SEMARAK ILMU
PUBLISHING
202103268166(003316878-P)

Journal of Advanced Research in
Micro and Nano Engineering

Journal homepage:
https://semarakilmu.com.my/journals/index.php/micro_nano_engineering/index
ISSN: 2756-8210

Synthetization and Characterization of Yttria Stabilized Zirconia Powder from Amang Zircon Sand

Nur Fatinhanani Fatimah Norsham[1], Istikamah Subuki[2,*]

[1] School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia
[2] Circular + Industrial Research Laboratory, School of Chemical Engineering, College of Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

ARTICLE INFO

Article history:
Received 2 September 2024
Received in revised form 9 October 2024
Accepted 14 November 2024
Available online 31 December 2024

ABSTRACT

Zirconia is widely used in industry as it exhibits high strength and amazing properties. However, pure zirconia powder is often unstable to be used in high temperature applications due to phase transformation that will lead to fracture and crack. Hence, zirconia is doped with yttrium oxide in order to enhance zirconium content and stability as it helps to hinder the phase transformation of zirconia. Locally available Amang zircon sand from tin tailing industry in Malaysia can be an economical alternative to synthesize zirconia due to the high zirconium content. Sadly, the study on utilizing Amang zircon sand is scarce despite decomposition of zircon sand to zirconia has been intensely done by other countries. Therefore, a study on Amang zircon sand was done with alkali sodium hydroxide fusion to obtain zirconium oxychloride. Zirconium oxychloride precursor was then stabilized with yttrium oxide in varied concentration using coprecipitation method with ammonia solution. Amang yttria stabilized zirconia powder was then characterized in order to study the effect of yttrium oxide concentration on Amang yttria stabilized zirconia powder. X-ray florescence analysis confirmed the high zirconium content of 71.01 wt.% after increasing the yttrium oxide concentration to 5 wt.% with lesser silicon impurity of 2.94 wt.%. The diffraction patterns satisfied monoclinic yttria stabilized zirconia with minor tetragonal and cubic phase. The peaks became sharper as concentration increased due to the stabilization of zirconia powder. Tetragonal and cubic phase increased along with the concentration, but the concentration of yttrium oxide was insufficient to fully stabilized the synthesized zirconia powder. Morphology analysis showed yttria stabilized zirconia powder was angular and pyramidal with large grain size in which led to high tendency of monoclinic phase.

Keywords:
Alkali fusion; amang zircon sand; yttria stabilized zirconia; zirconia

1. Introduction

Zircon ($ZrSiO_4$) also known as zirconium silicate is a non-magnetic material that usually contains high zirconium dioxide with traces of other elements such as silicon dioxide (SiO_2), titanium dioxide (TiO_2) and iron oxide (Fe_2O_3) [1]. Zircon sand exists from a by-product of weathered rocks or from a

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[*] *Corresponding author.*
E-mail address: istikamah@uitm.edu.my

https://doi.org/10.37934/armne.28.1.105117

by-product of extraction and mining industries [2]. The high composition of zirconium in zircon sand makes it as an amazing economical alternative to synthesize zirconia [3].

Amang zircon sand is a by-product from tin tailing industry in Malaysia. Tin tailing used to be a promising industry with processing plants densely located at Perak, Selangor, Pahang and Johor [4]. Unfortunately, the global market suddenly dropped in 80s' and tin processing plants in Malaysia was severely hit. Subsequently, this problem incited the abandonment and accumulation of tin ore minerals and its by-products.

Zirconium dioxide (ZrO_2) which is also known as zirconia is a type of ceramic that is typically derived from zircon sand. It has been nicknamed as "ceramic steel" due to brilliant wear resistant [5], amazing biocompatibility [6], low thermal conductivity and high melting and boiling point [7]. These excellent properties allow zirconia to be used in broad applications [8]. For examples in dentistry [9], catalyst support [10], pigments for white glazed tile [11], thermal barrier coating [12], ceramic membrane in separation process [13], solid oxide fuel cell [14-16] and gas sensor [17].

Polymorphic zirconia exists in three structures namely monoclinic, tetragonal and cubic [18]. Consequently, the three different structure of zirconia exhibit different properties and applications as phase transformations occurred upon cooling [19]. Monoclinic zirconia has strong covalent bonds between zirconium and oxygen atoms which made monoclinic zirconia thermodynamically stable at room temperature and up to 1170 °C [20]. Monoclinic zirconia also shows a high resistance to cracking and exhibits outstanding properties.

