

Synthesis and Characterization of Nanosilica from Rice Husk with Applications to Polymer Composites

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Abstract The present study deals with preparation of Silica nanoparticles (SiO₂NPs) from rice husk ash and to be used as nanofiller in Epoxy/SiO₂ –nanocomposites. Pure amorphous Silica (SiO₂) was prepared from rice husk ash (RHA) by thermal treatment of the rice husk at 400°C followed by acidic treatment and then thermal treatment of the ash at 650°C. The produced Silica was then characterized by X-ray fluorescence and X-ray diffraction to confirm the purity and crystalline of silica. The obtained pure amorphous Silica was used to produce Silica nanoparticles. Process parameters such as: NaOH mixing time and drying time, were studied and the particle sizes were confirmed by Atomic force microscopy (AFM). The best average particle diameters of SiO₂ NPs were (53nm) using the best mixing time 14 hours of Silica particles with 2.5 N NaOH at best drying time (48 hours) of silica solution. Fourier Transformation infrared Spectroscopy (FTIR) showed that broadband appeared at about 3429.43cm⁻¹ which represent the presence of hydroxyl groups on the SiO₂ NPs surface and no further functionalization is needed. Tensile test of Epoxy - (0, 0.25, 0.5, 0.75, 1, 2, 3, 6, 9, 12 and 14 wt%) SiO₂ nanocomposites results showed that the best improvements in tensile strength of epoxy resin at a loading of 0.5 wt % SiO₂ NPs due to better dispersion.

Keywords Silica Nanoparticles, Rice Husk, AFM, Epoxy, Nanocomposite

1. Introduction

A new technology involves waste utilization and cost reduction in industrial processing by using rice husk as a resource material is created due to Fast growth in industrialization and population (Kumar et al., 2013) [1]. One of the major food products in the world is Rice. Its production generates an equally large amount of waste in the world, namely rice husk (RH). Rice husk (RH), is an inexpensive waste material that was used to produce silica and nano silica (Rafiee et al., 2012) [2].

Most of researches utilized different recycled wastes to improve composites properties with the environmental friendly reinforcing phase. Among various reinforcement filler used, rice husk (RH) is an agricultural residue by product abundantly available (Saravanan and Kumar., 2013) [3] and (Moosa and Awad, 2016) [4].

At burning temperatures below 700°C, ash, which is rich in amorphous silica, will form. The formed rice husk ash may contain several metallic impurities like men, Fe, Na, K, Ca etc. that can decrease rice husk silica purity and surface area (Dominic et al., 2013) [5]. Eliminating these metallic impurities is required to obtain high purity silica (above 99%)

by hydrochloric or sulfuric acid leaching of rice husk followed by controlling combustion (Abu Baker et al., 2016) [6].

Rice husk (RH) is an agricultural biomass, which provides an abundant silica source (Adam et al., 2011) [7]. A wide range of silica products is manufactured for different applications. But it is mainly used as reinforcing, thickening and flattening material (Lazaro et al., 2010) [8]. Nano-engineering is the techniques of manipulation of the structure at the nano scale to produce a new generation of multifunctional materials with superior mechanical performance and durability. There are several methods to synthesis Nano silica as: Sol –Gel method (Rahman et al., 2012) [9], Precipitation Method (Premaratne et al., 2013) [10], In situ emulsion polymerization (Yazdimamaghani et al., 2013) [11].

The SiO₂NPs act as a filler or reinforcement in advanced composite materials with improved thermal, mechanical, physical and chemical properties. These composites have attracted strong interest in many industries such as: coatings, flame-retardant materials, optical devices, photoresist materials Photoluminescent conducting film, pervaporation membrane and sensors, (Zou et al., 2008) [12].

Polymer nanocomposites are complex systems with two or more phases (continued and dispersed) such that the dispersed phase is characterized by at least one dimension in the nanometer scale (Ajayan et al., 2004) [13]. The properties of polymer nanocomposites combine the

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advantages of the inorganic filler material (I. e., rigidity, thermal stability) and of the organic polymer (I. e., flexibility, ductility, processability). The addition of nanofillers leads to a large increase in the interface area as compared with the ordinary composites. This large interfacial area, then creates a large volume fraction of the interfacial polymer with the properties different from the bulk polymer even at low nanofiller percentage. Composites (Conradi 2013) [14], (Krishnamoorti and Vaia 2007) [15]. Polymer/Silica Nanocomposites exhibit markedly an improved properties when compared to their traditional composites due to their nano sizes inorganic filler (Tyagi and Tyagi 2014) [16].

Nanosilica with amino and epoxide functional groups shows strong interfaces with epoxy, which suppress large aggregations and enhance resin-wettability, hence enhancing the fracture toughness of epoxy composites. Due to their commercial availability, the low-cost of the raw material and simple fabrication method, those nanocomposites have the potential for large-scale applications (Han et al., 2014) [17]. The aim of this work is to prepare Silica nanoparticles (SiO_2NPs) by a simple chemical method using rice husk and. Study different parameters that affect the Silica purity and crystalline structure such as temperature, time and acidic addition. Also studying different parameters that affect the SiO_2NPs Average diameter such as NaOH mixing time and drying time and to prepare Epoxy/ SiO_2NPs nanocomposites with various amounts of SiO_2NPs using "Direct Mixing" technique as a simple, environmentally friendly, low-cost technique. Tensile of the nanocomposite will be measured. The best wt% of SiO_2NPs in Epoxy/ SiO_2 nanocomposites will be selected.

