



Zinc–diethanolamine complex: synthesis, characterization, and formation mechanism of zinc oxide via thermal decomposition

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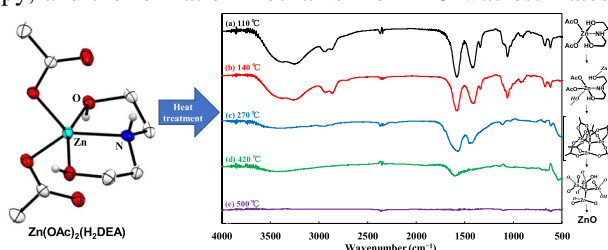
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Abstract

$\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was synthesized by the reaction of zinc acetate dihydrate ($\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$) with diethanolamine (H_2DEA), and was characterized using single-crystal X-ray structural analysis, nuclear magnetic resonance spectroscopy, Fourier-transform infrared (FT-IR) spectroscopy, and elemental analysis. $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ had a trigonal bipyramidal geometry comprised of one zinc atom, two acetate groups, and one H_2DEA as a neutral tridentate ligand to form two five-membered rings. The states of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ heated at various temperatures were determined by FT-IR spectroscopy. At 270 °C, the H_2DEA ligand dissociated and was removed. The absorption bands assigned to Zn–O stretching vibration of Zn_4O core such as the zinc-oxo cluster appeared. When heated at 500 °C, the absorption bands of μ_4 -oxozincate and the acetate group disappeared completely and hexagonal wurtzite structural ZnO was formed at 550 °C. A possible thermal decomposition pathway from $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ to ZnO was proposed. The ZnO film was highly transparent and formed by the deposition of ZnO nanoparticles with size ~40 nm.

Graphical Abstract

$\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was synthesized and characterized. The states of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ heated at various temperatures were determined by FT-IR spectroscopy, and the formation mechanism of ZnO was estimated.



Highlights

- Zinc–diethanolamine complex was synthesized by the reaction of zinc acetate with diethanolamine.
- Zinc–diethanolamine complex was isolated and characterized.
- The formation mechanism of ZnO was estimated by FT-IR spectra.

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Keywords Zinc–diethanolamine complex • Single-crystal X-ray structural analysis • FT-IR spectra • Thermal analysis

1 Introduction

Zinc oxide, ZnO, has attracted attention for its application in sensors, transparent conductors, etc. [1–7]. ZnO films are prepared by sputter deposition, chemical vapor deposition, sol–gel process, etc. [3, 8, 9]. The sol–gel process is a good technique because it is an easy process, of low cost, and easily coats large surface areas. Therefore, many researchers prepare ZnO films using the sol–gel process. ZnO precursor is prepared by the sol–gel reaction of zinc salts (e.g., zinc acetate, zinc nitrate, and zinc alkoxides) with alkanolamines such as monoethanolamine (HMEA), diethanolamine (H₂DEA), and triethanolamine (H₃TEA). ZnO thin films are deposited by spin-coating or dip-coating on a substrate followed by heat treatment [2, 10–17].

The formation mechanism of ZnO suggests that ZnO colloids or gels are formed by the polymerization of zinc hydroxide and/or zinc-oxo acetate clusters from ZnO precursors [12, 17]. The structures of ZnO precursors were proposed by Znaidi and Nehmann. Znaidi et al. [12, 13] proposed that Zn–monoethanolamine and Zn acetate complexes were present in equilibrium when mixing Zn(OAc)₂ and HMEA. Nehmann et al. [18] proposed that the Zn–diethanolamine complex Zn(DEA) was formed as a ZnO precursor by ligand exchange reaction between Zn(OAc)₂ and H₂DEA. However, these ZnO precursors could not be isolated and characterized and the authenticity of ZnO precursors remains uncertain. Recently, Conterosito et al. [19] reported the synthesis of the Zn–triethanolamine complex Zn(OAc)₂(H₃TEA) by mixing Zn(OAc)₂ with triethanolamine (H₃TEA). The complex was characterized by single-crystal X-ray structural analysis and transferred to cluster compounds (Zn₄(OAc)₄(HTEA)₂) by adding water. One of the zinc–alkanolamine complexes was thus revealed.

