

Asian Journal of Applied Research

DOI: http://dx.doi.org/10.20468/ajar.2018.01.02

Research Article

Preparation and Photocatalytic Activity of Aluminum Oxide (Al₂O₃) Nanoparticles

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ABSTRACT

Aluminum oxide (Al_2O_3) nanoparticles photocatalyst was successfully synthesized by urea decomposition method using metal nitrate as precursors in the presence of sunlight. The as-synthesized samples were characterized by X-ray diffraction (XRD), Fourier transform infrared (FTIR), and ultraviolet-visible spectroscopy. The XRD pattern indicated that as-synthesized sample had a crystal size with finest particle size of the catalyst (30.096 nmappr.) was obtained at 600°C calcination temperature. FTIR spectra confirmed the presence of hydroxyl group and Al-O bond vibration in the catalyst. Experimental result of the Al_2O_3 photocatalyst calcined at 600°C for 2 h, exhibited photocatalytic activity of under sunlight irradiation, the constants of malachite green dye degradation. In this study, the synthesized nanoparticles used to for the degradation of the dye by direct sunlight exposer.

INTRODUCTION

Industries have used different types of dyes resulted in the release of large amounts of toxic compounds into the environment. In general, Approximately 35-45% of these dyes remain in the wastewaters.^[1] Presence of these dyes diminishes the photosynthesis and causes many serious health problems for humanity.^[2] To overcome these problems, the wastewater from those industries must be treated before their discharge. Various physical and chemical methods have been used for toxic removal from wastewaters.^[3] One of these methods is metal oxides photocatalysis, and it has proven to be an effective in treating wastewater. The search for low cost and efficient photocatalyst is still continuing. Among many organic pollutants, malachite green (MG) is one of the pollutant color for environment undesirable which effects on esthetic of the environment.^[4] Thus, environmental contamination by these toxic chemicals has emerged as a serious global problem. On the contrary, bleached dye after degradation of solution is relatively less toxic and almost harmless.^[5] Second, dye-containing colored water is almost no practical use, but if this colored solution is bleached to give colorless water, then it may be used for some useful purposes such as washing, cooling, irrigation, and cleaning.^[6] Recently, photocatalytic reactions induced by illumination of semiconductors in suspension have been shown to be one of the most promising processes for the wastewater treatment.^[7]



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Keywords: Al_2O_3 , Nanoparticles,

Photocatalytic Received: 15th December 2017 Accepted: 26th January 2018

Published: 31th January 2018

Nano-sized metal oxide such as Al_2O_3 , TiO₂, ZnO, and Fe₂O₃ is often used as catalytic agents because of their high stability, low costs, high efficiency, and no toxicity.^[8] Among various metal oxide nanoparticles photocatalysts, aluminum oxide (Al_2O_3 ,) exhibit promising photocatalytic activities due to their environmental friendly behavior, low catalyst cost, high specific surface area, high crystallinity, and solar energy application and thus, could be an alternative material for environmental application and wastewater treatment.^[9] Al_2O_3 was used as photocatalyst under visible radiation for degradation of MG.

EXPERIMENTAL

Synthesis of photocatalyst

The Al_2O_3 , nanoparticles powder was prepared by urea decomposition method. The urea was corresponded to total volume ratio of metal nitrate, ratio of 1:2. In each case, aluminum nitrate dissolved in stoichiometric amounts of water, 10% then mixed with vigorous stirring at room temperature (55°C). The prepared slurry was left to stand for the formation of solid. After the solidification was completed, the solid was kept for 2 days at room temperature, and the sample was dried at 75°C for 36 h. After grinding the dried samples, they were calcined at 600°C for 2 h. Nano-sized materials of the catalyst were analyzed.

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Photocatalytic degradation studies

Photocatalytic study of the synthesized Al₂O₂, nanoparticles powder was evaluated by decolorization of MG dye in aqueous solution. The experiments were carried out in the presence of visible light irradiation without any catalyst (blank), with the catalyst in the dark and in the presence of Al₂O₂, photocatalyst. The photocatalytic reaction carried out in the glass beaker with sunlight direct exposer for 5 h. Reaction was set up by adding 0.3 g of Al₂O₂, Nanoparticles powder was mixed into 100 mL of MG solution, and this suspension was magnetically stirred in the dark for 20 min to obtain adsorption/desorption equilibrium before irradiating the sunlight in the beaker. Then, the suspension was exposed to sunlight; 9 mL of the sample was withdrawn for 1-h time interval over irradiation. The suspension was centrifuged at 1000 rpm for 10 min and filtered to remove the catalyst particles before measuring absorbance. The absorbance of the clear solution was measured at a λ max of 660 nm for quantitative analysis. Percentage degradation of MG dye was calculated using the following relation:

% degradation =
$$\frac{A_0 - A_t}{A_0} \times 100a$$

Where A_0 is absorbance of dye at initial stage A_t is absorbance of dye at time t.

Characterization

Fourier transform infrared (FTIR) is carried out from our institute using Perkin Elmer FTIR instrument ranges from 400 cm⁻¹ to 4000 cm⁻¹. X-ray powder diffraction (XRD) analysis was performed with Goniometer Ultima IV using a Cu K α radiation ($\lambda = 1.54060$ Å) operating at 40 kV and 40 mA. Absorbance carried out by spectrophotometer.

