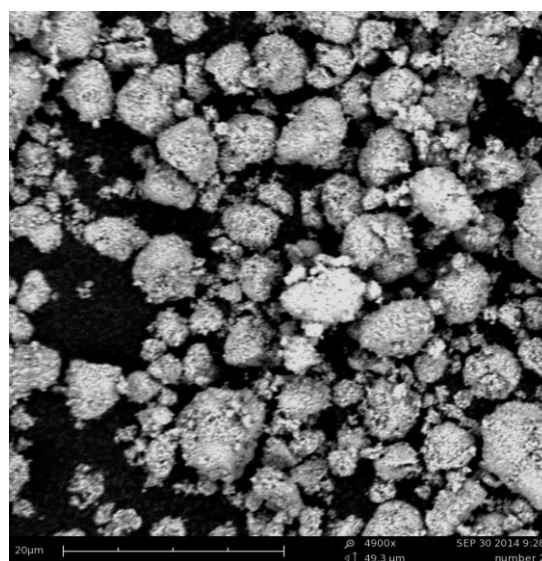


Fig.(3) UV-vis diffused reflectance spectra of (a) undoped TiO₂ and (b) N-dopedTiO₂.

A



B



Fig.(4) SEM images for the (A) anatase TiO_2 ; (B) N-TiO_2 .

3-3 Control experiments

Several control experiments were conducted to check the optimum conditions that essentially effect on efficient decolorization and degradation. The experimental results in this study have reached several conclusive statements: (1) the semiconductor and peroxide played a key role in photocatalytic degradation of dyes, (2) the self-degradation of methyl orange was almost negligible under visible illumination solely and (3) Photocatalytic degradation of dye was negligible when N-TiO_2 was used without light (there is no change in the concentration of the dye with time in dark), thus the color removal of the dye was truly photocatalytic and not just due to photolysis and/or due to ordinary adsorption. Fig.(5) displays photodegradation experiments for the control tests listed below;

- a = M.O.(10 ppm)+Vis light+duration 4h
- b = M.O.(10 ppm)+2 milliliter(50% wt/wt) H_2O_2 +duration 4hrs in dark (absence of light).
- c = M. O.(10 ppm)+2 milliliter (50% wt/wt) H_2O_2 +Vis light+duration 4hrs
- d = M.O.(10 ppm)+50 mg N-TiO_2 +2 milliliter (50% wt/wt) H_2O_2 +pH=2+duration 4hrs (absence of light).
- e = M.O.(10 ppm)+50 mg N-TiO_2 +Vis light + pH=2+duration 4hrs.
- f = M.O.(10 ppm)+50 mg N-TiO_2 +pH=2 + duration 4hrs in dark.

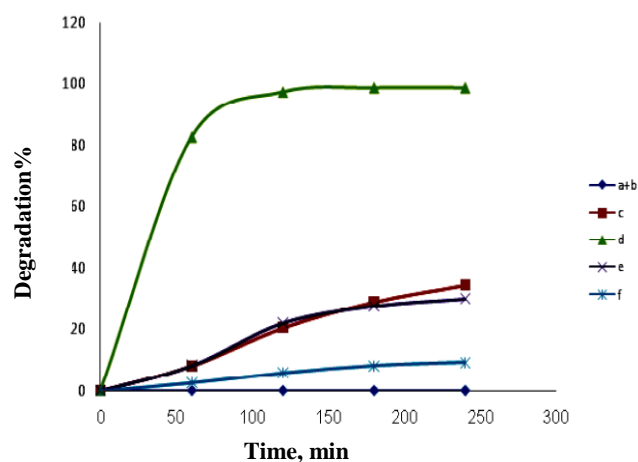


Fig.(5) Control experiments for the photodegradation of methyl orange.

3-4 Effect of initial methyl orange concentration on its degradation

The effect of initial M.O. concentration in water on the photocatalytic effectiveness of the process was examined. The photocatalytic decomposition of the dye was studied by varying the initial concentration from 5 to 30 mg.L^{-1} at pH=2.5 and with catalyst loading 50 mg. For M.O concentrations of ≤ 10 mg/L , complete discoloration of the solutions were observed (98%) after 60 min of illumination and the solutions turned completely 'transparent'. At initial dye concentration of 30 mg/L , the rate of elimination reached is approximately 50% for a total time of 60 min. Beyond 20 mg/L , degradation becomes slow. The rate of degradation relates to the probability of $\cdot\text{OH}$ radicals formation on the catalyst surface and to the probability of $\cdot\text{OH}$ radicals reacting with dye molecules [1]. Once the concentration of dye is increased, firstly, the adsorption amounts of catalyst attain the saturation for superfluous dye and secondly, the mutual screens among dye molecules also increase along with the concentration rising of dye. Thus, once the concentration of dye is increased, it also causes the dye molecules to adsorb light and the photons never reach the photocatalyst surface and therefore less $\cdot\text{OH}$ are formed [16-18], thus resulting in less degradation percentage as presented in Fig.(6).

