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EXPERIMENTAL SYNTHESIS OF ZIRCONIA NANOPARTICLES USING VARIOUS CAUSTIC FUSION TEMPERATURES DERIVED FROM NATURAL MATERIAL ZIRCON SAND IN INDONESIA

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ABSTRACT

The purpose of this research was to analyze the effect of caustic fusion temperature on the size, and impurity of Nano-zircon produced from zircon sand. Caustic fusion of zircon sand using NaOH was conducted with varied temperatures of 400-600 °C, followed by water leaching, acid leaching, precipitation, and calcination to produce zirconia nanoparticle powder. The synthesized zirconia nanoparticle was analyzed using FT-IR and SEM. The yield of the product was also determined and the correlation between varied caustic fusion temperature and yield and diameter product was determined using the least square equation. From the FT-IR spectrum, it is shown that the presence of -OH hydroxyl groups at a wavelength of 3417.725 cm^{-1} indicates the presence of ≡Si-OH groups. At wave 2258.504 cm^{-1} indicate the presence of a Si-H group in the form of ≡Si-O-OH groups. The correlation between the temperature of caustic fusion and yield follows the equation $y = -0.00184x + 1.244$ and $R^2 = 0.9888$ indicating that the higher the temperature of caustic fusion the higher the yield of zirconia nanoparticle product. The optimum temperature was found at 450 °C with a yield of 0.42% and a size of 32.984 nm.

Keywords: Caustic Fusion, Temperature, Zircon Sand, Zirconia Nanoparticle.

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INTRODUCTION

Zircon sand is a natural mineral that is extensively distributed in America, Australia, China, India, Indonesia, Mozambique, South Africa, and other nations. It is utilized in a variety of industries.[1] It can be converted into a nanomaterial, which has piqued the interest of researchers due to its unusual features associated with its size of 1 - 100 nm. Zircon sand can be synthesized into zirconia nanoparticles (ZrO_2), zircon nanopowder ($ZrSiO_4$), and silica nanopowder.[2] Because of their extraordinary toughness, strength, and hardness, zirconia nanoparticles have piqued the interest of researchers for both synthesis and application.[3] It leads to several utilisations in ceramics glazing because of its excellent mechanical, thermal, and chemical resistance[4], catalyst due to its inert, promoting surface sites and coordinative unsaturated surface sites [5], fuel cell material due to its strong conductivity qualities[6], nanofluids are used in heat transfer applications because they are heat resistant and increase thermal conductivity[7-8,30-35], as an adsorbent because of their large surface area[9], in dentistry because they are colorless, biocompatible, and have excellent strength and toughness[10], and in other biomedical applications because they are composite materials.[3,25-29] The application is varied for different crystal forms. Zirconia nanoparticles also known as zirconium nanoparticles exist in three different crystal forms e.g. mono, tetra, and cubic form. That presents the varied temperature stable (1100 – 2370 °C), with the highest temperature in cubic form. However, the cubic and tetragonal forms cannot be unstable in low temperatures and require high temperatures in interstate form. It can be stabilized using a stabilizer agent in a divalent or trivalent structure.[11] This crystal form can be maintained at low temperatures and pressure.[12] In hence, the addition

