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# The Photocatalytic Activity Under Sunlight Illumination of Organic Dyes Over the Spinel $MgAl_2O_4$

Djilali Mohamed Amine<sup>1,2\*</sup>, Mellal Mounir<sup>1</sup>, Mekatel Elhadj<sup>2</sup>, Boudiaf Salim<sup>3,1</sup>

<sup>1</sup>Laboratory of Recovery and Recycling of Matter for Sustainable Development, Faculty of Mechanical Engineering and Process Engineering, USTHB, BP 32, Algiers, Algeria.

<sup>2</sup>Laboratory of Transfer Phenomena, Faculty of Mechanical Engineering and Process Engineering, USTHB, BP 32, Algiers, Algeria.

<sup>3</sup>Laboratory of Reaction Engineering, Faculty of Mechanical Engineering and Process Engineering, USTHB, BP 32, Algiers, Algeria.

\*Corresponding author's email: mohamedaminedjilali91@gmail.com

## ABSTRACT

We report in this study, green, expedient, cost effective and environment friendly synthesis of  $MgAl_2O_4$  spinel with a nitrate route and their potential application in the field of Photocatalytic degradation of organic pollutant. The synthesized spinel was characterize by XRD, SEM, EDX, UV-Visible spectra, and BET for obtaining the structural, morphological, optical, and textural properties with an energy gap  $E_g = 3.1$  eV. The sunlight driven photocatalytic degradation efficiency of this catalyst was performer on a widely used industrial dye Methyl orange (MO) which is a major contaminant that is being mixer in water resources. The contamination could be degrade up to 89.23% when its concentration is 15 mg/L at pH 6, by the spinel of 0.5 g/L.

**Keywords:**  $MgAl_2O_4$ ; Methyl orange; Photodegradation; nitrate route.

## 1 Introduction

The production of textiles, cosmetics, paints and leather yields large amounts of organic contaminants that contain dyes. Producers in many countries routinely discharge these contaminants in wastewater, which then pollutes natural water resources. The rapid growth in the output of these industries is exacerbating this environmental problem. Wastewater containing dye molecules and other organic contaminants is treat with several techniques. Some treatment methods use physical processes such as adsorption [1], and other methods use chemical processes such as coagulation-flocculation and oxidation [2]. In recent years, wastewater treatment with photo catalysts has attracted attention because the photocatalytic process is a practical, environmentally friendly, sustainable and economical technique [3]. A variety of materials has been investigate in attempts to improve photocatalytic wastewater treatment, and interest in the unique properties of semiconductors. The chemical, thermal, dielectric, mechanical and optical properties of  $MgAl_2O_4$  spinel powder make this particular material one of the most interesting metal oxide semiconductors. The interest in  $MgAl_2O_4$  spinel powder has led to its use as a photo catalyst in the treatment of wastewater containing dye pollutants. They found that phase purity, specific surface area, light absorption ability, synergistic effect, charge transfer and recombination of photo-excited electron-hole pairs affected the photocatalytic activity of the composite.

## 2 Experimental

### 2.1 Preparation of the spinel $MgAl_2O_4$

An effective powder in photo catalysis must be former by crystallites as small as possible with more or less regular shapes. Stoichiometric amounts of  $Mg(NO_3)_2 \cdot 6H_2O$  and  $Al(NO_3)_3 \cdot 9H_2O$ , were dissolved in distilled water, under magnetic stirring for 120 min to ensure the homogenization of the mixture. The latter was treat at 100 °C for a gradual evaporation, followed by heating at 350 °C for the nitrates elimination.



Finally, the amorphous white powder was ground in an agate mortar and calcined at 1000 °C in a programmed muffle furnace with a heating rate of 10 °C min<sup>-1</sup> for 240 min. This operational process was repeated three times to have a single phase.

## 2.2 Characterization

The XRD pattern was recorded with a PANalytical diffractometer using mono-chromatid Cu K line ( $\lambda = 0.15418$  nm). The morphology of the spinel MgAl<sub>2</sub>O<sub>4</sub> was observed by scanning electron microscopy (SEM, Philips XL30 ESEM-EDS). The specific surface area ( $S_{\text{BET}}$ ) was determined by using an ASAP-2010, Micrometrics V 5.01 G equipment. The powder was degassed at 140 °C overnight and the  $S_{\text{BET}}$  value was computed from the BET equation.