2. Experimental Work

2.1. Materials

The rice husk (RH) was obtained from local Iraqi mill to be used as a silica source for the synthesis of nanosilica particles by chemical methods. Epoxy resin that was used as the matrix material is Sikadur 52 (Sika company, Switzerland), which is solvent-free, low viscosity injection liquid, based on high strength epoxy resin. It consists of component "A" which it's a modified epoxy resin of the diglycidether bisphenol type A which contains suitable viscosity control agents. The other component is "B" Component which is amine that blend with an epoxy resin containing suitable viscosity control agents, pigments, and accelerators.

2.2. Rice Husk Ash Preparation

The obtained rice husk contains some rice grains and sand particles. In order to separate rice grains from rice husk, the rice husk washed with tap water several times then sun dried for 24 hours. The rice husk was then burned for 3hrs at 400°C then left for 12 hours to cool down. The ash was then milled to get fine particles.

2.3. Preparation of Silica from RHA

A 100g of the cleaned RHA was refluxed with 800ml, (2N) HCl at 70°C for one hour to remove metallic impurities. After the reaction is completed, the acid was removed from the RHA by washing with distilled water many times using filter paper with pore size $0.45\mu\text{m}$. It was then dried in an electric oven at 50°C for 6 hrs. The treated RHA was then heated in an electrical box furnace a heating rate of $10^\circ\text{C} / \text{min}$ at 650°C for 3 hours to remove carbonaceous materials and then furnace cooled. High purity white silica was obtained. The rice husk ash treated at 400°C has a black color, while the heat treated rice husk ash at 650°C has a white color as shown in Figure 1. The silica was then collected for XRF and XRD analysis.

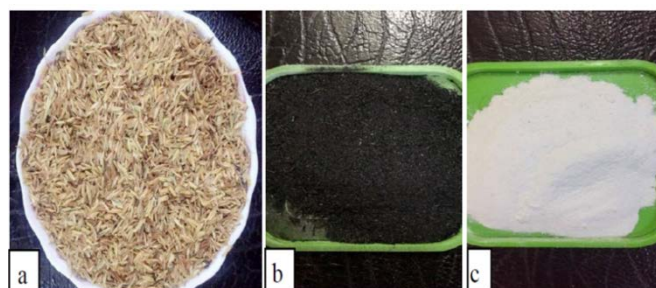


Figure 1. (a) Rice Husk (RH); (b) RHA heated at 400°C ; (c) RHA heated at 650°C for 3 hrs

2.4. Preparation of Nanosilica from Silica

A 10g of the obtained silica was refluxed with 80ml, (6N) HCl at 65°C for 3 hours. After the reaction is completed, the acid was removed from the silica by washing with distilled water several times and then vacuum filtered using filter paper with pore size $0.2\mu\text{m}$. The silica was dried in an electric oven at 50°C for 6 hrs. The silica was then magnetically stirred with 2.5N NaOH solution for 14 hours to form sodium silicate. H_2SO_4 was then added drop wise to sodium silicate solution with constant stirring until the pH equal to 8 and then a nanosilica gel was formed. The gel was then washed with double distilled water until the pH equal to 7 and then dried in an oven at 50°C for 48 hours to form a silica nanoparticles powder as shown in Figure 2. The obtained nanosilica was then collected for analysis.

2.5. Preparation of Epoxy/ SiO_2 Nanocomposite

The Epoxy/ SiO_2 nanocomposites were prepared by direct mixing of epoxy with (0.1, 0.2, 0.5, 0.75, 1, 3, 6, 9 and 12wt %) silica nanoparticles (SiO_2NPs). The mixture was firstly hand mixed by a glass rod to avoid volatilization of (SiO_2NPs) and then mixed using a mechanical mixer at 1500 (rpm) for 10 min to achieve macro-dispersion of SiO_2NPs in epoxy. Micro-dispersion of SiO_2NPs into an epoxy resin was achieved by high shear forces that formed by a high shear mixer at 5000 (rpm) for 15 min. After mixing processes had been completed, the curing agent (hardner) was added into Epoxy/ SiO_2NPs with a ratio 2:1 (resin: hardener) and mixed carefully by hand using a glass rod.

Then, the mixture was degassed for 5 minutes to remove the bubble using a vacuumed system and then cast in a silicon rubber mold (ASTM D-638 type I) as shown in Figure 2 then left for 24 hours for curing process. The cast Epoxy/SiO₂ NPs samples were then removed from the mold and heated in an oven at 40°C for 1 hour for post-cured.



Figure 2. (a) Nanosilica before drying; (b) Nanosilica after drying for 48 hours at 50

2.6. Characterization and Instruments

The X-Ray Florescence type Spectro/Ametek, (Model: XEPOS, Germany) was used to determine the elements and the oxides amount in the RHA. The diffractometer type Shimadzu (Model: XRD-6000, Japan, Cu target, 40 Kv and 30 mA, wave length 1.54060 Å) were used to characterize the sample. The scanning process is done with speed of 5°/min. The diffraction angle ranged from 10° to 80°. Fourier-transform infrared (FTIR) Spectroscopy type (IRTracer-100 / Shimadzu Co. / USA), were carried out to identify the functional groups that's present on the SiO₂NPs surface. AFM were used to characterize SiO₂NPs average diameter. The tensile properties of epoxy nanocomposite samples were measured by using (Tinius Olsen, model 50ST, Redhill, United Kingdom) with loading rate 5 mm/min at room temperature.

