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# Analysis of Crystalline Phase and Functional Groups of ZnO from *Pineapple* Peel Extract

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**Abstract**

*The use of pineapple peel has not been paid attention to because of the lack of public knowledge regarding the content and benefits of pineapple skin. However, it can use pineapple skin for herbal drinks and animal feed. Pineapple peel has phytochemical compounds and bioactive compounds such as ZnO. Therefore, this study was conducted to determine ZnO's crystalline phase and the functional group from pineapple peel extract. Synthesis of ZnO from pineapple peel extract used the green synthesis method, with the following steps: pineapple peel was pounded and stirred by heating at 80°C, and then ultrasonicated. The solution was added with NaOH and centrifuged at 4000 rpm. This result is heated at a temperature of 120°C with a 6- and 12-hour holding time. After that, it cooled to room temperature. The results of XRD analysis obtained the optimal ZnO crystalline phase at a holding time of 12 hours according to the pdf cards database (96-210-7060) and nanocrystalline size of 17.55 nm. The FTIR results on the ZnO sample had an absorption peak of 4000-400cm$^{-1}$ with functional groups O-H alcohol and phenol, -C=C alkene, C=O, C-O, C-N, and Zn-O. It is hoped to be applied later in the medical and industrial fields by obtaining ZnO samples from pineapple peel extract.*



## Introduction

Zinc oxide (ZnO) is an inorganic semiconductor in the form of a powder that has a wide band gap compared to other metal oxides [1-3]. In addition, ZnO also has unique physical properties [4], capable of absorbing radiation [5], high catalyst activity, and non-toxic properties [5]. With these properties, ZnO can be applied in industry, optics [6], sensors [7] and medical fields such as anticancer, antifungal, and antibacterial [8]. Zinc oxide has three crystal structures: hexagonal wurtzite, cubic zincblende, and cubic rock salt [6]. Hexagonal wurtzite is the most stable structure among the three structures compared to the cubic zincblende structure grown on substrates with a cubic lattice structure. Likewise, rock salt cubic structure is rarely observed [1]. ZnO could be obtained from plant extracts such as aloe vera leaf extract [9], *rambutan* peel extract [10], *polygala tenuifolia* root extract [11], *rosa canina* fruit extract [12], ginger rhizome extract [13], *Garcia mangostana* fruit extract [14] and *pineapple* peel extract [15,3,2]. *Pineapple* fruit is easy to obtain, and the price is low, so the more *pineapple* production

increases, the more waste is generated. During this time, it is often found that *pineapple* peel waste is not optimal, so the *pineapple* skin is thrown away and only used as syrup, wound medicine, and animal feed. In addition to the fruit, roots, and leaves, *pineapple* peel extract can be used to manufacture ZnO nanoparticles. *Pineapple* skin contains bioactive compounds such as saponins, flavonoids, tannins, anthocyanins [2], vitamin C, carotenoids, bromelain enzymes, water, crude fiber, reducing sugars, carbohydrate, es, and proteins [16]. Several methods of synthesizing ZnO such as aerosol-gel, hydrothermal [17], precipitation [18], solvothermal [14], thermal decomposition [14], sonochemistry [1] and green synthesis. The green synthesis method is easy to do because the method is environmentally friendly, natural materials are easy to obtain, and the process is simple and requires relatively low costs.

Mirgane et al. (2020) reported that ZnO was obtained from *pineapple* peel extract by green synthesis at 120°C calcination for 6 hours. These results are shown from XRD data, where the ZnO phase is a high-intensity peak and is identified as a crystal. However, at an angle between 20-30°, a diffraction peak still appears even though the intensity is low and not identified. In their research, [1] showed that ZnO with a hexagonal wurtzite crystal structure was obtained by calcining at 60°C for 24 hours and centrifuging at 15000 rpm for 5 minutes. Likewise, in [2] study, ZnO was formed from a biosynthetic method with *pineapple* peel extract as a capping and reducing agent. However, the results of his research still found some impurities in the XRD data. Based on the above studies, in this study, ZnO was synthesized from pineapple peel extract using the green synthesis method with a calcination temperature of 120°C and holding times of 6 and 12 hours. With this variation in holding time, it is hoped that the optimal ZnO crystalline phase. Likewise, the ZnO of functional groups will be obtained. Therefore, ZnO can be applied in the medical and industrial fields.

## Experimental Method

Materials and tools that need to be prepared before synthesizing ZnO include *pineapple* peel, water distillation, 1 M NaOH (Merck), zinc acetate (Merck), filter paper, pH meter, beaker, Erlenmeyer tube, digital balance, magnetic stirrer, mortar and pestle, dry oven, centrifuge, and ultrasonic cleaner.

