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# Synthesis and Characterization of Zirconium Oxychloride from Bangka and Belitung Zircon Sand

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## Abstract

Although zircon sand, a byproduct of tin mining in Bangka and Belitung, is abundant and rich in zirconium content, it remains underutilized despite its potential for conversion into value-added products. This study aims to explore the feasibility of using Bangka and Belitung zircon sand as raw materials for the synthesis and characterization of zirconium oxychloride octahydrate ($ZrOCl_2{\cdot}8H_2O$), and to evaluate its properties relative to a commercial reference. A hydrometallurgical process involving alkaline fusion and acid leaching was employed to convert zirconium silicate ($ZrSiO_4$) into $ZrOCl_2{\cdot}8H_2O$. Comprehensive characterization was conducted using X-ray fluorescence, X-ray diffraction, Fourier-transform infrared spectroscopy, scanning electron microscopy, and particle size analysis. The results revealed that Bangka zircon sand contains a higher zirconium content (79.12%) than Belitung sand (76.97%). Both sources exhibited lower zirconium purity and higher chlorine content compared to a reference. Despite these limitations, the total Zr and Cl content reached 88.12% for the Bangka sample and 86.08% for the Belitung sample, indicating promising potential. Both products shared similar structural and morphological features in different sizes. Belitung-derived $ZrOCl_2{\cdot}8H_2O$ exhibited a higher $ZrOCl_2{\cdot}8H_2O$ phase, more defined morphology, and smaller size, suggesting it has better properties as a precursor in zirconium-based applications. This study supports the valorization of mining byproducts and highlights the potential of local zircon sand as a raw material for zirconium-based applications.

## 1. Introduction

The Indonesian government's pursuit of sustainable green energy is intrinsically linked to mineral utilization in emerging technologies, including electric vehicles, energy storage, and electronic devices. However, the transition to renewables and net-zero emissions hinges on mineral availability and efficient processing [1]. Although mining is necessary to obtain these minerals, it inevitably damages the environment and generates substantial mineral waste.

Tin tailing sand is one of the major mineral waste products generated by the tin processing industry, particularly in the Bangka Belitung Islands of Indonesia. This region, known for its extensive tin mining activities, produces over 1.2 million tonnes of tin tailings annually [2]. The environmental impact of these tailings goes beyond their chemical content; improper handling and disposal can lead to severe environmental degradation. Dust emissions from dry tailing piles contribute to air pollution, while accidental releases during mining and processing operations pose risks to surrounding ecosystems and communities [3].

On the other hand, tin tailings contain a variety of associated minerals, one of the most valuable being zirconium, which is primarily found in the form of zircon sand. Zircon occurs as zirconium dioxide ($ZrO_2$) with concentrations of up to 65%, and more commonly as zirconium silicate ($ZrSiO_4$), with concentrations ranging from 88% to 94% [4, 5, 6]. Despite its abundance, zircon sand remains largely underutilized, even though it holds significant potential as a precursor for high-value zirconium-based compounds [7].

Zirconium oxychloride ($ZrOCl_2{\cdot}8H_2O$), a water-soluble derivative of zirconium, has garnered attention for its extensive applications in industries ranging from catalysis, energy storage, and environmental remediation due to its unique properties and ability to form stable complexes [8, 9]. At present, $ZrOCl_2{\cdot}8H_2O$ is primarily synthesized from high-grade zircon sources through energy-intensive and costly processes. This approach not only increases production costs but also puts pressure on primary zircon resources. With growing interest in sustainable resource management and waste valorization, there is a strong incentive to explore alternative sources and methods for producing zirconium-based product [10].

Zircon sand is the main raw material of $ZrOCl_2{\cdot}8H_2O$ production. This resource is a naturally abundant mineral primarily composed of zirconium silicate ($ZrSiO_4$) [11]. Poernomo *et al.* [12] reported that $ZrOCl_2{\cdot}8H_2O$ from West Kalimantan zircon sand had 38.39% $ZrO_2$ content by magnetic separator to increase $ZrO_2$ purity and hydrometallurgical process, while Liu *et al.* [13] succeeded with 58% by using zircon sand concentrate via alkali fusion method. This may be attributed to the method employed and the source of zircon sand utilized. However, the comprehensive characterization of $ZrOCl_2{\cdot}8H_2O$ synthesized directly from Bangka and Belitung zircon sand via a hydrometallurgical method has not been thoroughly explored, while they have more zirconium content than previous work and are underutilized.