3. Results and Discussion

3.1. Rice Husk Ash Characterization

Extracting high purity silica from RHA was executed using chemical treatment and thermal treatment. Four RHA samples were used and treated as in Table 1. These samples were treated with or without acidic treatment at two different temperatures 650 and 750°C. The acidic treatment was done using hydrochloric acid (HCl, 37%). Then the best treatment will be selected that gave amorphous and high purity silica.

Table 1. RHA treatment

Sample No	Chemical Treatment	Thermal Treatment
1	Without acidic Treatment	650 °C for 3hrs
2	Acidic Treatment, HCl	650 °C for 3hrs
3	Without acidic Treatment	750 °C for 3hrs
4	Acidic Treatment, HCl	750 °C for 3hrs

The chemical composition was investigated using XRF Figures 3-6 as shown in Tables 2-5. From Table 2-5 the major oxide in the treated RHA is silica. Other inorganic oxides such as CaO, Fe₂O₃, Na₂O, MgO, K₂O are also present with different amount depending on the treatment. The existence of these oxides will affect the silica purity. Trace amounts of Al₂O₃, MnO and TiO₂ oxide were also detected. It is clear from Table 2-5 that acidic treated RHA followed by thermal treatment gives high purity silica which is better than chemical treatment or thermal treatment alone. The chemical treatment with HCl is very important in reducing CaO content which is the second major oxide in RHA while the thermal treatment is effective in reducing the carbounsios materials. Although, the silica purity at 650 and 750°C are close, but structurally they are different. RHA treatment at 650°C for 3hrs gives high purity amorphous silica.

Table 2. Sample 1 chemical composition of Treated RHA

Element	Sample 1 (Wt%)	Sample 2 (Wt%)	Sample 3 (Wt%)	Sample 4 (Wt%)
SiO ₂	73.52	95.5	75.2	96.3
C	4.47	1.1	4.3	0.9
CaO	12.76	0.73	15.9	0.8
Fe ₂ O ₃	5.54	0.49	1.6	0.51
Na ₂ O	1.34	0.63	0.4	0.59
MgO	0.67	0.61	0.184	0.13
K ₂ O	1.11	0.613	0.34	0.17
MnO	0.15	0.008	0.052	0.08
TiO ₂	0.33	0.082	0.1	0.08
Al ₂ O ₃	0.07	0.02	0.02	0.019
others	0.031	0.012	0.061	0.002

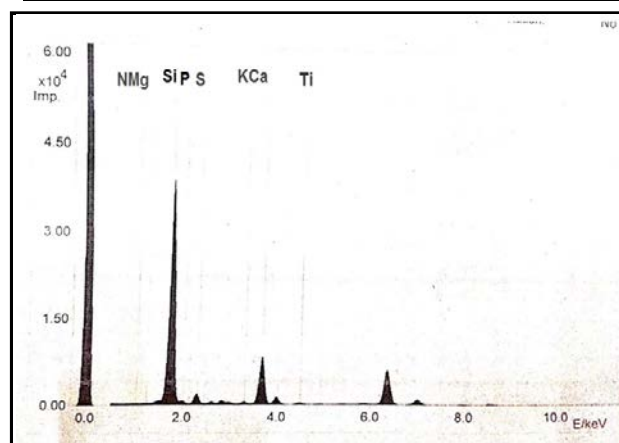


Figure 3. XRF for sample 1 (RHA thermally treated at 650°C for 3 hours)

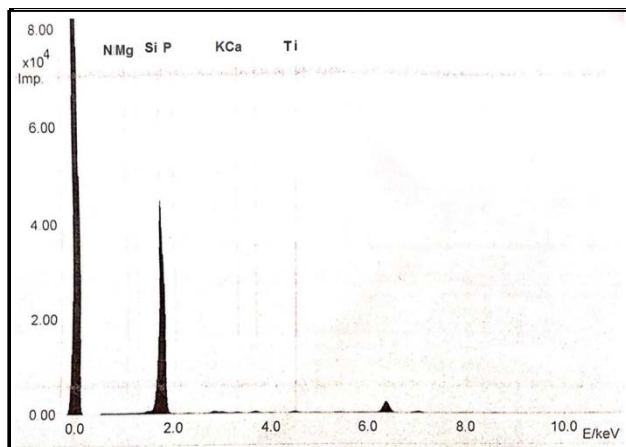


Figure 4. XRF for sample 2 (RHA acid and thermal treatment at 650°C for 3 hours)

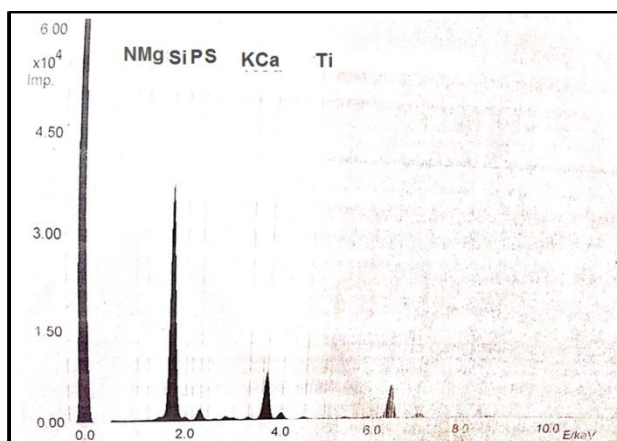


Figure 5. XRF for sample 3 (RHA thermally treated at 750°C for 3 hrs)