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of organic precursor can alter the crystal transition phase.[3] Many researchers reported the synthesis of zirconia nanoparticles. Arshad *et al.* (2022) reported that different variations of synthesis method led to different crystal forms.[3] The common synthesis methods of nanoparticles are top-down and bottom-up methods. The top-down methods include mechanical milling. The bottom-up method approaches chemical methods such as sol-gel, hydrothermal, precipitation, and alkali fusion methods.[2,8,9,13,14] The Alkali fusion method generated two monoclinic phases of zirconia[14], sol-gel method can produce a cubic dan monoclinic form of zirconia nanoparticles.[13] Low thermal treatments and mechanical milling were done to synthesize amorphous, tetragonal, and monoclinic zirconia nanopowder.[2] Furthermore, the precipitation method developed a tetragonal structure of zirconia nanoparticles.[9] The other method ultrafast laser ablation was investigated to perform zirconia nanoparticles for optical applications with size distribution 12 – 15 nm[15] the latest method ultra-short pulsed laser ablation was reported to produce composite material Zr-Al.[16] The sol-gel method has the lowest temperature compared to other methods; however, it is not an economical process due to its chemical consumption and waste treatment for industry scale. The laser ablation produced lower chemical waste although, the precipitation method can produce high-quality zirconia nanoparticles in purity and size, the product is expensive.[9] To overcome this problem, caustic fusion is the simplest method to synthesize the path of zirconia.[17,18] Caustic fusion has the advantage of lower temperature and pressure than hydrothermal methods.[12] It is a combination method of the caustic method and fusion method, generally effective in generating zirconia from zircon sand or another zircon mineral.[6,13,14] The chemical used in this method is alkali such as KOH, NaOH, $CaCO_3$, and Na_2CO_3.[14,19] Although KOH was reported more effective due to its low negative energy Gibbs related to its spontaneous reaction, it is more expensive and high exothermic heat.[19] NaOH was the suggested chemical used because it is a cheaper and stronger alkali than KOH and also less harmful than Na_2CO_3 and $CaCO_3$ for environmental no CO_2 released, although it showed no significant effect of KOH and NaOH in crystal size of zirconia.[20] The research aims to find out the effect of caustic fusion temperature on the yield and size of Zirconia nanoparticles from the synthesis process using zircon sand that comes from local material sources in Indonesia.

EXPERIMENTAL

Materials

The raw material, Zircon sand, was achieved from West Kalimantan, Natrium Hydroxide 96 – 98% used for caustic fusion was obtained from Sigma-Aldrich, and Chloride Acid 32% for acid leaching was obtained from PT. Katalis Datesa Prima, Ammonia 25% from Merck, and distilled water were obtained from the laboratory of the Chemical Engineering Department, Faculty of Engineering, Universitas Muhammadiyah Jakarta, Indonesia.

Procedures of Preparation

There are five procedures in the synthesis of zirconia nanoparticles in this research: caustic fusion, water leaching, acid leaching, precipitation, and calcination to get zirconia nanoparticle. The procedure was modified from the method by Athifah *et al*.[9]

Caustic Fusion

Caustic fusion of zircon sand was conducted using NaOH as an alkali. Zircon sand and NaOH with a ratio of 1:8 (50 gr zircon sand and 180 gr NaOH) were mixed and ground for 2 min. The mixture was placed in a nickel cup until ¾ full to avoid the spoil at the fusion furnace. The furnace with temperature varied of 400, 450, 500, 550, and 600 °C was used to conduct the variation of temperature alkali fusion for 1 hr. After cooling, the caustic fusion product (frit) was ground until smooth.

Water Leaching

The 58 gr of frit was leached by 200 ml distilled water to remove silica contained in a beaker glass. It was mixed and heated in a hotplate at 100 °C for 15 min. Then the solution was stand until it settled. The residual was separated from the solution by washing using distillate water. The washing process was repeated 6 times to measure the removal of silica content. Then the water content was removed and the precipitate residue overnight.

Acid Leaching

To achieve a pH of 6-7, HCl 5 M was added to the residue, and then the solution was heated in a heating furnace at 200 °C until it was dry and cracked and then it was cooled. 10 gr dried residue was placed in a beaker glass and 100 ml of 5 M HCl solution with a ration 1:10. The solution was heated and stirred in a hotplate of 100 °C for 30 min. Then it was settled in the acid room for 1 night until it turned into jelly. The jelly product was mixed and stirred for 10 min with 100 ml of 5 M HCl and cooled in the acid room for 1 night.

Precipitation

The precipitation was started with filtration using Whatman filter paper 120. The 100 ml filtrate was mixed with 100 ml of distilled water and heated in a hotplate for 30 min at 100 °C. Then it was poured gradually 150 ml ammonia. Then it was washed using 100 ml distilled water 6 times until it was not salty. The solution was settled in the acid room for 1 night. The water contained was removed to reduce the soluble impurities and the final step was calcination.

Calcination

The calcination was done at 200 °C until the residue was dried or cracked and taken carefully into place.