Before synthesizing ZnO, wash the *pineapple* skin first with distilled water to remove impurities adhering to the skin and then weigh using a digital balance. A total of 10 grams of *pineapple* peel was pounded with a mortar pestle and formed a yellow paste solution. The paste solution was mixed with distilled water and stirred at 80°C. After that, the filtering process is carried out, and the result is a filtrate. Then the filtrate was added with 4 grams of zinc acetate and 10 ml of distilled water and then ultrasonicated at 40°C for 1 hour. The result was added with NaOH solution to pH 12 and centrifuged at 4000 rpm to form a white precipitate. Furthermore, the residue was heated at a temperature of 120°C with a holding time variation of 6 hours and 12 hours.

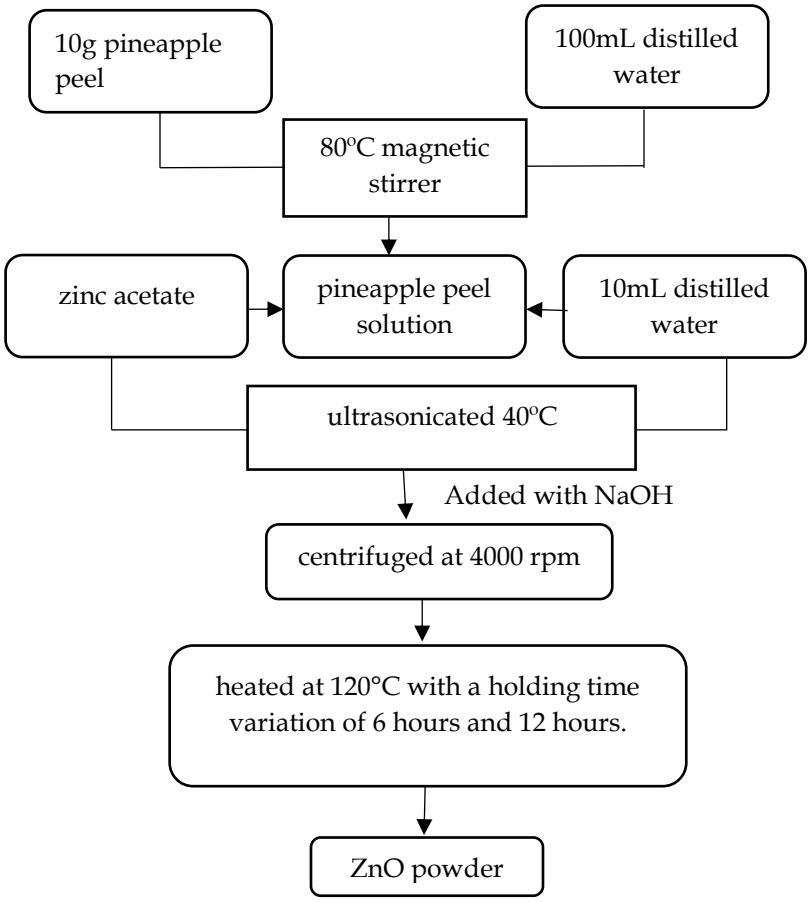


**Figure 1**. ZnO flow chart of pineapple peel extract

The collecting data on the ZnO sample using an XPert PRO type XRD (X-Ray Diffraction) characterization tool with a Cu radiation source of 30 mA, 40 kV, a wavelength of 1.54060 Å, and an angle of 2theta 10°-80°. From the XRD data obtained, to calculate the average diameter of the crystallite (d) can use the Debye-Scherrer equation [3], namely

$$\left(d = 0.94\frac{\lambda}{\beta cos\theta}\right) \text{ nm} \quad (1)$$

where the X-ray wavelength (0.15418 nm) is the peak intensity FWHM maximum at (101) and is the diffraction angle. Identification of the ZnO phase using QualX software by matching the XRD test data with the database from the software. To determine the absorption peak of the ZnO sample using the Shimadzu FTIR-8400 type FTIR (Fourier Transform Infrared) characterization tool with a wave number range of 4000 to 400 $cm^{-1}$. The data from the FTIR characterization is in the form of a graph of wave number and light transmission, where each absorption peak shows the chemical bonds of atoms.

## Result and Discussion

### XRD Characterization

The XRD characterization results for the ZnO sample were identified by phase using the QualX software, namely by matching the XRD test data with the existing database in the software. The following diffraction peaks for ZnO samples with holding times of 6 hours and 12 hours are shown in Figure 2.