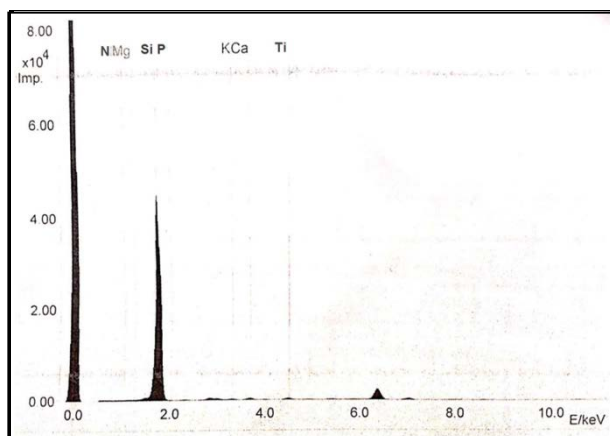


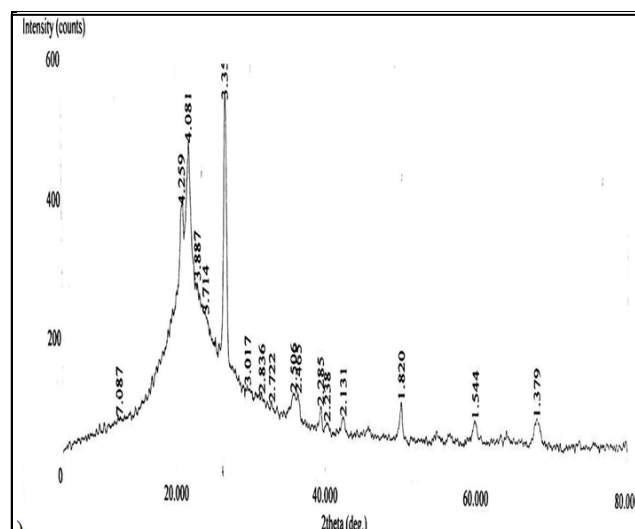
Figure 6. XRF for sample 4 (RHA acid and thermal treatment at 750°C for 3 hrs)

Figures 3 and Figure 5 show XRF of thermally treated RHA at 650 and 750°C. It is clear that silica contains large amount of metallic impurities. These impurities are reduced by acidic treatment as shown in Figures 4 and 6.

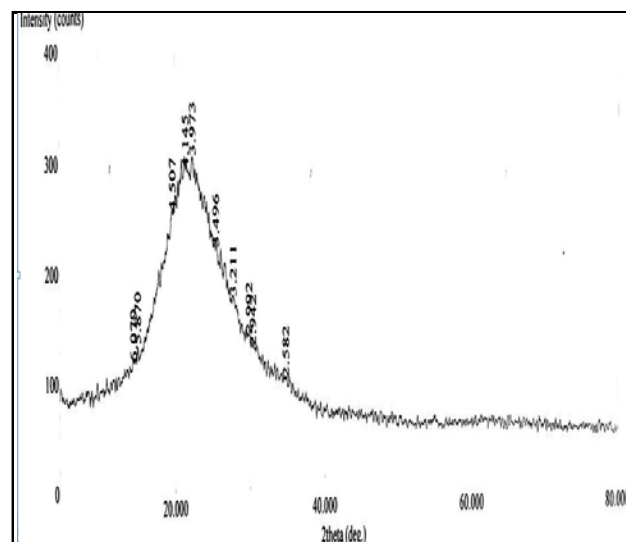
3.2. X-Ray Diffraction of RHA-Silica

X-ray diffraction of treated rice husk ash at two temperatures 650 and 750°C was taken by scanning the ash

sample for 2θ from 10° - 80° to examine the silica phases in the ash as shown in Figure 7. The XRD pattern of RH nanosilica particles treated at 650°C for 3 hours shows a broad peak at 2θ = 22° which confirms the amorphous nature of RH nanosilica. The silica in the rice husk ash firstly exists in a porous amorphous form at temperature 650°C but when the temperature increases to 750°C the silica will transform to alpha quartz which it is the only stable state of crystalline silica that found at such temperatures. The burning out temperature and time are two important factors to define whether the silica remains amorphous or become crystalline. The same results were reported by (Ramezani pour et al., (2009) [18]. They found that burning rice husks at temperature below 650°C produces amorphous crystals of rice husk ashes. The combination of burning temperature at 650°C and 60 minutes burning time seems to give the optimized solution resulting in noncrystallize RHA.



a)



b)

Figure 7. XRD of Heat Treated RH: a) at 650°C for 3 hours and b) at 750°C for 3 hours

The average crystallite size of nanoparticles was calculated using the Debye–Scherrer diffraction equation for the two temperatures

$$D(2\theta) = \frac{K\lambda}{\beta \cos \theta}$$

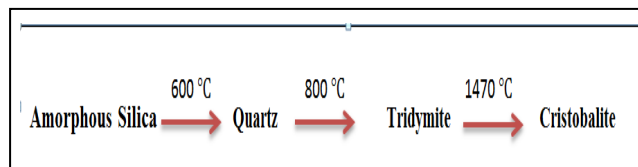
- D is the average particle size (nm)
- β is FWHM (full width at half maximum) in radian.
- θ is the Bragg angle (in degrees).
- K is a dimensionless shape factor, with a value close to unity. The shape factor has a typical value of about 0.9, but varies with the actual shape of the crystallite.

