International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 07 Issue 06 June 2024
DOI: 10.47191/ijcsrr/V7-i6-83, Impact Factor: 7.943
IJCSRR @ 2024
www.ijcsrr.org

Synthesis and Characterization of Bis-Acetylacetonatozink (II) [$(Zn(acac)_2(H_2o)]$]

Laode A. Kadir[1], Thamrin Azis[1], Imran[1], La Ode Kadidae[1], Ida Usman[2], Agusriyadin[3]
[1] Department of Chemistry, Universitas Halu Oleo, Indonesia
[2] Department of Physics, Universitas Halu Oleo, Indonesia
[3] Department of Chemistry, USN Kolaka, Indonesia

ABSTRACT: The synthesis of the bis-acetylacetonate zink (II) monohydrate complex $[Zn(acac)_2(H_2O)]$ has been conducted. FTIR analysis, TGA-DSC, and SEM micrographic analysis performed complex characterization. The yield of the complex compound $[Zn(acac)_2(H_2O)]$ was 36.24% using NaOH as a pH regulator. Based on the FTIR characterization of the typical absorption complex $[Zn(acac)_2(H_2O)]$ appears at 763.88 cm^{-1} for Zn-O, C-H at 933.9 cm^{-1} and 1020 cm^{-1}, C=C and C=O at 1516 cm^{-1}, 1610 cm^{-1} and OH at 3450 cm^{-1} respectively. Thermal analysis of the complex shows that the decomposition temperature was obtained at 186^{o}C. Based on the micrographic results, the complex has a non-uniform surface relief. The complex has an even distribution of pores with non-uniform pore sizes. Zn particles are deposited on the complex surface with various sizes to produce pores on the solid surface.

KEYWORDS: acetylacetonate, bis-acetylacetonates (II), complex compound.

INTRODUCTION

The advancement of research on complex compounds or coordination compounds has emerged as a compelling subject, given the broad-ranging applications of these compounds across various domains. For instance, in healthcare, complex compounds serve as contrast agents in Magnetic Resonance Imaging (MRI), thereby aiding in the enhancement of tissue visualization (Chen et al., 2022). In magnetic materials, complex compounds find utility as thin-film-free sensors (Baran et al., 2020). Concurrently, complex compounds are harnessed within the industrial sector for corrosion inhibition purposes (Zaidi et al., 2023).

Complex compounds can be synthesized by reacting ligands, which are bases with free electron pairs, with metals, which act as electron pair acceptors donated by the ligands. Acetylacetone (2,4-pentanedione) is a β-keto compound that can ionize as a weak acid, forming the acetylacetonate anion, forming six-membered ring bidentate complexes with oxygen donor ligands. Oxygen is commonly used as an organic ligand in synthesizing complex compounds (Saria et al., 2012).

Based on research conducted by inorganic researchers, transition metals have been extensively studied and synthesized into complex compounds (Soofivand et al., 2014). This is attributed to transition metals' inert and stable nature in forming complex compounds with various ligands, one of which is the acetylacetone ligand. Several previous studies have successfully synthesized Metal-acetylacetone complexes (M-acac), such as bis-acetylacetone nickel (II) complex (Mintari et al., 2015), bis-acetylacetone copper (II) complex (Paramitha et al., 2014), cobalt acetylacetonate complex (Saria et al., 2012), tris-aluminium acetylacetonate (Borker et al., 2010), tris-bis(acetylacetone) cobalt (II) complex (Gnanasoundari & Natarajan, 2005), and modified zinc/MgO-acetylacetone complex (Matsuhashi, 2022).

The synthesis technique of complex compounds is relatively more straightforward than inorganic materials or organic compounds. Unlike several previous studies, for the synthesis of metal acetylacetonate complexes, techniques such as the volatile phase technique (Feng et al., 2022), precipitation method (Petrović et al., 2015), Ultrasonic Chemical Spray (Biswal et al., 2014), and hydrolysis (Petrović et al., 2014) have been employed. However, in this study, the synthesis process was conducted solely through conventional chemical reactions and ligand-metal complexation processes, forming complex compounds. This research focuses on synthesizing zinc complex compounds with bidentate acetylacetonate ligands. Furthermore, FTIR, SEM, and TGA-DSC analysis characterize the synthesized complex compounds.

