Plasma-catalytic Efficiency on the Discolouration of Sunset Yellow FCF in a Glidarc Plasma Reactor in Presence of WO_3 Used as Catalyst

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ABSTRACT

In this work, we highlight the plasma-catalytic effect of gliding arc discharge on the bleaching of azo dye sunset yellow FCF in aqueous solution. The effect has been studied in the presence of tungsten oxide as a catalyst in the glidarc reactor. The catalyst has been synthesized via acid precipitation process. The product obtained was characterized by X-ray powder diffraction (XRD), Fourier Transform Infrared (FTIR) and Nitrogen Physisorption. The results showed firstly monoclinic structure of WO_3 catalyst. The maximum bleaching rates obtained are 17% and 52.5% after 30 min for initial dye concentration of 25 mg.L^{-1} for plasma alone and plasma-catalyst, respectively. These results have clearly shown that gliding arc discharge has a double effect, in on hand as a source of hydroxyl radical and on other hands as a source of radiation able to excite the catalyst. The effects of initial pH and photocatalyst loading have been studied.

Keywords: Discolouration, gliding arc discharge, plasma-catalytic, WO_3 .

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I. INTRODUCTION

Dyes are responsible for about one-tenth of environmental pollutants. Dyes from most agro-food industries, textures, pharmaceuticals, etc., which are directly diverted into the environment without prior treatment [1]. Different methods are used for the treatment of waters containing dyes, including photocatalysis [2], which has received an interest in recent years due to the possibility of destroying the organics pollutants in water or air. Among the different types of photocatalysts, tungsten oxide (WO_3) has shown interesting properties. The tungsten oxide (VI) is a n-type semiconductor oxide with a band-gap energy approximately 2.5–2.8 eV [3], [4]. The different properties of tungsten oxide (VI) have recently generated significant interests for a wide variety of applications in dye-sensitized solar cells, water splitting photocatalyst as main catalyst, photocatalysis, microelectronics, colloidal quantum dot LEDs, detecting of hazardous pollutants such as NO_x and other alcohol gases, surface acoustic wave gas sensor, photochromic, electrochromic [5]–[11]. To synthesize WO_3 different approaches have been used, thermal decomposition, ion-exchange methods, pyrolysis, sol-gel, colloidal processes,

precipitation [6], [10], [12]–[18], among other methods. During the last 10 years, WO_3 has attracted significant scientific interest, the number of research articles related to WO_3 increased. This rising shows that WO_3 is becoming a more and more important in the field of photocatalytic application. Laroussi et al. and Brisset et al. showed that the gliding arc plasma (GAP) is able to generate the ultraviolet radiation [19], [20], which can activate the catalyst surface and thus generate active dissociated electron hole pairs. The coupling of GAP with photocatalysis has been largely studied to improve organics dyes removal [21]–[25].

The goal to reach in this study is to investigate the efficiency of gliding arc plasma (GAP) to enhance the bleaching rate of Sunset Yellow FCF (SY-FCF) in presence of acid assisted synthesis monoclinic tungsten oxide in the glidarc reactor. The bleaching skill of the dye solution treated by Glidarc was compared to that of the solution treated by the Glidarc- WO_3 system.

II. MATERIAL AND METHOD

A. Material

Sunset yellow FCF ((SY-FCF), ($C_{16}H_{10}N_2Na_2O_7S_2$)) or acid-6-hydroxy-5-[(4-sulphophenyl)azo]-2-naphthalene-sulfonic disodium salt, molar mass: 452.37g was purchased from Sigma-Aldrich and was used as target dye with absorption maxima at 482 nm. SY-FCF stock solution (500 mg/L) was prepared in distilled water and kept in a dark condition before experiments. SY-FCF chemical structure is shown in Fig. 1, sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$, 99%) was purchased from Merck-Darmstadt, sulfuric acid 98 % obtained from Riedel-de Haën Germany, and distilled water was used throughout the sample's preparation.

Fig. 1. Structure of sunset yellow FCF (SY-FCF).