Where λ is wave length of X-Ray (0.1541 nm) for Cu-K α .

The average crystallite size for RH treated at temperature 650°C found to be 3.4(nm) while for RH treated at temperature 750°C found to be 6.5 (nm) which is higher as calculated from Figure 7. The crystallite size determines either the sample is crystalline or amorphous. For RH treated at 650°C it was found that the crystallite size was less than the RH treated at 750°C which means that the amorphous regions are predominant while for RH treated at 750°C the crystalline regions are predominant. This is in agreement with the results reported by (Ramezaniapour et al., (2009) [18]. At 650°C they obtained amorphous crystals of rice husk ashes.

The same results were reported by (Dominic et al., 2013) [5] where RHA exposed to a temperature below the crystallization temperature (700°C) of the silica it will still in amorphous form. But if it subjected to high temperature (more than 700°C) the silica cristobalite form in the rice husk will become in crystalline form. RHA Silica crystallization starts with formation of cristobalite crystals followed by tridymite crystals (Rozainee et al., 2008) [19]. The formation mechanism of Cristobalite was explained as (Gorthy and Pudukottah 1999) [20]:

At high temperatures (above 600°C) Si–O bonds will be liberated from long polymeric Chains which they are (Si–O–C / Si–O–Si) bonds. Upon further cooling, these Si–O bonds have little or no tendency to crystallize. Upon further cooling. Then more Si–O and C–O/C–C will be released when the pyrolysis temperatures are increased, leading to gradual transformation to crystalline silica (cristobalite) and crystalline carbon (graphite). (Chakraverty et al., 1988) [21] reported that the minimum silica crystallization temperature in RHA into cristobalite is 700°C. A sharp peaks at 2θ angle of 22°, Figure 7 (b), which confirm the presence of ordering crystalline structure at 700°C in the prepared RH-Silica. This peak is absent in the prepared RH-Silica which confirm the absence of ordering crystalline structure. This is in agreement with the work of (Adam et al., 2011) [7]. Transformation of Rice husk ash from purely amorphous to crystalline at temperature above 600°C was also reported by (Onojah et al., 2013) [22]. They explained. The phase transformation of rice husk ash as shown below:



3.3. Silica Nanoparticles Characterization

The extracted high purity amorphouse silica from RHA by acid treatment followed by thermal treatment at 650°C for 3 hours was used to prepare SiO₂NPs. This high purity silica was refluxed with HCl followed by basic reaction with NaOH solution then treated with (H₂SO₄) to form nanosilica gel.

3.3.1. FTIR of Silica Nanoparticles

FTIR measurements were carried out to identify the functional groups that's present on the SiO₂NPs surface. FTIR spectrophotometer (IRTracer-100, Shimadzu Co. Japan) was used and the measurements were carried out in the range from 4000-400 cm⁻¹. The FTIR spectra for silica nanoparticles show several peaks and functional groups as shown in Figure 8. The broad band at about 3429.43 cm⁻¹ can be attributed to hydroxyl groups (stretching mode) which will participate in hydrogen bonding with proton-donor and proton-acceptor sites at the polymer matrix and will contribute to a better dispersion of the nanosilica filler (Torki et al., 2012) [23]. The peak that about 1072.42 cm⁻¹ can be attributed to C=O stretching vibrations groups. A strong and broad peaks of Si–O was observed at 1161.15 cm⁻¹ (Kang et al., 2001) [24]. Also sharp peaks at 802.3 cm⁻¹ and attributes to O–Si–O is stretching and bending vibrations (Abu Bakar et al., 2016) [6].

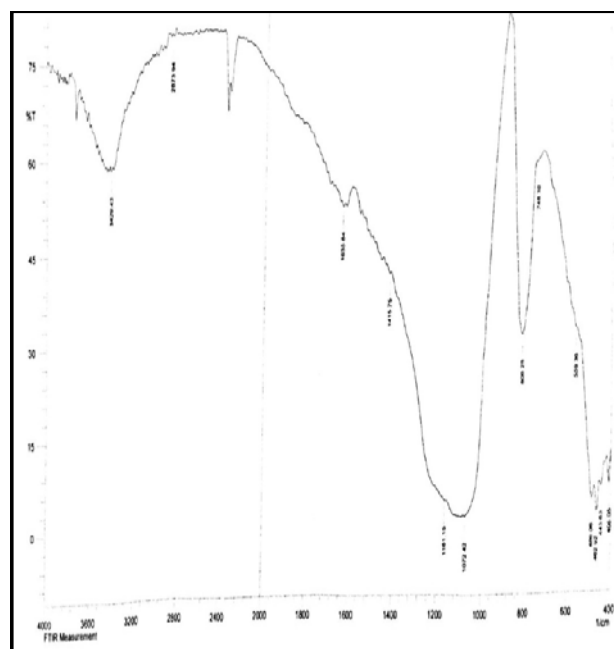


Figure 8. FTIR spectra of Silica Nanoparticles

3.3.2. AFM of Silica Nanoparticles

The diameter of chemically produced silica nanoparticles was affected by two important parameters: Silica mixing time with NaOH and SiO₂NPs drying time. NaOH (2.5 N) were mixed with the HCl reacted silica in order to form sodium silicate. The selection of 2.5 N NaOH treatment gives the highest SiO₂ content as reported by (Monshizadeh

et al., 2011) [25]. The mixing time of silica with NaOH (2.5 N) solution was selected at (10, 12, 14, 16) hours. AFM was used to characterize SiO₂NPs average diameter at each selected time as shown in Figure 9 with scanned area (1053 * 1053) nm of samples and a high-resolution (512 * 512) pixel for the topography of the nanoparticles.