In this work, Zn–diethanolamine complex, which is useful and is one of the zinc–alkanolamine complexes, was synthesized by the reaction of Zn(OAc)₂•2H₂O with diethanolamine (H₂DEA) in EtOH (Scheme 1). The Zn–diethanolamine complex Zn(OAc)₂(H₂DEA) was isolated and characterized by single-crystal X-ray structure determination, nuclear magnetic resonance (NMR) spectra, Fourier-transform infrared (FT-IR) spectrum, and thermal

analysis. Moreover, the formation mechanism of ZnO through the complex was studied via FT-IR spectra.

2 Experimental section

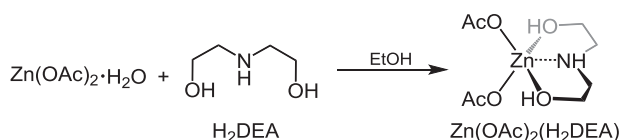
2.1 Reagents

The solvents Zn(OAc)₂•2H₂O and H₂DEA were purchased from Wako Pure Chemical Industries Ltd., Tokyo, Japan, and used as received. Zn₄(μ₄-O)(OAc)₆ was prepared according to the literature [20], and the method and characterization were described in supporting information. Silicon wafer (4" polishing wafer) was purchased from GlobalWafers Co., Ltd. The substrate was cut to 2.5 cm × 2.5 cm and cleaned with a neutral detergent, EtOH, and ethyl acetate.

2.2 Measurements

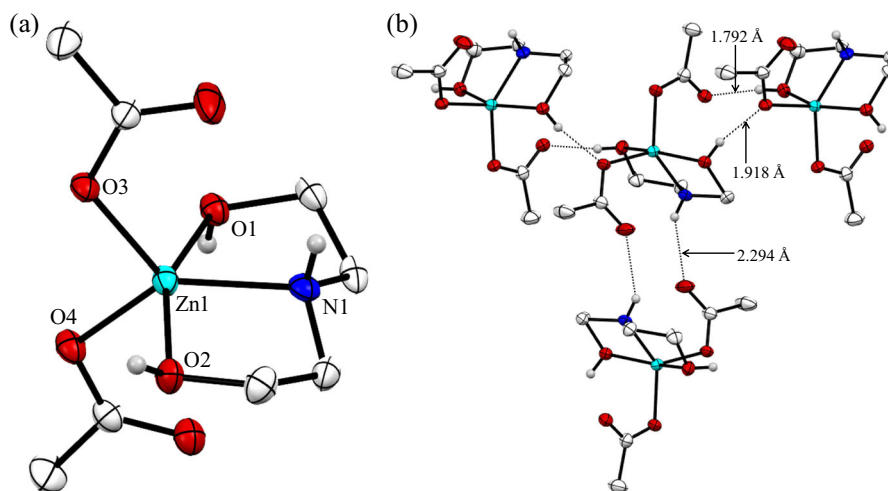
All NMR spectra were recorded on a JEOL JNM-ECP500 spectrometer (JEOL, Akishima, Japan; ¹H at 500.16 MHz, ¹³C at 125.77 MHz) at ~23 °C. The ¹H NMR and ¹³C{¹H} NMR spectra were recorded using tetramethylsilane as internal standard. FT-IR spectra were recorded on a JASCO FT/IR-6100 spectrometer (JASCO, Hachioji, Japan) using the KBr method or after coating on silicon wafer. Elemental analysis was performed using a Perkin Elmer 2400II Elemental Analyzer. Melting points were recorded using a Bibby Stuart Scientific SMP3 instrument; the reported melting points were uncorrected. Thermogravimetric-differential thermal analysis (TG-DTA) was performed using a NETZCH JAPAN TG-DTA 2000SE (Netzsch Japan, Yokohama, Japan). Samples were heated to 800 °C at the rate of 10 °C/min under airflow. X-ray diffraction (XRD) spectra were recorded using an IC Vario (PANalytical, Tokyo, Japan) with monochromatic Cu-K_α radiation as X-ray source. Atomic force microscope (AFM) observations were obtained using an SPM-9700 (Shimadzu, Kyoto, Japan). Transmittance spectra were recorded using a JASCO V-670 spectrophotometer equipped with an integrating-sphere photometer ISN-470 type (JASCO, Hachioji, Japan) in the wavelength range of 300–800 nm by coating films on glass substrates.