Detection Method

Yield Percentage for Nanoparticle

The yield percentage was calculated using equation (1) to analyze the efficiency of the process to produce the product.[21] The equation to calculate yield:

$$\text{Yield} = \frac{\text{Product mass}}{\text{Raw material mass}} \times 100\% \qquad (1)$$

FTIR (Fourier Transform Infrared Spectroscopy) Analysis

Zircon nanoparticles' FTIR spectra were acquired using a Fourier Transform Infrared (FTIR) Spectrometer Carry 630 FTIR (Fig.-1). KBr pellets were used to test samples in the 4000-400 cm^{-1} range. Previous studies.[25] used the same procedure to determine and identify the characterization of the zircon nanoparticles produced.

Fig.-1: Carry 630 FTIR for Check Sample of Silicate Nanoparticles

SEM of Zircon Nanoparticles

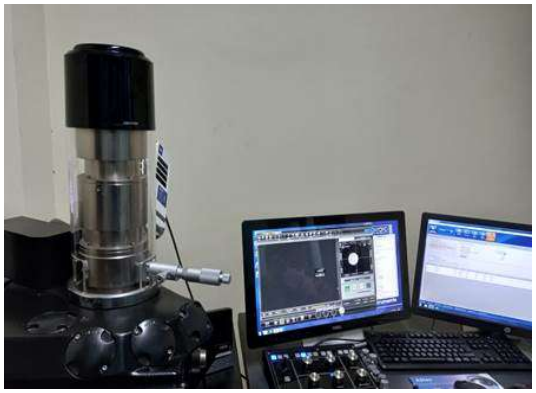
To study the morphology of Nano-Zircon, a scanning electron microscope (SEM) series JSM-IT300 at 20.0 kV was used. The image of Nano-Zircon morphology was captured at a magnification of 3,000X. Due to the unique chamber configuration, the sample was mounted on the sample holder and imaged directly without pretreatment, even when it was hydrated. ImageJ software was used to calculate the diameter based on SEM images. This SEM analysis was conducted by the National Research and Innovation Agency(Fig.-2). The process of testing this characterization is carried out in the same way.[21-24]

RESULTS AND DISCUSSION

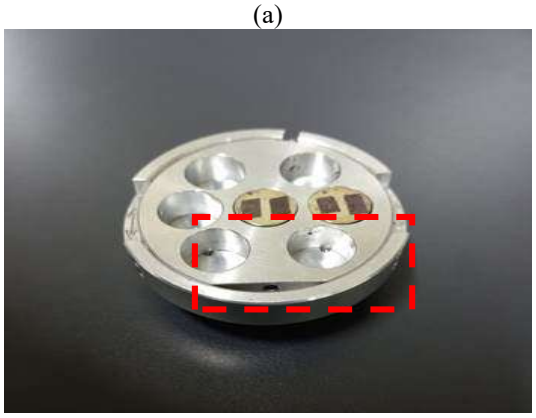
Effect of Caustic Fusion Temperature on Yield Percentage

Yield percentages were calculated using Equation (1). Yield percentage shows the effective process of achieving the zirconia nanoparticle product. This study was conducted to analyze the effect/ correlation

between Caustic fusion temperature on yield percentage. To calculate the yield, it is required the mass product and raw material. The weight of the zircon sand used was 50 grams. In this process, before the burning/fusion process at the furnace, grinding, and mixing of zircon sand and NaOH samples is carried out. In the same way to find out the yield percentage of the amount of nanoparticles obtained in the research that has been done.[13-15] The result of the yield percentage of varied temperatures of Caustic fusion is presented in Table-1.