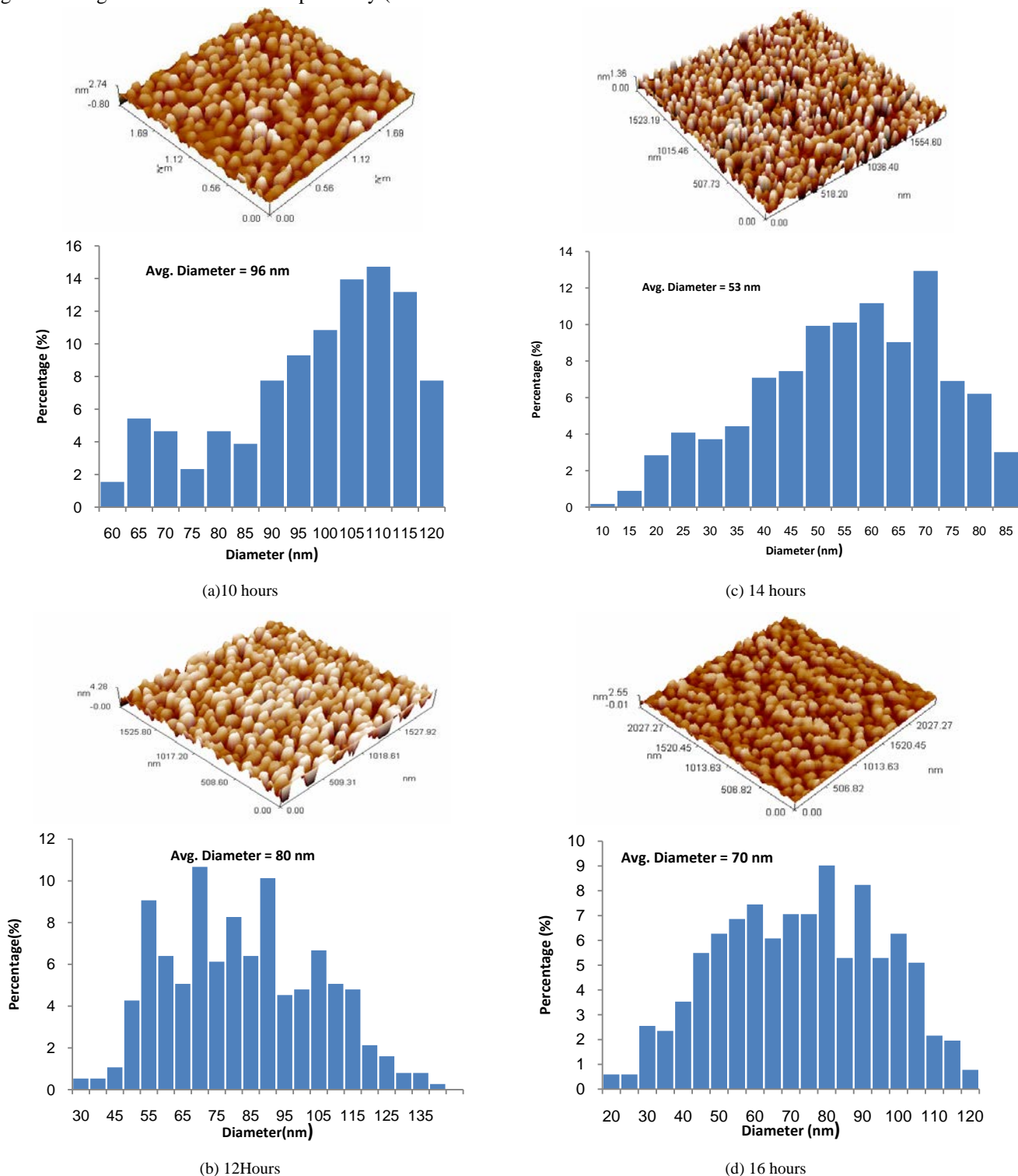


Figure 9. AFM 3-D images of SiO₂NPs size distribution at different mixing time of SiO₂NPs with NaOH: a) 10 hrs; b) 12 hrs; c) 14 hrs; d) 16 hrs

The variation of SiO₂NPs average diameter with mixing time is shown in Figure 10. The average diameter decreases with increasing mixing time with NaOH solution up to 14 hours and then start to increase with minimum diameter occurs at mixing time of 14 hours. The average diameter started to increase after 14 hours due to particle agglomeration since most of the NaOH were reacted with SiO₂NPs so no further reaction are possible. Then the best mixing time was selected as 14 hrs.