This monoclinic form then transformed to tetragonal as the temperature raised up to 2370 °C. The transformed tetragonal zirconia has an improved mechanical property than monoclinic zirconia and considered as the best structure out of all three crystallographic structures [21]. However, this structure will transform back to monoclinic during cooling as pure tetragonal zirconia is highly unstable especially in the presence of moisture [22]. Moreover, the phase transformation is reversible and lead to fracture, crack and surface roughening [23]. Hence, this reversible phase transformation of zirconia will limit the application [24].

As the demand and interest on zirconia increased, methods such as sol-gel, thermal dissociation and alkali fusion have been studied to produce zirconia powder from zircon sand [22,23]. However, alkali fusion method with sodium hydroxide (NaOH) is more effective in decomposing zirconium in zircon sand and economical compared to other methods [24,25].

Stabilizing zirconia with dopants such as yttrium oxide (Y_2O_3), magnesium oxide (MgO) and cerium oxide (CeO_2) were introduced in 1929 to limit phase transformation as a stable tetragonal zirconia was difficult to achieve without any addictive. The stabilized zirconia shows higher thermal expansion coefficient, low thermal conductivity and good mechanical properties [29].

Yttrium oxide is typically used to stabilize zirconia as it can retain tetragonal phase more effectively [30]. According to Chen *et al.,* [31] tetravalent ions Zr^{4+} are replaced by Y^{3+} and oxygen vacancies are formed when charge neutralization occurred. Then, yttrium zirconium hydroxide is formed in the reaction between alkalis and inorganic salts when dissolved in water for coprecipitation method during stabilization process of zirconia with yttrium oxide [32].

The phase of stabilized zirconia is highly affected by the concentration of yttria and sintering temperature [33]. A monoclinic yttria stabilized zirconia is naturally obtained at lower yttria content. Higher concentration of yttrium will result in a more stable zirconia. Nevertheless, the practice of stabilizing zirconia with higher yttria concentration is rarely done due to economic factors and high difficulty to obtain the right composition.

Economical value, availability and stability of zirconia are impending concerns of zirconia. Successful synthetization of zirconia from zircon sand has been done in Brazil, Indonesia, India and China [31-33]. However, there is no comprehensive study on zirconia synthesized from abundantly available Amang zircon sand in Malaysia despite the high potential of Amang zircon sand as a zirconia precursor [26]. Therefore, an elemental analysis on raw Amang zircon sand was conducted and synthetization of zirconia powder from Amang zircon sand with stabilization in varied concentration of yttria oxide has been studied.

2. Methodology

The purpose of this research is to study the suitability of local Amang zircon sand as a zirconia precursor. Therefore, an analysis on raw Amang zircon sand was conducted and synthetization of yttria zirconia powder in varied concentration using alkali fusion method has been studied.

2.1 Materials and Chemicals

Malaysian Amang zircon sand obtained from Amang Onn Sdn. Bhd. Onn Mineral Company in Kampar, Perak was used as received without further purification as a zirconia precursor. Analytical grade of solid sodium hydroxide (NaOH), hydrochloric acid (HCl), ammonium hydroxide (NH_4OH) from R&M Chemicals and ethanol (C_2H_5OH) from Systerm was used to synthesize zirconia powder and yttrium (III) oxide (Y_2O_3) from Sigma-Aldrich was used for stabilizing zirconia powder in this research.

2.2 Method

Zirconia powder from Amang zircon sand was prepared by using alkali fusion method with caustic soda [27]. Amang zircon sand was mixed with 6M sodium hydroxide (NaOH) in an iron crucible based on the reaction in Eq. (1).

$$ZrSiO_4 + 6NaOH \rightarrow Na_2ZrO_3 + Na_4SiO_4 + 3H_2O \quad (1)$$

Then, a sequence of thermal treatment was conducted starting from 100 °C before increased to 200 °C and finally to 670 °C. The temperature was being held for 1 hour in the first two temperature while 2 hours holding time was implemented for the last temperature to obtain higher zirconium composition [17,31].

