

NANO-SILICA DERIVED FROM POWER GENERATION RICE HUSK ASH WASTE BY PRECIPITATION

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Rice husk ash (RHA) from a power generation plant has not been fully utilised although this substance contains a high amount of active silica. In this research, RHA was utilised as the precursor for the extraction of nano-silica using a simple precipitation method. The extraction of nano-silica contained three main steps; pre-treatment, dissolution and neutralising precipitation. The effects of the pre-treatment conditions, types of neutralisation acid, impurity (Na and Cl) removal method, solvent modification and surfactant (Tween80) addition on the characteristic properties of the extracted nano-silica, such as the morphology, size distribution and chemical composition, have been carefully studied in detail. By pre-treatment with hot 2M HCl (90 °C), neutralisation with 2M HCl, ageing the neutralised gel for 24 h, and finally washing the precipitate with hot water (90 °C) three times, the resulting nano-silica was ~43 nm in size and had 99.75 % purity. Using the ethanol/water solvent system with an addition of 0.2 – 0.3 % Tween80 could remove the gel ageing step with final nano-silica particles of ~36 nm.

INTRODUCTION

Nano-silica has found an increasing demand in a wide range of fields and industries, such as polymers, electronics, catalysis, pharmaceuticals, dental materials, and others [1-3]. Nano-silica powder is generally prepared by using vapour-phase reaction, sol-gel, and thermo-decomposition methods, mainly using chemicals as a raw material [4-7]. With more concerns about the environmental impact and resource scarcity, there is an urgent need of alternative raw materials for the preparation of nano-silicas.

Rice husk (RH) is a well-known, abundant agricultural by-product in rice-producing countries such as China, India, Indonesia, Bangladesh, Vietnam and Thailand [8-9]. The annual global production of rice husk and rice husk ash (RHA) were around 120 – 150 million tonnes and 30 million tonnes, respectively [9-10]. Due to the high silica content (~ 15 – 28 wt. %) in rice husk [11], the extraction of amorphous silica has been extensively studied [12-17]. In general, RHA or the primary nano-silica could be prepared from rice husk using pyrolysis at 600 – 700 °C for a few hours [18]. Combustion-derived RHA contains less than 95 wt. % of SiO₂, and the remaining part comprises different alkali oxides and impurities (C, K, Na, Fe) [19]. Refining the RHA or RH before the chemical conversion to the final nano-silica powder may, thus, be necessary [20, 21].

Refluxing the primary silica or silica gel derived from the first step in a chemical solution have been reported [18, 22-23]. The silica produced by the precipitation method is of high purity [24], but the process is a rather complex, having a massive reagent consumption and high cost [8]. By contrast, pyrolysis of the pretreated rice husk is more attractive due to its simple preparation process and low cost [25]. A small portion of the silica, however, might be covalently bonded to the organic compounds and can withstand very high temperatures [11]. In the chemical precipitation or sol-gel synthesis of the nano-silica, agglomeration and coarsening of the nanopowder could result [26]. To control the morphology and the agglomeration states of the resulting material, chemical additives (surfactants) were added during the gelation or precipitation steps [26]. The nano-silica was prepared from RH using the sol-gel method with two types of surfactants, surface-active and cationic surface-active substances. The results showed that using surface-active substances, the size distribution of the silica particles is more uniform [26].

With the growing demand for alternative energy to replace oil, rice husk has been used as a primary fuel in power generation plants in many counties including Thailand [27]. The thermochemical conversion of rice husk to fuel could be achieved via gasification technologies, such as fluidised bed gasification at a temperature of around 800 – 850 °C. The power generation gasification system may be slightly

modified to fit the raw materials. Stoker-type gasification is one of the technologies used for RH power generation. In this process, the RH is milled before combustion with two types of RHA resulting, fly ash and bottom ash. The bottom ash is coarser in size and a lot smaller in quantity than the fly ash. Preparation of nano-silica from RHA disposed from a biomass power plant by the precipitation method for use in Belite cement preparation has been reported [28]. The nano-silica was highly reactive due to the high surface area and pore volume.

From the literature, numerous works have reported on the extraction of nano-silica from RH, but only a few works used RHA as the starting material. In this research, the authors attempted to study the effects of the synthesis parameters (neutralisation acid, pre-treatment, washing method, and surfactant) on the characteristic properties of the resulting nano-silica extracted from the waste RHA obtained from the power generation plant. The effects of the neutralisation acid, pre-treatment, and washing method were first studied so these parameters were optimised before the extraction was performed using the final process. Tween 80, (polysorbate 80, polyoxyethylene sorbitan monooleate) is a non-ionic surfactant widely used as an emulsifier in cosmetics, pharmaceuticals and food products [29]. Tween 80 was selected for its availability, low cost and non-ionicity. The non-ionic surfactant was reported to offer better performance than ionic-type surfactants [26].

