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Production of Nano-Silica from Water Glass

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ABSTRACT

This research focusing on the improvement of physical properties of silica by acidic precipitation from water glass and optimize the cost of this production. One of the prominent applications of silica nanoparticles is as a filler or reinforcement in advanced composite materials. A nanosilica was characterized by many tests such as particle size analyzing, XRD, FTIR, TEM, SEM and EDS. The results show that morphous SiO2 particles with an average size less than 100 nm, with 100% purity with very fine white powder, and density of 0.2817 g/ml.

Keywords: Water glass, Precipitated method, amorphous nano-silica, Particle size.

INTRODUCTION

Silica gel is extensively used as a catalyst and as an adsorbing agent. It adsorbs moisture from the air in the enclosed space and stop rusting .To prevent rusting during shipment or storage, machine parts and other equipment are packed in moisture-proof containers, together with small porous bag containing silica gel [1]. Silica has been employed in material sciences and engineering for many years, which is a major and natural component of sand and glass. Due to the variety of available chemical and physical modifications that are available, It is a versatile material [2].

The white color of silica and their fine primary particle size, give rise to a remarkably high reinforcing efficiency compared with other white fillers in the production of light-colored products. Silica is considered to be a highly polar and reactive filler, because it has a number of surface silanol groups (Si-OH), on the surface which brings about strong filler–filler interactions [3].

After incorporating filler to polymers, The degree of reinforcement or improvement of mechanical behavior depends on strong bonding at the matrix–particle interface [4]. To improve the tear strength and heat aging resistance in a wide variety of manufactured rubber goods, including hoses, conveyor and power transmission belts, bumper pads and rubber rollsis, motor and dock mounts, used silica as filler in matrix. It is also used in shoe soles for improving the resistance to wear and tearing [5]. Decreasing the particle size to the nano-size dimension influences the macroscopic properties of the polymer [6,7]. silica is used in refractories, lumber processing, cements, textile, automobiles and passive fire protection [8]. Yasushige Mori and Yuusuke Fukumoto using electrostatic atomization to produce fine silica particles. With uniformly sized droplets. Diluting the water glass with pure water resulted in silica particles having a small mean volume diameter and a narrow particle size distribution [9]. Sang-Wook Ui, et al. Used low cost sodium silicate(waterglass) instead of high cost alkoxide or organo-metallic compound to synthesize nano-sized silica powders with very high specific surface area and low density. Showing as the molar ratio of HCl:H_2O

increased the particle size decreased [10]. A. Lázaro And H. J. H. Brouwers produced nano-silica in large quantities, for low prices by sustainable method. It may replace cement in the mix, in concrete to improve strength and durability. Due to the small particle size and the large surface area (up to 30 m_2/g), it acts both as a microfiller that reduces the porosity and as a pozzolan, it improves the properties in hardened state [11]. L. P. Singh, et.al prepared Spherical and amorphous silica nanoparticles by the hydrolysis reaction of tetraethoxyorthosilane (TEOS) in ethanol using sol-gel method. observed that the addition of small quantity of n- SiO_2 significantly improves the morphology and mineralogy of the cementitious materials. imparting higher strength and durability to cementitious materials [12]. S. Musić et al Produced amorphous SiO2 particles by the precipitation by neutralization of sodium silicate solution (water glass) with H_2SO_4 solution. the results show amorphous SiO2 particles with specific surface area up to 130 m2g-1, with partical size ~ 15 to ~ 30 nm and did not show a contamination of precipitated SiO_2 with sulfate or other ions [13].

In this research, Preparation of pure silica from water glass of chemical composition (Na_2O. $XSiO_2$) for using as a filler to improve the physical properties of rubber recipe. particle size analyzer, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Transmission electron microscopy (TEM), scanning electron microscope (SEM) , Energy-dispersive x-ray spectroscopy (EDS) were carried on.

EXPERIMENTAL

Preparation of silica

Silica prepared from water glass with a chemical composition of 9.01% Sodium oxide (wt % Na_2O), 29.49% Silica, and 61.5% H_2O weight percent, with specific gravity at 20 C̊ is 1.408 and viscosity at 20 C̊ is 330 centipoise by slowly addition of hydrochloric acid until silica deposited and the solution become acidic. The complete process showed in chart1.

