

hasil-Fabrication of ZnAl₂O₄ from corresponding metals nitrate in the co-existence of natural SiO₂ and it application as adsorben of mercury nitrate

by Agus Martono

Submission date: 23-Apr-2022 09:19AM (UTC+0700)

Submission ID: 1817811776

File name: Fabrication_of_ZnAl₂O₄_AIP_Conf_Proc.pdf (1.6M)

Word count: 196

Character count: 11188

Fabrication of ZnAl_2O_4 from corresponding metals nitrate in the co-existence of natural SiO_2 and its application as adsorbent of mercury nitrate

Cite as: AIP Conference Proceedings **1823**, 020115 (2017); <https://doi.org/10.1063/1.4978188>
Published Online: 17 March 2017

Agus Martono Hadi Putranto, Salprima Yudha S., Morina Adfa, et al.



View Online



Export Citation

ARTICLES YOU MAY BE INTERESTED IN

[Iron sand - ZnO based materials of natural origin for dye decolorization under sunlight irradiation](#)

AIP Conference Proceedings **1823**, 020114 (2017); <https://doi.org/10.1063/1.4978187>

[Fabrication of hollow coaxial \$\text{Al}_2\text{O}_3/\text{ZnAl}_2\text{O}_4\$ high aspect ratio freestanding nanotubes based on the Kirkendall effect](#)

Journal of Vacuum Science & Technology A **38**, 013402 (2020); <https://doi.org/10.1116/1.5130176>

[Enhanced green emission from UV down-converting \$\text{Ce}^{3+}\$ - \$\text{Tb}^{3+}\$ co-activated \$\text{ZnAl}_2\text{O}_4\$ phosphor](#)

Journal of Vacuum Science & Technology B **30**, 031401 (2012); <https://doi.org/10.1116/1.3696720>

Lock-in Amplifiers
up to 600 MHz



Zurich
Instruments



AIP Conference Proceedings **1823**, 020115 (2017); <https://doi.org/10.1063/1.4978188>

1823, 020115

© 2017 Author(s).

Fabrication of $ZnAl_2O_4$ from Corresponding Metals Nitrate in the Co-existence of Natural SiO_2 and its Application as Adsorbent of Mercury Nitrate

Agus Martono Hadi Putranto, Salprima Yudha S.^a, Morina Adfa, Totok Eka Suharto, Zulfikri Achid Mardlia

Department of Chemistry, Faculty of Mathematics and Natural Sciences, The University of Bengkulu, Jalan Raya W.R Supratman, Kandang Limun 38371A, Bengkulu, Indonesia

^aCorresponding author: sp.yudha.s@gmail.com

Abstract. In situ fabrication of $ZnAl_2O_4$ from precursor of zinc nitrate and aluminium nitrate aqueous solutions was carried out in a rice husk's SiO_2 suspension. The reaction's mixture was heated at 100 °C in open air under stirring and the solvent was evaporated to form white gel. The gel was cooled to room temperature and a solid material was obtained, subsequently heated at 1100 °C for 5 hours. The obtained material was applied as adsorbent for mercury ions at room temperature

INTRODUCTION

It was known and confirmed that the differences on the $ZnAl_2O_4$ preparation give different morphology, acidity and also in some cases will affect the application of the produced materials. Recently, some synthetic methods of $ZnAl_2O_4-SiO_2$ including their applications was emphasized. Previously, $ZnAl_2O_4/SiO_2$ containing $89SiO_2-6Al_2O_3-5ZnO$ was synthesized [1]. More recently the $ZnAl_2O_4@SiO_2$ was prepared via sol-gel processes from zinc nitrate, aluminium nitrate and tetraethylorthosilicate, including their application for catalyst of acetylation of alcohols, phenols and amines without any solvent [2]. Based on the recent information, $ZnAl_2O_4$ was used in many purposes, therefore many researchers are working in the preparation and some modification in this ceramic material. For example synthesis of $ZnAl_2O_4$ as a transparent by using of development optic method [3]. On the other hand synthesis of $ZnAl_2O_4$ by different methods and used as catalytic support of platinum metal was reported recently [4]. The $ZnAl_2O_4$ was also used as a filler in $LiClO_4$ -PEO polymer electrolyte system [5]. Moreover, the preparation of cobalt ion supported by $ZnAl_2O_4$ was carried out and their properties was investigated [6]. Currently, research development on preparation of $ZnAl_2O_4$ as a supporting material for iron compound by solid-state reaction has been created and applied as a catalyst in a transesterification reaction of triglyceride. Unfortunately the catalytic system did not give the desired product. Therefore, more efforts should be developed to increase the activity of the material as a catalyst [7]. Furthermore, a facile synthesis of highly thermostable mesoporous $ZnAl_2O_4$ with adjustable pore size was developed [8]. The research result give a new information for the formation new material to host for some metals to increase the reactivity of $ZnAl_2O_4$ itself.