Therefore, this study explores the potential of Bangka and Belitung zircon sand, a byproduct of tin processing in Bangka and Belitung, as a valuable product. The objective is to synthesize and characterize $ZrOCl_2{\cdot}8H_2O$ from Bangka and Belitung zircon sand, and to evaluate its characteristics by comparing it to the reference. The findings are expected to inform the suitability of these materials as precursors for zirconium-based compounds.

## 2. Experimental

### 2.1. Materials

The materials used in this study were zircon sand sourced from the Bangka and Belitung Islands (samples were byproducts of tin processing), distilled water, hydrochloric acid (HCl; analytical grade), sodium hydroxide (NaOH; analytical grade), filter paper, and zirconium oxychloride ($ZrOCl_2{\cdot}8H_2O$) for analysis (Sigma Aldrich).

### 2.2. Experiment

#### 2.2.1. Zircon Sand Preparation

The preparation of zircon sand involved washing, drying, and filtering processes using a 100-mesh sieve to produce a fine powder. The resulting powder was subsequently analyzed to determine its composition, structure, phase, morphology, and particle size.

#### 2.2.2. Synthesis of Zirconium Oxychloride from Zircon Minerals

The prepared zircon sand powder was melted with NaOH 1:1.25% (w/b) in an open container at 700°C for 3 hours. After that, the mixture was allowed to stand for a while at room temperature and washed with distilled water for 3 hours. It was then filtered to separate the water-soluble sodium silicate ($Na_2SiO_3$) from the insoluble sodium zirconate ($Na_2ZrO_3$). The dry insoluble sodium zirconate is leached with 12 M hydrochloric acid (HCl) to produce zirconium oxychloride ($ZrOCl_2{\cdot}xH_2O$), which is converted into a crystalline structure by cooling the solution. The crystals were isolated from the mother liquor by decantation, followed by filtration and further dried in air to obtain a white powder of $ZrOCl_2{\cdot}8H_2O$ [14]. This product was compared with the reference (commercial $ZrOCl_2{\cdot}8H_2O$).

### 2.3. Material Characterization

Elemental content was measured using X-ray fluorescence (XRF; Shimadzu 8201 PC) for zircon sand, synthesized $ZrOCl_2{\cdot}8H_2O$, and commercial $ZrOCl_2{\cdot}8H_2O$. Structural and phase analyses were conducted using X- ray diffraction (XRD; Shimadzu XRD-7000), while the functional groups of the samples were determined using Fourier Transform Infrared spectroscopy (FTIR; Spirit). Surface morphology was examined by Scanning Electron Microscopy (SEM; SU500), and the particle sizes of zircon sand and $ZrOCl_2{\cdot}8H_2O$ were measured using a Particle Size Analyzer (PSA; BIOBASE BK-802N).

## 3. Results and Discussion

### 3.1. Characteristics of Bangka and Belitung Zircon Sand

The characteristics of Bangka and Belitung zircon sand were carried out through several analyses, namely composition analysis, structure and phase analysis, functional group, and morphological analysis. Compositional analysis was conducted using XRF instrumentation, the results of which are presented in Table 1.

**Table 1**. Chemical composition of Bangka and Belitung zircon sands

| Element | Content (%) | |
|---|---|---|
| | Bangka zircon sand | Belitung zircon sand |
| Zr | 79.12 | 76.97 |
| Si | 5.98 | 3.68 |
| Fe | 0.56 | 0.445 |
| Ti | 2.87 | 0.386 |
| Al | 0.254 | 0.467 |

