

Synthesis and Characterization Of CaMgO₂ Nanoparticles Photocatalyst For the Decolorization Of Orange G Dye

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ABSTRACT

A series of heterogeneous basic catalysts of CaO, MgO and CaMgO₂ at different calcination temperature were synthesized via solution combustion method. Different characterization techniques have been carried out to investigate the structure of the produced catalysts i.e. X-ray diffraction (XRD), particle size analyzer, morphology by atomic force microscope (AFM) and reflection using UV-VIS diffuse reflectance spectra. The particles size analyzer revealed that the mixed oxide catalysts calcined at different calcination temperature possess smaller nano size particles compared to pure CaO. Moreover, the energy band gap was calculated based on the results of diffuse reflectance spectra. The energy band gap was reduced from 4.1 to 3.6 eV for the CaMgO₂ catalyst calcined at 400 and 600 °C respectively compared to pure oxide catalysts. The higher decolorization efficiency was 100% after 60 min of photocatalytic reaction for CaMgO₂ calcined at 600 °C compared to CaO and MgO with catalytic activity of 58% and 27% respectively.

Key wards: mixed oxide catalyst, photocatalytic reaction, calcium oxide, magnesium oxide

تصنيع وتشخيص اكاسيد الكالسيوم/المغنيسيوم النانوية كعامل مساعد ضوئى لازالة صبغة الاورانج ج

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الخلاصة

تهدف الدراسة الى تحضير مجموعة من العوامل المساعدة ذات الصفة القاعدية مثل اوكسيد الكالسيوم، اوكسيد المغنيسيوم و خليط من اكاسيد الكالسيوم / المغنيسيوم عن طريق عملية الحرق الحراري. تم تشخيص بنية المواد المصنعة باستخدام عدة تقنيات ومنها اشعة الحيود السينية (XRD)، جهاز قياس حجم الجزيئة، وقياس فجوة الطاقة بواسطة AFM. spectra و مجهر القوة الذرية AFM.

اثبتت نتآئج اشعة الحيود السينية للعامل المساعد المصنع من خليط اوكسيد الكالسيوم/المغنيسيوم بوجود كل من طور اوكسيد الكالسيوم واكسيد المغنيسوم والذي يثبت نجاح عملية تحضير العامل المساعد . اثبتت نتائج قياس حجم الجزيئة بانه مجموعة العوامل المساعدة المصنعة والمعاملة حراريا بدرجات حرارة مختلفة تمتلك حجوم نانوية اصغر مقارنة باوكسيد الكالسيوم. تم احتساب قيم فجوة الطاقة من خلال نتائج diffuse reflectance spectra واظهرت قيم فجوات الطاقة بانها تما لي الى على ع 3.6 eV والم الموانة المسببة لخليط اوكسيد الكالسيوم/ما والزيا بدرجة حراريا بدرجات حراريا بدرجة حراريا بدرجة حراريا والم المواني معام المواني من 4.1 eV المواني معام المواني معام المواني معام المواني من 4.1 eV المواني معام المواني معام المواني معام المواني من 4.1 eV المواني المواني معام المواني المواني المواني معام المواني من 4.1 eV المواني معام المواني من 4.1 eV المواني المواني معام المواني المواني معام



بالاكاسيد الاحادية المصنعة. بعد استكمال عملية التشخيص للعامل المساعد المصنع تم دراسة كفاءة العامل المساعد على ازالة الملوث المتمثل بصبغة الاورانج ج وكانت اعلى كفاءة للعامل المساعد قد وصلت الى 100% بعد 60 دقيقة من وقت التفاعل الضوئي لخليط اوكسيد الكالسيوم/ المغنيسيوم المعامل حراريا بدرجة 600 درجة مئوية مقارنة بأوكسيد الكالسيوم واوكسيد المغنيسيوم الذي اظهر كفاءة 58% و 27% على التوالي.

الكلمات الرئيسية : خليط من اكاسيد المحفز ات، تفاعلات ضوئية بوجود العامل المساعد، اوكسيد الكالسيوم، اوكسيد المغنيسيوم

1. INTRODUCTION

Dyes cause damage to the environment because they prevent sunlight and oxygen penetration. Therefore, they can significantly affect photosynthetic activity in aquatic systems **Dizge, et al., 2008**. A wide range of treatment methods have been developed for the removal of dyes from water and wastewater to eliminate their harmful effect on environment.