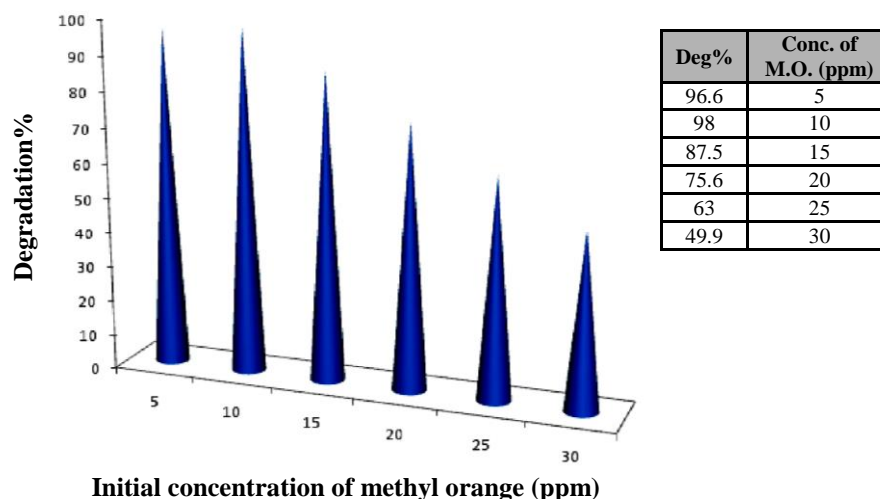


Fig.(6) Effect of initial methyl orange concentration on photodegradation efficiency.

3-5 Effect of the dose of semiconductor

The effect of catalyst dosage on photocatalytic degradation of M.O. under an irradiation of a 500W halogen-lamp within 60 min is illustrated in Fig.(7) When the irradiation was employed lonely, the self-degradation of M.O. was almost negligible under visible light illumination. In The presence of TiO₂ catalyst the degradation has been significantly enhanced. After 60 min, the degradation efficiency increased with increasing dose of TiO₂ and reached a maximum of 98% at a dose of 50 mg, which was followed by a decrease with a further increase in catalyst. Generally, the dye degradation increases with increasing catalyst concentration, which is characteristic of heterogeneous photocatalysis. The reason generally advanced for this, is that the increase in the amount of catalyst increases the number of active sites on the photocatalyst surface, which in turn increase the number of hydroxyl radical which can take part in actual discoloration of dye solution. Again, when the concentration of the catalyst increases above the optimum value, the degradation rate decreases due to the excess catalyst prevent the illumination. •OH radical, a primary oxidant in the photocatalytic system decreased and the efficiency of the degradation reduced accordingly [19,20]. Furthermore, A reduction in degradation rate was obviously observed at photocatalyst overdose due to the opacity caused by excess photocatalyst clusters (agglomeration of catalyst particles) then the solution becomes turbid and thus blocks radiation for the reaction to proceed and therefore percentage

degradation starts decreasing [18,21]. In other words, this supposedly an increased opacity of the suspension, brought about as a result of excess of photocatalyst particles [17]. On the basis of above results, the photodegradation rate of M.O. would also impacted seriously due to the increase in N-TiO₂ dosage as it is explicitly illustrated in Fig.(8).

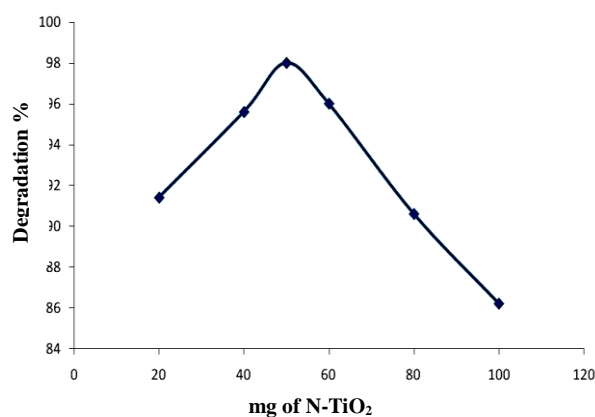


Fig.(7) Effect of N-TiO₂ loading on the removal of dye.