(a)



(b)

Fig.-2:(a) JSM-IT300 Scanning Electron Microscope (SEM), (b) Silicate Nanoparticle Samples

Table-1: The Yield Percentage of product at Varied Caustic Fusion Temperature

Temperature of furnace in Caustic fusion (°C)	Mass Raw material (gr)	Mass Product (gr)	Yield (%)
400	50	0.27	0.54
450	50	0.32	0.42
500	50	0.92	0.34
550	50	0.12	0.24
600	50	0.19	0.18

Table-1 shows that high to low-temperature variations in the furnace in the acoustic fusion process can cause a decrease in the yield of nano-silicate obtained. As for the amount of returns, there are increases and decreases due to the influence of temperature variations. As obtained in research by.[13,15] To evaluate the influence or correlation between the variation of caustic fusion temperature and Yield is presented in Fig.-3. Based on Fig.-3, the yield decreased at a temperature of 600°C and increased significantly at a temperature of 400°C amounting to a yield of 0.54%. This is caused by the occurrence of a caustic fusion reaction, thus proving that variations in temperature result in greater yield. Research evidence by.[14]

FT-IR Spectrum analysis

The FT-IR spectrum of zirconia nanoparticle is presented in Fig.-4 at different temperatures. The presence of -OH hydroxyl groups at a wavelength of 3417.725 cm^{-1} indicates the presence of ≡Si-OH groups. At wave 2258.504 cm^{-1} could be the presence of a Si-H group in the form of ≡Si-O-OH groups. The peak is

similar to the result of Subuki *et al.*[14] He suggested the peak at 3418 and 1624 cm^{-1} related to the stretching and bending vibration of the -OH atom. This is due to the strong band between -OH from NaOH with Zr groups creating the energetically neutral bond states. The peak of 680 cm^{-1} corresponds to zircon crystal.

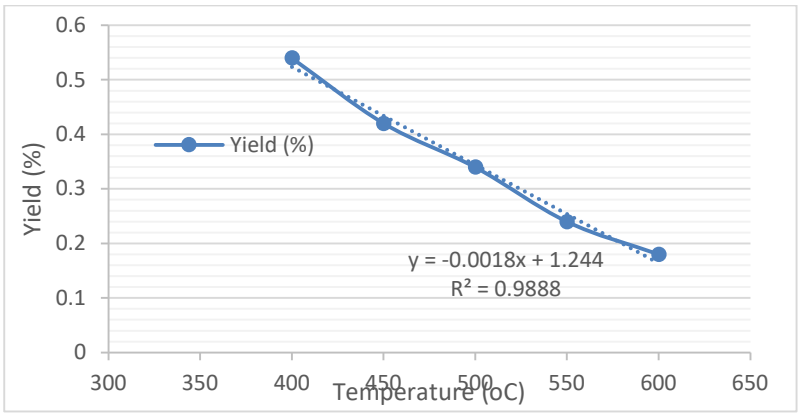
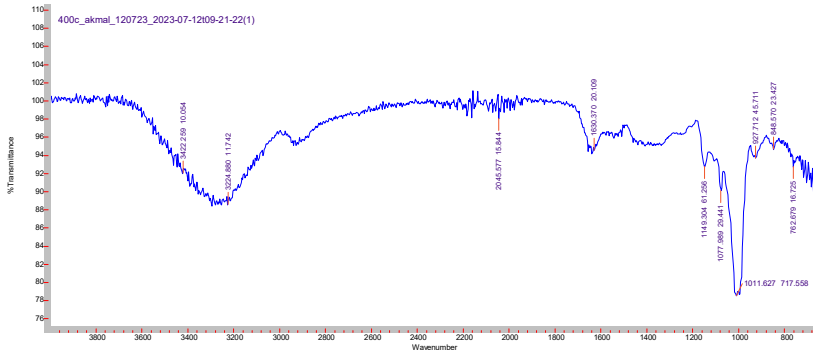
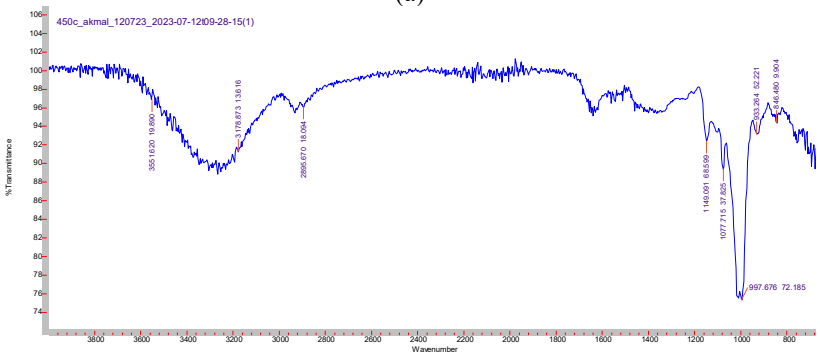