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 07 Issue 06 June 2024
DOI: 10.47191/ijcsrr/V7-i6-83, Impact Factor: 7.943
IJCSRR @ 2024
www.ijcsrr.org

METHODS

Materials and Equipment

The instruments utilized in this research included a Fourier Transform Infrared Spectrometer (FT-IR spectrometer; Perkin Elmer) with a resolution of 4 cm^{-1} and a KBr pellet with a wavenumber range of 400-5000 cm^{-1}. Scanning Electron Microscope (SEM), EVOMA 10, was employed to observe the morphology of the synthesized products. Thermal analysis was conducted using TGA PT 6000 and DSC PT 1600. The materials used in this study consisted of ZnSO4.7H_2O (Merck), acetylacetone (Sigma Aldrich), NaOH (Merck), and ethyl acetate.

Synthesis of Bis-acetylacetonate Zinc (II) Complex

The bis-acetylacetonate zinc (II) complex was synthesized by modifying the method (Brahma & Shivashankar, 2015). A solution of acetylacetonate was added with NaOH as a pH regulator and reacted with a solution of $ZnSO_4 \cdot 7H_2O$. This mixture was then heated at 60°C for 1 hour. Subsequently, the solution was cooled until a precipitate formed. The precipitate was then filtered and subjected to crystallization with ethyl acetate. The bis-acetylacetonate zinc (II) complex appeared as white crystalline solids. The procedure was repeated using methanol and acetone as pH regulators to determine the optimal yield. The product with the highest yield was further characterized.

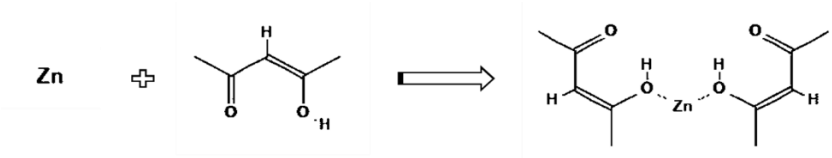

Figure 1. Scheme of Bis-acetylacetonate zinc (II) Complex Formation

RESULTS AND DISCUSSION

The synthesis of zinc metal with acetylacetonate ligand in NaOH, methanol, and chloroform solvent yields a white crystalline precipitate. The yield of the bis-acetylacetonate zinc (II) complex compound [$Zn(acac)_2(H_2O)$] can be seen in Table 1. The percentage yield of the complex in NaOH solvent is the highest in this study, at 36.24%. This is to the previous report by (Hayami et al., 2014) that bis-acetylacetonate zinc (II) complex is a white crystalline material that can be synthesized with NaOH as a pH regulator with a %yield reaching 88%. The yields of bis-acetylacetonate zinc (II) synthesis using different pH regulators are presented in Table 1.

Table 1. yields of bis-acetylacetonate zinc (II)

No	Compounds	% Yield
1	Bis-asetilasetonatozink (II)@ NaOH	36.24
2	Bis-asetilasetonatozink (II)@Metanol	24.21
3	Bis-asetilasetonatozink (II)@Kloroform	18.96

The FTIR analysis of the complex compound [$Zn(acac)_2(H_2O)$] is conducted in the wavenumber range of 500-4000 cm^{-1} to determine the functional groups of the complex compound and the interactions that occur between Zn and acetylacetonate. The indication of the formation of the complex compound [$Zn(acac)_2(H_2O)$] is the appearance of characteristic absorptions of the functional groups of the complex [$Zn(acac)_2(H_2O)$], such as Zn-O absorption at the wavenumbers of 650-760 cm^{-1} (Ambrožič et al., 2010), aliphatic C-H absorption at the wavenumbers of 927 cm^{-1} and 2979 cm^{-1} (Chesnokov & Rostovtseva, 2019), C=C and C=O absorptions at the wavenumbers of 1400 cm^{-1}, 1650 cm^{-1}, and broad hydroxyl group absorption at the wavenumber of 3500 cm^{-1} (Brahma & Shivashankar, 2015). From the FTIR analysis results of the synthesized complex compound, as shown in Figure 2, it can be observed that there is a broad absorption from O-H appearing at the wavenumber of 3450 cm^{-1}, indicating that the bis-acetylacetonate zinc (II) complex binds water molecules. According to the research (Musić et al., 2010), the Zn-acetylacetonate complex will bind one water molecule, forming a pentacoordinate structure adopted from a square pyramidal structure.