EXPERIMENTAL

Materials

All the chemicals were reagent grade and used without further purification. The fly-RHA was received from the power generation plant, in Pichit Province, Thailand (Figure 1a). This power plant has the highest generation capacity (20 MW) among other RH power generation plants in Thailand. The RHA, mixed with both white and black particles, was produced in a Stoker-type gasifier. The preliminary analysis for the composition, using an X-ray fluorescence technique, the loss on ignition (LOI), using a thermogravimetric analysis, of the RHA is shown in Table 1. The scanning electron microscopy picture of the RHA (Figure 1b) showed a high surface porosity.

Table 1. Chemical analysis of the as-received RHA.

Element (wt. %)													
Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Mn	Fe	Zn	Br
0.04	0.36	0.26	94.43	0.87	0.14	0.32	2.26	0.89	0.01	0.17	0.21	0.02	<0.01
LOI at 1025 °C 5.62 %													

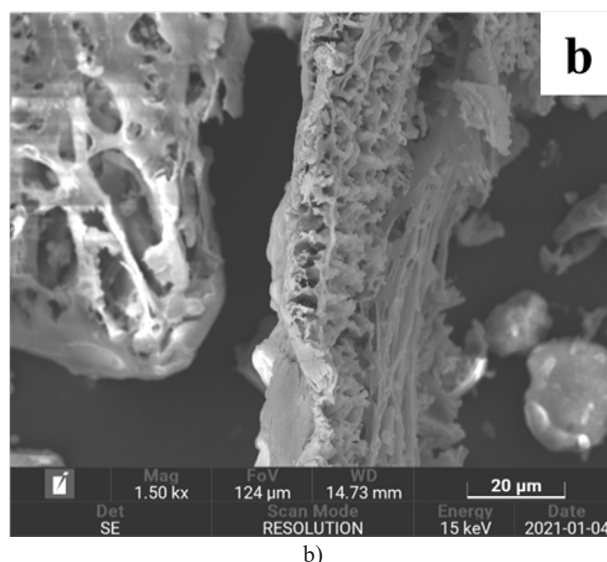
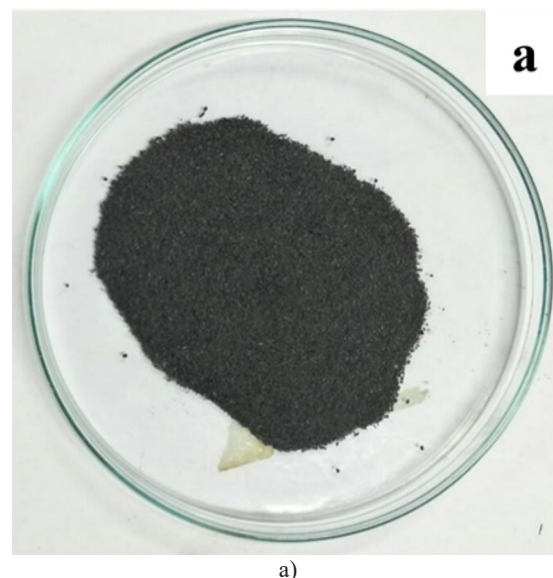


Figure 1. (a) As-received RHA and (b) SEM picture.

The preparation of nano-silica in this research, in general, had four steps; pre-treatment or acid (HCl) leaching, dissolution of RHA to form a sodium silicate gel with NaOH solution, neutralisation of a sodium silicate gel with an acid, and finally gel ageing to form a nano-silica powder.

Effects of acid neutralisation and ageing time

The as-received RHA powder (15 g) was placed in a beaker and treated with 2M HCl (RHA was added to the acid at 1 to 10 by weight) by soaking

for 12 hours at an ambient temperature (average 27 °C). The leached RHA was washed with deionised water many times until a pH of 7 was obtained. The RHA was then boiled in 3 M NaOH (1 to 10 by weight) at 95 °C for 4 hours until a sodium silicate gel (Na_2SiO_3) was obtained. The gel was subsequently neutralised with 6M HCl or 6M H_3PO_4 using the direct mixing method until the precipitate formed. The direct mixing method was reported to have a high production rate with an acceptable size and dispersion of the silica nanoparticles [30]. The precipitate was aged for 0, 12, 18 and 18 hours at an ambient temperature. The precipitate was filtered and washed with deionised water many times. The precipitate was washed with normal deionised water many times and finally dried in an electric oven at 90 °C for 24 hours.