Based on the these results, the development of synthetic method of $ZnAl_2O_4$ in the presence of a natural based compound such as silica from rice husk was carried out to create a material that could give new characteristics and further application development. In almost cases the SiO_2 was obtained in the reported reactions were from tetra-orthosilicates as precursors at same time of the $ZnAl_2O_4$ formation [1-2]. Since the SiO_2 could be obtained from nature, a study on the preparation of $ZnAl_2O_4$ from metals nitrates in the co-existence of SiO_2 from rice husk via sol-gel method has been carried out. The study was also continued to the application for adsorption of mercury ions in a solution.

MATERIALS AND METHOD

$\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were obtained from Merck and used as received. SiO_2 was isolated from rice husk of *Oryza sativa* var glutinose following reported procedures with a little modification [9]. H_2O in this research is demineralized water. Synthesis of ZnAl_2O_4 in the presence of SiO_2 was carried out as follow: 25 mmol (6,536 gram) $\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ and 50 mmol (18,76 gram) $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were added to an erlenmeyer containing SiO_2 (25 mmol, 1,502 gram) suspension in demineralized water (100 ml), stirred until all metal nitrates were dissolved well. The reaction mixture was heated at 100°C until the solvent was in small amount and the transparent white gel formed in the bottom of erlenmeyer. The gel was removed to crucible and heated using oven at 110°C for 3 hours and subsequently heated at 1100°C for 5 hours. The product was characterized using XRD, IR spectrophotometer, DTA and TGA. A representative procedure for adsorption study is as follow: mercury nitrate solution (29,2 ppm, determined using cold vapour atomic absorption spectrophotometry) was added to an erlenmeyer (200 ml) and followed by addition of $\text{ZnAl}_2\text{O}_4\text{-SiO}_2$ to the solution. The mixture was shaken using automatic shaker for 1 hour and kept for 12 hour. The aliquots were taken from the reaction mixture around 50 ml and measured using atomic absorption spectrophotometer (AAS) cold vapour system.

RESULTS AND DISCUSSIONS

The in situ fabrication of ZnAl_2O_4 from Zinc (II) nitrate and aluminium (III) nitrate under heating at boiling point of water in the presence of natural SiO_2 without any addition of basic compounds was performed. During the heating of reaction mixture in a household, annitrogen oxide smell was observed by nose. It was assumed that nitrous gas was released during the reaction. The white powder was analyzed using XRD and the result is shown in Fig 1.

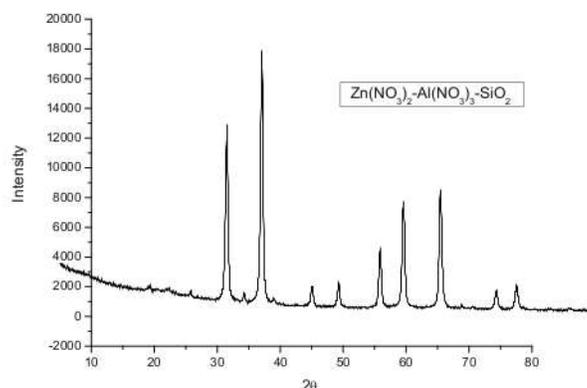


FIGURE 1. XRD patterns-prepared ZnAl_2O_4 in the presence of natural SiO_2

The XRD pattern of the obtained material as shown in Fig 1, revealed that the desired ZnAl_2O_4 was obtained in the presence of SiO_2 (broad peak around $2\theta = 20^\circ$). Small amount of impurity of ZnO was also observed. This result gives another alternative for preparation of ZnAl_2O_4 . In some reports, the presence of basic compounds such as ammonium hydroxide, NaOH were essential for synthesis of ZnAl_2O_4 [10] or ammonia [11], but in the present report, the presence of such compounds were not needed. Further characterization using DT/TG analysis is shown in Fig. 2. The DT/TG analysis were carried out in the normal atmospheric pressure under air condition