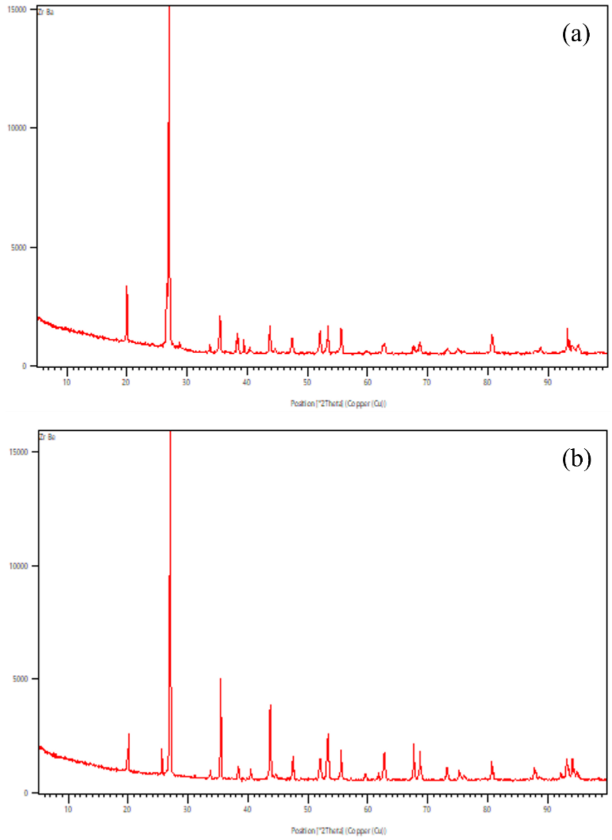


**Figure 1**. XRD diffractograms of zircon sand from (a) Bangka and (b) Belitung

Based on Table 1, the Bangka zircon sand exhibits the highest zircon content at 79.12%, while the Belitung zircon sand is slightly lower by 2.15%, at 76.97%. Both samples also contain other elements, including silicon (Si), iron (Fe), titanium (Ti), and aluminum (Al). These results indicate that zircon sand from Bangka and Belitung possesses a high zirconium percentage, making it suitable for conversion into zirconium-based derivatives. Further characterization was performed through phase analysis using XRD, as shown in Figure 1.

The dominant crystal phase in the zircon sand samples from both Bangka and Belitung is identified as the $ZrSiO_4$ phase. This is indicated by diffraction peaks at 2θ angles of 26.97°, 35.42°, 43.66°, and 53.37°, corresponding to the ICDD 01-081-0588 reference for $ZrSiO_4$. These results align with the chemical composition data, confirming the presence of both zirconium and silicon, consistent with $ZrSiO_4$.

Functional group analysis using FTIR is shown in Figure 2. Bangka and Belitung zircon sand exhibit similar absorption spectra, with characteristic peaks in the fingerprint region. The minor absorption peaks at 3745 $cm^{-1}$ and 3740 $cm^{-1}$ are attributed to the stretching vibration of the -OH group of water molecules, further supported by the absorption band at 2377.82 $cm^{-1}$, which corresponds to the stretching vibration of zirconium hydroxyl (Zr–OH). The functional group of zircon sand was identified at 820 $cm^{-1}$ and 821 $cm^{-1}$, indicating the presence of the $SiO_4^{2-}$ functional group. Additionally, peaks observed at 590 $cm^{-1}$ and 582 $cm^{-1}$ correspond to Zr–O stretching vibrations in the $ZrO_2$ phase [15].