Effects of pre-treatment of the RHA

An acid-leaching or pre-treatment step using hot hydrochloric acid (HCl) solution was carried out to remove the metallic impurities. To determine the effectiveness of the acid pre-treatment procedures, the as-received RHA powder (15 g) was placed in a beaker and leached with 2M HCl (RHA was added to the acid at 1 to 10 by weight) using four conditions, soaking for 12 and 24 hours at an ambient temperature (average 27 °C), and heated at 90 °C for 1 and 3 hours. The leached RHA was washed with deionised water many times until a pH of 7 was obtained. The RHA was then boiled in 3M NaOH (1 to 10 by weight) at 90 °C for 4 hours until a sodium silicate gel (Na_2SiO_3) was obtained. The gel was subsequently neutralised with 6M HCl until the precipitate formed. The precipitate was filtered and washed with normal deionised water many times and finally dried in an electric oven at 90 °C for 24 hours.

Effects of final washing for impurity removal

In this experiment, the as-received RHA powder (15 g) was pre-treated by boiling it with 2M HCl for 3 hours. The leached RHA was washed with deionised water many times until a pH of 7 was obtained. The RHA was then boiled in 3M NaOH (1 to 10 by weight) at 90 °C for 4 hours until a sodium silicate gel (Na_2SiO_3) was obtained. The gel was subsequently neutralised with 6M HCl until the precipitate formed. The precipitate was aged for 12 hours before washing with hot deionised water (90 °C), at a ratio of solid to the water of 1:10, once, twice and three times. The solid was filtered and dried in an electric oven at 90 °C for 24 hours.

Effects of solvent modification and surfactant addition

In this experiment, the as-received RHA powder (15 g) was pre-treated with hot 2M HCl (90 °C)

for 3 hours. The pre-treated RHA was washed with deionised water many times until a pH of 7 was obtained. The washed RHA was then soaked in 3M NaOH (1 to 10 by weight) at 95 °C for 4 hours until a sodium silicate gel (Na_2SiO_3) was obtained. The sodium silicate gel was then mixed with water and ethanol at a weight ratio of 1:1:0.1 ($\text{Na}_2\text{SiO}_3\cdot\text{H}_2\text{O}:\text{C}_2\text{H}_5\text{OH}$). Tween 80 was slowly added at 0, 1, 2, 3 % by weight of the Na_2SiO_3 gel. Subsequently, 6M HCl was slowly added to the gel until a precipitate formed. Finally, the precipitate was filtered and washed with hot deionised water (90 °C) three times. The solid-to-water ratio was 1:10.

Characterisation

The microstructure of the polished surfaces was observed under a Scanning Electron Microscope (FE-SEM, Tescan, model Mira 4) equipped with Energy Dispersive Spectroscopy (EDS, Oxford Instruments, Model Ultim MaX) at a 20 kV accelerating voltage while the phase development was examined using X-ray diffraction (XRD) method with $\text{CuK}\alpha$ radiation at 15 kV, 20 mA. The chemical composition was analyzed using Energy Dispersive X-ray Fluorescence (EDXRF, Bruker, M4 Tornado).

RESULTS AND DISCUSSION

Effects of neutralising acid and ageing time

In this experiment, two types of acid solutions (HCl and H_3PO_4) were selected as the neutralising agents. Pre-treatment was performed by soaking with 2M HCl for 12 hours at an ambient temperature. Figure 2a shows that, when using HCl, the precipitate was thick with a milky-like feature and very fine. With the use of H_3PO_4 , however, a clear colourless gel was obtained (Figure 2b).



a) HCl

Figure 2. The resulting products with use of (a) HCl and (b) H_3PO_4 as the neutralising agents.

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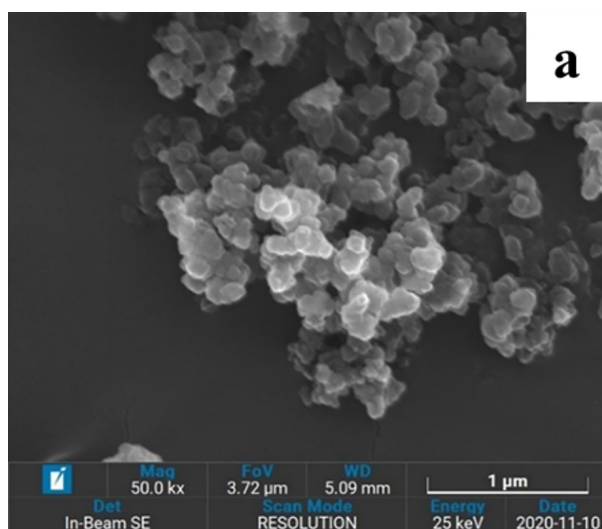
b) H_3PO_4

Figure 2. The resulting products with use of (a) HCl and (b) H_3PO_4 as the neutralising agents.