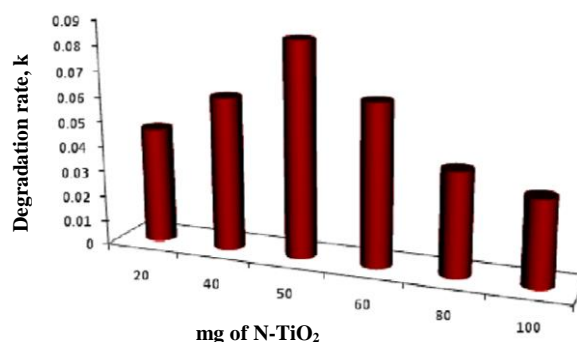
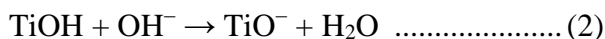
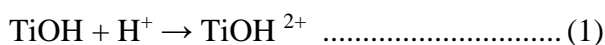


Fig.(8) Influence of N-TiO₂ dose on the rate of photocatalysis.

3-6 Influence of initial pH of photocatalysis solution

According to the literature, pH value is the major factor influencing the rate of photocatalytic process [6,22]. The examined range of pH was from 1 to 9. In all the experiments, pH was adjusted by adding appropriate amount of NaOH or HNO₃ solutions. The results are depicted in Fig.(9). It could be noticed that the best results were obtained in acidic solution: 82.7% (pH=2.5), 74.6% (pH=3) and 57% (pH=4) after 1hour of irradiation. At neutral and alkaline pHs, the degree of decomposition of M.O. for similar photocatalysis period were 15.2% (pH=7) and 6.9% (pH=9.01), respectively. The possible explanation for these results is related with the significant role of the surface property of the photocatalyst TiO₂; the acid-base property of the metal oxide surface has a considerable influence on the photocatalytic activity with varying pH [23, 24]. Under acidic or alkaline condition the surface of titania can be protonated or deprotonated, respectively according to the following reactions.



Where TiOH₂⁺, TiOH, and TiO⁻ are the positive, neutral, and negative surface hydroxyl groups, respectively. Thus titania surface will remain positively charged in

acidic medium and negatively charged in alkaline medium [25,19]. The point of zero charge (pzc) of TiO₂ is about 6.3. At pH values lower than 6.3 the catalyst surface is positively charged whereas at higher pH values it is negatively charged. Electrostatic attraction or repulsion between the catalyst surface and the organic molecule takes place depending on the ionic form of the organic compound (anionic or cationic) and consequently enhances or inhibits the photodegradation efficiency [26, 27]. Since M.O. is an anionic dye, it is easier to adsorb onto the surface of catalysts to promote reaction at lower pH. Therefore, the highest degradation efficiency (82.7%) was obtained in the solution of pH 2.5. Thus it can be suggested that the influence of pH on photocatalysis is due to the amount of dye adsorbed on catalysts, and the degradation takes place at or near to the catalyst surface, rather than in the bulk solution [28].

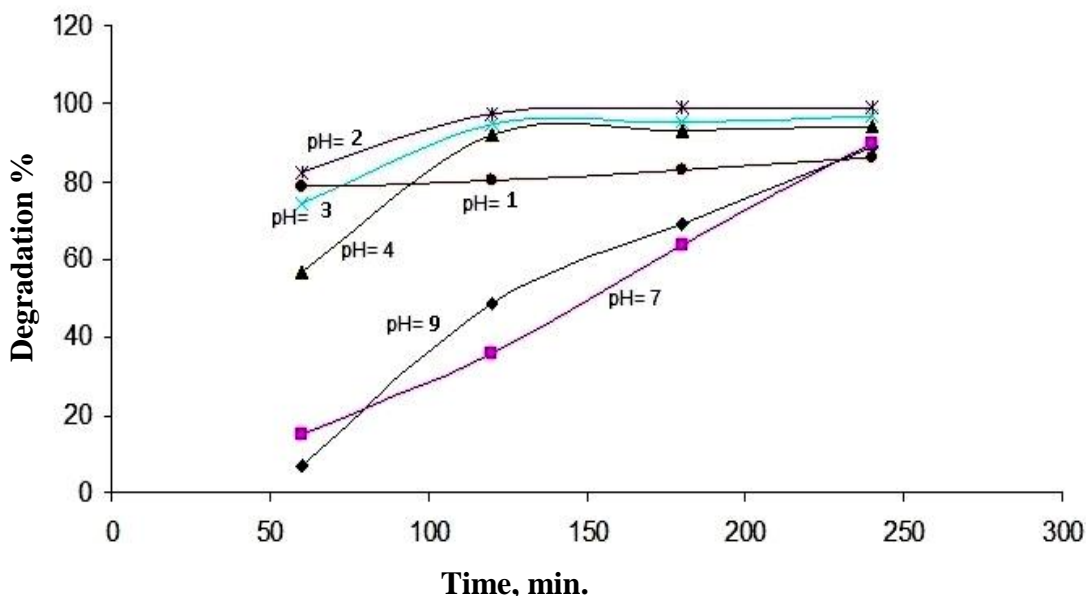


Fig.(9) Influence of pH on the degradation of M.O: Co 10 mg/l; N- TiO₂ loading 0.5 g/l; 2ml of 50% H₂O₂.