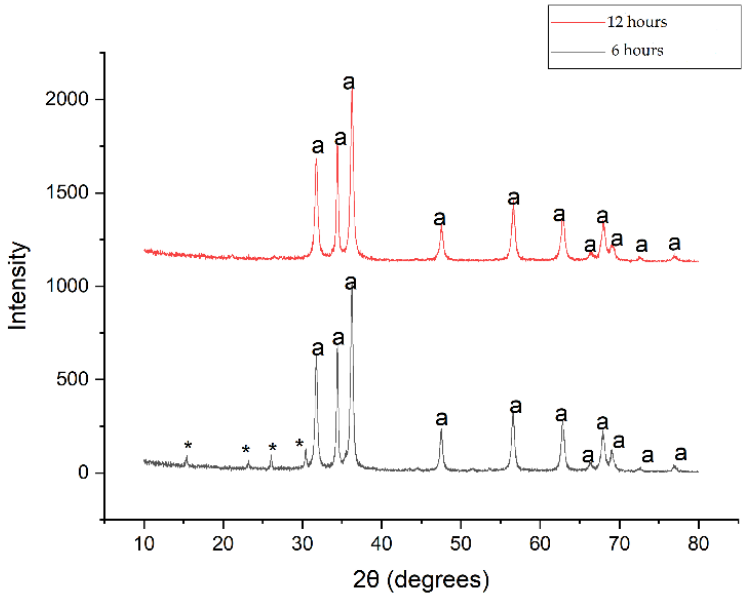


**Figure 2**. Results of XRD characterization of ZnO samples for holding times of 6 and 12 hours, (a) ZnO and (*) Impurity.

The results of XRD characterization in Figure 2 show the formation of the optimal phase of ZnO, namely Zincite, with a hexagonal structure identified in the sample with a holding time of 12 hours. A diffraction peak characterizes it with maximum intensity at an angle of 36.28° with a Miller index (101). The diffraction peak at this angle corresponds to the results of research conducted by Ahmad et al. (2019), which is 36.245° (101), and also in the study of Sari et al. (2021), which is at the diffraction peak with an angle of 36.28° [2]. Other diffraction peaks detected in the Zincite (ZnO) phase were 31.88°, 34.56°, 47.68°, 56.76°, 63.06°, 66.51°, 68.14°, 69.30°, 72.90°, 77.23° with Miller index (100), (002), (102), (110), (103), (200), (112), (201), (004), (202) which correspond to the data pdf cards number 96-210-7060. No impurities were found in the sample with a holding time of 12 hours. Therefore, it was formed in the ZnO phase at the optimal. In contrast to the 6-hour holding time sample, impurities were still found with minimum intensity at angles of 15.61°, 23.24°, 26.24°, and 30.24°. The presence of impurities may be derived from the pineapple peel content in the ZnO sample [2]. Based on the Debye-Scherrer formula, the ZnO crystallite sizes of the samples to representatives for the 6 and 12 hours holding times were 20.68 nm and 17.55 nm, respectively. Both samples have

nanocrystalline characters, namely crystallite size below 100 nm [19]. For more details related to the size of the ZnO crystallites, see Table 1 below.

**Table 1. ZnO crystallite size using the Debye-Scherrer formula**

| No | Sample | d-spacing (Å) | FWHM | Average crystallite size (nm) |
|---|---|---|---|---|
| **1** | ZnO 6 hours | 2.816135 | 0.438174 | 20.68 |
| **2** | ZnO 12 hours | 2.81499 | 0.518771 | 17.55 |

**Table 2. ZnO 12 hours**

| No | Peak position 2 theta | FWHM | Crystallite size | Average crystallite size (nm) |
|---|---|---|---|---|
| **1** | 31,76224 | 0,48937 | 16,87791062 | |
| **2** | 34,41748 | 0,33332 | 24,95078617 | |
| **3** | 36,25299 | 0,48824 | 17,12095799 | |
| **4** | 47,55552 | 0,50753 | 17,10476275 | 17,55458414 |
| **5** | 56,61637 | 0,54799 | 16,46636066 | |
| **6** | 62,87436 | 0,5654 | 16,46804359 | |
| **7** | 67,99366 | 0,6897 | 13,89326724 | |

**Table 3. ZnO 6 hours**

| No | Peak position 2 theta | FWHM | Crystallite size | Average crystallite size (nm) |
|---|---|---|---|---|
| **1** | 31,74897 | 0,41787 | 19,76516875 | |
| **2** | 34,41071 | 0,28784 | 28,89259271 | |
| **3** | 36,23417 | 0,41523 | 20,13025851 | |
| **4** | 47,52225 | 0,4014 | 21,62448993 | 20,68356195 |
| **5** | 56,58368 | 0,48316 | 18,67293454 | |
| **6** | 62,85415 | 0,49651 | 18,75093808 | |
| **7** | 67,94583 | 0,56521 | 16,94855115 | |

From the pineapple peel ZnO experiment, it can be concluded that at 12 hours the crystalline size of ZnO is smaller than the crystalline size of ZnO at 6 hours, but the impurities are less so that with the longer drying time of ZnO pineapple peel (more than 12 hours) the crystalline size is getting smaller.