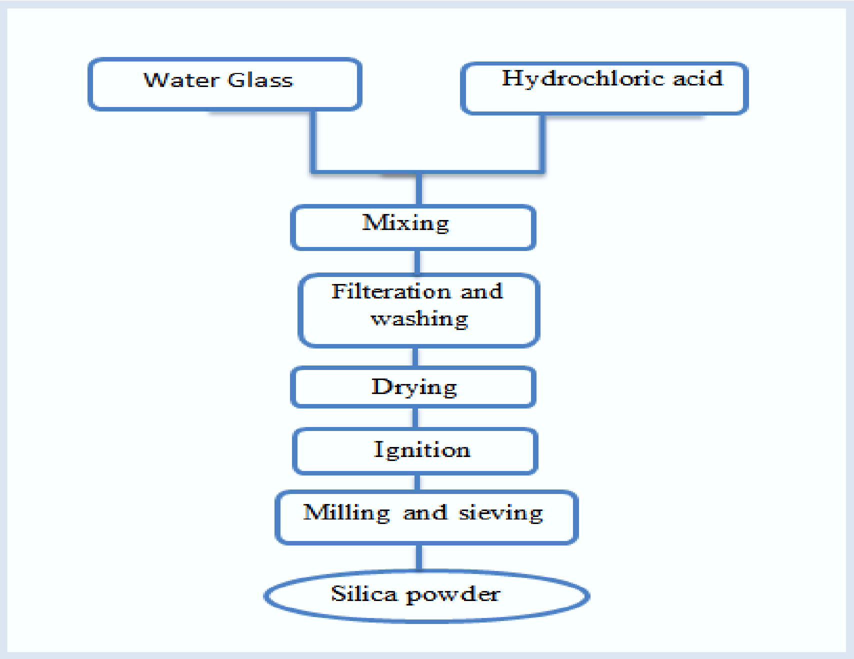

Chart 1. Silica preparation

Characterizations

Transmission Electron Microscopy (TEM):

The morphologies of the powder were measured by using transmission electron microscopy type (EM 208, phlilps Holland). Samples of TEM analysis were prepared by placed on a copper grid coated with carbon after dispersing by ultrasound for few minute.

Scanning Electron Microscopy (Sem):

The surface morphology of powder characterized by using SEM type (Tescan,Chic). The surface of the sample was coated with gold. Then tested at different magnification.

Energy Dispersive X-Ray Spectroscopy (Eds):

Energy-dispersive x-ray spectroscopy (EDS) is important for microanalysis, particularly for heavier elements, for identifying the chemical purity of silica. Which is carried on 3746 X of magnification with accelerating voltage 20.00 kV.

Particle Size Analyzer:

The average sizes and distribution of powders were obtained by Better size2000 laser particle size analyzer.

X-Ray Diffraction Analysis (Xrd):

The phase of powders studied with XRD analysis type (XRD-6000, Shimadzu, Japan) in the bragg angle (2Θ) range from 5° to 50°, using Cu Kα source, with a wave length of 1.54060 Å.

Fourier Transform Infrared Spectroscopy (FTIR):

FTIR spectrum was carried on using 8400S - FTIR-Perkin-Elmer spectrophotometer. The Sample of FTIR analysis in the powder form carried on KBr as a tablet.

RESULTS AND DISCUSSION

The Purity of prepared silica test

A qultitive test is carried on by addition of hydrofluoric acid to the prepared silica. the results show no residue remain after the test, which indicate high purity ~100% silica obtained. as the equation shows:

$$SiO_2 + 4HF \longrightarrow SiF_4 + 2H_2O$$

Transmission Electron Microscopy (TEM)

The TEM micrographs of prepared silica particles is observed that the average particles size in all photo in **Figure 1**, with approximate sizes less than 100 nm. As shown it is easily to agglomerate due to the silica particles have irregular spherical shapes which have high surface area.

Scanning Electron Microscopy (SEM)

Figure 2 shows different magnifications of silica surface that microparticles and nanoparticles were measured. The results of this analyses show that the diameters of nanoparticles in the range of 25-43 nm and spherical, highly agglomaration are observed by this examination. This agree with the results of Musić, et.al [2].