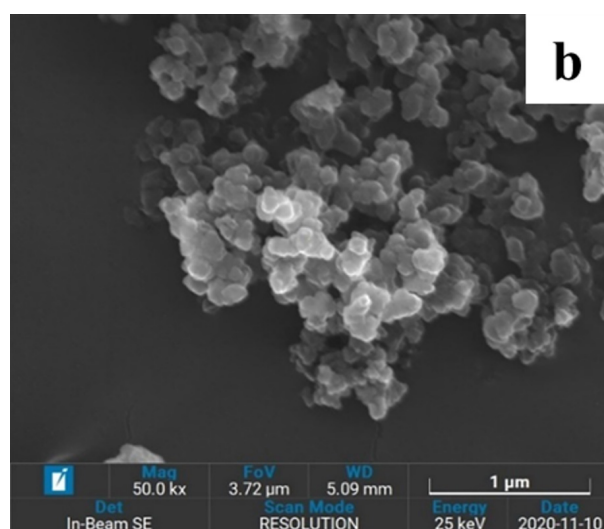
The SEM micrographs for the extracted nano-silica powder using 2M HCl as the neutralising agent (Figure 3) showed that the particle size of the silica powder at 0 hour ageing (~100 nm) became slightly larger when the ageing time was increased to 12 hours before getting smaller with a further increasing ageing time. At 18 hours of ageing, the size of the nano-silica was reduced to around 40 nm. The average particle size and the size range of the nanoparticles analysed using image a software (Image J) are displayed in Table 2.

Table 2. Effects of ageing time on the size of the extracted nano-silica.

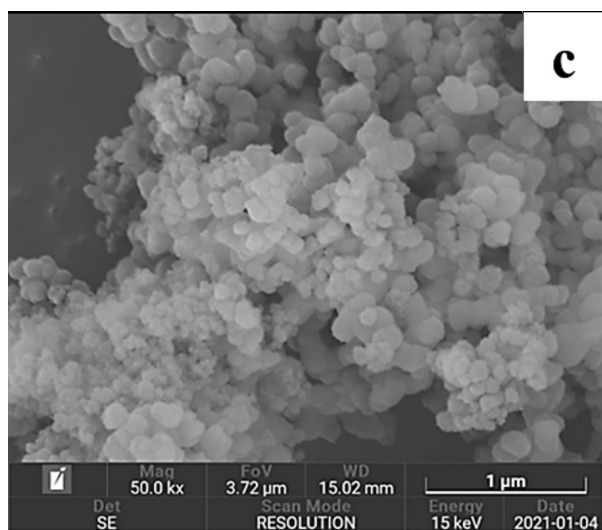
Ageing time (h)	Average particle size (nm)	Particle size range (nm)
0	93.95	56 – 145
12	103.30	51 – 165
18	75.74	29 – 130
24	43.35	32 – 61



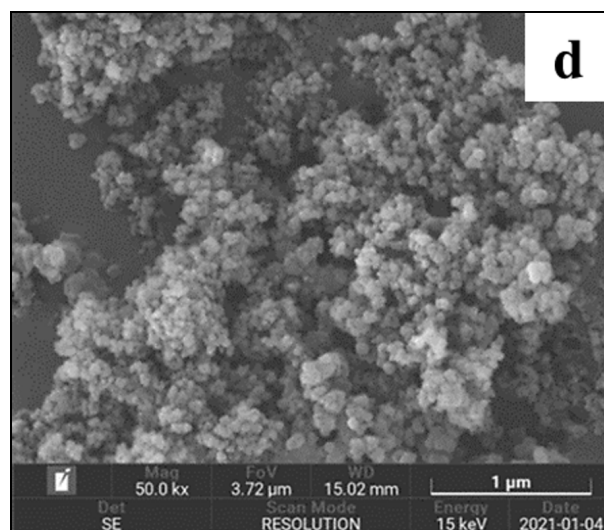
a) 0 h



b) 12 h



c) 18 h



d) 24 h

Figure 3. SEM micrographs for the extracted silica powders with 2M HCl as the neutralising agent and ageing for (a) 0, (b) 12, (c) 18 and (d) 24 hours.