Crystal data were collected using a Bruker AXS SMART APEX CCD X-ray diffractometer equipped with monochromatic Mo-K_α radiation (0.7107 Å). Empirical absorption corrections using equivalent reflections and Lorentzian polarization corrections were performed using the SADABS



Scheme 1 Synthesis of Zn(OAc)₂(H₂DEA)

Fig. 1 ORTEP drawings of **a** the molecular structure of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ and **b** interaction with thermal ellipsoids at 50% probability level. Crystal solvents are omitted for clarity. Color code: gray, H; white, C; red, O; blue, N; light blue, Zn



program [21]. All data were collected with SMART and Bruker SAINTPLUS (Version 6.45) software packages. The structures were solved using the SHELXA-97 program [22] and refined against F^2 using SHELEXL-97 [23].

The deposition number was CCDC-1567248 for $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$. Free copies of the data can be obtained via <http://www.ccdc.cam.ac.uk/conts/retrieving.html> (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK; Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

2.3 Synthesis of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$

$\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ (0.66 g, 3.0 mmol) and H_2DEA (0.31 g, 3.0 mmol) were dissolved in 5 mL of EtOH, and the mixture was refluxed overnight. The solution was concentrated to about half the volume, and kept standing at -18°C for 1 h. A white powder was obtained from the solution by reprecipitation with hexane. The obtained powder was purified by recrystallization with tetrahydrofuran (THF), and $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was obtained as colorless crystals (0.50 g, 69%).

M.p. (dec.) = $130.4\text{--}132.5^\circ\text{C}$. Elemental analysis: calcd. for $\text{C}_8\text{H}_{17}\text{NO}_6\text{Zn}$ C 33.29, H 5.94, and N 4.85; found C 33.66, H 5.86, and N 4.90. ^1H NMR (500 MHz, CD_3OD): δ = 1.96 (s, 6 H, CH_3), 2.95 (t, J = 5.0 Hz, 4H, $\text{CH}_2\text{-N}$), 3.74 (t, J = 5.0 Hz, 4H, O-CH_2), and 4.91 (s, 3H, NH and OH) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (126 MHz, CD_3OD): δ = 23.01 (CH_3), 50.05 ($\text{CH}_2\text{-N}$), 59.51 (O-CH_2), and 181.01 (COO) ppm. FT-IR (KBr, cm^{-1}): 3412, 2969, 2889, 1577, 1409, 1341, 1099, 1057, 1023, 933, 899, 678, and 619.

2.4 Preparation of ZnO thin film

ZnO thin film was prepared by spin-coating (500 rpm for 10 s followed by 1000 rpm for 40 s) using 10 wt% $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})/\text{EtOH}$ solution (200 μL) dropped on a substrate and drying at 100°C for 10 min. This process was repeated twice. The coating film was heated at various temperatures, at the rate of $10^\circ\text{C}/\text{min}$, followed by keeping at a chosen temperature for 30 min.

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3 Results and discussion

3.1 Synthesis of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$

$\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was synthesized by the reaction of $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ with H_2DEA in EtOH, and purified by reprecipitation with hexane followed by recrystallization from hot THF. After 1 d, single crystals of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ appeared. $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was easily soluble in alcohol and hot THF, and insoluble in hexane, toluene, and chloroform. The structure of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was stable even in the presence of a small amount of water, unlike that of $\text{Zn}(\text{OAc})_2(\text{H}_3\text{TEA})$.

3.2 Characterization of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$

The structure of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was measured by single X-ray structural analysis, as shown in Fig. 1. $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ had a trigonal bipyramidal geometry comprised of one zinc atom, two acetate groups, and one H_2DEA as a neutral tridentate ligand to form two five-membered rings (Fig. 1a). The distances of these bonds were Zn1-O1 (2.176(3) Å), Zn1-O2 (2.202(3) Å), Zn1-O3 (1.990(3) Å), Zn1-O4 (1.988(3) Å), and Zn1-N1 (2.059(4) Å); therefore, these bonding distances increased in the order $\text{Zn-OAc} < \text{Zn-N}$ of $\text{H}_2\text{DEA} < \text{Zn-O}$ of H_2DEA . The structural geometry, bonding distances, and angles of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ were similar to that of $\text{Zn}(\text{OAc})_2(\text{H}_3\text{TEA})$ [19]. Interestingly, $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ showed the formation of hydrogen bonding