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 07 Issue 06 June 2024
DOI: 10.47191/ijcsrr/V7-i6-83, Impact Factor: 7.943
IJCSRR @ 2024
www.ijcsrr.org

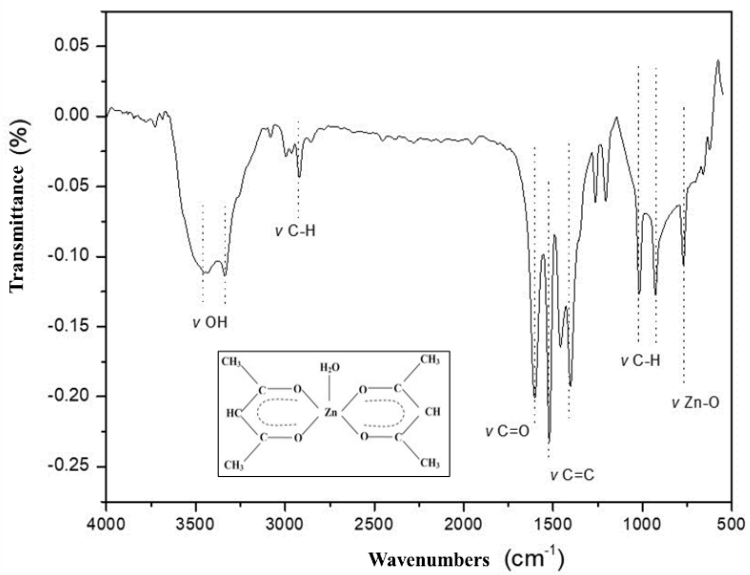

Figure 2. FTIR Spectrum of Bis-acetylacetonate zinc (II) Complex

The Zn-O absorption appears at the wavenumber of 763.88 cm^{-1}, and C-H absorption occurs at 933.9 cm^{-1} and 1020 cm^{-1}. Meanwhile, C=C and C=O absorptions appear at 1516 cm^{-1} and 1610 cm^{-1} respectively.