Two phase products of sodium silicate (Na_2SiO_3) and sodium zirconate (Na_2ZrO_3) were obtained after thermal treatment. Excess sodium hydroxide, unreacted zircon and sodium silicate was removed by leaching the soft frit with deionized water at room temperature for 1 hour. Water insoluble sodium zirconate was filtrated and dried for 24 hours before leached with hydrochloric acid (HCl) in order to dissolve alkali zirconium and precipitate silicate by using Eq. (2) and Eq. (3).

$$Na_2ZrO_3 + 4HCl \rightarrow ZrOCl_2 + 2NaCl + 2H_2O \quad (2)$$

$$Na_2ZrSiO_5 + 4HCl \rightarrow ZrOCl_2 + H_2SiO_3 + 2NaCl + H_2O \quad (3)$$

The acid leaching process was done at 100 °C with pH 3.8 for 4 hours. In order to obtain zirconium oxychloride ($ZrOCl_2$), the temperature was reduced to 80 °C for 30 min before being filtered and dried for 24 hours.

In order to stabilize zirconia powder from Amang zircon sand, zirconium oxychloride was mixed with yttrium nitrate hexahydrate. Yttrium nitrate (III) hexahydrate ($Y(NO_3)_3$) was prepared by dissolving yttrium (III) oxide (Y_2O_3) in nitric acid (HNO_3) using a hot plate.

Next, zirconium oxychloride and deionized water solution was mixed with the prepared yttrium nitrate hexahydrate as ammonium hydroxide was titrated into the mixture until the pH reached 10-10.5 to produce an aqueous solution according to Eq. (4) in varied concentration from 3 wt.%, 4 wt.% and 5 wt.% to study the purity and phase of synthesized zirconia powder.

$$ZrOCl_2 + Y(NO_3)_3 + 5NH_4OH \rightarrow ZrO(OH)_2 + Y(OH)_3 + 2NH_4Cl + 3NH_4NO_3 \qquad (4)$$

Coprecipitation gel produced from the hydrolysis process needed to be filtered and washed with deionized water and ethanol (C_2H_5OH) for a few times. The gel was dried overnight at room temperature before calcined at 800°C for 4 hours to obtain yttria stabilized zirconia powder.

2.3 Characterization

Characterization of yttria stabilized zirconia (YSZ) powder was done by using X-ray fluorescence (XRF) to evaluate the elemental composition of zirconium in zircon sand and zirconia powder using Malvern Panalytical Epsilon 3XLE, from Britain Kingdom. X-ray diffractometer (XRD) Rigaku Ultima IV was used to analyse the phase using CuKα radiation with wavelength (λ) of 0.15 nm and scanning range of 10-80°. The diffraction spectra were then matched with database of powder diffraction file from ICDD while the crystalline size was calculated using Debye-Scherrer's equation as in Eq. (5). Scanning electron microscopy (SEM) from Hitachi SU3500 was used to study the morphology and average particle size of the synthesized zirconia powder and the particle size and particle size distribution slope parameter (S_w) was analysed by using particle size distribution (PSD) analysis from Malvern Panalytical Mastersizer 2000, Britain.

$$D = K\lambda/(\beta \cos \theta) \qquad (5)$$

whereby D is the particle size, K is Scherrer constant of 0.9, λ X-ray wavelength, θ Bragg's diffraction angle and β of full width at half maximum (FWHM) in rad.

3. Results

This chapter explained the results obtained from elemental analysis of raw Amang zircon sand that had been carried out. Then, the synthetization and characterization of Amang zirconia by using alkali fusion method was discussed. The effect of varied yttrium oxide concentration was carefully observed.

3.1 Raw Amang Zircon Sand

As seen in the elemental composition from x-ray florescence (XRF) analysis in Table 1, 76.07 wt.% of zirconium oxide (ZrO_2) was recorded in the raw Amang zircon sand. This composition was higher compared to other studies done previously with zircon sand from Brazil, Australia and China which were recorded at 62.43 wt.% (ZrO_2), 66.90 wt.% and 64.53 wt.% (ZrO_2) respectively [19,25,31]. This analysis showed that zirconium was the major element in Amang zircon sand.