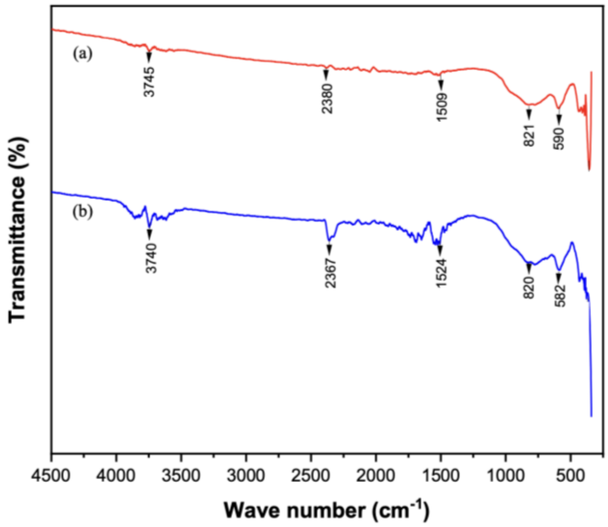


**Figure 2**. FTIR spectra of zircon sand sourced from (a) Belitung and (b) Bangka

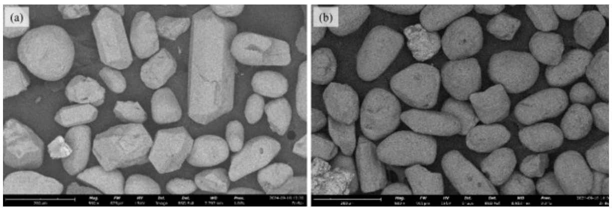


**Figure 3**. SEM images of zircon sand at 500× magnification: (a) Bangka and (b) Belitung

Morphological analysis was conducted using SEM, and the zircon sand morphology is shown in Figure 3. The images reveal that zircon sand samples from Bangka and Belitung exhibit non-uniform, irregular lump shapes. Within these irregular forms, several euhedral zircon crystals are observed, characterized by well-defined and regular crystal planes. This observation is consistent with previous studies reporting that natural zircon minerals typically display euhedral morphologies of varying sizes [16]. Additionally, particle size analysis reveals that the particle sizes of Bangka and Belitung zircon sands are 964.80 nm and 809.65 nm, respectively.

### 3.2. Characteristics of $ZrOCl_2 \cdot 8H_2O$ from Bangka and Belitung Zircon Sands

The $ZrOCl_2 \cdot 8H_2O$ derived from Bangka and Belitung zircon sand was characterized to determine its composition, structure, phase, functional groups, morphology, and particle size. Compositional analysis was performed using XRF, with results shown in Table 2.

The $ZrOCl_2 \cdot 8H_2O$ samples from Bangka and Belitung exhibit a dominant $ZrO_2$ content, although their purity is lower than that of the commercial product. This is likely due to the natural origin of the raw materials, which are derived from tin mining tailings. As a result, the composition of compounds and elements in the raw materials is highly varied, including significant levels of impurities. Nevertheless, the $ZrO_2$ content in the Bangka and Belitung samples, at 38.39%, is higher than the levels reported by Poernomo *et al.* [12]. The $ZrOCl_2 \cdot 8H_2O$ content, calculated from the Zr and Cl levels in the samples, is presented in Table 3.

**Table 2**. Composition of $ZrOCl_2 \cdot 8H_2O$ derived from Bangka and Belitung zircon sand compared with a commercial product

| Compound | Content (%) | | |
|---|---|---|---|
| | Bangka $ZrOCl_2 \cdot 8H_2O$ | Belitung $ZrOCl_2 \cdot 8H_2O$ | *Commercial $ZrOCl_2 \cdot 8H_2O$ |
| $ZrO_2$ | 50.096 | 48.654 | 78.32 |
| $TiO_2$ | 0.567 | 0.561 | 0.741 |
| $Fe_2O_3$ | 0.23 | 0.246 | 0.223 |
| $SiO_2$ | 4.182 | 4.384 | 2.156 |
| $Al_2O_3$ | 1.83 | 1.95 | 1.816 |
| CaO | 1.468 | 1.54 | 1.466 |

*Commercial refers to a manufacturer's product

As shown in Table 3, the commercial sample has the highest Zr content at 79.29%, with a relatively low Cl content of 10.22%. In contrast, the synthesized samples from Bangka and Belitung show noticeably lower Zr content (50.55% and 48.84%, respectively) and higher Cl content (37.58% and 37.24%). The higher chlorine levels and lower zirconium percentages in the Bangka and Belitung samples may indicate incomplete conversion or residual byproducts from the synthesis process [17].

Despite this, the results still illustrate that zircon sand from both Bangka and Belitung can be successfully converted into $ZrOCl_2 \cdot 8H_2O$, with the total chemical composition of Zr and Cl reaching 88.12% and 86.08%, respectively. The production process begins with a sintering reaction, during which the zircon structure is broken down to facilitate the preliminary separation of zirconium and silicon. This is followed by a series of chemical treatments that yield intermediate products. However, the high thermal and chemical stability of zircon ($ZrSiO_4$), attributed to the strong coordination of bisdisphenoid $ZrO_8$ within a tetragonal structure linked to $SiO_4$ tetrahedra, necessitates the use of aggressive reaction conditions and reagents to decompose the zircon crystal lattice or dissociate zircon into zirconia and silica [18]. Furthermore, the structure and phase of $ZrOCl_2 \cdot 8H_2O$, as determined from the XRD patterns of Bangka and Belitung samples, are presented in Figure 4.