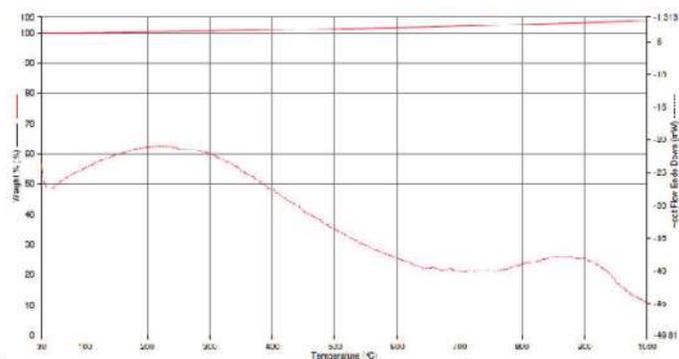


FIGURE 2. DTA - TGA as-prepared $ZnAl_2O_4$ in the presence of natural SiO_2 (the measurement have been done at air condition)

As shown in Fig. 2, the absence of any significant exothermic peak on the DTA curve of the mixed material shows that no recrystallization process occurs at temperature up to 1000 °C. This result suggested that the material could be used in some reactions at high temperature due to their thermal stability. Furthermore, to know the composition and morphology of the obtained materials, the product was subjected to EDX (Figure 3).

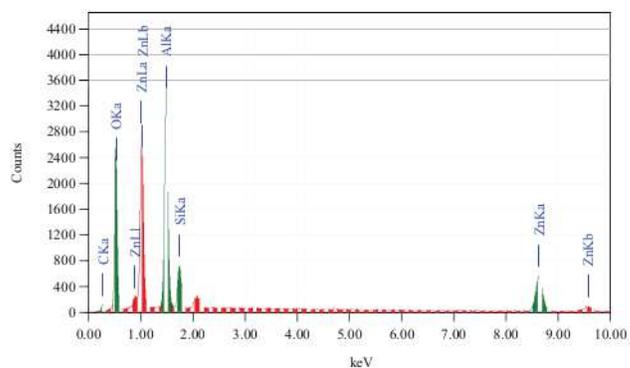


FIGURE 3. Energy Dispersive X-ray analysis result of the as-prepared material

Fig. 3 shows the presense of Zn, Al and Si metals as major element in the materials and it was in line with evidence of the obtained XRD profile for $ZnAl_2O_4$ in the presence of natural SiO_2 . Scanning Electron Microscopy (SEM) analysis was performed to investigate the morphology of the obtained materials and the results are shown in Figure 4.

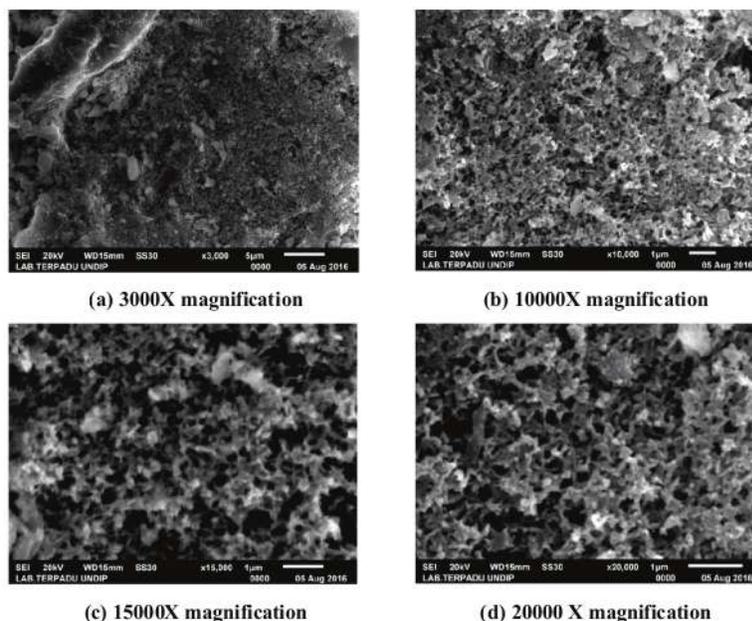


FIGURE 4. SEM analysis results of $ZnAl_2O_4-SiO_2$

Fig. 4 show that the current material was dominated by the porous particles (part b, c and d). This result will give an opportunity to use the material as adsorbent. The application of the obtained material as adsorbent of mercury nitrate solution is summarized in Table 1.