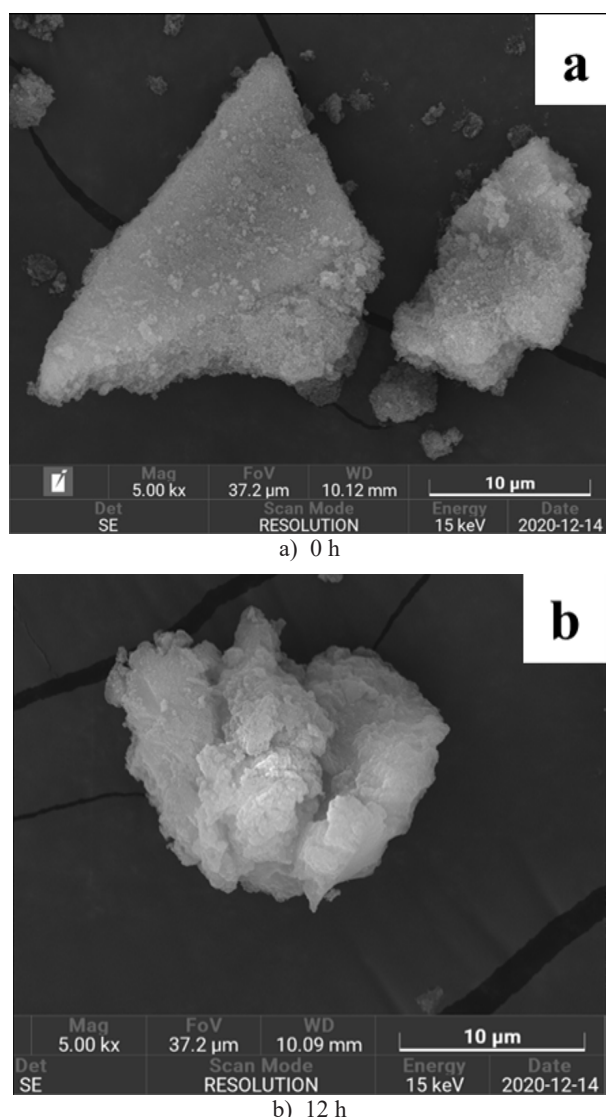


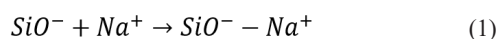
Figure 4. SEM micrographs for the extracted silica powders with 2M H_3PO_4 as the neutralising agent and ageing for (a) 0 and (b) 12 hours.

The SEM micrographs for the neutralised product using H_3PO_4 as the neutralising agent are shown in Figure 4. At all the ageing times (18 and 24 hours not shown), the resulting products did not form a dispersed powder, but rather a packed solid lump. This result indicated that HCl was a better neutralising agent to use in the next step.

Pre-treatment method

The chemical analysis result of the extracted nano-silica powder prepared using the different pre-treatment conditions is shown in Table 3. The result revealed that the main impurity was sodium, with Cl being another main impurity found in No. 1–3. The original RHA powder contained only a minor amount of Na (0.04 %) and Cl (0.32 %), so the greater impurity levels found later in the extracted silica powder arose from the added chemicals (NaOH and HCl). The result indicated that insufficient washing occurred in the final stage. The extracted silica based on 90 °C for 3 hours showed a relatively smaller content of Cl, Fe, and Zn impurities than the other conditions.

Sodium ions (Na^+) absorbed onto the negatively charged surface of silica can be explained by Equation 1 which led to the negative value of the zeta potential [31].



The presence of electrolytes caused enhanced aggregation of the primary silica nanoparticles and stabilised aggregates and agglomerates [31]. It is, thus, imperative to remove the Na^+ as much as possible.

The SEM micrographs for extracted silica based on different pre-treatment conditions are shown in Figure 5. The results indicated that, by prolonging soaking at an ambient temperature from 12 (Figure 5a) to 24 hours (Figure 5b), the particles' coalescence to a larger size could be observed. Some of the fine particles were still observed, suggesting a wider particle size distribution than the 12 hours of soaking. Soaking the RHA at 90 °C for 1 and 3 hours (Figure 5d) resulted in nano-silica powders with a better size uniformity than at an ambient temperature. According to the EDXRF and SEM results, the pre-treatment condition selected for the next step was 90 °C for 3 hours.

Effects of final washing

When HCl is used as the neutralising agent in the final step, sodium chloride would be the by-product of this reaction. As discussed in Section 3.2, the presence of Na^+ resulted in a stable aggregate of particles. The effectiveness of the final washing process investigated using the X-ray diffraction technique is shown in Figure 6. The results indicated the disappearance

Table 3. EDXRF chemical analysis of the extracted silica with different pre-treatment conditions.