Amang zircon sand also consists of other elements that were considered as impurities such as silicon (Si), ferum (Fe) and hafnium (Hf) which were recorded at 5.22 wt.%, 0.52 wt.% and 2.05 wt.%. These amounts of impurities were considered low. Lestari *et al.,* [34] mentioned that zircon typically have a weight ratio 2.92:1 of zirconium to silicon. However, Zr to Si weight ratio in raw Amang zircon sand was almost 4 times more from the previous finding with Indonesian zircon sand. This stressed the high composition of zirconium in the Amang zircon sand in which denoted it as a suitable precursor to synthesize zirconia powder.

Table 1
Chemical composition of raw Amang zircon sand

Element	Composition (wt.%)
ZrO_2	76.07
Si	5.22
Y	0.48
Hf	2.05
Fe	0.52
Ti	0.53
Ca	0.20
Ce	0.82
Nd	0.26
Th	0.59

Other elements recorded were also corresponding to previous research on the presence of silicon, hafnium and ferum in zircon sand. Attallah *et al.,* [35] mentioned that hafnium was typically found on the surface of zircon in 1-3 wt.% while titanium (Ti) was typically found together with zircon in the form of rutile (TiO_2), ilmenite ($FeTiO_3$) and titanite ($CaTiSiO_5$). Low amount of titanium at 0.53 wt.%. was recorded in raw Amang zircon sand in accordance to the statement by previous research.

0.20 wt.% of calcium (Ca) was also detected due to the presence of limestone in tin deposit in Malaysia. Tin (Sn) was also present in a small amount of 2.32 wt.% as Amang zircon sand was a by-product of tin mining industry in Malaysia. Varied sizes tin ores obtained in the tin mining industry in Malaysia was treated and left with a by-product known as “Amang”. The presence of tin in the analysis was justified because it was difficult to completely strip tin from the by-product in tin mining industry.

According to Apriany *et al.,* [36] a commercial industrial grade zircon usually composed of 65 wt.% mixture of ZrO_2 and HfO_2 with 33 wt.% of SiO_2. As seen in Table 1, higher composition of ZrO_2 and HfO_2 was recorded at 78.39 wt.% while SiO_2 was only at 12.86 wt.% compared to commercially used zircon. Thus, further confirmed the suitability of utilizing local Amang zircon sand as the precursor in synthesizing zirconia.

Based on the analysis, radionuclide elements such as cerium (Ce), thorium (Th) and neodymium (Nd) were also detected due to the monazite minerals in the Amang zircon sand. However, 0.59 wt.% of thorium found in the raw Amang zircon sand was determined to be low to trigger any radioprotection standard [37]. Thus, concluded that Amang sand from the location it was mined was safe to be used.

X-ray diffraction (XRD) analysis of Amang zircon sand was shown in Figure 1. The pattern showed several sharp peaks belonging to zircon sand and showed that the main crystalline phase is zirconium silicate, $ZrSiO_4$. All the peaks obtained satisfied the peaks of zircon in ICDD 00-002-0474. The highest peak at 43.94° with crystallite size of 65.96 nm belongs to raw zircon.

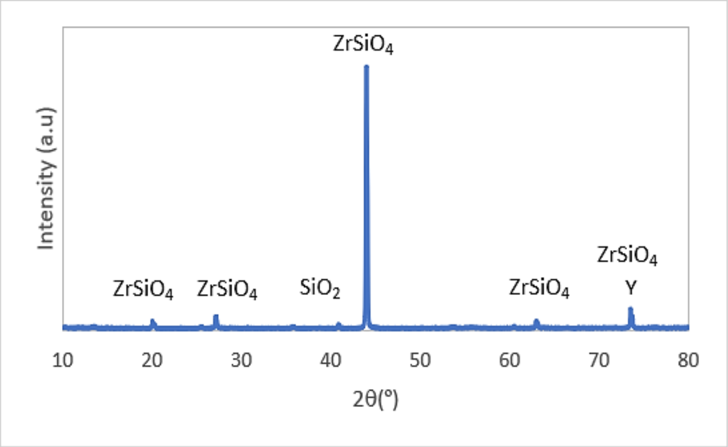


Fig. 1. XRD pattern raw Amang zircon sand

Silicon dioxide (SiO_2) was also detected at 36.68° and 40.84° (ICDD 01-089-3609). This pattern supported the composition analysis in Table 1 that mentioned zirconium and silicon as the main elements in Amang zircon sand. The presence of silicon dioxide in smaller peaks was due to the minor composition of silicon as detected in elemental analysis. The same applied to yttria (Y) at small peak of 73.52° (ICDD 01-074-1828). Raw Amang zircon was reported to have high crystallinity of 98.77%. This indicated that raw Amang zircon sand has a good crystallinity due to the strong bond of zirconium and silicon packed closely together in zircon sand [38].