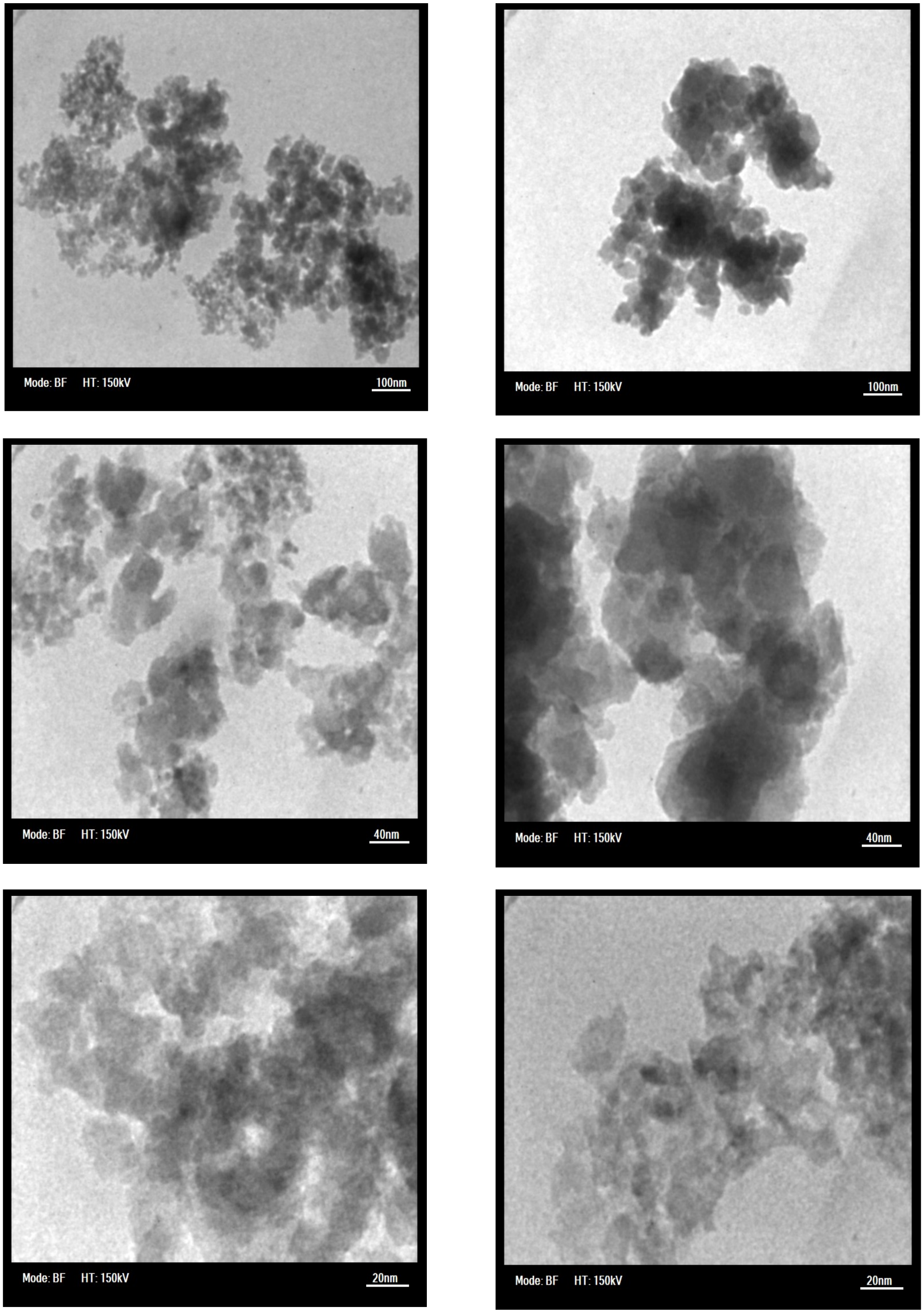

Fig. 1. TEM images of precipitated nanosilica particles

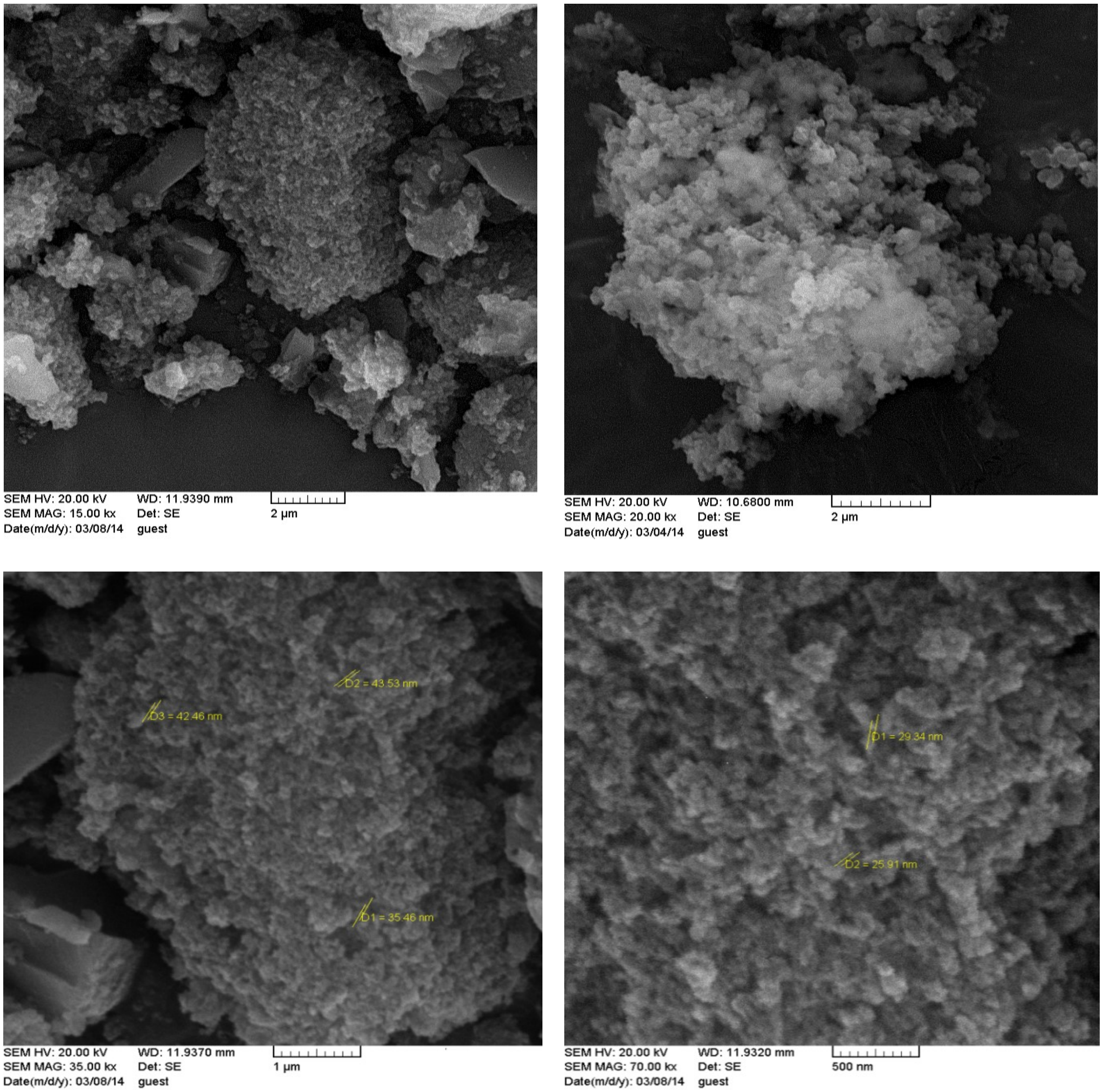

Fig. 2.SEM micrographs images of precipitated SiO_2

Particle Size Analyzer

From **figure 3** the distribution of particle size of prepared silica shows that the silica powder with particle size range of (0.7 µm - 105µm) with maximum% size (43µm). The results of this text appears that particle size of silica in micro-size instead of nano-size due to the high surface area of fine product tends to agglomerate, beside that the test require a distilled water, which enhance conglomerate particles.

