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Electrochemical preparation of aluminum oxide nanoparticles and using for remove chromotrope-E122 dye

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***Abstract*---**A photocatalyst aluminum oxide Al2O3 Nanoparticles was effectively prepared by electrochemical method using electrolysis cell which contains stainless steel (1cm 2) and aluminium plate (1cm2) were worked as a cathode and anode electrodes respectively. The synthesized Al2O3 Nanoparticles sample was identified by Fourier transform infrared (FTIR), X-ray diffraction (XRD), and Field Emission Scanning Electron Microscope (FE-SEM) with (EDS) analysis. The XRD pattern specified that prepared Al2O3 sample had a crystal structure for γ-Al2O3with average crystal size (3nm) was achieved at (1000°C) calcination temperature. FTIR spectra indicated the existence of O–Al–O bonds vibration and hydroxyl group in the sample. The FESEM images gave morpholgy of Al2O3 nanoparticles powder after calcination to 1000°C was spherical shape and the particles were attached to each other, leading to an increase in particle size. The EDS measurements indicated the presence of Al with O peaks. The experimental results of the Al2O3 photocatalyst calcined at 1000°C for two hours showed photocatalytic efficiency to degradation Chromotrope -E122 dye under UV light.

***Keywords*---**photocatalyst, aluminium oxide nanoparticles, sunset yellow dye.

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Introduction

Al_2O_3 is dielectric material with a broad band gap about 6 eV at 300 k , Al_2O_3 can occure in varied crystalline shapes like delta, gamma, alpha and theta phases, every phase has its properties with other applications. Increasing temperatures of calcination lead to the phases sequence: $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ of l_2O_3 [1]. Ther are different methods to produce Al_2O_3 nano sizid such as laser ablation, chemical vapor deposition in flame, hydrothermal, spark plasma sintering , sol-gel , electrochemical reduction and mercury mediated technique . Among these methods, the electrochemical method is preferred due to easy installation, obtain sample has high purity, time budget, without formation side product and easy isolation. Al_2O_3 nanostructured like mesoporous alumina, rod, tube, belt, wires and fiber have been synthesized by electrochemical reduction method [2]. γ- Al_2O_3 and α- Al_2O_3 considered as the strongest absorber for most photocatalytic reactions because it has a large surface area [3]. Nano-sized metal oxide Al_2O_3 is often used as catalytic agents because of their high efficiency, no toxicity, high stability, low costs and due to their high given surface area, environmental friendly behavior , application of solar energy and high crystallinity has promising photocatalytic activities and might be an alternate material for wastewater treating and environmental application [4] .

Malachite green dye solution was photodegraded by used Al_2O_3 nanoparticles as photocatalyst [5,6]. Alumina nanoparticles was used for photodegradation of methyl red, methylene blue and congo red dyes by Rishu Katwal et al. Al_2O_3 exhibit a good catalytic efficiency to photodegradation of congo red about 94 % and lower efficiency toward methyl red 19% [7]. Carmoisine E122 (Disodium4-hydroxy-2-[(E)-(4-sulfonato-1-naphthyl) diazenyl] naphthalene-1-sulfonate) dye is one of the synthetic azo food's dyes that wide consumed in food industries to obtain on red colour yields. Since of this dye is not toxic and not carcinogenic , it is used as coloring agents in ice cream, confectionary, gelatine desserts drinks, cosmetics and in medicine [8] . On the other hand, at high amounts of carmoisine cause harm and damaged in the human body vital organs like the kidneys and liver and by reaction DNA with this dye the gens may be changed [9]. For this Many various chemical, biological and physical processes were done to treat these colored effluents, such as chemical degradation , chemical adsorption , sonochemical process , electrochemical process , photo degradation , biodegradation , bio-adsorption , using Fenton and Photo-Fenton reaction and extraction. Among these treatments is photodegradation, which they have appeared as powerful active processes to destroy the manufactures organic pollution in water [10].

