JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

PREPARATION AND CHARACTERIZATION OF ZINC CHLORIDE THROUGH HYDROTHERMAL SYNTHESIS

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Abstract

This study comprehensively examines and presents the elemental, structural, morphological, and optical characteristics of Zinc Chloride ($ZnCl_2$) through the utilization of Electron Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), and Ultraviolet-Visible Spectroscopy (UV-vis). The preparation and characterization of zinc chloride through hydrothermal synthesis involves the creation of zinc chloride crystals using a hydrothermal reaction process. This yielded high-purity products with well-defined crystal structures. EDS analysis revealed that the molecular weight of zinc chloride ($ZnCl_2$) constitutes 79.76% of its total weight, while it contains 20.24% oxygen an impurity or contaminant within zinc chloride ($ZnCl_2$). Furthermore, SEM captures the rod-like crystal morphology of zinc chloride ($ZnCl_2$). UV-visible results illustrate a reduction in absorbance from an initial intensity from 3.4 to 0.9 within the wavelength range of 227 nm to 400 nm. Subsequently, the absorbance intensifies from 2.4 to 3.4, culminating in maximum absorbance at 227 nm wavelength. Notably, the hygroscopic characteristic of zinc chloride is verified following exposure to atmospheric conditions. This research firmly establishes zinc chloride ($ZnCl_2$) as a hygroscopic compound, showcasing its promising potential as a preservative and desiccant across diverse applications.

Keywords: Zinc chloride, Characterization, SEM-EDS, SEM, and UV-visible.

Introduction

In recent times, the global community has grappled with the pressing challenge of preserving items while ensuring their adequate security. This has spurred a significant interest in the potential of nanomaterials to effectively address these challenges. Nanomaterials, characterized by their size ranging from 1 to 100 nanometers, are prevalent in nature and are subjects of study across various scientific disciplines including Chemistry, Physics, Geology, and Biology (5).

One noteworthy inorganic compound is Zinc Chloride, pivotal as a source of zinc and renowned for its diverse industrial applications (3). Regrettably, it lacks comprehensive and precise physicochemical, thermal, and spectral characterization. This research endeavors to bridge this gap by utilizing contemporary analytical techniques to thoroughly characterize Zinc Chloride. The compound finds utility as a condensing agent, disinfectant, dehydrating agent, and deodorant. Additionally, it serves as a flux and cleaning agent for soldering, effectively dissolving oxide layers on metal surfaces (1). Given its deliquescent nature and hygroscopic properties, it possesses the capacity to attract and absorb moisture from the environment, necessitating protection from such moisture (9).

Zinc Chloride manifests in five distinct hydrate forms with the general formula $ZnCl_2(H_2O)n$, where n can take on values such as 1, 1.5, 2.5, 3, and 4. This compound also exhibits polymorphism, adopting various crystal structures including tetragonal, monoclinic, and orthorhombic (6). The bonding within Zinc Chloride, involving the zinc cation and chloride anions, holds both ionic and covalent characteristics, leading to its modest melting point and solubility in ethereal solvents. A comprehensive understanding of these fundamental attributes is pivotal to unlocking Zinc Chloride's versatile potential across a multitude of industries.

Zinc Chloride, acting as a solvent, demonstrates the ability to dissolve high molecular weight bacterial cellulose. However, this cellulose-dissolving capacity is significantly influenced by the water content, particularly in the case of $ZnCl_2$ hydrates (2). Studies indicate that the zinc chloride-to-water molar ratio exerts a notable influence on product yield, which rises from 6.3% to 12.0% as the hydrogen extent shifts from R=3.78 to 3.36 under constant reaction conditions (10). This increase in product yield is attributed to enhanced cellulose dissolution in the solvent with higher Zinc Chloride concentration.

The synthesis of diverse carbon materials has involved hydrothermal carbonization and subsequent $ZnCl_2$ activation, employing varied mass ratios of the activating reagent. Physical and electrochemical characterization of resulting powder materials in a two-electrode cell configuration, utilizing 1-ethyl-3-methylimidazolium tetrafluoroborate ionic liquid, revealed a pronounced dependence on the activation process for materials' porosity (7). However, the effectiveness of $ZnCl_2$ activation is fundamentally contingent upon activation conditions and precursor materials used (8).