B. Apparatus

The experimental apparatus used for this study is represented in (Fig. 2); it has been described by several author [22], [24], [25]. The formed species in gliding arc plasma reactor such as $OH^\bullet$ radicals H_2O_2 play an important role for the strong oxidizing effects [26]. Equation 1 to 11 represent the formation of reactive oxygen and nitrogen species in liquid through liquid/gas interface and develop in the liquid target.

Fig. 2. Experimental setup of gliding arc discharge.

C. Synthesis of photo-catalyst

10 g of $Na_2WO_4 \cdot 2H_2O$, 98% purity were added in 500 mL distilled water followed by stirring to obtain the aqueous transparent solution. The concentrated Sulfuric acid (32 %) was dropped slowly into the solution until a yellowish tungstic solution was thoroughly formed. The precipitate was allowed to settle for 3 hours; afterward, the sediment was settled down for natural dryness. Then the formed yellowish precipitate was washed several, after that the product was dried in the oven (100 °C). Finally, the material obtained was calcined at 800 °C for 3 h in a tube furnace to obtain the yellowish coloured WO_3 .

The pictures a, b, c, and d show by figure 3 represent the different steps of WO_3 synthesis. Initially the salt solution of $NaWO_4 \cdot 2H_2O$ is colourless, then we added sulfuric acid 4M with 30 s rapid stirring, the colour of solution turns to yellowish. After 3 hours of aging, we obtained yellow precipitate hydrated of tungsten oxide ($WO_3 \cdot nH_2O$). Finally, the yellowish powder of tungsten oxide is obtained by calcination of $WO_3 \cdot nH_2O$ at 800 °C.

Fig. 3. Pictures of different steps of WO_3 synthesis: a) aqueous solution of $NaWO_4 \cdot 2H_2O$, b) a + Sulfuric Acid stirring 30 s, c) a + Sulfuric Acid 3h, d) WO_3 after calcination at 800 °C.

D. Characterization

The X-ray powder diffraction (XRD), Fourier Transform Infrared (FTIR), and Nitrogen Physisorption were performed as described in the following article [23], [24].

E. Experimental Procedure and Analytical Methods

An exact volume of 420 mL of dye solution of Sunset Yellow FCF (25 mg. L^{-1}) were filled in the gliding arc plasma reactor and the air flow rate was set at $13.33 L \cdot min^{-1}$. The treatments of the liquid dye are evaluated in function of the treatment time $t^*(min)$ by keeping fix. The treatment times were respectively: 5, 10, 15, and 30 min. The solutions were exposed to the discharge without and with WO_3 added (at 0.5, 1, 1.5, 2, and 2.5 $g \cdot L^{-1}$ concentration) with the aim of optimizing the quantity of catalyst, before analysis, the aqueous samples were centrifuged to remove WO_3 agglomerates. UV-vis analyses were performed on a UV-vis spectrophotometer (AQUALITIC) to follow the fading process for each treatment type. The residual absorbance of

Sunset Yellow FCF after each exposure time was determined by spectrophotometry at $\lambda_{\max} = 482$ nm. The decreased dye absorbance was measured and bleaching efficiency (BE) was calculated using the equation:

$$BE = \frac{(A_o - A_i)}{A_o} \times 100 \quad (12)$$

where A_o represents the input absorbance of the dye solution and A_i represents the output absorbance (after the reaction).

III. RESULTS AND DISCUSSION

A. Structural characterization

The XRD of the synthesized WO_3 powders at the temperature of 800°C is depicted in Fig.4, the main peaks at $2\theta = 23.1^\circ$, 23.7° , and 24.4° are assigned to Miller indices (002),(020), and (200), respectively and all diffraction lines were correctly assigned to the monoclinic polymorph of WO_3 (m- WO_3) according to the JCPDS Card No. 01-083-0950. The average crystallite size (D) of the WO_3 sample was evaluated by using the below formula [27]:

$$D = \frac{0.90 \lambda}{\beta \cos \theta} \quad (13)$$

where λ (0.1548 nm, $\text{CuK}\alpha$) is the X-Ray wavelength, β is the breadth of the diffraction lines in radian and θ is the diffraction angle in degree. The reflecting peak at the crystal plane of (200) for the sample is chosen to estimate the average size of the WO_3 , the crystallite size estimated is about 35.05 nm.