The particle size distribution analysis for raw Amang zircon sand was done and the average particle size was calculated at 384.17 µm which was bigger compared to the average particle size of raw zircon from China at only 22.565 µm [28]. Consequently, the specific surface area was reduced to 0.0161 m^2/g. Based from previous research, it is advisable to mill the raw Amang zircon sand into a smaller particle size for industrial sintering. This will ensure higher increase of zirconium recovery and less sodium and silicon formation along with increasing the rate of reaction. However, the particle size should be bigger than 95 µm in order to control the formation of sodium silicate [27].

The value of particle size distribution slope parameter for raw Amang zircon, which was detected to be higher than 7, which was at 9.472. This implied a very narrow distribution [39]. A narrow distribution indicated monodispersed distribution in which the nanoparticles were in the same sizes. This was reflected in Figure 2 in which a monodispersed distribution was observed.

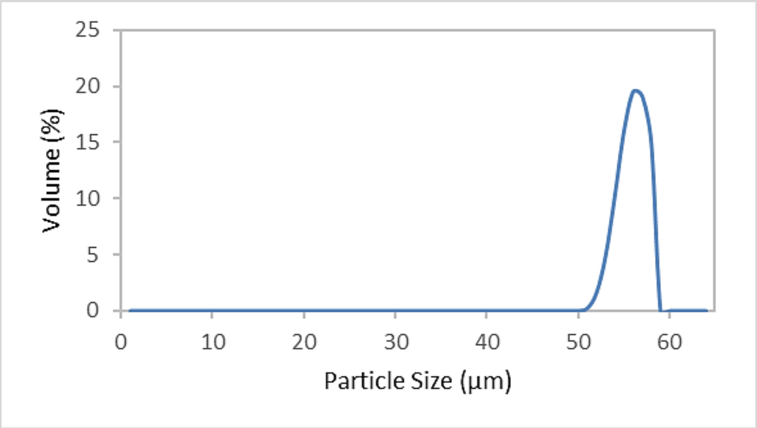


Fig. 2. PSD raw Amang zircon sand

3.2 Yttria Stabilized Zirconia Powder

Zirconia has to be stabilized with the addition of a dopant in order to hinder phase transformation in high temperature. Thus, doping of zirconia with yttrium in varied concentration ranging from 3-5 wt.% was carried out using zirconium oxychloride with ammonium hydroxide (NH_4OH). Coprecipitation gel was observed in Figure 3 after the pH reached 10-10.5 as the viscosity of the solution increased. Lastly, a mixture of zirconium hydroxide and yttrium hydroxide were formed, then calcined at 800 °C for 4 hours.

Fig. 3. Coprecipitation gel

As the samples were doped with yttria, the elemental composition of yttria stabilized zirconia (ZrO_2 +Y_2O_3) was observed in Table 2. The highest chemical composition was recorded with 5 wt.% of yttria at 71.01 wt.%, followed by 4 wt.% and 3 wt.% with readings of 70.62 wt.% and 69.91 wt.% respectively. Thus, concluded that the composition positively increased as the concentration of yttrium increased. Obviously, the composition of yttrium (Y) in the analysis increased as the concentration of yttria doped into zirconia powder increased. It was evidently from the low yttria composition of 0.48 wt.% in raw Amang zircon sand.

Table 2
Chemical composition of yttria stabilized zirconia (YSZ)

Element	3 wt.%	4 wt.%	5 wt.%
$ZrO_2 + Y_2O_3$	69.91	70.62	71.01
Si	3.83	3.36	2.94
Y	10.37	15.07	19.82
Hf	2.11	1.9	1.72
Fe	0.69	0.62	0.59
Ti	0.76	0.53	0.49
Cl	0.06	0.19	0.18
Ca	0.21	0.48	0.19
Ce	2.89	2.68	2.60
La	1.30	1.19	1.12
Nd	0.91	0.88	0.85
Th	1.13	1.07	0.99

It was observed that silicon (Si) was at lowest when stabilized with 5 wt.% yttria and highest at 3 wt.% yttria with readings of 2.94 wt.% and 3.83 wt.% respectively. However, it could be observed that silicon composition was even higher in raw Amang zircon sand which was at 5.22 wt.%. The addition of yttrium supported the reduction of silicon in the zirconia powder. Nguyen *et al.,* [11] reported that the presence of silicon dioxide in zirconia powder assist the abrasion resistance in ceramic coating application as long as the amount of silicon dioxide did not exceed the threshold as it may also lead to cracking if the amount was abundant.