A great interest has been given to advanced oxidation processes (AOPs) due to the alternative destructive treatment in which the chemical species are reduced into smaller fragments and even to the point of mineralization. AOPs such as ozonation, photocatalysis, ultrasonic reaction, Fenton and a combination of photo Fenton, UV/O_3 and UV/H_2O_2 are novel technologies that have been widely developed to enhance their ability to generate the high reactive hydroxyl radicals ('OH) that have a high efficiency to oxidize organic matters **Jamalluddin**, and Abdullah, 2011. However, the high running cost is the main drawbacks of these processes. Thus, there is a need to use a catalyst with such a process to increase the efficiency and reduce the coast effect.

Among these advanced oxidation processes, the use of heterogeneous photocatalytic technology is more attractive process for the degradation of organic dyes contrary to physical process; it can facilitate the complete mineralization of organic compounds to carbon dioxide, water and mineral acids **Gopalappa**, et al., 2012. As a heterogeneous photocatalyst, metal-oxides are a promising materials because of the wide variety of physical properties they possess, which make them attractive for a wide range of applications such as photovoltaic devices, gas sensors, micro-electronics and corrosion protection devices. The synthesis of metal and metal oxide materials has attracted considerable attention in physical, chemical, biological, medical, optical, mechanical and engineering sciences where novel techniques are being developed to probe and manipulate single atoms and molecules **Prabhavathi, et al., 2014**.

Metal- oxide including semiconductors such as calcium oxide are inexpensive, non-toxic and capable of extended use without substantial loss of photocatalytic activity **Gaikwad**, et al., 2012. The activity and stability of CaO can be improved by mixing with other metal oxides such as MgO, La_2O_3 and CeO₂ Teo, et al., 2014. The combination between two semiconductors provides a promising way to select the efficient charge transfer between them, further to reduce the rate of electron-hole recombination Hassan, et al., 2014. Recently, many studies have been focused on the synthesis of mixed oxide catalyst and therefore various methods have been used to synthesize. These methods include, sol-gel, hydrothermal, flame spray pyrolysis, chemical gas phase deposition, combustion and aqueous wet chemical. However the morphology and



characteristic of the materials are differing and depend largely on the synthesis method plus processing condition **Badar**, et al., 2012.

In the present study the synthesis of $CaMgO_2$ and its application on color removal for Orange G dye solution as a catalyst under UV irradiation has been reported. The CaO, MgO and CaMgO₂ have been synthesized using thermal combustion method. In comparative studies of photo degradation efficiency of these materials, the CaMgO₂ is found to be more efficient than CaO. Different characterization techniques were conducted including characterization technique using X-ray diffraction, FTIR, diffuse reflectance spectra and AFM.

2. MATERIALS AND METHODS

The pure metal calcium and magnesium nitrate were purchased from GPR and BDH respectively. Meanwhile, hydrogen peroxide (30% analytical grade) was supplied from Analar. The urea was purchased from Merck. The pollutant model Orange G dye was obtained from Fluka and its properties are listed in **Table 1**.

2.1 Preparation method

Pure Calcium and magnesium oxide in addition to their mixed oxide catalyst $CaMgO_2$ were prepared by thermal combustion method **Gopalappa**, et al., 2012. A known amount of $Ca(NO_3)_2.4H_2O$ and $Mg(NO_3)_2.6H_2O$ were dissolved in minimum quantity of water along with urea in a silica crucible. The mixture was introduced into muffle furnace which was preheated at 500 °C. In both experiments the reaction mixture undergoes dehydration followed by decomposition with the release of CO_2 , N_2 and H_2O . The final product of combustion was a solid powder CaO, MgO and CaMgO2 respectively. The obtained catalysts were crushed and grounded separately to make them amorphous. According to propellant chemistry the reactions are as shown.

 $3Ca(NO_3)_2 + 5NH_2CONH_2 \longrightarrow 3CaO + 5CO_2 + 10H_2O + 8N_2$ $3Ca(NO_3)_2 + 3Mg(NO_3)_2 + 10NH_2CONH_2 \longrightarrow 3CaMgO_2 + 10CO_2 + 20H_2O + 16N_2$

3. CHARACTERIZATION TECHNIQUES

The characterization of the catalysts with different analytical techniques was performed on the catalysts. X-ray diffraction patterns were obtained by means of XRD (Philips Goniometer PW 1820) diffractometer. The FTIR spectroscopic analysis was carried out using Bruker Alpha spectrophotometer. Meanwhile, the reflectance of the solid samples was determined using UV-Vis spectrophotometer (Perkin Elmer) in a wavelength range between 190 and 1,300 nm. The roughness of the catalyst surface was checked using AFM technique. The particle size analyzer was carried out by Brookhaven instrument.