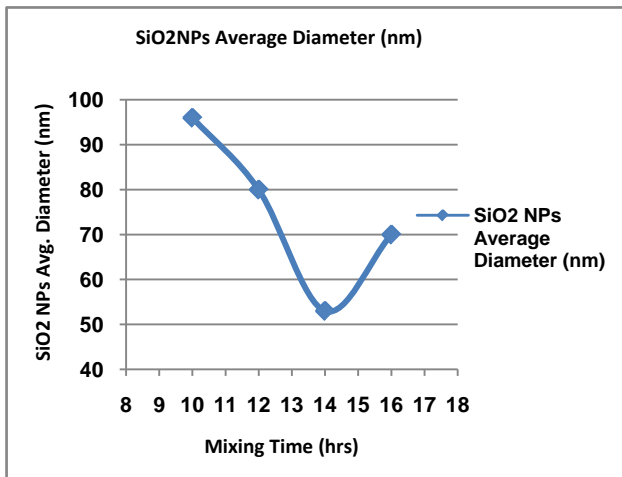
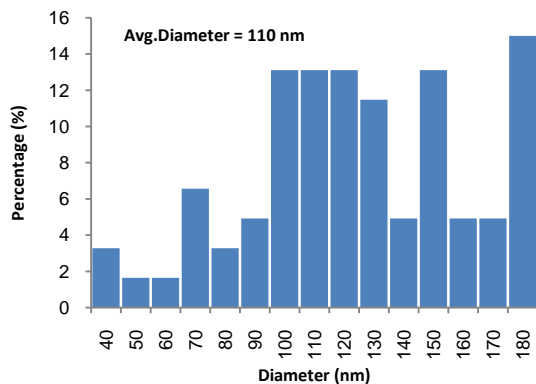
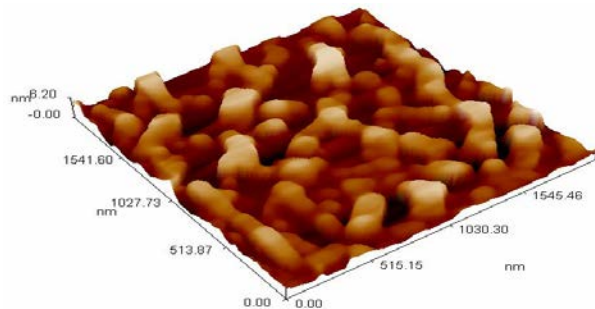
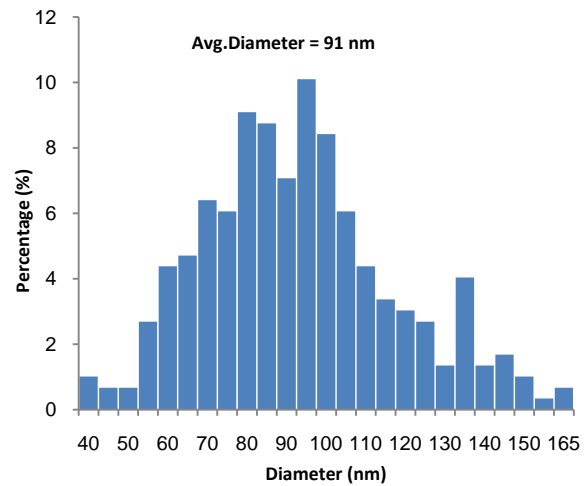
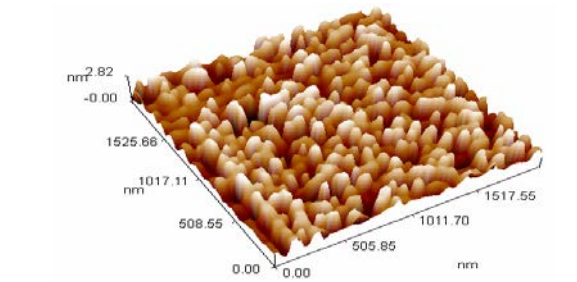


Figure 10. SiO₂NPs average diameter (nm) variation with mixing time of Silica with NaOH (2.5 N)

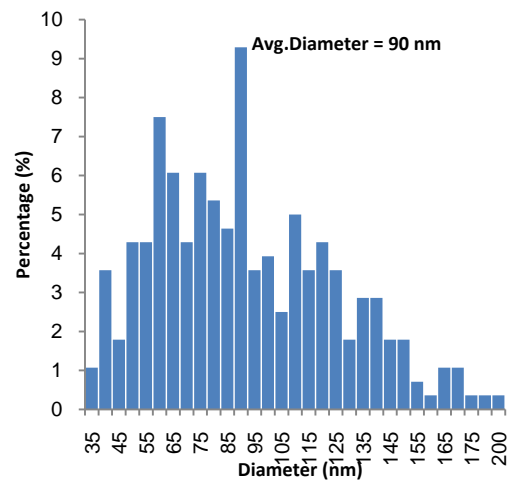
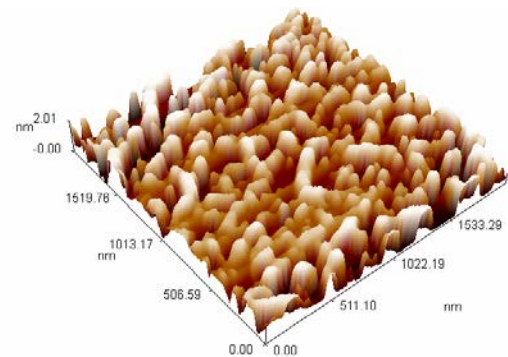
The SiO₂NPs drying time in the oven was the other parameter that was studied by taking three different drying times (24, 48 and 72) hrs. The AFM was used to characterize SiO₂NPs average diameter variation as shown Figure 11.



(a) 24 hours



(b) 48 Hours



(c) 72 hours

Figure 11. AFM 3-D images of SiO₂NPs size distribution at different drying time: a) 24 hrs; b) 48 hrs; c) 72 hrs

Figure 12 shows SiO₂NPs average diameter (nm) variation at different drying time of SiO₂NPs. The average diameter starts to decrease with increasing drying time and then reach a constant value of 90 nm at 48 hours and that's due to reduction in moisture content and grain volume shrinkage when the drying time is increased.