Fig. 4. XRD patterns of WO_3 sample synthesized.

B. FTIR Spectroscopy

To confirm the monoclinic tungsten oxide, the powder was characterized by FTIR method. This analysis is a good way to provide the absorption peaks which agree the frequencies of vibrations of the sample. Fig. 5 shows the FTIR spectra of the synthesized tungsten oxide calcined at 800°C (wavelength range $400\text{--}4000\text{ cm}^{-1}$). The tungsten oxide vibrations are found in the infrared region of $1200\text{--}600\text{ cm}^{-1}$, which correspond to W–O stretching [28], [29], bending and lattice mode [30]. The peak 1624 cm^{-1} may be attributed to W–OH vibration. The figure also shows a strong band at 815 cm^{-1} for W–O–W bridging mode also the presence of a broad W–O–W bridging stretching band centred at 658 cm^{-1} is noticed [30]. The peak at 815 cm^{-1} is attributed to the O–

W–O stretching vibration mode of the bridging oxygen atoms [29], [31]. The absorption band in the range of 3429 cm^{-1} can be due to the symmetric stretching vibrations and intercalated water molecules ($\text{WOH}\dots\text{H}_2\text{O}$).

Fig. 5. FTIR spectra of tungsten oxide.

C. Nitrogen Physisorption Analysis

The nitrogen adsorption/desorption isotherms and the BJH pores distribution curve of prepared tungsten oxide were explored by nitrogen physisorption analysis (Fig. 6). The results exhibited an isotherm with a hysteresis loop, typical of type IV isotherm and illustrating the presence of mesopores, moreover, the hysteresis loop is more shifted to high relative pressure (0.9–1), suggesting the presence of macropores ($>50\text{ nm}$) [21], [23] (Fig. 6a). The pore distribution (Fig. 6b) shows a single band, typical of monomodal distribution and illustrating the homogeneity of spores sizes. The specific surface area (S_{BET}) value obtained from the BET (Brunauer–Emmett–Teller) analysis reveals that the synthesized monoclinic tungsten oxide has values of $5.2\text{ m}^2\cdot\text{g}^{-1}$, 22.3 nm and $0.35\text{ cm}^3\cdot\text{g}^{-1}$ respectively for surface area, pores diameter and pores volume.

Fig. 6. N_2 adsorption–desorption isotherms (a) and the pore size distribution curve (b) of WO_3 .

D. Catalytic activity of WO₃ sample

1. Catalytic efficiency of WO₃ synthesized

To investigate the catalytic effect of synthesized m-WO₃ on the dye bleaching, the solutions were treated in the plasma reactor without catalyst (plasma alone), with catalyst added (plasma + m-WO₃) and use of catalyst like adsorbent (m-WO₃ alone). The Fig. 7 shows the sunset yellow FCF beaching rate with 10.5, 16.9 and 52.5% for m-WO₃ alone, plasma alone and plasma + m-WO₃ respectively after 30 min of treatment. These results (Fig.7) show that the plasma treatment alone is not sufficient to obtain a high dye bleaching rate; the plasma treatment alone has given us a bleaching efficiency of 16.9% after 30 min treatment, this lower value of bleaching efficiency can be due to the quantity of oxidizing species generate by the gliding arc discharge inside the dye solution. Moreover, the bleaching efficiency obtained when the glidar discharge is coupled with catalyst (plasma + m-WO₃) is highest than the total of plasma alone and catalyst alone. Nevertheless, the gliding discharge alone is able to fade the dye. The increasing of dye bleaching rate after the coupled treatment (plasma + m-WO₃) could be due to radiation from gliding arc discharge. Benstaali et al., [32] showed that the light generated by the gliding arc discharge is rich in ultraviolet radiations and the emergence of a photo-sensitive catalyst such as WO₃. The UV radiations on WO₃ might also enhance the formation of active $\cdot\text{OH}$ due to the ability of tungsten oxide to dissociate adsorbed water molecules. Equations 14-21 show the mechanism for degradation of dye using WO₃ under radiation. In the mechanism, hole and electron pairs are generated by light irradiation.

where h^+ refers to a positive hole.