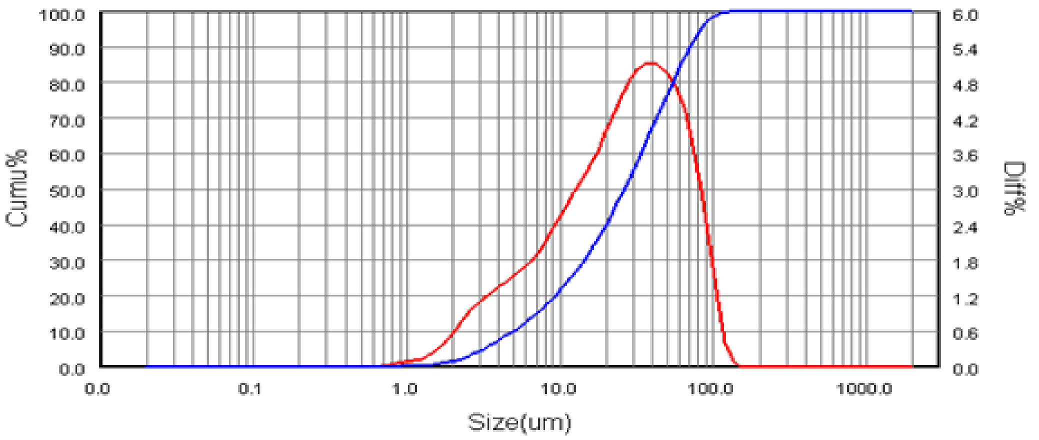

Fig.3. shows particle size analyzer of nano silica

Energy Dispersive X-Ray Spectroscopy (EDS)

Figure 4 shows that spectrum of amorphous SiO_2 particles. The purity of silica is 100% and element analysis agree with table 1. The result of this test agree with **Musić, et.al** results [13].

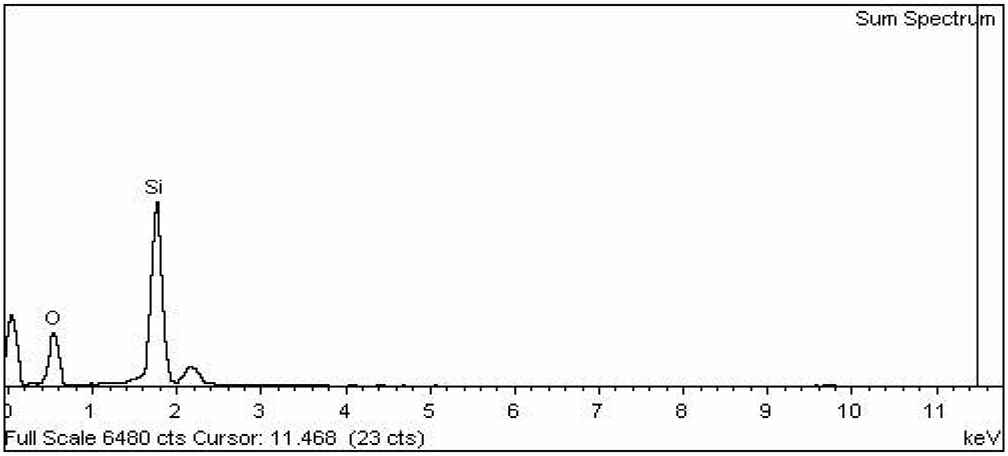

Fig. 4. EDS spectrum of precipitated SiO_2

Table 1. Elementl analysis of SiO_2

Elament	*App*	*Intensity*	*Weight%*	*Weight%*	*Atomic%*
	Conc.	*Corrn.*		*sigma*	
O K $_{\text{orbital}}$	98.40	0.9626	54.86	0.67	68.08
Si K $_{\text{orbital}}$	82.10	0.9757	45.14	0.67	31.92
Totals			100.00		

X-Ray Diffraction Analysis (XRD)

From the X-ray test of nano silica powder at diffracted angle (5° to 50°), a weak crystalline peak appeared which indicate semi-amorphous structure at ($2\theta = 23°$) as figure (5a). Also, apeak for sodium chloried (by product) appeared with very low concentration at (2θ =

31.569, 2θ= 45.174) result from neutralization of water glass with hydrochloric acid and this disappear with extra washing as figure (5b).This indicates that amorphous material is prepared which agree with the results of Sang-Wook Ui, et al [10].