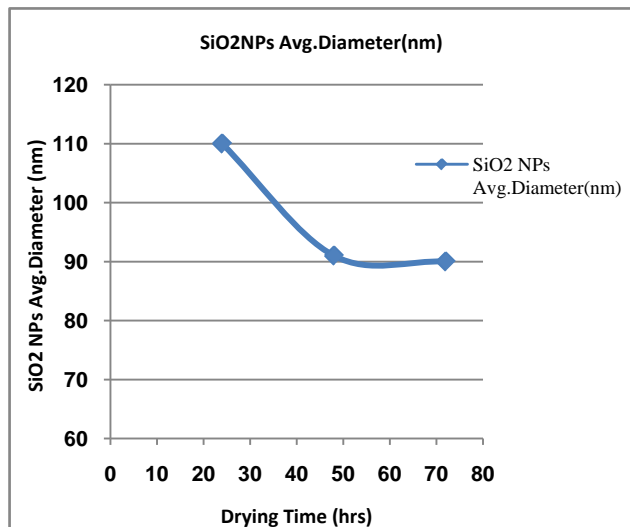


Figure 12. SiO₂NPs average diameter (nm) variation at different drying time of SiO₂NPs

3.4. Tensile Test for Epoxy/SiO₂ Nanocomposites

Epoxy/SiO₂ Nanocomposites were made by mixing Different percentage of SiO₂NPs with epoxy. Tensile test was made to choose the best SiO₂NPs percentage to be used later in Epoxy/SiO₂ Nanocomposites and compared it to the neat epoxy sample. The tensile strength of Epoxy/SiO₂ nanocomposites with SiO₂NPs (0, 0.25, 0.5, 0.75, 1, 2, 3, 6, 9, 12 and 14 wt. %) are shown in Figure 13.

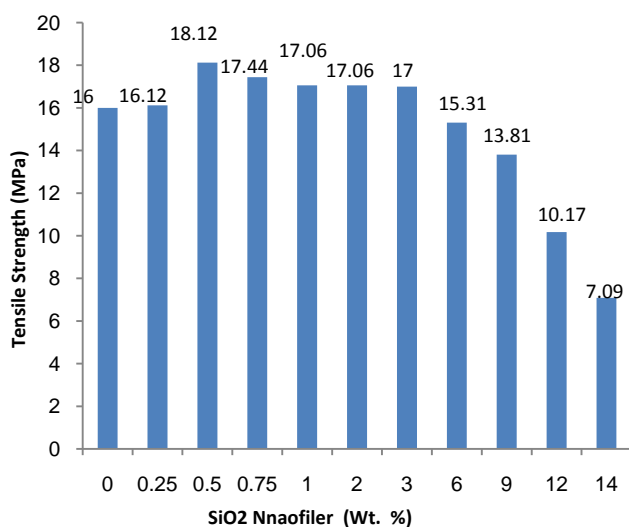


Figure 13. Tensile strength of Epoxy/SiO₂NPs nanocomposites

The maximum tensile strength of neat epoxy is (16 MPa). With the addition of SiO₂NPs of 0.25%, there is no change

in the tensile strength. The maximum tensile strength (18.12 MPa) occurs at 0.5 wt % SiO₂NPs. At SiO₂NPs of (0.75, 1, 2, 3 wt. %) the tensile strength remain almost constant at about 17 MPa. The tensile strength starts to drop sharply at SiO₂NPs greater than 3% due to the agglomeration of nanoparticles. The drop in the tensile strength is due to crack initiation in the agglomerated regions of silica nanoparticles. The same result was reported by (Eqra et al., 2015) [26] where the strength of nanocomposite decreased is significantly owing to the crack initiation in the agglomerated regions of graphene nanosheets. SiO₂NPs agglomerations reduce the surface areas of dispersed SiO₂NPs in epoxy matrix, and this will decrease the interfacial areas between the SiO₂ and epoxy matrix. The reduction in the surface area led to decreasing the mechanical properties (Kim et al., 2010) [27]. Uniform dispersion of nanofiller mainly contributes to the high tensile modulus (Moosa et al., 2017) [28]. In this work the best tensile strength was found at 0.5 wt% SiO₂NPs as a nanofillers due to better dispersion.

4. Conclusions

High Purity Silica (96% purity) with amorphous structure can be prepared from rice husk ash (RHA) by acid treatment with HCl for 1 hour, Followed by thermal treatment at 650°C for 3 hours. Amorphous Silica can be obtained at a temperature below 650°C but when the temperature increase the Silica polymorphous will change to crystalline. Silica nanoparticles could be prepare from pure amorphous Silica by a simple chemical method that involve Silica refluxing with HCl acid for 1 hour, Followed by mixing Silica with (2.5N) NaOH solution and then H₂SO₄ adding to neutralize the solution. SiO₂NPs best average particle size (53nm) Was achieved at 14 hours mixing time of Silica particles with 2.5N NaOH at best drying time (48 hours) of silica solution. SiO₂NPs that synthesis by this chemical method contain a hydroxyl group on their surface so no further functionalization is needed. Epoxy resin. Epoxy/SiO₂NPs nanocomposites were prepared by direct mixing method and the best SiO₂NPs loading was at 0.5 wt% which gives the best improvement in the ultimate tensile strength. This is due to uniform dispersion and no agglomeration.

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