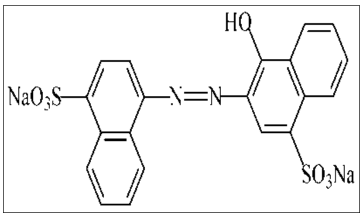

Figure.1 structure of (Carmoisine E122) dye[11]

In the present investigation, Al2O3 nanoparticle was prepared by electrochemical method. The resulting samples were characterized by FTIR, XRD and FESEM. This synthesis alumina is used for the removal of Carmoisine E122 dye from wastewater by photocatalysis method.

Chemicals and Methods

Electrochemical synthesis of alumina nanoparticle

For electrochemical synthesis of alumina nanoparticles we have used electrochemical cell which consists 50 mL of SDS (0.01g) aqueous solution as the stabilizer to electrolyte, stainless stee l(10× 10) mm and 99.5% purity aluminium plate (10 × 10) mm were used as cathode and anode an elecrodes respectively. A precipitate of aluminum hydroxide was formed by applying a constant voltage 5 V for one hour and it was monitoring by observing the turbidity of the solution. The precipitate is collected, washed with deionized for several times, dried by hot air at 80 °C for 48 hours, dried precipitate was calcined for two hours under a 1000 °C to obtain γ-Al_2O_3 nanoparticles.

Photocatalytic degradation

Al_2O_3 photocatalyst 0.165 g was added to100 mL of 10ppm E122 dye solution and the result suspension was exposed to UV light. After 5 minutes, 3ml of radiation dye solution was drawn off with a syringe and subjected to a centrifuge 2000 rpm for time 5min and the absorption of the dye solution was measured with a spectrophotometer at 555 nm. The photo degradation effective (P.D.E.) was calculated as follows equation[12]:

$$P.D.E = \frac{A_0 - A}{A_0} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots..(1)$$

Where A is the absorption of dye after irradiation and A_0 is the primary absorption of dye before irradiation

Result and Discussion

FTIR Analysis

Figure 2 shown the FTIR spectra of the Al_2O_3 samples. The broad absorption band from 500-1000 cm^{-1} shows the formation of Al_2O_3 , the peak at 578 cm^{-1} characterizes the Al-O-Al bond and the stretching vibration of Al-O noted at 758 cm^{-1} [13]. The absorption band at 1627 cm^{-1} agreed with the existence of the OH group in the synthesized sample which shows the bending mode of water molecules . Stretching vibration of hydroxylates group that is bonded to Al(III) appear as broad absorption band at 3456 cm^{-1} [14]

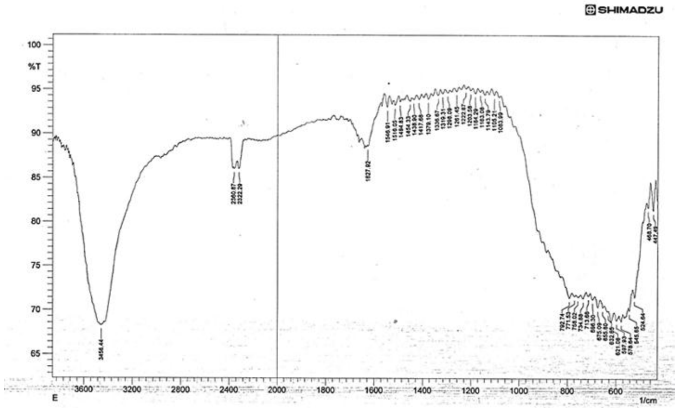

Fig. 2. FTIR Spectra of Al_2O_3

XRD Analysis

The XRD patterns of the prepared γ-Al_2O_3 nano size shown three distinct peaks at 2θ = 37.5^0, 2θ = 45.87^0 and 2θ = 66.55^0 are defined to (111), (200) and (220) reflections of γ-Al_2O_3. By using the Debye- Scherrer the average crystallite sizes were also calculated (Eq.1) was found to be 3 nm for γ-Al_2O_3 :

$$D = \frac{K\lambda}{\beta cos\theta} \qquad (1)$$

where, λ = 0.15406 nm , K = 0.95, β=(FWHM), θ : diffraction angle.

