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Abstract. ZnAl2O4: Mn4+ particle were synthesize using co-precipitation using NaOH and PVA as precipitating agent and surfactants, followed by microwave-assisted thermal treatment. The morphology properties and absorption spectral of particles were investigate using SEM and UV-Vis spectrometer. The results showed that the $ZnAl_2O_4$ particles has a spinel structure with various particle size. Absorption spectral exhibits that particle has a potential photo catalytic properties at visible light spectra, especially for the pollutant degradation.

1. Introduction

Zinc aluminate (ZnAl2O4), related to the electronic structure Fd3m [1], is a semiconductor material with a wide energy band gap (3.8-3.9 eV). ZnAl2O4 material has superior semiconductor properties, such as low surface acidity levels, high thermal stability, electronic and optical properties, and hydrophobic. Therefore, these materials could be develop as electroluminescence material, sensor, or a heterogeneous catalyst [2, 3].

In recent years, these various physical and chemical processes have been used to synthesize ZnAl2O4, such as sol-gel method [3], solid state reaction [4], hydrothermal [5], deposition [6] etc. Compared with other methods, the deposition method is faster, high levels of chemical homogeneity, easy in setting the composition and structure, and setting the particle size and morphology. However, this method requires a sintering process in order to produce the proper oxide particles.

In this paper, $ZnAl_{2-x}O_4$:xMn^{4+} (x=1%) have successfully synthesized using the co-precipitation method.

Moreover, the process of microwave heating were use as part of the process of sintering material ZnAl2O4:Mn. Microscopic structure, morphology and optical properties of the sample of interest to be investigate and discussed closely.

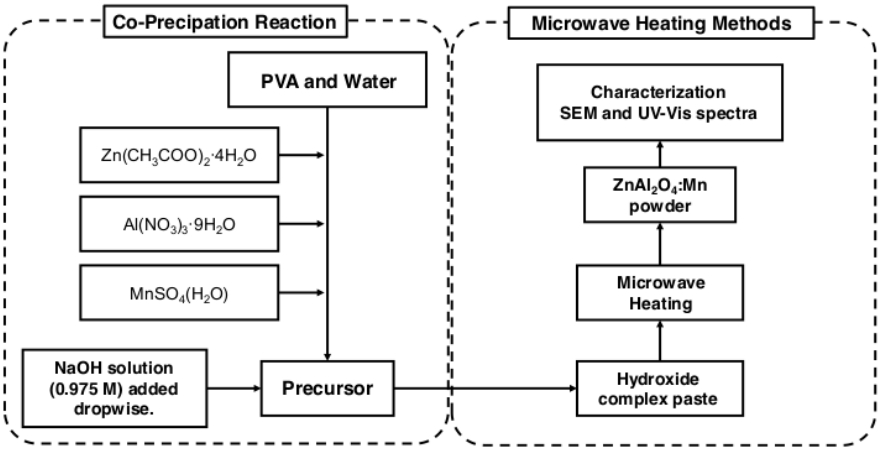

Figure 1. Synthesis scheme of ZnAl2O4:Mn particle using co-precipitation methods with microwave thermal assisted.

2. Experiment

Manganese doped Zinc Aluminate (ZnAl2O4:Mn) synthesized using 0.5 g zinc acetate tetrahidrate (Zn(CH3COO)2•4H2O, BM= g/mol), 1.708 g aluminum nitrate nonahidrate (Al(NO3)3•9H2O, BM= g/mol) dan 0.0025 g mangan sulfat hidrate (MnSO4(H2O), BM= g/mol) as zinc, aluminum and mangan source, respectively. All of these chemicals will produce a material with the following composition ZnAl1,99O4: 1% Mn4+. First, 0.01 g of PVA (polyvinyl alcohol, BM = g / mol) as a surfactant dissolved in 100 ml of pure water. After that, all basic materials are dissolve to obtain a clear precursor solution. Then, a solution of NaOH (0.78 g NaOH in 20 ml of water solvent) is slowly dripped into a precursor solution (drop wise). From this process,coloured solution became white and a precipitate formed. Subsequently, the precipitate is separated from the solvent water using filter paper until obtain white paste. The white paste is a complex particles hydroxide ($Zn(OH)_2$, $Al(OH)_3$, and $Mn(OH)_2$). To determine the effect of microwave heating on particle morphology $ZnAl_2O_4$:Mn, 1g complex hydroxide paste sample were heated using the microwave (800 watts) for 0 - 2 minutes, until white ceramic powder produced. The morphology and absorbance spectra of $ZnAl_2O_4$:Mn particle characterized using Scanning Electron Microscope (JEOL JCM-6000 NeoScope Benchtop SEM, Japan) and UV-Visible Spectroscopy (Home-Made Spectroscopy from LD-Didactic).

