UNIVERSITI TEKNOLOGI MARA

SYNTHESIS OF YTTRIA-STABILIZED ZIRCONIA FROM LOCAL SANDS

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ABSTRACT

Amang zircon sand is abundant in Malaysia due to the large tin mining industry and resources in Malaysia especially around Perak and Selangor. However, no comprehensive study has been dedicated to Amang zircon sand. This study transformed the by-product in tin mining industry into something valuable like zirconia. The study aimed to evaluate the influence of temperature on zirconia powder synthesised from Amang zircon sand and the effect of yttrium oxide concentration as the doping agent in stabilizing Amang zirconia powder. The raw Amang zircon sand obtained from Kilang Amang Onn Sdn. Bhd. (Onn Mineral Company) at Kampar, Perak was analysed with Malvern Panalytical Epsilon and the analysis showed major zirconium composition of 60.17 wt.% with 5.22 wt.% silicon and impurities. Alkali fusion of Amang zircon sand with 6M sodium hydroxide (NaOH) was employed in this study followed with thermal treatment at 670 °C for 2 hours. Sodium zirconate and sodium silicate obtained from the fusion was subjected to leaching with deionized water and 8M hydrochloric acid (HCl). Sodium silicate and unreacted NaOH and zircon were leached out, leaving zirconium oxychloride ($ZrOCl_2$) for further hydrolysis process. Zirconium oxychloride was neutralized and hydrolysed by ammonium hydroxide at pH of 10-10.5 to produce zirconium hydroxide ($Zr(OH)_4$). Zirconium hydroxide powder was then calcined at varied temperature of 400-800 °C for 4 hours to obtain Amang zirconia. The effect of temperature on Amang zirconia was observed in term of increased zirconium composition at 81.28 wt.% coupled with low impurities when calcined at high temperature of 800 °C. Agglomerated monoclinic zirconia powder with a small fraction of tetragonal and cubic phase, particle size of 150.2 μm and specific surface area of 0.314 m^2/g was observed by Rigaku Ultima IV diffractometer, Brunauer-Emmett-Teller, Malvern Panalytical Mastersizer and Hitachi SU3500 microscopy. Subsequent stabilization of zirconia with yttrium oxide as doping agent at concentration of 3-5 wt.% revealed an enhanced crystallinity of monoclinic yttria stabilized zirconia powder with smaller particle size of 88.97 μm. The composition of yttria zirconium oxide increased with the addition of yttrium oxide. 71.01 wt.% of zirconium composition when stabilized at 5 wt.% demonstrated the material's potential for locally synthesised yttria-stabilized zirconia. In conclusion, this comprehensive study highlighted the potential of zirconium oxychloride ($ZrOCl_2$) produced from Amang zircon sand as precursor to synthesized yttria stabilized zirconia powder at designated parameters. The finding not only highlighting the potential of locally available Amang zircon sand but also opening the probability to utilized Amang zirconium oxychloride and zirconia for tailored application in various industries such as textile, oil and also nuclear.

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CHAPTER ONE
INTRODUCTION

1.1 Research Background

Zirconia or zirconium dioxide (ZrO_2) is a polymorphic material that was accidentally found in 1789 by a chemist named Martin Heinrich Klaproth (Slobodyan, 2021). It has been dubbed as "ceramic steel" because of the similarity of mechanical properties with stainless steel (Manicone et al., 2007; Ong et al., 2024). This element is abundant in nature, and it is usually conjugated with other element such as silica in a pure state. Zirconia is naturally derived from zircon and baddeleyite which is also known as zirconia ore (Blanchart, 2018; Gauna et al., 2015). Nevertheless, baddeleyite is reported to contain a higher amount of silica compared to zircon and this leads to higher difficulty in processing baddeleyite into zirconia (Naher & Haseeb, 2006).

Zircon or zirconium silicate ($ZrSiO_4$) usually contains traces of other elements. It is a non-magnetic heavy metal with heavy minerals such as biotite, garnet, ilmenite, magnetite, kyanite, monazite and xenotime (Amalia et al., 2020). However, the aforementioned valuable heavy minerals in zircon sand might also reduce the effectiveness of zircon decomposition (Biswas et al., 2010). The high zirconium content in zircon sand makes it as the dominant source for zirconia. Zircon naturally exist in a form of sand from a by-product of weathered rocks or from a by-product of tin mining industry known as Amang in Malaysia (Azreen et al., 2018; Kumar et al., 2015). Zircon also typically contains radionuclides (Taftazani et al., 2022). Hence, it is important to ensure the composition of radionuclides presence in the sand is under the allowable composition and the synthesisation process must be done strictly following the Atomic Energy Licensing Act 1984 (Act 304).

Zirconia is economically attractive in various application such as in polycrystal ceramic, pigment, and refractory due to the low reactivity, chemical stability, high hardness, high thermal conductivity, and resistance to erosion (Ebrahimi et al., 2022). Zirconia is also less expensive and has higher melting point than alumina (Guo et al., 2021). However, all undesired elements that exist in zircon such as silica must be removed before it can be used as a raw material for zirconia as the impurities may lead to brittleness of zirconia synthesized from zircon sand.