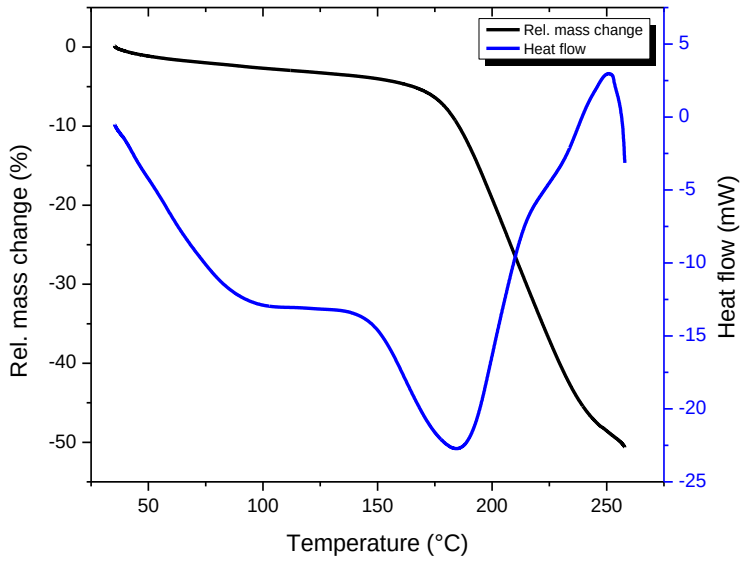

Figure 3. Thermal analysis of bis-acetylacetonate zinc(II) complex using TGA-DSC

Thermal analysis using TGA (Figure 3) reveals a single-stage mass decomposition originating from the precursor complex $[Zn(acac)_2(H_2O)]$. Mass decomposition initiates with the loss of water molecules at temperatures ranging from 40 to 120°C. Significant mass decomposition occurs at 150°C with a total percentage mass loss of 46.4%. This phenomenon indicates that the $[Zn(acac)_2(H_2O)]$ complex exhibits considerable stability against heat. Based on the DSC analysis results (Figure 3), an exothermic peak is observed at 186°C, corresponding to the decomposition temperature of the $[Zn(acac)_2(H_2O)]$ complex. These findings align with previous research, which reported the decomposition temperature of the $[Zn(acac)_2(H_2O)]$ complex to be within the range of 136-220°C (Brahma & Shivashankar, 2015).

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 07 Issue 06 June 2024
DOI: 10.47191/ijcsrr/V7-i6-83, Impact Factor: 7.943
IJCSRR @ 2024
www.ijcsrr.org

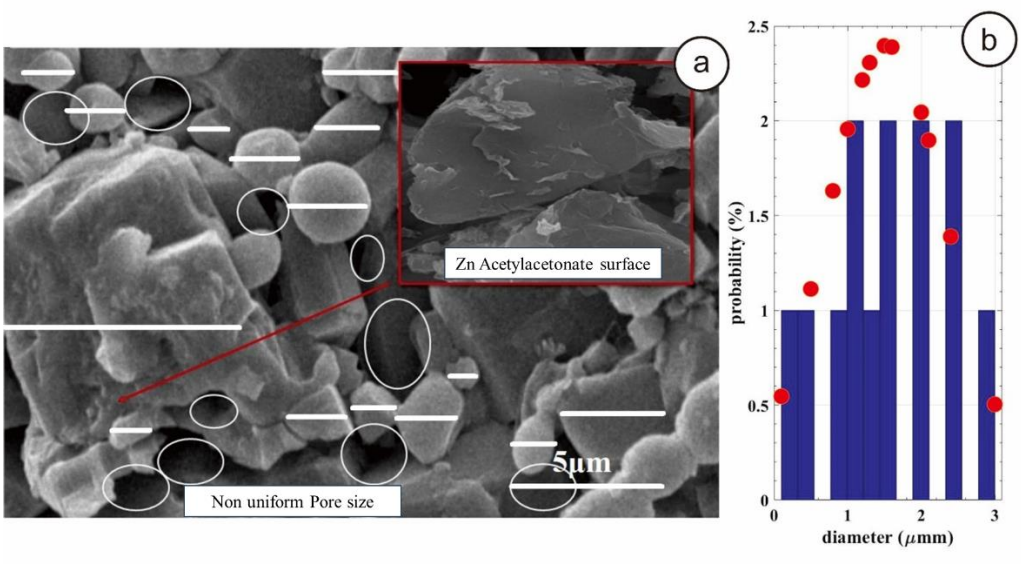

Figure 4. Micrograph analysis using SEM of bis-acetylacetonate zinc(II) complex

Continuing from the micrograph analysis results aided by SEM (Figure 4), it is observed that the $[Zn(acac)_2(H_2O)]$ complex exhibits a non-uniform surface relief. The complex demonstrates a uniform distribution of pores with non-uniform pore sizes. Zinc particles are deposited on the surface of the complex with varying sizes, thus resulting in surface pores on the solid. This condition allows the $[Zn(acac)_2(H_2O)]$ complex to possess low stability and susceptibility to hydrolysis. Numerous researchers have exploited these properties to utilize the $[Zn(acac)_2(H_2O)]$ complex as a precursor material in zinc oxide synthesis (Šarić et al., 2019).