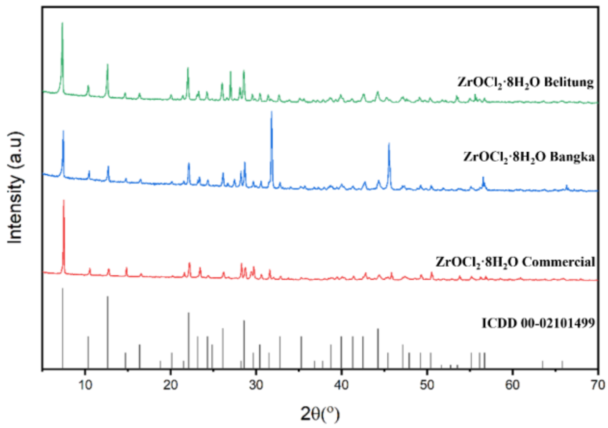


**Figure 4**. XRD diffractograms of $ZrOCl_2 \cdot 8H_2O$ derived from (a) Bangka and (b) Belitung zircon sand

**Table 3**. Zr and Cl content of $ZrOCl_2 \cdot 8H_2O$ samples

| Element | Content (%) | | |
|---|---|---|---|
| | Bangka $ZrOCl_2 \cdot 8H_2O$ | Belitung $ZrOCl_2 \cdot 8H_2O$ | Commercial $ZrOCl_2 \cdot 8H_2O$ |
| Zr | 50.549 | 48.841 | 79.288 |
| Cl | 37.576 | 37.244 | 10.215 |

Figure 4 shows the formation of the $ZrOCl_2 \cdot 8H_2O$ phase from the reaction of Bangka and Belitung zircon sand, exhibiting a tetragonal structure. The $ZrOCl_2 \cdot 8H_2O$ phase from Bangka zircon sand is observed at 2θ values of 7.33°, 12.58°, 16.34°, 21.99°, 26.01°, and 28.53°, while the corresponding phase from Belitung zircon sand appears at 2θ values of 7.33°, 12.61°, 16.36°, 22.02°, 26.03°, and 28.57°, consistent with the standard $ZrOCl_2 \cdot 8H_2O$ phase (ICDD 00-021-1499) and the commercial $ZrOCl_2 \cdot 8H_2O$. The peak intensity of $ZrOCl_2 \cdot 8H_2O$ from Belitung zircon sand is higher than that from Bangka, indicating greater crystallinity. However, the Bangka sample exhibits additional high-intensity peaks due to residual $ZrSiO_4$ (zirconium silicate) that has not fully transformed into $ZrOCl_2 \cdot 8H_2O$ [17], as well as impurity phases such as $Al_2O_3$ and CaO, as confirmed by XRF analysis. Furthermore, the functional groups of $ZrOCl_2 \cdot 8H_2O$ are shown in Figure 5.

Figure 5 demonstrates that the $ZrOCl_2 \cdot 8H_2O$ absorption peaks from Bangka and Belitung zircon sand exhibit similar characteristics. The absorption bands at 3109 $cm^{-1}$ and 1621 $cm^{-1}$ in Belitung $ZrOCl_2 \cdot 8H_2O$, and 3110 $cm^{-1}$ and 1617 $cm^{-1}$ in Bangka $ZrOCl_2 \cdot 8H_2O$, correspond to the absorption of the hydroxyl (-OH) group. Additionally, the absorption peaks at 908 $cm^{-1}$ and 916 $cm^{-1}$ are attributed to Zr–O–Zr vibrations, indicative of $ZrOCl_2 \cdot 8H_2O$. The characteristic vibration of Zr=O is generally observed within the range of 896–915 $cm^{-1}$, and a shift to 1017 $cm^{-1}$ is likely influenced by the presence of Cl. The peaks in the fingerprint region at 416 $cm^{-1}$ and 411 $cm^{-1}$ confirm the tetragonal structure of zirconia [19, 20]. Furthermore, the surface morphology of $ZrOCl_2 \cdot 8H_2O$ is analyzed in Figure 6 using SEM characterization.