Several researchers have conducted X-ray structure determinations of $ZnCl_2$ complexes in concentrated aqueous solutions, culminating in a summary of their findings by Richards and Williams (1970). Notably, the highest complex identified is $(ZnCl_4)^{2-}$, exhibiting a tetrahedral structure. This study, in contrast, undertakes an exhaustive investigation of $ZnCl_2$ through diverse analytical methods, including Electron Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), and UV-visible Spectroscopy (UV-vis), to comprehensively analyze the elemental, structural, morphological, and optical properties of Zinc Chloride.

Methodology

Sample Preparation of Zinc Chloride

The Zinc chloride sample was meticulously prepared in the Chemistry laboratory of Kogi State University. A total of 20 grams of pure zinc granules were accurately weighed into a beaker. Subsequently, 70ml of hydrochloric acid was added gradually to the beaker containing the zinc granules. The dissolution process took approximately 60 minutes to ensure complete dissolution of the zinc granules. Throughout this process, hydrogen gas was released into the atmosphere.

The interaction between the zinc granules and hydrochloric acid generated a hydrous form of zinc chloride. The chemical equation representing this reaction is as follows:

$$Zn_{(s)} + 2HCl_{(aq)} \longrightarrow ZnCl_{2(aq)}$$

To obtain anhydrous zinc chloride, the solution in the beaker was subjected to evaporation by heating it on a hot plate. The resulting sample was precisely weighed and transported to Rolab Research and Diagnostic Laboratory in Ibadan for further experimental analysis, which included Energy Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), and UV-visible Spectroscopy.

Results and Discussion

SEM-EDS Analysis

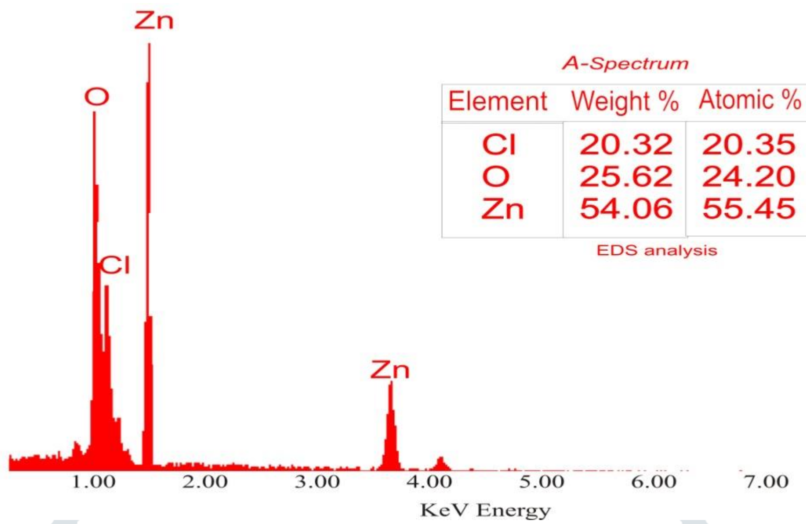

Figure 1: Energy Dispersive X-ray Spectroscopy (EDS) Analysis

The EDS analysis of zinc chloride displayed in Figure 1 indicates that zinc constitutes 74.38wt.% of the total elements present. Further breakdown reveals that zinc contributes 54.06wt.%, chlorine accounts for 20.32wt.%, and oxygen, serving as an impurity, makes up 25.62wt.%. Additionally, the atomic percentages of zinc, chlorine, and oxygen are found to be 54.06, 20.32, and 24.20 respectively.