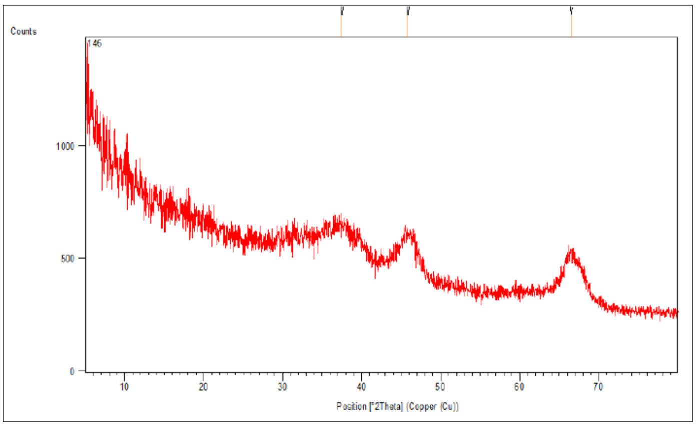

Fig 3. XRD patterns of γ-Al_2O_3

Field Emission Scanning Electron Microscopy

Figure 4 shown the FE-SEM figures and EDS patterns of the Al_2O_3. FESEM images be seen a porous and irregular morphology of prepared Al_2O_3 Nanoparticles sample with various pore shapes and sizes. The particles contain spheres particles and highly porous round-shaped agglomerates of, with a average particle size distribution between 22 to 261 nm due to the coalescence and the densification of the preliminary particles. The EDS pattern (Figure 4B) confirms the presence of Al (59.8%), O (29.4%), and (10.8%) C is due to utilization of carbon coating through the FE-SEM analysis.

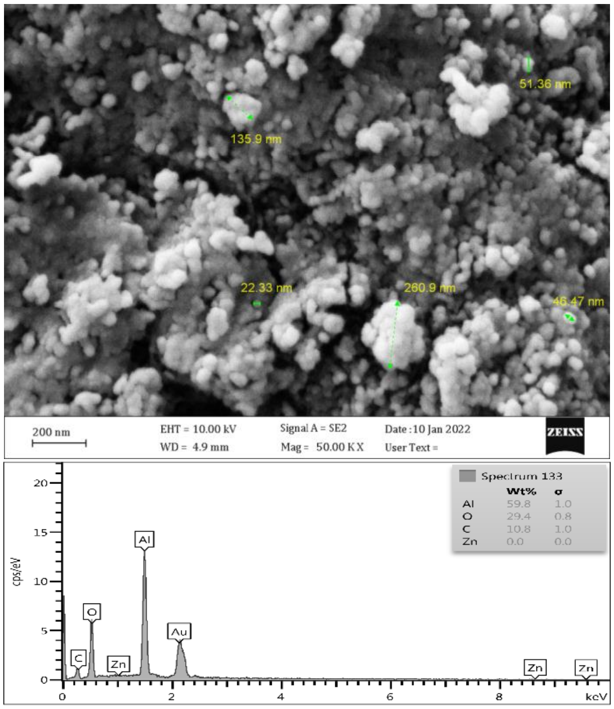

Fig. 4. XRD SEM images and EDX patterns of γ-Al_2O_3

Photocatalytic degradation of E122 dye solution

The photocatalytic activities of Al_2O_3 nanoparticles are measured through the degradation effect of Chromotrope -E122 dye as typical reaction in UV irradiation , and the results are shown in figure 5. We found that the dye was not affected by the presence of light alone, as it was stable in light, but when Al_2O_3 nanoparticles was added to the dye solution and exposed to UV-light, the photocatalytic efficiency was 78.84% after 60 minutes, in dark reactions, the removal efficiency was 73% after one hour of stirring in the presence of Al_2O_3 nanoparticles alone without UV light .This indicates that the rate of degradation of Chromotrope - E122 dye solution on Al_2O_3 nanoparticles is influenced by the adsorption process [15, 16].