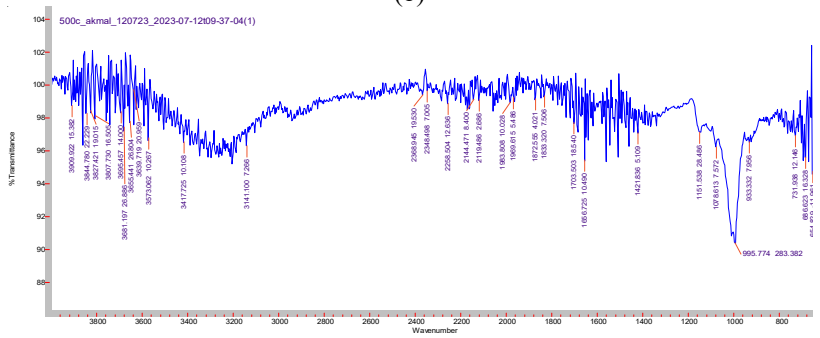
Fig.-3: The Correlation between Caustic Fusion Temperatures on Yield Percentage



(a)



(b)



(c)

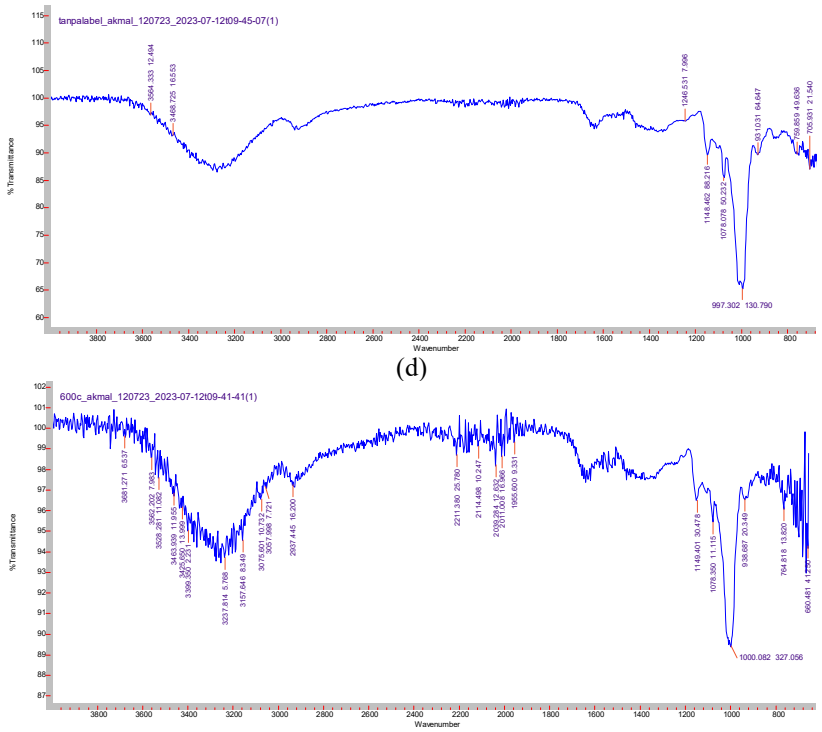


Fig.-4: The FT-IR Spectra of Zirconia Nanoparticles at (a) 400°C, (b) 450°C, (c) 500°C, (d) 550°C, and (e) 600°C

Figure-4 Shows the Caustic Fusion of Zircon Minerals Using NaOH with a Stoichiometry of Reaction:

$$ZrSiO_4 + 4\ NaOH \rightarrow Na_2ZrO_3 + Na_2SiO_3 + 2\ H_2O$$

To facilitate the interpretation of the FTIR spectrum, the FT-IR was carried out at wave numbers 4000 – 2000 cm^{-1} which was tested at waves 2000 – 600 m^{-1}. Figure-4 presented that at peak 3417.725 cm area is stretching of the H_2O molecule from hydroxyl bond in the form of ≡Si-OH groups on the surface of the product.[21] The ≡Si-OH groups are visible at 500 °C, however, a temperature of 600 °C indicates more -OH groups that are equivalent to ≡Si-OH on the surface.[22] At peak of 2258,504 cm^{-1} at a temperature of 500 °C and 600 °C. According to[23] this peak is in the form of a Si-H group, and this form is the result of hydrolysis of the ≡Si-O-H group which binds to Hydrogen atoms forming ≡Si-Zr≡ on the surface. Furthermore, a peak appeared in the region of 1421.836 cm^{-1} stretching vibration of the O-C-O group from the carbonation reaction of Na_2CO_3. The carbonation reaction occurs due to the presence of NaOH which doesn't react in delignification but reaches with CO_2 from the air and is detected as an O-C-O group at the peak of 1450 cm^{-1}. This appears at a variable temperature of 500 ºC, but no presence in another temperature variable. This shows the presence of silanol groups in the form of Si-O-X (X = metal) due to at other temperatures presence of a peak of 1151.538 cm^{-1}. It can be said as an asymmetric stretching vibrational bond of Si-O(Na) or the formation of Na_2SiO_3 compounds. While the formation of Na_2ZrSiO_5 occurs at all varied temperatures which forms the following reaction:

$$ZrSiO_4 + 2\ NaOH \rightarrow Na_2ZrO_5 + 2\ H_2O$$

Effect of Caustic Fusion Temperature on Diameter Nanoparticles Using Scanning Electron Microscope (SEM) Analysis

The diameter of the synthesized zirconia nanoparticles was determined by SEM examination. Using an electron microscope designed to directly see the surface of a material with a magnification of 10-3,000,000 times, a depth of field of 4-0.4 mm, and a resolution of 1-10 nm. SEM is an electron microscope that uses an electron beam to describe the surface form of a synthetic zirconia nanoparticle. Table-2 shows the size obtained. Results of the SEM image in Table-2 revealed that the nanoparticle size decreased at a temperature of 450°C while increasing at 500 °C at 73.753 nm. It is bigger than Nuryadin and Triwikantoro [20] reported. The crystal size of ZrO_2 was 50.2913 nm at the same temperature but

longer fusion time. It resulted in tetragonal zirconia. On the other hand, the size is almost the same between temperatures of 400 °C and 550 °C. The largest size is obtained at 600 °C. It is shown that caustic fusion temperature does not affect significantly to size of the nanoparticle. Temperature above 450 °C will increase the nanoparticle size even though it is still in nano size except at 600 °C. It is due to the endothermic reaction of fusion which required a high temperature to gain the product and the product does not reverse to reactant, however the temperature above 550 °C achieved size > 100 nm. It is to the result of Murti [et al][24] that the temperature at 700 °C resulted in 143 nm – 260 nm at varied pH. Although others reported the fusion temperature varied from 200-700 °C.

Table-2: The Diameter of the Product at Varied Caustic Fusion Temperature

Temperature of furnace in Caustic fusion (°C)	Magnification (times)	Diameter zirconium nanoparticle average (nm)
400	30.000	41.983
450	30.000	32.984
500	30.000	73.753
550	30.000	41.720
600	30.000	157.79