3.1 Reaction procedure and analytical method

The photo-efficiency of all synthesized catalysts was tested towards degradation of orange G dye which was used as a pollutant model in this reaction. The experimental runs were carried out in a 250 mL conical flask as a reactor equipped with continuous mechanical stirrer. Thereafter, the dye solution was exposed to UV light with constant stirring for 1 h after the addition of solid catalyst and hydrogen peroxide . The samples were taken at periodic intervals i.e. (10 min).

The maximum absorption of the dye was detected using spectrophotometric analysis with maximum absorption of the dye at 478 nm. The concentration of the dye solution was calculated based on the maximum absorbance wavelength using Beer-Lambert law. The decolorization efficiency of the catalyst was calculated according to the following equations:

$$DE \% = \left[1 - \frac{c_t}{c_*}\right] \times 100 \tag{1}$$

where, DE% is the decolorization efficiency, C_{o} (mg/L) is the initial concentration of dye, C_{t} (mg/L) is the concentration of dye at certain reaction time and t (min).

3.1.1 X-ray diffraction

The XRD pattern of CaO, MgO and CaMgO₂ calcined at 400 °C, 600 °C and 700 °C respectively, are shown in **Fig.1**. The parent CaO and MgO in **Fig. (a)** and **(b)** shows high crystalline structure where the highest peaks intensity were at 20 of 37.08° , 43.01° and 62.41° for MgO catalyst. Meanwhile, the CaO catalyst shows diffraction peaks at 18.10° , 34.20° , 47.25° and 50.97° respectively. The diffraction peaks of CaO match well the reference database number of 96-100-8782. Watcharathamrongkul, et al. 2010 reported that these peaks were ascribed to the portlandite phase. **Galván-Ruiz1**, et al., 2016 reported that the presence of portlandite is due to the spontaneous reaction between CaO and moisture.

For MgO the peaks match well the MgO phase (periclase) with reference database number of 96-100-0054. This result was also similar to that of **Tamilselvi, et al., 2013**. On the other hand **Fig. 1 (b)** shows the CaMgO₂ calcined at different temperature, i.e. 400 °C, 600 °C and 700 °C the results shows high crystalline structure for all the samples. As the temperature was above 400 °C the structure was more crystalline and the diffraction peaks were almost the same at 600 °C and 700 °C at 18.18°, 34.20°, 43.01° and 62.33° which was ascribed to a mix between calcium and magnesium oxide phases. The existence of both phases improves the success of preparation method.

3.1.2 Particle size analyzer

Fig. 2 presents the particles size analyze for CaO mono catalyst and CaMgO₂ calcined at 400 °C, 600 °C and 700 °C respectively. The results showed that the mixing between the two metals clearly reduced the particles size for those calcined at different calcination temperature. At 400 °C the particles size was the larger i.e. 1530 nm due to the low calcination temperature that the reaction between the urea as fuel and calcium, magnesium nitrate may be uncompleted during the preparation method. This can be indicated from the nature of the product as foam structure produced after calcination temperature. **Gopalappa, et al., 2012** also reported the formation of foam structure



due to the reaction between urea and metals however, as the calcination temperature was increased the reaction was much faster and the product was as solid white powder. Here it should be highlighted that as the calcination temperature was increased the particle size of the produced catalyst was decreased due to the strong interaction between the materials. **Teo, et al., 2014** reported that the difference in ionic radius and molar ratio between the mixed metals will obviously affect on the crystalline size of the mixed oxide metals and subsequently affected its particle size.

3.1.3 UV-vis diffuse reflectance spectra analysis

The UV-VIS diffuse reflectance spectra was obtained for the developed catalysts in order to calculate the difference in energy gaps for mono and binary oxide catalyst which can give a clear indication about the successful mixing between these metals after the preparation methods as in **Fig.3**. The UV-vis reflection spectra were carried out for calcium and magnesium oxide catalysts as mono oxide materials in addition to CaMgO₂ catalyst at different calcination temperature as in **Fig.3** The mono oxide catalysts i.e. CaO and MgO calcined at 600 °C shows a reflection spectrum at 367 and 344 nm respectively. Meanwhile CaMgO₂ catalyst calcined at 400, 600, and 700 °C showed a reflection spectra at 331, 375, 364 nm respectively. The energies band gap for the prepared samples was calculated from their reflection spectra as in **Table 2**.

The band gap values were determined from the extrapolation of the straight line for the graph plotted between the square Ln $[(R_{max}-R_{min})/(R-R_{min})]$ versus hv (as abscissa) **kumar, et al., 1999**. **Table 2** lists the calculated values of the band gap energies for all the prepared samples based on the method suggested by **Kumar et al. (1999)**.