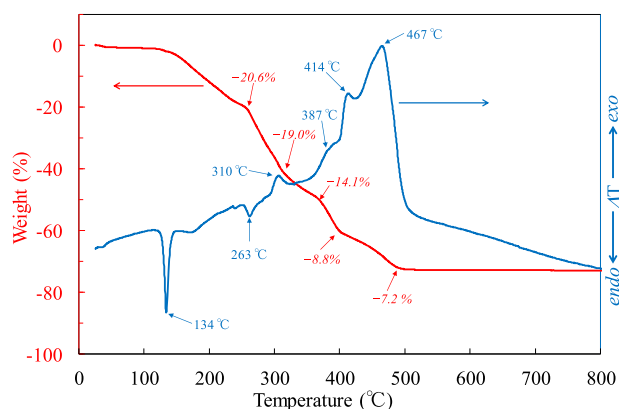


Fig. 2 TG-DTA traces of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ under airflow

between one central complex and three complexes, and the hydrogen atoms in H_2DEA interacted with acetate groups of other complexes (Fig. 1b). These hydrogen bond distances were in the order $\text{O}-\text{H}\cdots\text{O}=\text{C}$ (1.792 \AA) $<$ $\text{O}-\text{H}\cdots\text{O}-\text{Zn}$ (1.918 \AA) $<$ $\text{N}-\text{H}\cdots\text{O}=\text{C}$ (2.294 \AA).

^1H and ^{13}C NMR spectra of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ were obtained using CD_3OD . For both the NMR spectra, the signals assigned to H_2DEA ligand shifted slightly downfield compared to that of H_2DEA . In the FT-IR spectrum, the absorption bands were assigned to $\nu_{\text{O-H}}$ and $\nu_{\text{N-H}}$ at $3200\text{--}3500 \text{ cm}^{-1}$, $\nu_{\text{C-H}}$ at 2969 and 2889 cm^{-1} , $\nu_{\text{C=O}}$ at 1577 cm^{-1} , ν_{COO} at 1409 cm^{-1} , $\delta_{\text{C-H}}$ at 1340 cm^{-1} , $\rho_{\text{C-H}}$ at 1023 cm^{-1} , δ_{COO} at 670 cm^{-1} , π_{COO} at 617 cm^{-1} [24, 25], $\nu_{\text{C-N}}$ at 1099 cm^{-1} [26], $\nu_{\text{C-O}}$ at 1060 and 991 cm^{-1} [26, 27], and $\tau_{\text{O-H}}$ and $\tau_{\text{N-H}}$ at 899 cm^{-1} [26].

3.3 Thermal analysis and FT-IR spectra of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$

The thermal analysis of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ was operated by TG-DTA in airflow, as shown in Fig. 2. The temperature at $134 \text{ }^\circ\text{C}$ appeared as an endothermic peak and weight loss was observed. A small endothermic peak appeared at $263 \text{ }^\circ\text{C}$, and the weight loss value was 20.6%. The temperature was similar to the boiling point of H_2DEA . Exothermic peaks due to oxidation reaction appeared at 310 , 387 , 414 , and $467 \text{ }^\circ\text{C}$ with weight losses of 19.0, 14.1, 8.8, and 7.2%, respectively. Finally, the organic compounds were completely burnt, and ZnO was obtained as char ceramic (char yield 27.2%, calculated yield 28.2%). The obtained ZnO was derived from the wurtzite structure by the XRD spectrum (see Supporting information).

The states of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ at various temperatures were measured by FT-IR spectra, as shown in Fig. 3. The measured samples were prepared using $100 \mu\text{L}$ of 10 wt% $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ -ethanolic solution on silicon wafer by spin-coating at various temperatures for 30 min in air. At $110 \text{ }^\circ\text{C}$, the IR spectrum was similar to that of pure $\text{Zn}(\text{OAc})_2$

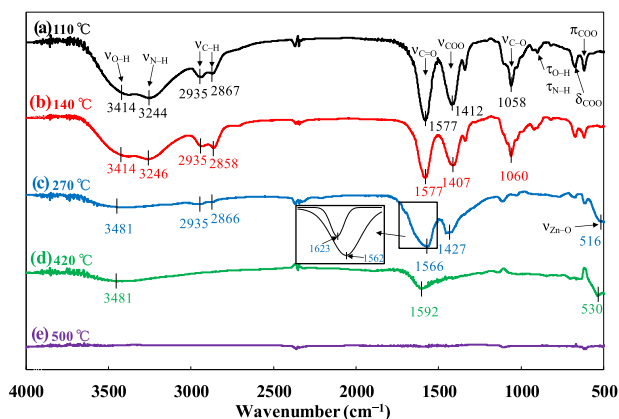


Fig. 3 FT-IR spectra of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ on heat treatment at a $110 \text{ }^\circ\text{C}$, b $140 \text{ }^\circ\text{C}$, c $270 \text{ }^\circ\text{C}$, d $420 \text{ }^\circ\text{C}$, and e $500 \text{ }^\circ\text{C}$ for 30 min