Chlorine (Cl) was not detected in raw Amang zircon sand. Yet, this element present and increased after doping in varied concentration. This was due to the zirconium oxychloride precursor and processing route as reported by Kocjan *et al.,* [40]. Besides, the composition of chlorine was enhanced by the doping process as the concentration increased from the residue in Eq. (4).

Radionuclides elements such as neodymium (Nd), cerium (Ce) and thorium (Th) were reduced after zirconia was stabilized with yttrium. This observation was vital as high radionuclides will trigger health and environmental hazard especially when the sintering process of Amang zirconia was done in large quantity. The same was also observed by Lubbe *et al.,* [41] in which a small amount of radionuclides was detected from zirconia synthesized from South African zircon sand. Moreover, the research declared that the waste produced from the process could be safely disposed of as the number of radionuclides was low.

Apart from that, lanthanum (La) which was absent in raw composition was found after doping with yttria at 800 °C. Lanthanum formed due to the conversion of monazite that was naturally present in raw Amang zircon sand when sufficient heat was applied to initiate the conversion. Nevertheless, lanthanum (La) was reduced to 1.12 wt.% as the concentration of yttrium increased to 5 wt.%. On the other hand, a small composition of lanthanum, neodymium and cerium in zirconia powder improved the wear characteristic and hardness of the synthesized powder [11].

Beside lanthanum, impurities such as hafnium, ferum and titanium were also reduced when concentration increased to 5 wt.% yttria with reading of 1.72 wt.%, 0.59 wt.% and 0.49 wt.% respectively. It could be said that impurities reduced after doping with yttria. The reduction of impurities in the stabilized zirconia would enhance the strength and properties of synthesized yttria stabilized zirconia powder.

Based on XRD analysis in Figure 4, zirconium and yttrium oxide were observed in the patterns. The highest peaks for all samples belong to monoclinic zirconium oxide corresponded to ICDD 00-003-0515. The monoclinic peaks were steeper as the concentration increased. This indicated that yttria increased the crystallinity of zirconia.

Monoclinic peaks at 27.14° and 55.64° were observed after zirconia was doped with 3 wt.% of yttria. Yttria was also observed when compared to ICDD 00-025-1200 and coexist with zirconia at some peaks. Compared to pattern in ICDD 00-002-0733, tetragonal zirconia was observed at the same peaks of cubic from ICDD 01-071-4810. This was due to the decomposition of tetragonal after cooling under the low temperature calcined at 800 °C.

Diffraction patterns in 4 wt.% and 5 wt.% of yttria were studied with the same ICDD and monoclinic peaks were observed at the same peaks. Yttria was observed alongside tetragonal. On the other hand, tetragonal and cubic zirconia phase coexist at some peaks. The formation of tetragonal and cubic zirconia was higher as the concentration increased. The monoclinic peak was also observed to be the sharpest at 5 wt.%. As seen in all diffraction patterns, yttria typically coexist with zirconia at some peaks which was mostly in tetragonal phase.

Most of the time, tetragonal and monoclinic phases can be found in the same peaks. Heshmatpour *et al.,* [42] also observed the same for tetragonal and cubic at 30.5° and the synthesized yttria zirconia powder tended to become monoclinic. This was explained by Leib *et al.,* [43] that yttria stabilized zirconia only remain stabilized at tetragonal phase when doped with 8 mol% of yttria. Supplementally, Wilk *et al.,* [44] stated that zirconia was predominantly in monoclinic phase at low yttrium oxide concentration. Hence, the presence of major monoclinic yttria stabilized zirconia and minor cubic yttria stabilized zirconia due to the inability of the tetragonal to remain metastable as the concentration increased.

Sharp peaks in the diffraction patterns showed the increased of crystallinity after doping process as peak width was inversely proportional to the crystallite size [45]. Crystallite size was affected by the concentration of dopant and reduced from 85.33 nm, 71.15 nm, to 47.43 nm which was at 3 wt.%, 4 wt.% to 5 wt.% respectively.