## FTIR Characterization

The results of FTIR characterization show an absorption peak with a wave number range of 4000 to 400 $cm^{-1}$, which indicates the chemical bonding of a sample. The data can be seen in Figure 2 for the ZnO sample with a holding time of 12 hours.

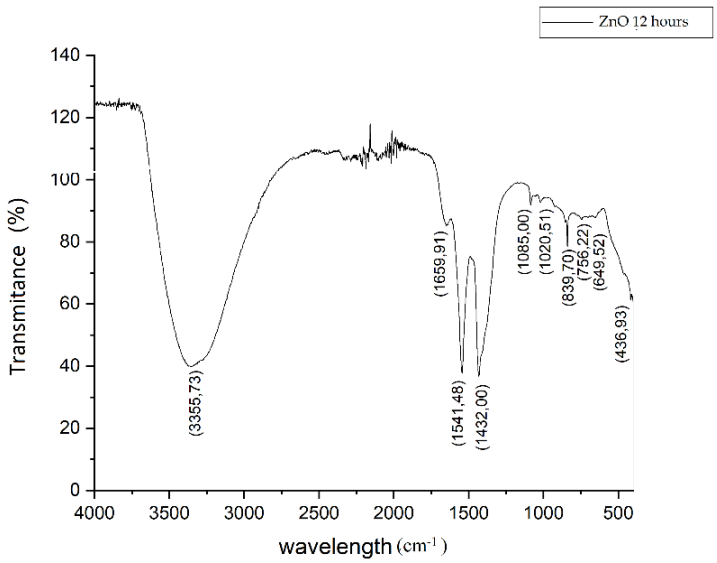


**Figure 3**. Results of FTIR characterization of ZnO samples

The results of the FTIR characterization in Figure 3 show the absorption peaks of ZnO synthesized from pineapple peel extract using the green synthesis method with a holding time of 12 hours, namely 3355.73 $cm^{-1}$, 1659.91 $cm^{-1}$, 1541.48 $cm^{-1}$, 1432.00 $cm^{-1}$, 1085.00 $cm^{-1}$, 1020.51 $cm^{-1}$, 839.70 $cm^{-1}$, 756.22 $cm^{-1}$, 649.52 $cm^{-1}$, and 436.93 $cm^{-1}$. The absorption peak at a wave number of 3355.73 $cm^{-1}$ shows the O-H bond of alcohol and phenol, and the peak is by research [11], namely at a wave number of 3380 $cm^{-1}$. The wave number 1659.91 $cm^{-1}$ indicates a chemical bond –C=C alkene. The C=O vibrational bond with the carboxylic acid group occurs at a wave number of 1541.48 $cm^{-1}$. Likewise, at 1432 $cm^{-1}$, there is a C-O bond, and at 1085 $cm^{-1}$, there is a C-N vibration. At the absorption peak of 839.70 $cm^{-1}$, the formation of tetrahedral coordination of Zn occurs, and the peak matching is to Jayarambabu et al. (2015) research at a wave number of 875 $cm^{-1}$ [4]. The ZnO functional group is found at wave numbers 756.22, 649.52, and 436.93 $cm^{-1}$. Basri et al. (2020) research that the ZnO functional group is at a wave number of 800-400 $cm^{-1}$ [1]. For more details, the FTIR spectrum synthesized can be seen in Table 4.

**Table 4. ZnO Functional Group Bonds**

| No | Synthetic ZnO ($cm^{-1}$) | Reference Peak ($cm^{-1}$) | Bond type | Reference |
|---|---|---|---|---|
| **1** | 3355.73 | 3380 | O-H alcohol and phenol | [5] |
| **2** | 1659.91 | 1660 | –C=C alkene | [6] |
| **3** | 1541.48 | 1562-1577 | C=O | [7] |
| **4** | 1432.00 | 1425-1446 | C-O | [7] |
| **5** | 1085.00 | 1075 | C-N vibration | [6] |
| **6** | 839.70 | 875 | Zn formation | [4] |
| **7** | 756.22, 649.52 dan 436.93 | 443 dan 546 | ZnO | [8] |
| | | 800-400 | | [1] |


**Figure 4**. ZnO Mechanism

## Conclusion

Based on the results and discussion in this study, it can be concluded that pineapple peel has phytochemical compounds and bioactive compounds such as ZnO. Synthesis of ZnO from pineapple peel using the green synthesis method, where the results of XRD characterization obtained the optimal ZnO crystalline phase at a holding time of 12 hours according to the pdf cards database (96-210-7060) and got a nanocrystalline size of 17.55 nm. The FTIR results on the ZnO sample had an absorption peak of 4000-400 cm-1 with functional groups O-H alcohol and phenol, -C=C alkene, C=O, C-O, C-N, and Zn-O.

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