According to the above results, the mixing between calcium and magnesium oxide clearly change its optical properties consequently affected the energy band gap. Furthermore, with increasing calcination temperature from 400 to 600 °C the band gap energy was decreased. This behavior could refer to the effect of calcination temperature on the particle size thus affected the optical properties. This was approved in the above section for particle size analyzer. However, as the temperature was increased from 600 to 700 °C the band gap was increased a gain to 3.9 eV. This increment in band gap energy was ascribed to the quantisazation effect as the particle size of the CaMgO₂ calcined at 700 °C was less compared to that CaMgO₂ calcined at 600 °C.

3.1.4. Atomic force microscopy (AFM)

The surface uniformity of the bare CaO and MgO in addition to its mixed oxide catalyst calcined at different temperatures i.e. $CaMgO_2$ calcined at 400 °C, 600 °C and 700 °C respectively were analyzed using AFM technique. **Fig. 4** presents the AFM images in the three dimensional (3-D) forms for all types of catalyst used. The results show that the CaO and MgO have an average roughness values of 0.857 and 0.928 nm, respectively. Meanwhile, as the calcination temperature was increased from 400 to 600 the average roughness was decreased from 1.26 nm to 0.901 nm. These results were in agreement with the results of particle size analyzer as the average roughness value was decreased with decreased in particle size. However, as the calcination temperature was



increased to 700, the average roughness value was slightly increased to 0.997 nm compared to that at 600. This slight increment could be referring to non uniformity of catalyst surface during the testing of AFM technique.

4. CONTROL EXPERIMENT

Photocatalytic or photolysis experiments were carried out as in **Fig. 5** to ascertain that the color removal of the Orange G dye was truly photocatalytic and not just due to photolysis process. The decolorization of Orange G dye was investigated by photolysis alone in the absence of catalyst with initial dye concentration of 10 mg/L, 1.5 g/l of catalyst loading, 0.1 ml/100 ml of reaction and 60 min of reaction at ambient temperature and the pH of the dye is 9. The results indicated that no color removal was obtained after 60 min of reaction. This low activity of the decolorization was ascribed to the insufficient amount of •OH radicals generated by photolysis alone since radicals in this case were generated only through the dissociation of water molecules. Furthermore, the short life time of these radicals could reduce their chance to react with the dye molecules thus, reducing the probability to obtain a complete decolorization efficiency **Jamalluddin, and Abdullah, 2011**.

A preliminary study for different heterogeneous catalysts i.e. MgO, CaO and CaMgO₂ was also performed in order to investigate the effect of mono or co-mixed oxide catalyst on the decolorization efficiency of Orange G dye in the presence of photolysis irradiation. The MgO and CaO catalysts showed an adsorption rate of 19 % and 12.7% respectively, after 30 min of mechanical stirring followed by 60 min of photocatalytic reaction with decolorization efficiency of 27 % and 58% respectively. On the other hand, the CaMgO₂ showed higher catalytic activity of 100% after 60 min of photocatalytic reaction due to the higher adsorption rate i.e. 39% compared to those of mono oxide catalysts CaO and MgO. These significant differences in adsorption rate and catalytic activity for CaMgO₂ compared to CaO and MgO were ascribed to the new crystalline structure formed after co-mixing between calcium and magnesium oxide. Furthermore, the enhancement in energy gap value of CaMgO₂ calcined at 600 °C(3.6 eV) was less compared to calcium and magnesium oxide i.e. 4 eV, 4.9 eV respectively as it was detected by diffuse reflectance spectra technique. **Al-Johani**, *et al.*, **2015** reported that the incorporation between two different metals results in a significant variations in the energy band gap, thus enhance

4.1 Effect of calcination

As the higher photocatalytic activity for decolorization of Orange G dye was with $CaMgO_2$ catalyst. Further experiment was carried out to investigate the effect of calcination temperature on the catalytic activity of the catalyst. The parameter of the reaction was fixed at an initial dye concentration of 10 mg/L, a catalyst loading of 1.5 g/L and H_2O_2 concentration of 0.1ml/100ml of reaction. Prior to the photocatalytic reaction, the solution was first stirrer for 30 min at room temperature to maintain a good dispersion of catalyst with the dye solution. As shown in **Fig. 6**, the absorption ability was increased from 14% for CaMgO₂ calcined at 400 °C to 38 % and 36 % for CaMgO₂ calcined at 600 °C and 700 °C respectively. Consequently, as the photocatalytic reaction



was started, the catalytic activity was higher at 400°C however, this catalyst was almost soluble during the reaction thus it behaves as homogeneous catalyst. This was ascribed to the effect of low calcination temperature as the catalyst was looks like foam structure. As such, it was more efficient to choose the CaMgO₂ catalyst calcined at 600 °C as the best catalyst with maximum catalytic activity 100% after 60 min of reaction.