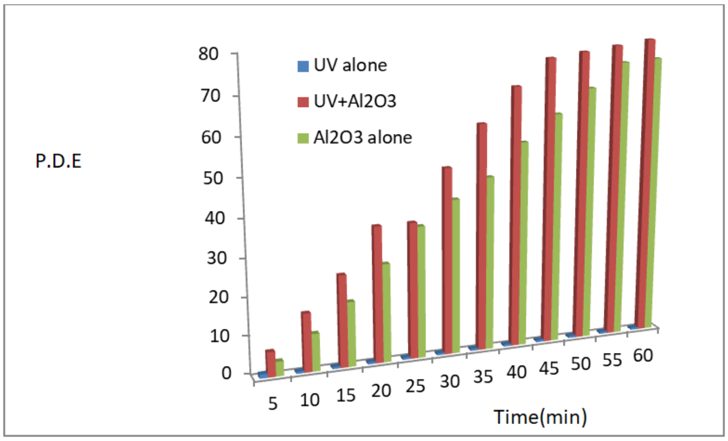

Figure 5. Effect of aluminum oxide nanoparticles on removing E122 dye in the absence and presence of UV-light

Fig. 6 shows the kinetic of photocatalytic degradation of the Chromotrope -E122 dye in the existence of Al_2O_3 NPs. We see that the presence of Al_2O_3 nanoparticles plays a role in the degradation of Chromotrope -E122 dye , we found that by using Al_2O_3 NPs, the photocatalytic degradation of E122 dye is well fit for the pseudo-first-order kinetics. The rate constants were calculated to be 0.025 min^{-1} and 0.0341 min^{-1} with correlation constant R^2 = 0.9848, 0.9842 for Al_2O_3 alone and Al_2O_3 with UV- light respectively

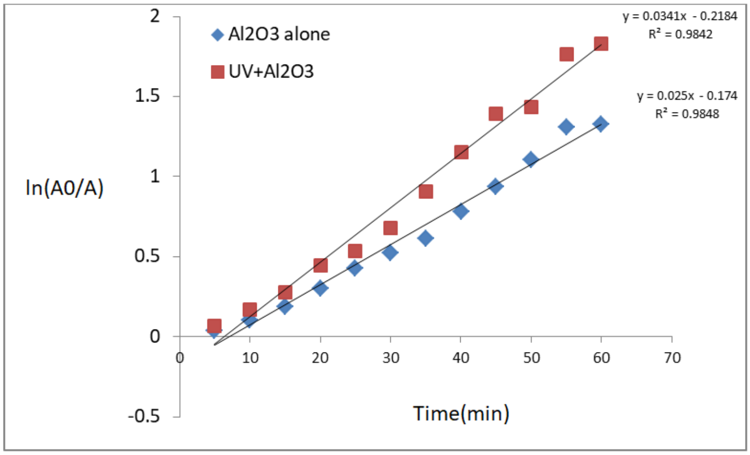

Figure 6. Kinetic analysis for the degradation of Chromotrope -E122 dye using Al_2O_3 alone and Al_2O_3 with UV- light

Conclusions

Aluminum oxide nanoparticles were successfully prepared by a simple electrochemical method, and the average crystal size calculated from the Debye Scherrer equation was 3 nm through the X-ray pattern. The efficiency of the prepared aluminum oxide nanoparticles as a photocatalytic agent was tested by degradation of Chromotrope -E122 dye, and the percentage of deradation was 78.84% within one hour, which is a good result indicating the possibility of using aluminum oxide nanoparticles as a photocatalyst for the removal of organic dyes.

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