## 2.3 Photocatalysis

The MO photodegradation on the MgAl<sub>2</sub>O<sub>4</sub> was performed in a double-walled Pyrex reactor (200 cm<sup>3</sup>); the temperature was fixed at 20 °C thanks to a thermostat bath. The reactor was exposed to the Sunlight source. The operational parameters such as (MO) initial concentration ( $C_0$ ) were studied. In order to obtain the optimum value for the parameter, we varied it while keeping the other constant. The aliquots were regularly withdrawn and centrifugation (HETTICH Zentrifugen Universel 320, 4,000 rpm, 10 min) to separate the solid particles from the supernatant. The residual MO concentration was titrated by UV-Visible spectrophotometry. The Photodegradation percentage of MO was determined according to the relation:

$$\text{photodegradation \%} = \left( \frac{C_0 - C_t}{C_0} \right) \times 100$$

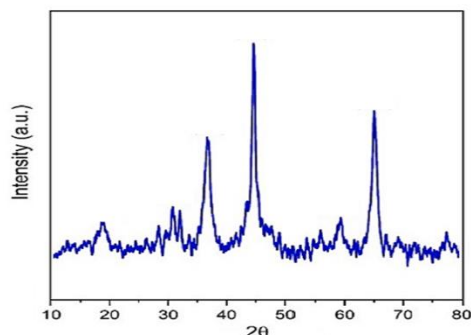
$C_0$  and  $C_t$  are the concentration of the Methyl orange solution at initial time and time ( $t$ ) respectively.

## 3 Results and Discussion

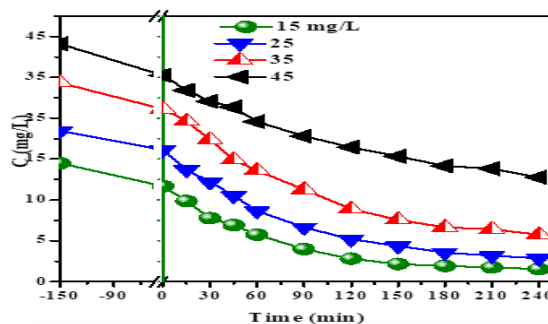
The x-ray diffractogram of material magnesium aluminate is depicted in **Figure1** with heat treatment at 1000°C. The major peaks resulting from pure MgAl<sub>2</sub>O<sub>4</sub> nanomaterial can potentially be visible in the diffractogram accordingly which are confirmed by employing **JCPDS card N° 01-075-1797** as the point of reference. The structural pattern displayed is consistent with MgAl<sub>2</sub>O<sub>4</sub> with type spinel, and the material that was treated exhibited peaks with good intensity.

### 3.1 Effect of initial concentration

The effect of initial concentrations ( $C_0$ ) is interesting to study and the photo-degradation of MO has been studied in the range (15 - 45 mg/L), keeping constant the catalyst dose MgAl<sub>2</sub>O<sub>4</sub> at 0.5 g/L and pH 6 (**Figure2**). As expected, MO degradation occurs rapidly for low  $C_0$  values (15 mg/L) and a photo-degradation of MO equal 89.23 %. This is due to the lack of adsorbed MO molecules and the availability of reactive species ( $h^+$ ,  $e^-$ ,  $O_2^{\cdot-}$ ,  $\cdot OH$  ...). On the contrary, increasing the concentration of  $C_0$  dye decreases the luminous flux, in accordance with the Beer-Lambert law, and weakens the number of photons reaching the surface of the catalyst thus producing fewer reactive radicals.



**Figure1.** The X-ray diffraction pattern of  $\text{MgAl}_2\text{O}_4$  prepared by nitrate route



**Figure2** Effect of the initial concentration on the MO Photodegradation onto the spinel  $\text{MgAl}_2\text{O}_4$  (pH ~ 6 and dose = 0.5 g/L)

#### 4 Conclusions

The present work has many advantages over conventional methods by having easy and environmentally benign procedures. The synthesis of the  $\text{MgAl}_2\text{O}_4$  spinel and the fast elimination of MO using the derived nanoparticles are reported for the first time. Through BET analysis, the mesoporous nature of the spinel was confirmed and a surface area was obtained, which was found to be 36.22 m<sup>2</sup>/g. The optical band gap energy of the material was found to be 3.1 eV. Through a UV–visible study. The crystalline planes obtained through XRD studies confirmed the cubic crystalline nature of spinel. In addition, SEM images showed the structure of the catalyst, which enabled superior activity in both Photodegradation and heterogeneous catalysis. Only 0.5 g/L of the spinel, even in 240 min, successfully removed 89.23 % of the 15 mg/L (MO) dye when the suspension was exposed to sunlight.

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