Scanning Electron Microscope (SEM) Images Analysis

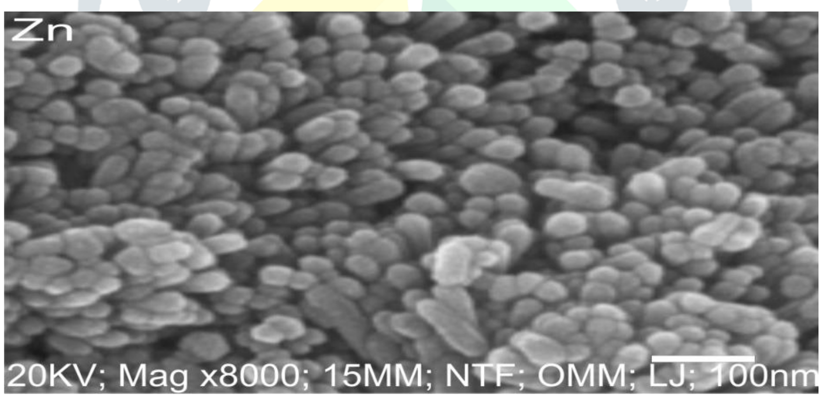

Figure 2: Micrograph of the Sample ($ZnCl_2$) at 100x Magnification

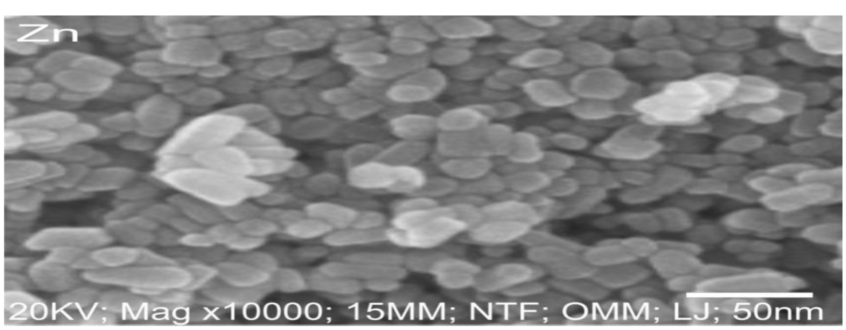

Figure 3: Micrograph of the Sample ($ZnCl_2$) at 50x Magnification

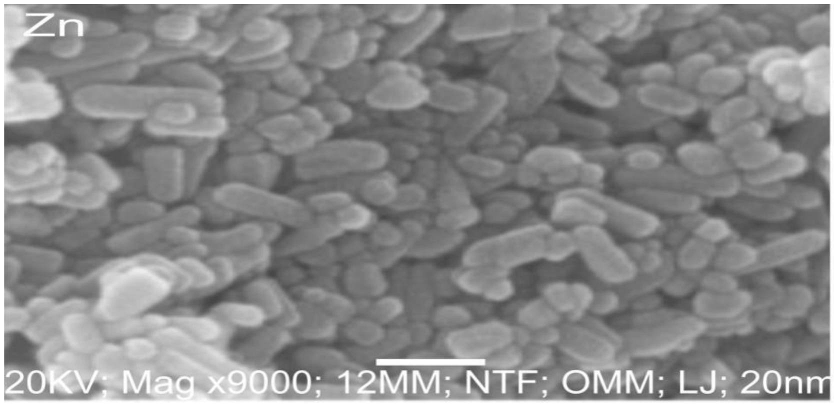

Figure 4: Micrograph of the Sample ($ZnCl_2$) at 20x Magnification

The Scanning Electron Microscope was employed to assess the morphological attributes of the zinc chloride sample. The SEM images reveal the shapes and distributions of the zinc chloride particles.

As depicted in Figures 2, 3, and 4, the particles exhibit a rod-like crystalline structure. The proximity of the molecules to one another is more pronounced when the wavelength is 20nm (Figure 4). At a wavelength of 50nm, the particles are smaller and sparsely dispersed, with noticeable pinholes (Figure 3). Conversely, at 100nm wavelength, a finer arrangement is observable, characterized by smaller and evenly distributed molecules (Figure 2). These variations in particle arrangement are influenced by the presence of other elements in the zinc chloride sample.

UV-Visible Analysis

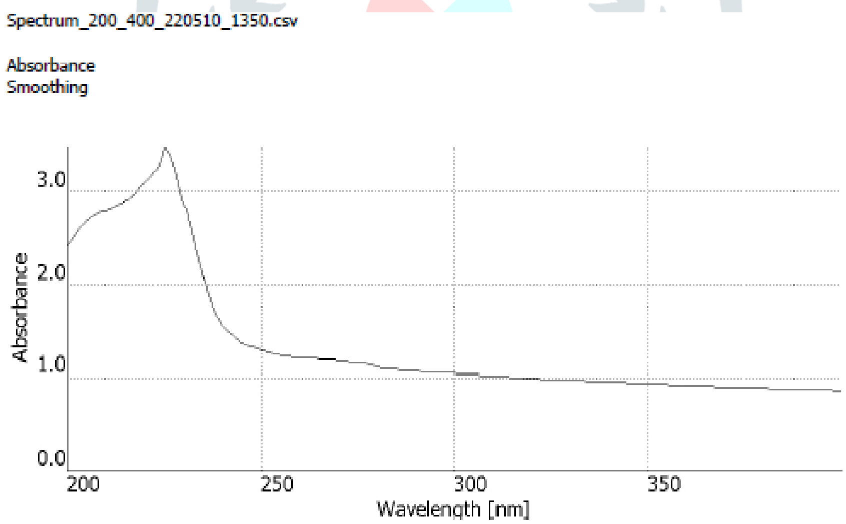

Figure 5: Absorbance Spectrum of the Sample ($ZnCl_2$)