CONCLUSION

The synthesis of bis-acetylacetonate zinc (II) complex monohydrate $[Zn(acac)_2(H_2O)]$ has been successfully achieved. The yield of the complex compound $[Zn(acac)_2(H_2O)]$ obtained is 36.24% using NaOH as the pH regulator. Based on the FTIR characterization, characteristic absorption bands of the $[Zn(acac)_2(H_2O)]$ complex appear at wave numbers of 763.88 cm^{-1} for Zn-O, absorption of C-H appears at wave numbers of 933.9 cm^{-1} and 1020 cm^{-1}, C=C and C=O absorption occur at wave numbers of 1516 cm^{-1} and 1610 cm^{-1}, respectively, while OH appears at a wave number of 3450 cm^{-1}. Based on TGA and DSC characterization, the zinc bis-acetylacetonate complex (II) monohydrate $[Zn(acac)_2(H_2O)]$ demonstrates sufficient thermal stability, with complex decomposition occurring at 186°C. Micrograph analysis using SEM reveals that the complex exhibits evenly distributed pores with non-uniform pore sizes.

ACKNOWLEDGMENTS

The authors thank the Department of Chemistry Laboratory, Institut Teknologi Bandung, Indonesia, for providing research resources and Department of Chemistry Laboratory Universitas Halu Oleo for supporting administration.

REFERENCES

1. Ambrožič, G., Škapin, S. D., Žigon, M., & Orel, Z. C. (2010). The synthesis of zinc oxide nanoparticles from zinc acetylacetonate hydrate and 1-butanol or isobutanol. *Journal of Colloid and Interface Science*, *346*(2), 317–323. https://doi.org/10.1016/J.JCIS.2010.03.001
2. Baran, S., Tyvanchuk, Y., Penc, B., Kalychak, Y., Hoser, A., & Szytuła, A. (2020). Crystal structure and complex magnetic properties of $R_{11}Pd_4In_9$ compounds (R = Y, Gd–Er). *Intermetallics*, *123*, 106837. https://doi.org/10.1016/J.intermet.2020.106837
3. Biswal, R., Maldonado, A., Vega-Pérez, J., Acosta, D. R., & Olvera, M. D. L. L. (2014). Indium Doped Zinc Oxide Thin Films Deposited by Ultrasonic Chemical Spray Technique, Starting from Zinc Acetylacetonate and Indium Chloride. *Materials 2014, Vol. 7, Pages 5038-5046*, *7*(7), 5038–5046. https://doi.org/10.3390/MA7075038

International Journal of Current Science Research and Review
ISSN: 2581-8341
Volume 07 Issue 06 June 2024
DOI: 10.47191/ijcsrr/V7-i6-83, Impact Factor: 7.943
IJCSRR @ 2024
www.ijcsrr.org