(H_2DEA). When heated at $140 \text{ }^\circ\text{C}$, the band assigned to ν_{COO} at 1407 cm^{-1} was slightly broader than that at $110 \text{ }^\circ\text{C}$. From ^1H NMR spectrum, the signals of methylene derived from H_2DEA appeared at two regions comprising different chemical environments, and the endothermic peak for TG-DTA appeared at $134 \text{ }^\circ\text{C}$. Therefore, the temperature was identified with the decomposition temperature of $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$. On additional heating to $270 \text{ }^\circ\text{C}$, the bands assigned to $\nu_{\text{N-H}}$, $\nu_{\text{C-H}}$, and $\nu_{\text{C-O}}$ (at 3414 and 3246 , 2935 and 2866 , and 1060 cm^{-1} , respectively) almost disappeared. The broad band at 1566 cm^{-1} overlapped with the bands due to $\delta_{\text{O-H}}$ at 1623 cm^{-1} and $\nu_{\text{C=O}}$ at 1562 cm^{-1} ; therefore, the H_2DEA ligand was completely dissociated and evaporated at $270 \text{ }^\circ\text{C}$. Moreover, a new band at 516 cm^{-1} appeared. The new band was assigned to Zn-O stretching vibrations derived from cluster-forming Zn_4O cores such as $\text{Zn}_4\text{O}(\text{OAc})_6$ [28, 29]. However, the IR spectrum was different from that of pure $\text{Zn}_4\text{O}(\text{OAc})_6$ (see Supporting information). Probably, the coating film contained a segment of μ_4 -oxozincate formation. When heated to $420 \text{ }^\circ\text{C}$, the bands assigned to $\nu_{\text{C=O}}$ and $\nu_{\text{C-O}}$ disappeared, and $\nu_{\text{Zn-O}}$ was unclear due to cleavage of the Zn_4O structure. Moreover, the band due to $\nu_{\text{O-H}}$ was assigned to the absorption of water and/or hydroxyl groups of Zn compounds. Finally, the bands of organic, hydroxyl groups, and Zn_4O core completely disappeared at $500 \text{ }^\circ\text{C}$.

Based on report [12], we propose the formation of ZnO through $\text{Zn}(\text{OAc})_2(\text{H}_2\text{DEA})$ as shown in Scheme 2. When heated at $140 \text{ }^\circ\text{C}$, a part of the coordinated hydroxyl group on H_2DEA was dissociated and/or re-coordinated to other zinc atoms as in polymerization (intermediate 1). At $270 \text{ }^\circ\text{C}$, most of the H_2DEA was removed and $\text{Zn}(\text{OAc})_2$ changed to ZnO colloidal formation from a few μ_4 -oxozinc compounds such as $\text{Zn}_4\text{O}(\text{OAc})_6$, $\text{Zn}_{10}\text{O}_4(\text{OAc})_{12}$, and $\text{Zn}_5(\text{OH})_8(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$ because the band at 516 cm^{-1} was assigned to Zn-O stretching vibrations of the Zn_4O core and these compounds were prepared by the hydrolysis–condensation of $\text{Zn}(\text{OAc})_2$ in the presence of a basic compound [30–32]. After heating at