Number	Pre-treatment condition (T, h)	Element (wt. %)									
		Na	Al	Si	Cl	K	Ca	Ti	Mn	Fe	Zn
1	Ambient, 12	7.02	0.28	86.07	5.84	0.55	0.09	0.03	0.03	0.05	0.04
2	Ambient, 24	3.22	0.33	93.81	2.04	0.13	0.33	0.02	0.01	0.04	0.01
3	90 °C, 1	7.46	0.31	85.20	6.39	0.37	0.18	0.02	0.01	0.00	0.00
4	90 °C, 3	6.51	0.22	91.76	0.89	0.89	0.37	0.02	0.01	0.00	0.00

Table 4. EDXRF chemical analysis of the extracted silica after the 2nd and 3rd wash.

Treatment	Element (wt. %)									
	Na	Al	Si	S	K	Ca	Ti	Mn	Fe	Zn
2 nd wash	2.45	-	97.55	-	-	-	-	-	-	-
3 rd wash	0.25	-	99.75	-	-	-	-	-	-	-

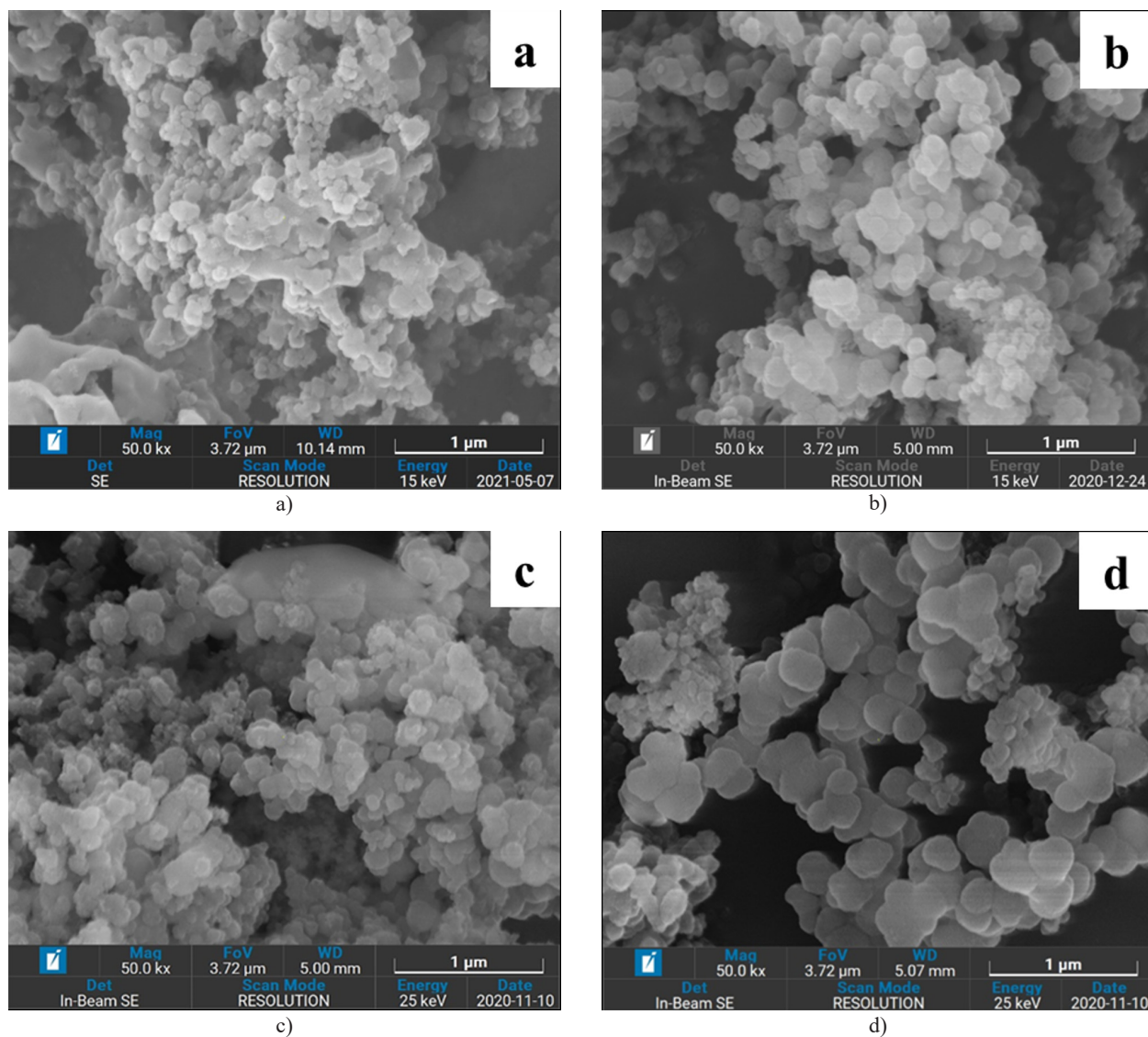


Figure 5. SEM micrographs for the extracted silica with the different pre-treatment conditions of (a) ambient temperature, 12 hours, (b) ambient temperature, 24 hours, (c) 90 °C, 1 hour and (d) 90 °C, 3 hours.

of NaCl after the 3rd wash. The EDXRF analysis (Table 4) was in good agreement with the XRD result, the SiO₂

content increased while the other impurities decreased dramatically with the increasing number of washings.