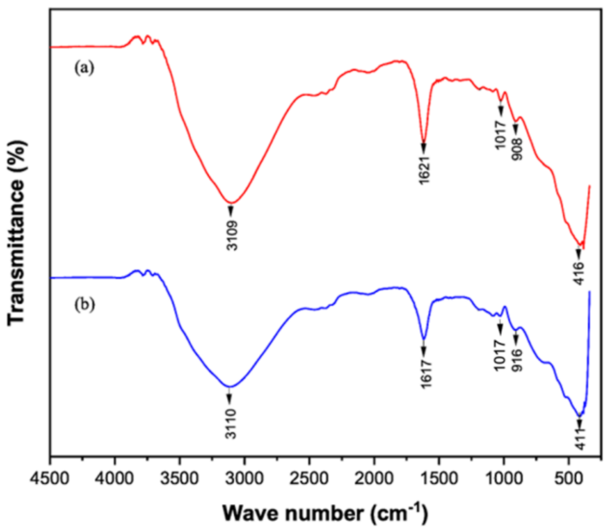


**Figure 5**. FTIR spectra of $ZrOCl_2 \cdot 8H_2O$ derived from (a) Belitung and (b) Bangka zircon sand

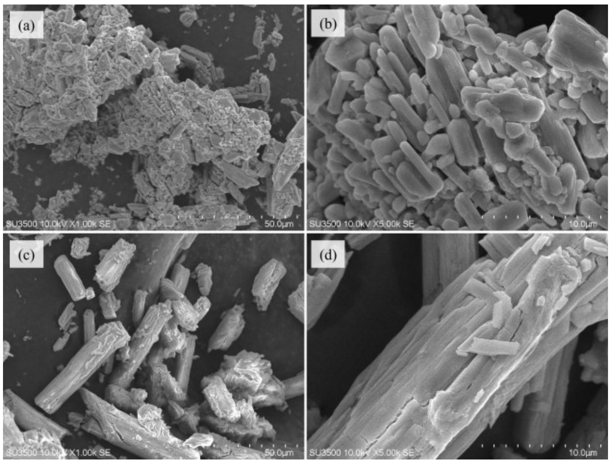


**Figure 6**. SEM images of $ZrOCl_2{\cdot}8H_2O$ surfaces: (a, b) Bangka zircon sand at 1000× and 5000× magnification, respectively; (c, d) Belitung zircon sand at 1000× and 5000× magnification, respectively

Figure 6 illustrates the morphology of $ZrOCl_2{\cdot}8H_2O$ synthesized from Bangka and Belitung zircon sand. Both samples exhibit rod-shaped structures with visible cracks, indicating non-compact and fragile characteristics. While the overall morphology is similar, $ZrOCl_2{\cdot}8H_2O$ particles from Bangka are larger and differ in shape compared to those from Belitung. Furthermore, the Bangka particles show agglomeration due to strong van der Waals forces [21], whereas the Belitung particles are more isolated with clearly defined boundaries. These observations are supported by particle size analysis, which shows average sizes of 19.77 nm for Bangka and 10.94 nm for Belitung.

## 4. Conclusion

This study aimed to address the underutilization of zircon sand from Bangka and Belitung by evaluating its potential as a local raw material for producing zirconium oxychloride ($ZrOCl_2{\cdot}8H_2O$). The results demonstrate that, although both zircon sand sources contain high zirconium content and were successfully converted via hydrometallurgical synthesis, the resulting products exhibited lower zirconium purity and higher chlorine content compared to commercial-grade material. This suggests incomplete decomposition of $ZrSiO_4$ and the presence of impurities, likely due to the heterogeneity of tin tailings and non-optimized processing conditions. Despite these limitations, the total Zr and Cl content of the synthesized products (88.12% for Bangka and 86.08% for Belitung) indicates their promising potential for further development. $ZrOCl_2{\cdot}8H_2O$ derived from Belitung showed a higher phase purity, more defined morphology, and smaller particle size, suggesting superior properties as a precursor for zirconium-based applications. These findings validate the feasibility of using Bangka and Belitung zircon sand as alternative sources for $ZrOCl_2{\cdot}8H_2O$, supporting the sustainable valorization of mining byproducts. Further optimization of synthesis parameters is recommended to improve purity and conversion efficiency to meet industrial standards.

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