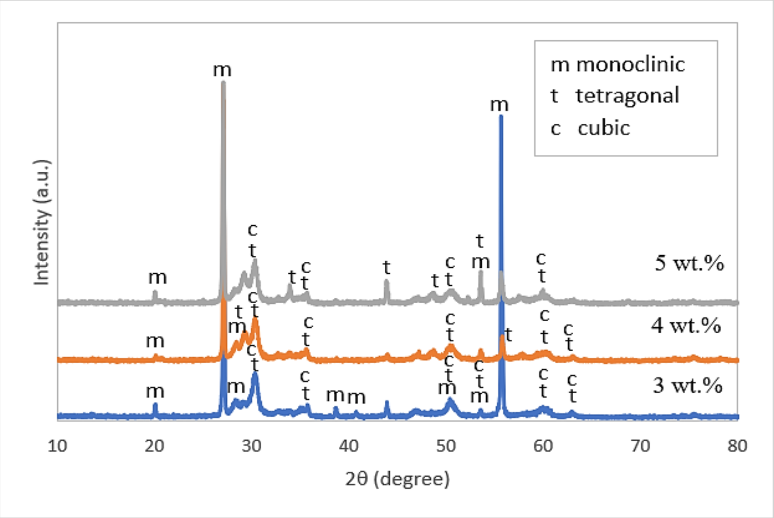


Fig. 4. XRD analysis of YSZ powders in varied concentration

Particle size distribution (PSD) analysis of yttria stabilized zirconia calcined at varied concentration in Table 3 observed a reduction from 384.17 µm once stabilized with yttria. The smallest particle size was determined to be 88.47 µm when doped with 4 wt.% and largest at 122.29 µm when doped with 3 wt.%. Yttria stabilized zirconia powder at 5 wt.% was slightly bigger than 4 wt.% which was recorded at 88.97 µm. This indicated agglomerated zirconia powder at 5 wt.% due to the conventional coprecipitation method of synthesizing yttria stabilized zirconia as mentioned by Nishihara *et al.,* [46]. Moreover, Lubbe *et al.,* [41] added that zirconia powder synthesized from alkali fusion of caustic soda was hygroscopic in nature.

Theoretically, smaller particle size was favoured as specific surface area would increase along with surface roughness when particle size was reduced. Thus, the rate of reaction increases and facilitate a better dissolution [30]. High specific surface area of 0.234 m^2/g was recorded when the particle size was reduced to 88.47 µm when doped at 4 wt.%. In high performance application, a large specific surface area is ideal as the efficiency of interparticle distance increased with specific surface area. In addition, Bumajdad *et al.,* [47] also reported that particle size of zirconia affected the stability of zirconia polymorphs and large particle would stabilize monoclinic zirconia.

Table 3
Particle size distribution analysis of yttria stabilized zirconia (YSZ)

Process	Raw	3 wt.%	4 wt.%	5 wt.%
Particle size (µm)	384.17	122.29	88.47	88.97
S_w	9.472	2.204	1.764	1.901
Specific surface area (m^2/g)	0.0161	0.122	0.234	0.195

As seen in Figure 5, the doping of zirconia with yttria showed monomodal distribution. Monomodal distribution indicated homogeneous dispersity with lower sizes in the system. Rahmawati *et al.,* [15] also reported a reduction in particle size and increased in homogeneity for yttria stabilized zirconia. The homogenous system was achieved as the stabilization of zirconia with yttria reduced the agglomeration typically occurred during the synthetization.