Table 5. Analysis of the particle size and the distribution of the extracted nanoparticles.

%Tween 80	Average single particle size (nm)	Single particle size range (nm)
0	60.18	36 – 91
0.1	56.65	32 – 78
0.2	36.52	12 – 67
0.3	36.40	15 – 60

Effects of solvent modification and surfactant

From our previous experiment, the best processing conditions were (i) pre-treatment soaking in hot HCl (90 °C, 3 hours) (ii) neutralisation with 6M HCl (iii) washing with hot deionised water three times. However, the results suggested that the particle size of the extracted nano-silica was still non-uniform, agglomerated with the size of >50 nm if the final precipitate was not aged for 24 hours. In this experiment step, the solvent system

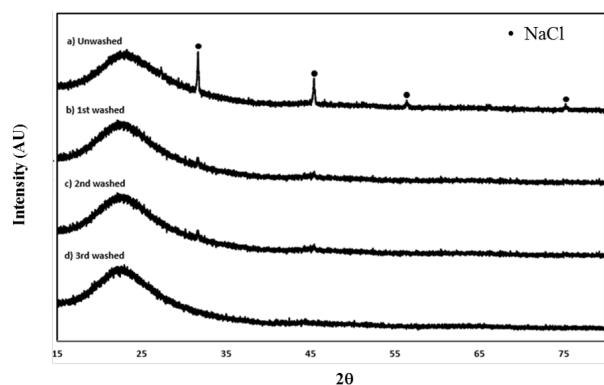
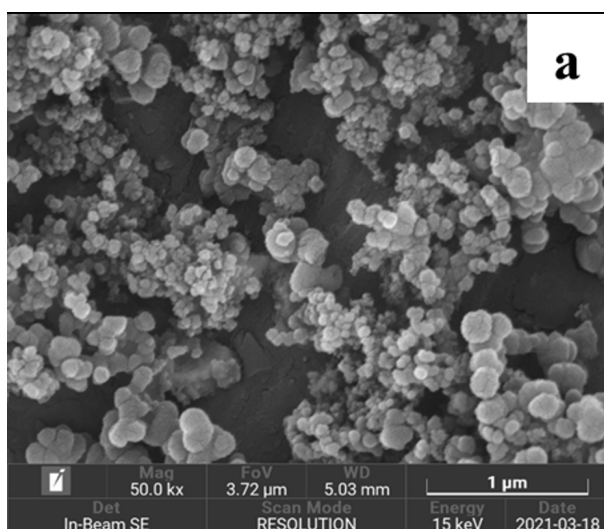


Figure 6. X-ray diffraction patterns for the extracted nano-silica after washing at different times.

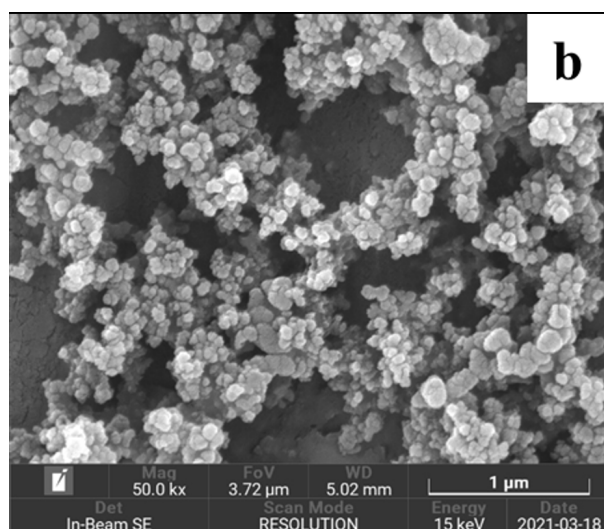
was, thus, further modified and the final ageing was removed. Ethanol ($\gamma = 21.6 \text{ mN}\cdot\text{m}^{-1}$ [32]) was added to reduce the surface tension of water ($\gamma = 72.8 \text{ mN}\cdot\text{m}^{-1}$ [32]), so the particle coarsening and agglomeration

could be reduced. The effect of ethanol as a co-solvent on the monodispersity and size of the silica particles was observed [33–34]. The effect of the concentration of the surfactant (Tween80) on the morphology of the extracted nano-silica is shown in Figure 7. The particle size analysis of the nano-silica is shown in Table 5.