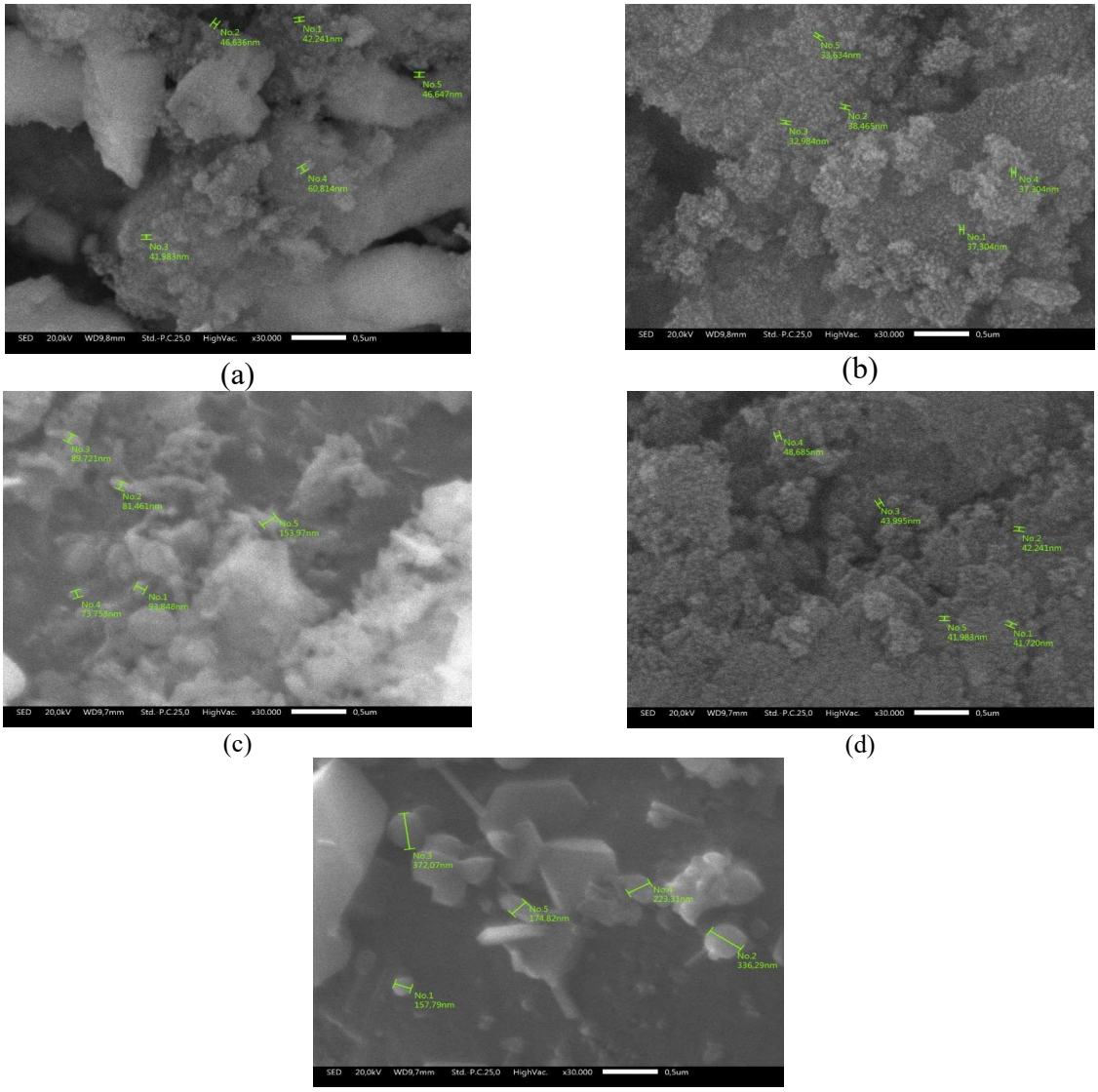


Fig.-5: The SEM Picture of Zirconium Product at Various Caustic Fusion Temperatures: (a) 400°C, (b) 450°C, (c) 500°C, (d) 550°C, and (e) 600°C

Figure-5 Presents that nano zirconia particle size at a temperature of 400 °C to 550 measures <100 nm; however, at a temperature of 600 °C, its size increases almost twice its size at a temperature of 500 °C. This is taken as the average nanoparticle size as a result of testing using SEM, as obtained from research.[21-25]

CONCLUSION

In this research, caustic fusion temperature does not affect the percentage of zirconia nanoparticle yield percentage. The correlation between the variation of temperature (x) to yield percentage (y) follows the equation $y = -0.00184x + 1.244$ and $R^2 = 0.9888$. The optimum temperature to get the highest yield is 400 °C of 0.54% yield. The result of the FT-IR spectrum shows the presence of the -OH group at 3417.725 cm^{-1} due to the presence of H_2O molecules at 500 °C. At the same time, the smallest size of zirconia nanoparticle is 32.984 nm at 450 °C. The average nanoparticle size was determined by testing using SEM.

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CONFLICT OF INTERESTS

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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