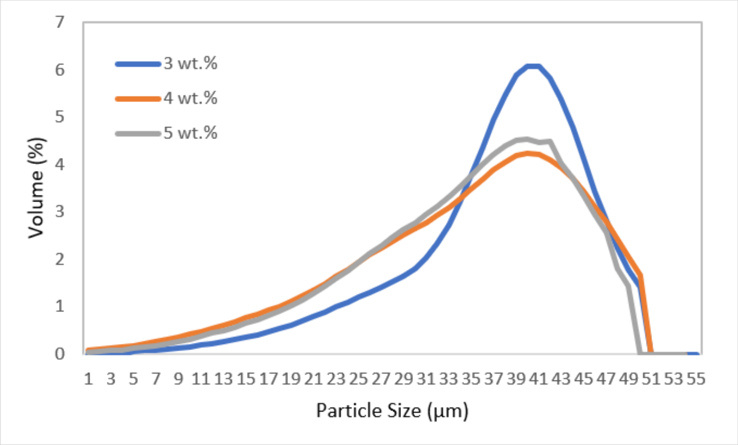
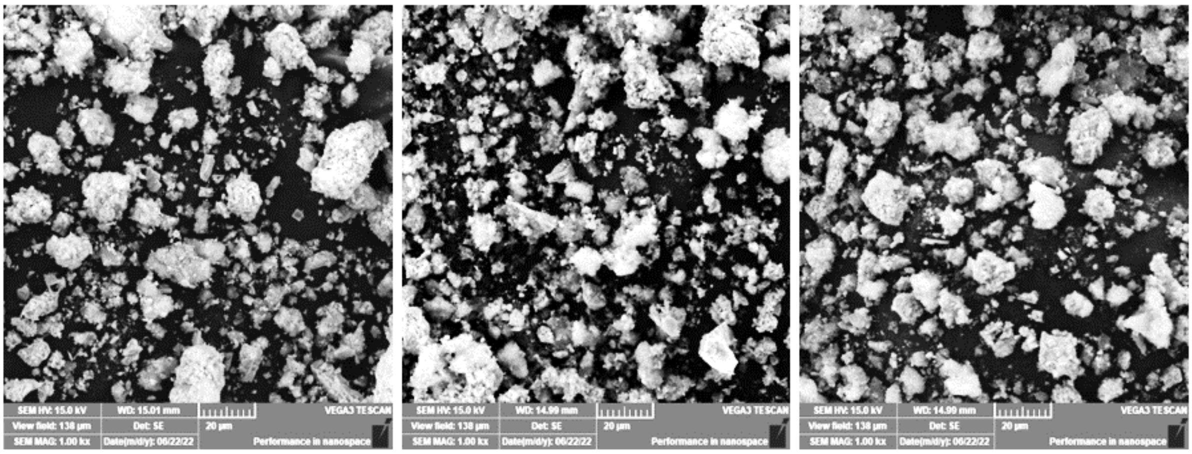


Fig. 5. PSD of zirconia powder in varied concentration

In Figure 6, SEM analysis at 1,000x magnification was done. Mixed grains of large and small sizes measuring not more than 20 µm were detected in all samples with angular and pyramidal shape. The grain size ranging from 13.06 to 16.09 µm for 3 wt.%, 9.58 to 11.86 µm and 9.50 to 16.02 µm when stabilized with 4 wt.% and 5 wt.% yttria respectively. The grain size was rather large. Thus, Amang yttria zirconia power could not be stabilized to tetragonal as seen in Figure 4. Besides, Yurdakul *et al.,* [48] explained that zirconia powder obtained from ammonia hydrolysis has the tendency to agglomerate due to the extremely high surface tension during calcination process [8].



(a) (b) (c)

Fig. 6. SEM photography of (a) 3 wt.% (b) 4 wt.% (c) 5 wt.% of YSZ

4. Conclusions

Raw Amang zircon sand was studied and proven to be able to be used as a precursor to synthesize zirconia. Yttria stabilized zirconia (YSZ) powder in varied concentration were synthesized successfully from Amang zircon sand using alkali fusion and coprecipitation method. The elemental composition confirmed the high composition of zirconium oxide and low impurities in the powder. The composition of zirconium increased as the concentration of yttria increased. Conversely, the impurities decreased. The phase was determined to be predominantly monoclinic yttria stabilized zirconia with minor tetragonal and cubic phase. Tetragonal and cubic phase increased along with concentration of yttrium. However, the phase transformation from monoclinic to tetragonal hindered the stabilization of tetragonal zirconia even after doping with yttria as the amount of yttrium was considered low. Based on analysis, yttria stabilized zirconia has high tendency for monoclinic due to the large particle size.

Acknowledgement

This research was funded under the Fundamental Research Grant Scheme (FRGS, 600-RMI/FRGS 5/3 (457/2019) from Universiti Teknologi MARA (UiTM) Shah Alam, Malaysia and authors would like to thank Amang Onn Sdn. Bhd. for providing the raw Amang zircon sand for this research.

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