4. Borker, V., Bensch, W., & Rane, K. (2010). Synthesis and structure of Tris (acetylacetonato) aluminum(III) Chalcogene Chemistry View project Polyoxovanadates View project. In *Article in Indian Journal of Chemistry Section a*. https://www.researchgate.net/publication/279595164
5. Brahma, S., & Shivashankar, S. A. (2015). Zinc acetylacetonate hydrate adducted with nitrogen donor ligands: Synthesis, spectroscopic characterization, and thermal analysis. *Journal of Molecular Structure*, *1101*, 41–49. https://doi.org/10.1016/J.Molstruc.2015.07.075
6. Chen, S., An, L., & Yang, S. (2022). Low-Molecular-Weight Fe(III) Complexes for MRI Contrast Agents. *Molecules (Basel, Switzerland)*, *27*(14). https://doi.org/10.3390/Molecules27144573
7. Chesnokov, V. V., & Rostovtseva, I. A. (2019). Adducts of Zinc(II) Acetylacetonate with Various Aminoquinolines: Synthesis, Structure and Luminescence. *ChemistrySelect*, *4*(9), 2607–2609. https://doi.org/10.1002/slct.201803840
8. Feng, J., Li, J., Feng, J., Wei, Z., Wang, Z., & Song, X. (2022). Synthesis of Aliphatic Polycarbonates from Diphenyl Carbonate and Diols over Zinc (II) Acetylacetonate. *Molecules 2022, Vol. 27, Page 8958*, *27*(24), 8958. https://doi.org/10.3390/Molecules27248958
9. Gnanasoundari, V. G., & Natarajan, K. (2005). Synthesis, characterization and catalytic studies of iron(III), cobalt(II), nickel(II) and copper(II) complexes containing triphenylphosphine and β-diketones. *Transition Metal Chemistry*, *30*(4), 433–438. https://doi.org/10.1007/s11243-005-1023-4
10. Hayami, R., Tsukada, S., & Gunji, T. (2014). Synthesis and properties of β-diketonato metal complexes as a catalyst for the synthesis of polyurethane. *Advance in Civil, Environmental, and Material Research*.
11. Matsuhashi, H. (2022). Synthesis of a Solid Base Catalyst Formed from Zinc(II) Acetylacetonate and Consisting of MgO Modified with ZnO. *Catalysis Research*, *2*(2), 1–1. https://doi.org/10.21926/cr.2202015
12. Mintari, N., Suhartana, S., & Sriatun, S. (2015). Pengaruh Variasi Jenis Pelarut pada Rendemen Sintesis Senyawa Kompleks Bis-Asetilasetonatodiaquonikel(II). *Jurnal Kimia Sains Dan Aplikasi*, *18*(1), 29–33. https://doi.org/10.14710/JKSA.18.1.29-33
13. Musić, S., Šarić, A., & Popović, S. (2010). Formation of nanosize ZnO particles by thermal decomposition of zinc acetylacetonate monohydrate. *Ceramics International*, *36*(3), 1117–1123. https://doi.org/10.1016/J.Ceramint.2009.12.008
14. Paramitha, L. D., Suhartana, S., & Pardoyo, P. (2014). Pengaruh Pelarut pada Rendemen Sintesis Senyawa Kompleks Bis-Asetilasetonatotembaga(II). *Jurnal Kimia Sains Dan Aplikasi*, *17*(2), 48–50. https://doi.org/10.14710/JKSA.17.2.48-50
15. Petrović, Ž., Ristić, M., & Musić, S. (2014). Development of ZnO microstructures produced by rapid hydrolysis of zinc acetylacetonate. *Ceramics International*, *40*(7 PART B), 10953–10959. https://doi.org/10.1016/j.ceramint.2014.03.095
16. Petrović, Ž., Ristić, M., Musić, S., & Fabián, M. (2015). Nano/microstructure and optical properties of ZnO particles precipitated from zinc acetylacetonate. *Journal of Molecular Structure*, *1090*, 121–128. https://doi.org/10.1016/J.Molstruc.2014.11.068
17. Saria, Y., Saria, Y., Lucyanti, L., Hidayati, N., & Lesbani, A. (2012). Sintesis Senyawa Kompleks Kobalt dengan Asetilasetonato. *Jurnal Penelitian Sains*, *15*(3), 2012. https://doi.org/10.56064/jps.v15i3.107
18. Šarić, A., Despotović, I., & Štefanić, G. (2019). Solvothermal synthesis of zinc oxide nanoparticles: A combined experimental and theoretical study. *Journal of Molecular Structure*, *1178*, 251–260. https://doi.org/10.1016/j.molstruc.2018.10.025
19. Soofivand, F., Tavakoli, F., & Salavati-Niasari, M. (2014). *Synthesis and Characterization of Zn(acac)2 One-dimensional Nanostructures by Novel Method* (Vol. 3, Issue 2).
20. Zaidi, K., Bouroumane, N., Merimi, C., Aouiniti, A., Touzani, R., Oussaid, A., Hammouti, B., Salim, R., Kaya, S., & Ibrahim, S. M. (2023). Iron-ligand complex, an efficient inhibitor of steel corrosion in hydrochloric acid media. *Journal of Molecular Structure*, *1284*, 135434. https://doi.org/10.1016/J.Molstruc.2023.135434

Cite this Article: Laode A. Kadir, Thamrin Azis, Imran, La Ode Kadidae, Ida Usman, Agusriyadin (2024). Synthesis and Characterization of Bis-Acetylacetonatozink (Ii) [(Zn(Acac)2(H2o)]. International Journal of Current Science Research and Review, 7(6), 4315-4319