RESULTS AND DISCUSSION

FTIR studies

The reaction of aqueous solutions of urea with $Al(NO_3)_3$, produces a clear white colored oxide, Al_2O_3 . The formation of this oxide on the heating of an aqueous mixture of $Al(NO_3)_3$. For the reaction mechanisms, an oxidation process for aluminum urea complex occurs during the decomposition of urea into ammonia, carbon dioxide, and hydrogen chloride gases. The infrared spectra of synthetic oxide product are shown in Figure 1. The infrared spectra of the obtained products show bands due to characteristic groups of urea (carbonyl and amide groups) at 1102 cm⁻¹ and 1629 cm⁻¹, the bands associated to the O-H are observed at 3429 cm⁻¹ is due to moisture absorbed during measurement of spectra.

XRD studies

The phase formation and orientation of Al_2O_3 nanoparticles were investigated using XRD in the ranges (20–80°). XRD patterns of nanoparticles with shown in Figure 2. It was found that the presence of Al_2O_3 at temperatures 600°C. The XRD results also reveal the structural results for work and the values obtained using the Scherrer equation: $D = k\lambda/\beta \cos\theta$ where D is the crystallite size, λ is the wavelength of the Cuk α radiation, k is a constant equal to unity, β is corrected peak width at half maximum intensity, and θ is peak position (68.69° used for all lines). Crystallite size of Al₂O₃-NPs increases. The decomposition process is highly affected by the molar ratio.

Photocatalytic degradation studies

The photocatalytic activity of Al₂O₂ nanoparticles was evaluated by the degradation of MG dye in aqueous solution. The decolorization of the MG dye was examined under three different conditions (treatments): Sunlight irradiation without any catalyst (blank solution), in the presence of catalyst without light irradiation (in the dark) and in the presence of Al₂O₂ nanoparticles photocatalyst under sunlight irradiation, respectively. For the blank experiment (in the absence of the catalyst) under sunlight irradiation, almost insignificant degradation of the dye was observed. In the presence of Al₂O₂ nanoparticles, the formation of electrons-holes pairs is responsible for enhancing the oxidation and reduction reactions with the MG dye, which might be adsorbed on the surface of the Al₂O₂ nanoparticles to give the necessary products. The experimental results show that when the dye solution is exposed to sunlight irradiation for 5 h in the presence of Al₂O₂ nanoparticles. The degradation of MG dye as a function of time under sunlight irradiation in the presence of Al₂O₂ nanoparticles as shown in Table 1. Accordingly, the degradation efficiency of MG dye under the sunlight was found to be much larger than the degradation efficiency as compare to blank and dark treatment. This enhancement under sunlight in the presence of Al₂O₂ nanoparticles, the first one could be the fact that the Al₂O₂ nanoparticles prepared by the urea decomposition method have a high specific surface area, that could give more active surface sites to absorb water molecules and to form active •OH

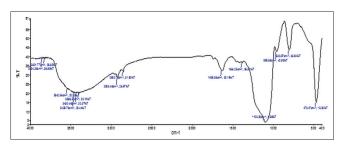


Figure 1: Fourier transform infrared of Al₂O₃ nanoparticles

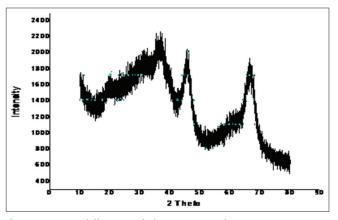


Figure 2: X-ray diffraction of Al₂O₃ nanoparticles

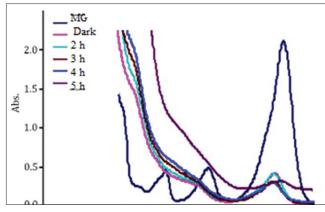


Figure 3: Graphical representation of absorption under ultraviolet visible

Table 1: Measurement of absorbance of suspension $(Al_2O_3 NPs and MG dye)$

Time in hours	Absorbance intensity (approximately)
2	2.0
3	0.4
4	0.3
5	0.2

and HOO• radicals by trapping the photogenerated holes. This free active radical drive the photodegradation reactions and eventually leads to the decomposition of organic pollutants in aqueous solution. Under sunlight irradiation, MG molecules are absorbed on the surfaces of nanoparticles and produced electrons. These electrons are captured by the surface adsorbed O_2 molecules to yield $O_2 \bullet -$ and $HO_2 \bullet$ radicals, which makes more chance to touch with dye molecules and giving a faster reaction speed then, the MG molecules could be mineralized in time by the superoxide radical ions. Therefore, it can be concluded that the smaller crystalline size of nanoparticles is favorable for the reduction of O_2 and oxidation of H_2O molecules by trapping electrons and holes, which improves the photocatalytic activity.

CONCLUSION

The phase of Al_2O_3 nanoparticles can successfully be synthesized by urea decomposition method using aluminum nitrate, at room temperature then the burnt product was calcined at 600°C for 2 h. The prepared sample was characterized using different tools; FTIR, XRD, and ultraviolet. Al_2O_3 NPs with average crystallite size 30.096 nm approximately was obtained at 600°C. The decomposition process is highly affected by the molar ratio. The produced Al_2O_3 NPs showed photocatalytic activity by degradation of 85% approximately of the MG dye, under sunlight irradiation, respectively, within 5 h in overall studies it is concluded that the Al_2O_3 NPs showed photocatalytic activity and it can be used as best degradation agent.

ACKNOWLEDGMENT

The authors are thankful to Solapur University, and S.A.P.D. Jain Pathshala's Walchand College of Arts and Science, Solapur, for providing access to instrumentation.

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Cite this article: Ali T, Mahesh CP, Venkatraman A. Preparation and Photocatalytic Activity of Aluminum oxide (Al_2O_3) Nanoparticles. Asian J Appl Res 2018;4(1):4-6

Source of Support: Nil, Conflict of Interest: None declared.