The UV-visible analysis indicated a decrease in absorbance from 3.4 to 0.9 across the wavelength range of 227nm to 400nm (Figure 5). Additionally, the intensity increased from 2.4 to 3.4 before reaching the maximum absorbance point. This maximum absorbance (λ-max) was determined to occur at a wavelength of 227nm. This value is significant in elucidating the molecular properties of zinc chloride.

Hygroscopic Proof

Table 1: Zinc Chloride Weight Measurements Over Five Days

S/N	Days	Mass (with Beaker) (g)	Mass (minus Beaker) (g)
1	1	147.92	52.92
2	2	149.36	54.36
3	3	151.68	56.68
4	4	153.10	58.10
5	5	155.80	60.80

Empty beaker weight = 95g Sample weight in beaker = 50g

The provided data, obtained from weighing the synthesized Zinc Chloride while exposed to air at room temperature, demonstrates the compound's hygroscopic nature. Over a five-day period, consistent daily measurements were taken at the same time interval (3:00pm). It was apparent that the sample's size increased daily, with a noticeable liquefying effect upon exposure to air. This observation firmly establishes the hygroscopic property of Zinc Chloride.

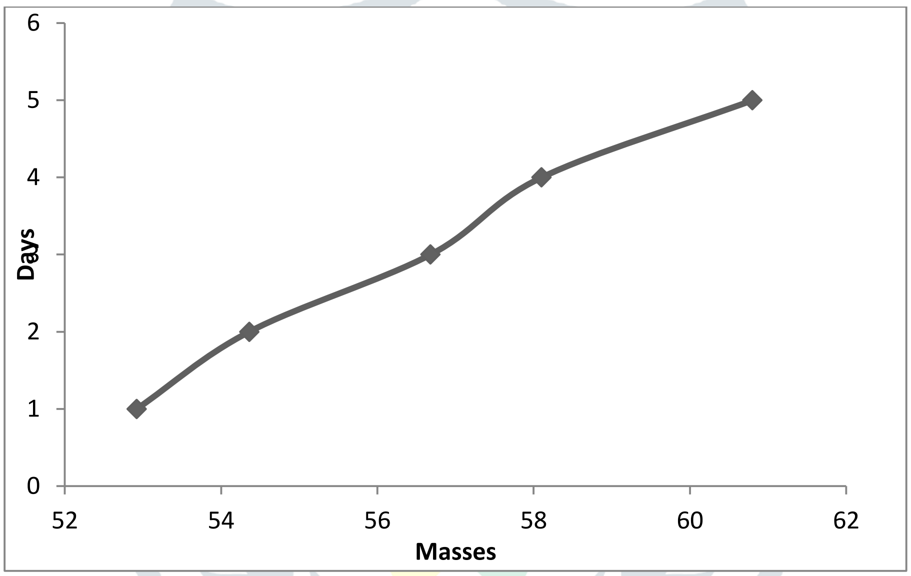

Figure 6: Graph Illustrating Zinc Chloride's Hygroscopic Nature

Conclusion

The findings derived from the energy dispersive X-ray spectroscopy (EDS), scanning electron microscope (SEM), and UV-visible spectroscopy analyses provide valuable insights. The EDS analysis highlighted that zinc chloride constitutes 74.38wt.% of the sample's elements, with impurities such as oxygen present. SEM images indicated rod-like crystalline shapes of the nanoparticles, with their distribution being influenced by the wavelength. The UV-visible analysis uncovered maximum absorbance at 227nm, shedding light on the compound's molecular properties. Furthermore, the hygroscopic nature of zinc chloride was effectively proven through consistent weight measurements over five days. Collectively, this research enhances our understanding of zinc chloride, facilitating its optimal utilization across various industrial processes.

Acknowledgment
The authors are grateful to Kogi State University, Staffs & management of Physics Department and Chemistry Department for their supports and cooperation.

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