3. Results and Discussion

Figure 2 is a SEM image of the sample ZnAl2O4: Mn for some time variations microwave heating. Results precipitation reaction in the form of a white paste that we call complex hydroxide particles has a size in the range of 400 nm. Figure 2 (a). SEM observation showed that the hydroxide particles covered by the polymer layer of PVA. These causes the particles are not mutually agglomeration, and produce a particle size that is small enough. Microwave heating causes the particles undergo localized heating process. This is due to the microwave energy is absorbed by the particle turns into heat which is sufficient for the oxidation processes. Therefore, microwave heating transform complex particles into particles ZnAl2O4: Mn. In addition, the length of time the heating causes agglomeration caused by high heat and energy loss due to the polymer layer decomposes during microwave heating process, see Figure 2 (b) and 2 (c). It is characterize by an increase in particle size $ZnAl_2O_4$:Mn as the length of time the microwave heating, from 1.6 μm to 2.3 μm (Figure 2 (d)). This observation indicates that microwave heating is very effective in the process of sintering and particle oxidation.

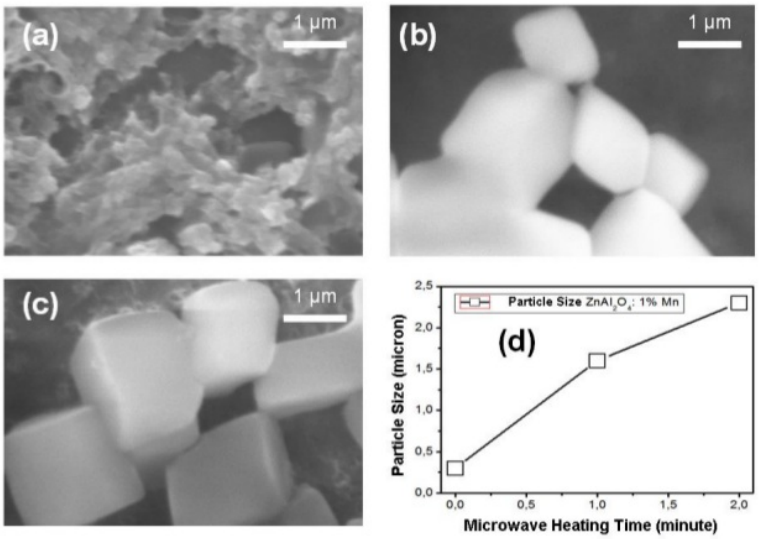

Figure 2. SEM image of ZnAl2O4:Mn paricle for various of microwave heating time, (a). 0 min, (b). 1 min, and(c) 2 min. While, (d).realtion of ZnAl2O4:Mn particle size distribution with heating time.

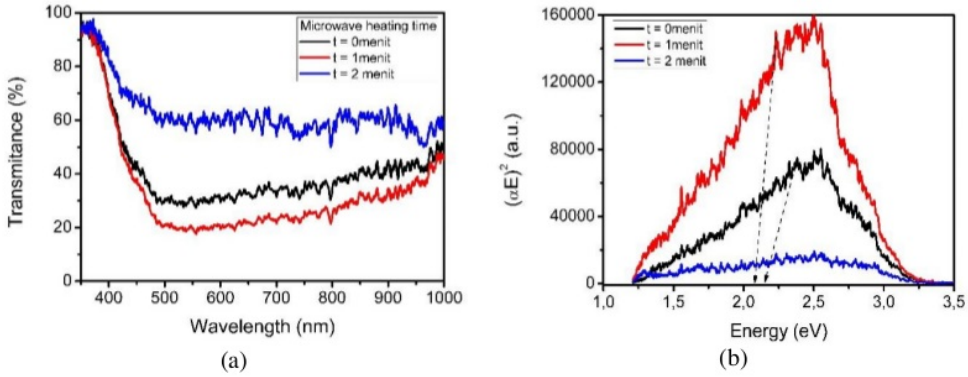

Figure 3. (a) Absorbance spectra, and (b) Tauc plot analysis of ZnAl2O4:Mn particle with various of heating time, all sample prepared with 0,01 g of ZnAl2O4:Mn particle at 5 ml of pure water.

Figure 3 is an absorbance spectrum of particle observations ZnAl2O4: Mn in the synthesized at some time variations synthesis. The results of these observations have managed to show that the sample absorbance spectrum of wide area, hingg 900 nm to 500 nm, with a peak at around 650 nm. Observations showed differences absorbance spectrum intensity for each sample time microwave heating. This probably caused by the quality of crystallization and particle size ZnAl2O4: Mn. The intensity of the absorbance spectrum in the wavelength range of blue-yellow (400-500 nm) is very low. This probably caused by the emission properties (luminescence) particle ZnAl2O4: Mn, this part needs to be studied further.

The optical band gap calculated using the Tauc plots relationship. Expressed by,

$$(\alpha hv) = C(hv - E_g)^n \quad (1)$$

where C is a constant, α is absorbance coefficient, E_g is the optical band gap of material, and n depends on the type of transition (n = 2 or 1/2 for direct band gap and indirect band gap) [????]. The calculated optical band gap of $ZnAl_2O_4$:Mn particle from Tauc plots was found to be 2.7 eV as shown in Fig. 3(b). The calculation results showed that all samples have the similar value of optical band gap.

4. Conclusions

Particle ZnAl2O4: Mn was synthesized using microwave heating assisted deposition. Observations show that the particle morphology occurs localized heating process that causes agglomeration or formation of large particles. Moreover, observations with absorbance, particle ZnAl2O4: Mn is estimate to have photocatalytic properties. Thus the particles ZnAl2O4: Mn has the potential to be develope as a photocatalytic material especially for liquid waste degradation.

5. References

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