Scheme 2 A possible thermal decomposition pathway from Zn(OAc)₂(H₂DEA) to ZnO

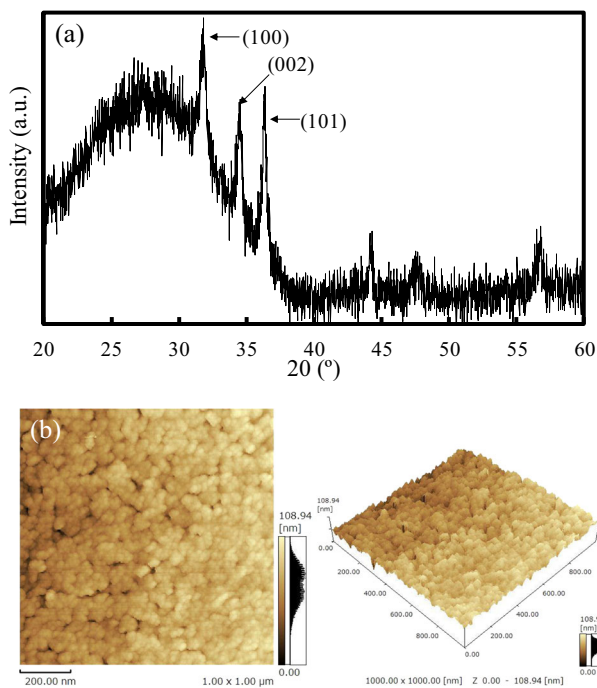
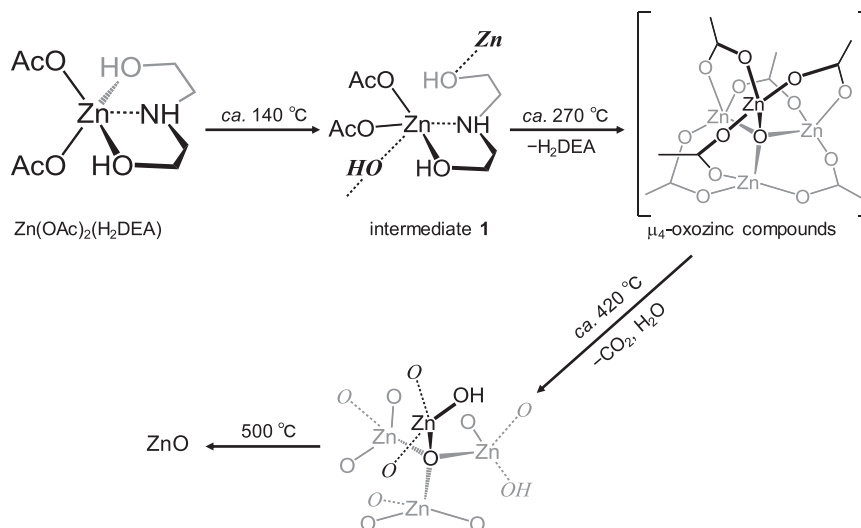


Fig. 4 a XRD spectrum and b AFM images of ZnO thin film

420 °C, the acetato groups were burnt and the hydroxyl group was formed; at 500 °C, ZnO was formed by dehydration–condensation between hydroxyl groups. From the result, it was clear that H₂DEA acted as a ligand and also as a basic compound to form μ₄-oxozinc compounds.

3.4 Characterization of ZnO thin film via heat treatment of Zn(OAc)₂(H₂DEA)

The ZnO thin film was prepared by heat treatment at 550 °C of Zn(OAc)₂(H₂DEA) coated on a glass substrate. The ZnO

thin film was characterized by XRD and AFM, as shown in Fig. 4. The XRD pattern of ZnO thin film appeared at the diffraction peaks of (100), (002), (101), (102), and (110) as derived from the hexagonal wurtzite structure [33, 34]. A broad peak between 20° and 40° was assigned to the amorphous glass substrate. Densely packed ZnO particles were observed with a size of approximately 40 nm using AFM. Finally, the transmittance was high (>90%) in the visible region.

4 Conclusion

Zn(OAc)₂(H₂DEA) was synthesized by the reaction of Zn(OAc)₂·2H₂O with H₂DEA in EtOH. The structure of the intermediate was revealed. It was characterized by single-crystal X-ray structural analysis, NMR and FT-IR spectra, and elemental analysis. The single-crystal X-ray structure comprised a zinc atom, two acetate groups, and one H₂DEA as a neutral ligand. From the TG-DTA and FT-IR study, Zn(OAc)₂(H₂DEA) decomposed at 140 °C. At 270 °C, most of the H₂DEA was removed and μ₄-oxozinc compounds were formed. After heating at 420 °C, the acetato groups burned to form the hydroxyl group. At 500 °C, ZnO was formed. The ZnO film formed from Zn(OAc)₂(H₂DEA) showed high transparency (>90%) and a hexagonal wurtzite structure. Densely packed ZnO particles were observed with a size ~40 nm.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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