Fig.7. Bleaching efficiency of SY-FCF as a function of treatment time (substrate dye concentration: 25 mg.L⁻¹, initial pH: 5, catalyst concentration: 1 g.L⁻¹).

The HO radicals photo-generated added to HO radicals generated by gliding arc discharge thus accelerate the degradation process.

2. Effect of initial pH of the dye solution

The influence of pH on the bleaching efficiency of SY-FCF (25 mg/L) was analyzed at a constant loading of catalyst (1 g/L) by changing the initial pH of the aqueous solution (3 to 9). Fig.8 depicts that, the bleaching efficiency of SY-FCF decreases by raising the initial pH. Indeed, the discoloration rate was decreased from 59.7 to 19.6% by increasing the initial pH from 3 to 9. The bleaching rate decrease with the increase in reaction pH and high bleaching rate was obtained at 3.0 pH as shown in Fig. 8, the greater bleaching efficiency at pH 3.0 can be explained by the isoelectric point (pHIEP) of the catalyst which corresponds to the zeta potential equal to zero. At pH values greater than pHIEP (pH > pHIEP), the catalyst surface becomes more negatively charged, the dye used in this study was anionic dye and was negatively charged under experimental conditions. The decreasing of the bleaching efficiency can also be explained by the direct competition between the OH $\cdot$ radicals produce on the surface of the catalyst (reaction of h^+ and adsorbed OH $^-$) also some negatively charged species [9], [15], [16], [33]. Meanwhile, in acid medium (pH<pHIEP) the catalyst surface becomes positively charged In such case, the electrostatic attraction between the positively charged surface and the anionic dye may result in an increased discoloration process [34].

Fig. 8. Effect of initial pH of substrate dye solution on bleaching rate (dye concentration: 25 mg.L⁻¹, catalyst concentration: 1 g.L⁻¹, time: 30 min).

3. Effect of catalyst concentration

The effect of the catalyst concentration on bleaching process of SY-FCF (25 mg/L) was studied at pH 5. As shown in Fig. 9 the bleaching rate increase from 25.04 to 52.56% when the catalyst dosage increases from 0.5 to 1 g/L, however for the catalyst dosage above 1 g/L the bleaching rate decreases. The bleaching efficiency up to 1 g/L can be explained by the fact that more photocatalyst leads to the reaction of more reactive species. In many cases due to the increase of the active sites as well as by the generation of free electrons in the conduction band during photocatalysis process, the rate of photocatalytic bleaching of organics pollutants increases with photocatalyst loading [21], [23]–[25]. Indeed, at lower catalyst dosage, more light is transmitted through the reactor to increase of hydroxyl radical produced from the irradiated catalyst. However, beyond the optimum amount of catalyst dosage, the discoloration efficiency can be expected to decrease due to the increase of the density of aqueous solution and thus be increasing the

light dispersion. Moreover, the agglomerations due to increasing of catalyst dosage reduce the number of surface adsorption sites and thus decrease the photocatalytic activity [35].

Fig. 9. The effect of photocatalyst dosage on the on bleaching rate of SY-FCF (substrate dye concentration: 25 mg.L⁻¹, initial pH: 5, time: 30 min).

IV. CONCLUSION

In this present work, the plasma-catalytic efficiency on the discoloration has been proven. The gliding arc discharge plays a double role during the bleaching process. The catalyst used in this work has been successfully synthesized via acid precipitation. The WO₃ obtained was used as photocatalysts for the discoloration enhancement of SY-FCF solution in glidarc plasma reactor. The pH of the solution and the catalyst concentration has an important role in the bleaching process. These experiments allow us to conclude that the as-synthesized catalyst presented efficiency in the discoloration of SY-FCF solutions and the plasma which is a rich medium of reactive species and sources of UV radiation could be used in the aim to improve the discoloration by coupling with m-WO₃ incorporated.

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