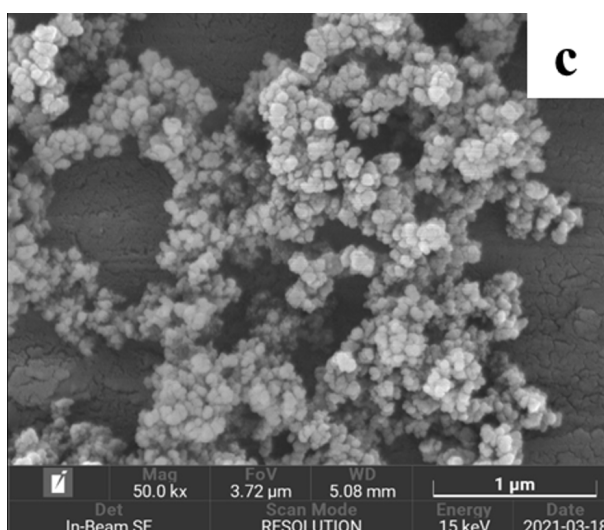
The SEM result showed that with the mixed ethanol-water solvent (Figure 7a, without the surfactant), the particles became more uniform with a smaller size, as compared to the pure water solvent (Figure 5d). The particle size of the extracted nano-silica had reduced further with a narrower size distribution when Tween 80 was added. At 0.2 – 0.3 % Tween 80, the size of the extracted nano-silica was not changed so this could be the saturation point. A previous study also reported the size reduction of nano-silica when Tween 80 was added [35].



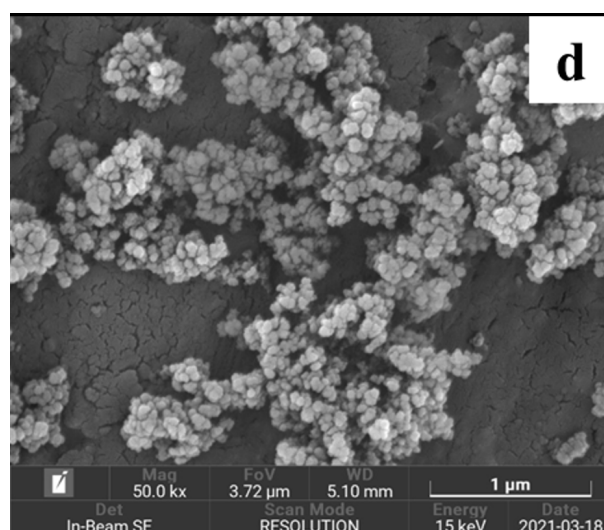
a)



b)



c)



d)

Figure 7. SEM micrographs for the extracted nano-silica particles using different amounts of surfactant (a) 0, (b) 0.1, (c) 0.2 and (d) 0.3 %.

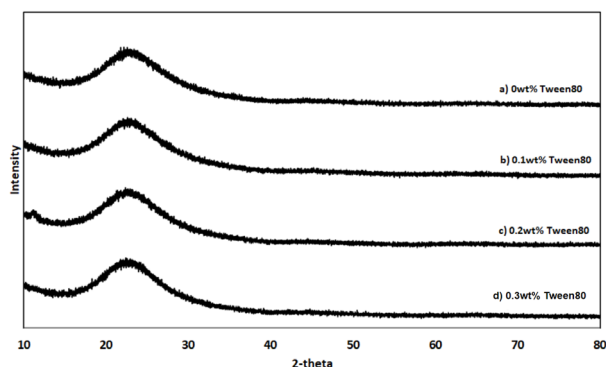


Figure 8. XRD analysis for the extracted nano-silica with a variety of Tween 80 concentrations.

The XRD results (Figure 8) revealed the amorphous nature of the extracted nano-silica in all the conditions suggesting that the surfactant did not affect the molecular structure of the nano-silica.

CONCLUSIONS

Nano-silica was successfully extracted from rice husk ash waste from power generation. The effective pre-treatment process for metal impurity removal from the as-received RHA was obtained by soaking with hot 2M HCl for 3 hours before the dissolution process by NaOH. The effective neutralisation acid was 2M HCl with an ageing time of 24 hours. The final nano-silica was ~43 nm. Sodium (Na^+) and (Cl^-) ions, the main impurities derived from the acid-base reagents used, could be removed by washing with hot water for at least three times. The further modification of the precipitation step to eliminate the gel ageing by mixing water with ethanol and the addition of Tween 80 was found to be successful. The extracted nano-silica had a mean size of ~36 nm and a purity of 99 %.

Acknowledgments

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