On the other hand, as the calcination temperature was increased to 700 °C, the decolorization efficiency was also 100% after 60 min of photocatalytic reaction however obviously the reaction was much slower compared to the reaction at 400 and 600 °C. This could be ascribed to the effect of higher the energy gap of 3.9 eV compared to that of CaMgO₂ calcined at 600 °C which has an energy gap of 3.6 eV. Although the energy gap of CaMgO₂ remains in UV region, however, light energy was sufficient to cause excitation of CaMgO₂ particles. Consequently it caused higher photocatalytic activity compared to calcium and magnesium oxide.

4.2 Effect of pH

Effect of initial pH of the solution on decolorization efficiency of Orange G dye was studied at different initial pH levels (7, 9 and 12). The parameter of the reaction was fixed at an initial dye concentration of 10 mg/L, a catalyst loading of 1.5 g/L, and a H_2O_2 concentration of 0.1 ml/100 ml or reaction volume. Prior to the photocatalytic reaction, the solution was first stirrer for 30 min at room temperature to maintain a good dispersion of catalyst with the dye solution. As shown in **Fig. 7**, the absorption capacity was increased with an increment in pH value i.e. 20%, 40% and 82% for pH 7, 9 and 12 respectively.

As the photocatalytic reaction was started, the maximum decolorization efficiency was higher at pH 9 with almost 100 % within 60 min of reaction time. However, lower catalytic activity was obtained at pH 7 and 12 with maximum decolorization efficiency off 7 and 36 % respectively. **Chang, et al., 2013** reported that the addition of HCl solution which was used to adjust solution pH value effect on reaction medium. At this operating condition, there were abundant amounts of high concentration chloride ions in the solution to play the role of free radical scavenger. Chloride ions competed with OG molecules to consume free radicals. Therefore, the decolorization rate of OG by CaMgO₂ system decreased at pH 7. Meanwhile the reason behind the reduction in catalytic activity at pH 12 could be ascribed to the effect of excess amount of OH⁻ species within solution that can play the role of radical's scavenger.

4. CONCLUSION

The results of this work revealed that the mixed oxide $CaMgO_2/H_2O_2/UV$ system could be efficiently used for the decolorization of azo dyes instead of mono oxide catalyst due to its higher catalytic activity. The mix between calcium and magnesium oxide enhance the optical properties of the produced catalyst i.e. $CaMgO_2$. Meanwhile, the particles size analyzer revealed that the mixed oxide catalysts calcined at different calcination temperature possess smaller nano size particles compared to pure CaO. The surface uniformity of the $CaMgO_2$ calcined at different calcination temperature was in good agreement with the particle size analyzer technique. In control experiment, the $CaMgO_2$ shows



higher catalytic activity compared to CaO and MgO. The maximum photocatalytic activity was for the decolorization of Orange G dye was 100% at initial dye concentration of 10 ppm, 0.1 ml of $H_2O_2/100$ ml of reaction, 1.5g/l of catalyst loading and pH 9.

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Molecular structure	OH NN SO ₃ Na SO ₃ Na
Other names	Acid Orange 10
Molecular weight	452.38 g/mol
Color Index	16230
λmax	478 nm

Table 1. The structure and properties of Orange G dye.

Table 2. List of energy gap values for different prepared catalysts.

Type of Catalyst	Energy gap eV
CaO	4.2
MgO	4.9
CaMgO ₂ 400 °C	4.1
CaMgO2 600 °C	3.6
CaMgO2 700 °C	3.9





Figure 1. Xrd of different prepared catalysts.





Figure2. Particle size analyzer.



Figure 3. UV-VIS diffuse reflectance spectra.





Figure 4. Atomic force microscopy of CaMgO₂ calcined at different temperature compared to CaO and MgO.



Figure 5. Control experiment study for decolorization of orange G dye under various conditions. (Initial dye concentration 10 mg/L, 1.5 g/l of catalyst loading, 0.1 ml/100 ml of reaction and pH 9).



Figure 6. Effect of calcination temperature for decolorization of orange G dye under various conditions. (Initial dye concentration 10 mg/L, 1.5 g/l of catalyst loading, 0.1 ml/100 ml of reaction and pH 9).



Figure 7. Effect of pH on the decolorization efficiency orange G dye at initial dye concentration 10 mg/l, 1.5 g/l catalyst loading and 0.1 ml H₂O₂/100ml reaction volume.