3-7 Effect of light intensity on the degradation of dye

Apparently, light irradiation plays an important role in all of the photocatalytic reactions and determines the number of created $e^- \cdot h^+$ pairs [29]. We observed that as light intensity increases the rate of degradation of dye molecules also increases. This change in the rate of degradation of dye molecules due to the variation in light intensity might be ascribed to the multiplication of emitted number of photons and consequently the

hydroxyl radicals and super oxide anions ($O_2^{\cdot-}$) yields are magnified. This magnification of oxidizing radicals would certainly increase the rate of degradation of dye molecules [30, 31]. Similar conclusion has been reported in our previous work [32]. Graphical representation of light intensity influence on M.O. photodegradation rate is shown in Fig.(10).

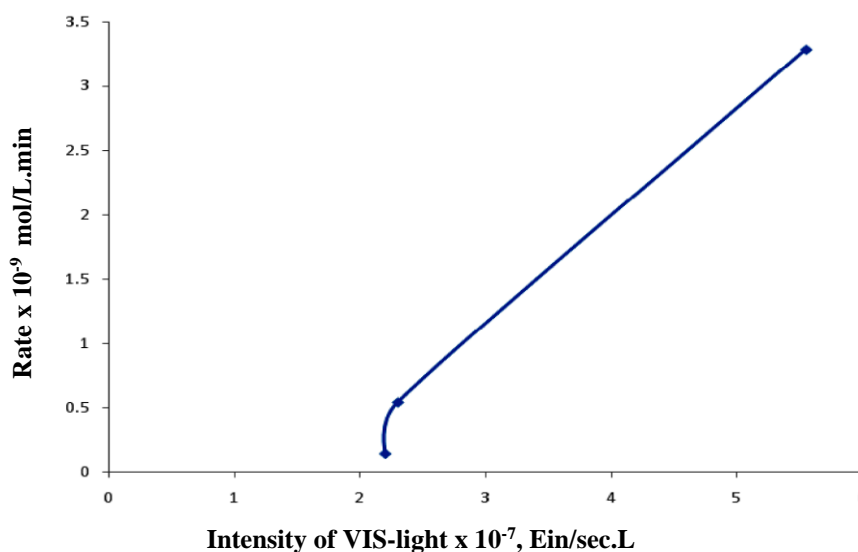


Fig.(10) Variation of the initial rates as function of light source intensity.

4. Conclusions

N-TiO₂ catalysts displayed enhanced visible light photocatalytic activities in methyl orange degradation. The photocatalytic degradation of methyl orange was influenced by its concentration in photocatalysis medium. The photocatalytic activities of the nanoparticles were greatly dependent on the solution pH where acidic pH values have been found to be favorable for the photocatalytic degradation of the target pollutant molecules in their anionic form. Titanium dioxide loading has significant effect on the degradation of methyl orange. The degradation efficiency increases with elevated concentration of TiO₂ suspension up to 50 mg, then the efficiency reduced slightly when the concentration is above 50 mg due to a light scattering effect. On optimal photodegradation conditions including initial pH 2.5, methyl orange concentration, 10ppm, catalyst loading 50 mg, irradiation time 60 min

with 500 W halogen lamp, the degradation efficiency attained 98%.

5. Acknowledgements

The authors are pleased to acknowledge the Department of Chemistry, College of Science, Al-Nahrain University and Iraqi Ministry of Higher Education and Bangor University for giving the opportunity to work in the school of Chemistry and Scientific Research for the financial support.

6. References

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الخلاصة

حُضر مسحوق ثنائي اوكسيد التيتانيوم النانوي المشوب بذرات النتروجين مختبريا باستخدام طريقة سول-جل وأختبرت كفاءته بإزالة لون صبغة الميثيل البرتقالي تحت الضوء المرئي. لقد أستخدمت تقنية المجهر الإلكتروني الماسح لدراسة طوبوغرافية السطح والتحليل الطيفي الانعكاسي في مجال الاشعة المرئية وفوق البنفسجية لحساب طاقة الفجوة والمساوية إلى 3.02 إلكترون فولت (410 نانوميتر) مقارنة بطاقة الفجوة لثنائي أوكسيد التيتانيوم القياسي (الانتيز) المساوية إلى 3.26 إلكترون فولت (380 نانوميتر). تم دراسة المؤثرات التشغيلية الرئيسة على عملية القصر مثل التركيز الابتدائي للصبغة ، كمية المسحوق النانوي، الدالة الحامضية للوسط وشدة الضوء المرئي. بينت النتائج الدور الايجابي للتشويب في عملية قصر الصبغة وصولا إلى تحطيم أكثر من 98% منها خلال فترة 60 دقيقة من التعرض للضوء المرئي. كما وجد إن هذه النتائج تتفق بشكل كبير مع نتائج البحوث الأخرى.