TABLE 1. Adsorption efficiency of mercury nitrate solution using $ZnAl_2O_4-SiO_2$

No	Adsorbent Amount (gr)	Final Concentration Mercury (mg/L)	% Efficiency*
1	0.3	27.00	7.53
2	0.5	25.10	14.04

*% efficiency was calculated from equation $[(C_0 - C_f)/C_0] \times 100$; where C_0 is initial concentration and C_f is final concentration

Table 1 show the ability of the synthesized material as adsorbent of mercury nitrate (29,2 ppm). The final concentration is lower than the mother solution after treatment using two variations of adsorbent weights (0,3 gram and 0,5 gram). These results clearly show that the current as-prepared materials become alternative material for heavy metal adsorbent.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support from Kementerian Riset, Teknologi dan Pendidikan Tinggi, Indonesia via Pengembangan IPTEKS Research Grant for fiscal 2015-2016. Z.A.M. Thank Head of Department of Chemistry to give opportunity as Research Assistant at Division of Inorganic Chemistry.

REFERENCES

1. X. Duan, D. Yuan, Z. Sun, H. Sun, D. Xu and M. Lu, *J. Crystal Growth* **252**, 4–8 (2003).
2. S. Farhadi and K. Jahanara, *Chinese J. Cat.* **35**, 368–375 (2014).
3. A. Goldstein, Y. Yeshurun, M. Vulfson and H. Kravits, *J. Am. Ceram. Soc.* **95**, 879–882 (2012).

4. A.D. Ballarini, S.A. Bocanegra, A.A. Castro, S.R. de Miguel and O.A. Sceiza, *Cat. Lett***129**, 293-302 (2009).
5. L. Wang, W. Yang, J. Wang and D.G. Evans, *Solid State Ionic*, 392-397 (2009).
6. T.T. Loan, N.N. Long and L.H. Ha, *Electronic J. Surf. Sci. Nanotech.***9**, 526-530 (2011).
7. S. Yudha, E. Angasa and E. Maryanti, "Synthetic application of ZnAl₂O₄-supported iron catalysts to transesterification of triacetin and conversion of glycerol," in *ITSF Seminar Book* (2011).
8. D. L. Ge, Y. J. Fan, C. L. Qian and Z. X. Sun, *J. Mater. Chem. A*, **1**, 1651-1658 (2013).
9. a) W. Wang, J.C. Martin, X. Fan, A. Han, Z. Luo and L. Sun, *ACS Appl. Mater. Interfaces***4**, 977-981 (2012).
9. (b) N. Liu, K. Huo, M. T. McDowell, J. Zhao and Y. Cui, *Nature***3**, 1919 (2013).
10. G. Sukul, D.V. Srinivasan and P.V. Balaramakrishna, *J. Nano. Adv. Mat*, 69-74 (2013).
11. S. Battiston, C. Rigo, E. da C. Severo, M.A. Mazutti, R.C. Kuhn, A. Gündeland and E.L. Foletto, *Material Research***17**, 3, 734-738 (2014).

hasil-Fabrication of ZnAl₂O₄ from corresponding metals nitrate in the co-existence of natural SiO₂ and it application as adsorben of mercury nitrate

ORIGINALITY REPORT

9%

SIMILARITY INDEX

0%

INTERNET SOURCES

9%

PUBLICATIONS

9%

STUDENT PAPERS

PRIMARY SOURCES

1

W A Rizal, K Nisa', R Maryana, D J Prasetyo, D Pratiwi, T H Jatmiko, D Ariani, A Suwanto. "Chemical composition of liquid smoke from coconut shell waste produced by SME in Rongkop Gunungkidul", IOP Conference Series: Earth and Environmental Science, 2020
Publication

9%

Exclude quotes On

Exclude matches Off

Exclude bibliography On