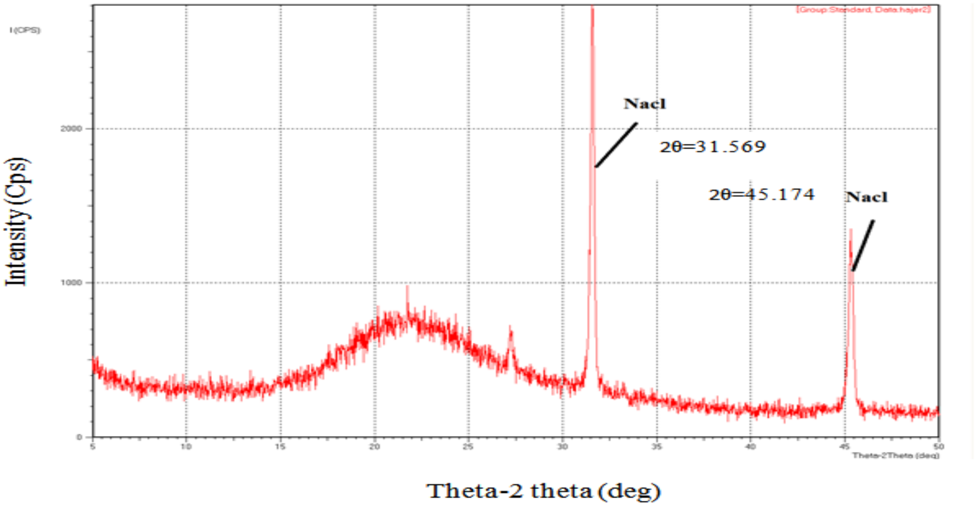

Fig.(5a). X-Ray Diffraction Analysis of precipitated SiO_2 (before washing)

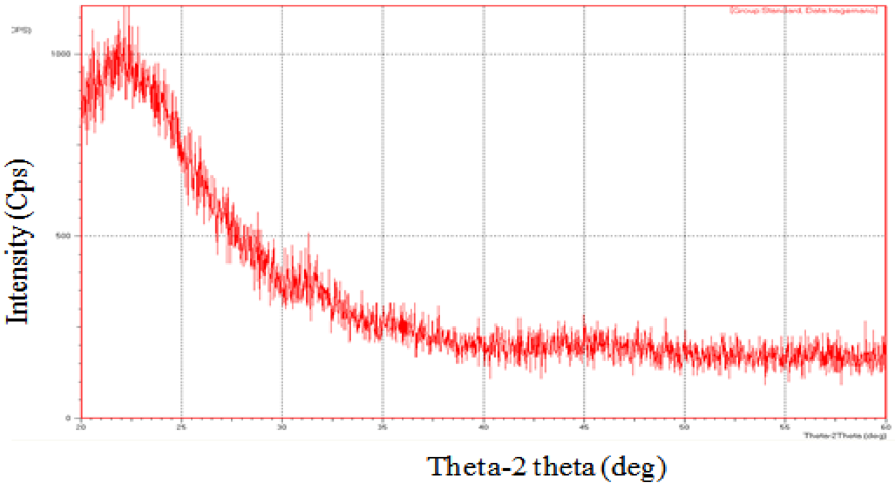

Fig.(5b). X-Ray Diffraction Analysis of precipitated SiO_2 (after extra washing)

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectrum of amorphous silica in Figure (6a) shows several absorption bands. The spectrum is matching with the standerd spectrum of amorphous silica and in a good agreement with Musić, et.al results as figure (6b) [13]. The IR bands at 3427 cm-1 and 1631 cm-1 is the intensity of water peak. The band at 2352 cm-1 of Si-OH groups for silica powder. The Si-O-Si bond stretching vibrations of the strong IR band 1111 cm-1 and 1184 cm-1. the (Si-OH/Si-O) groups apear at 952.48 cm-1.

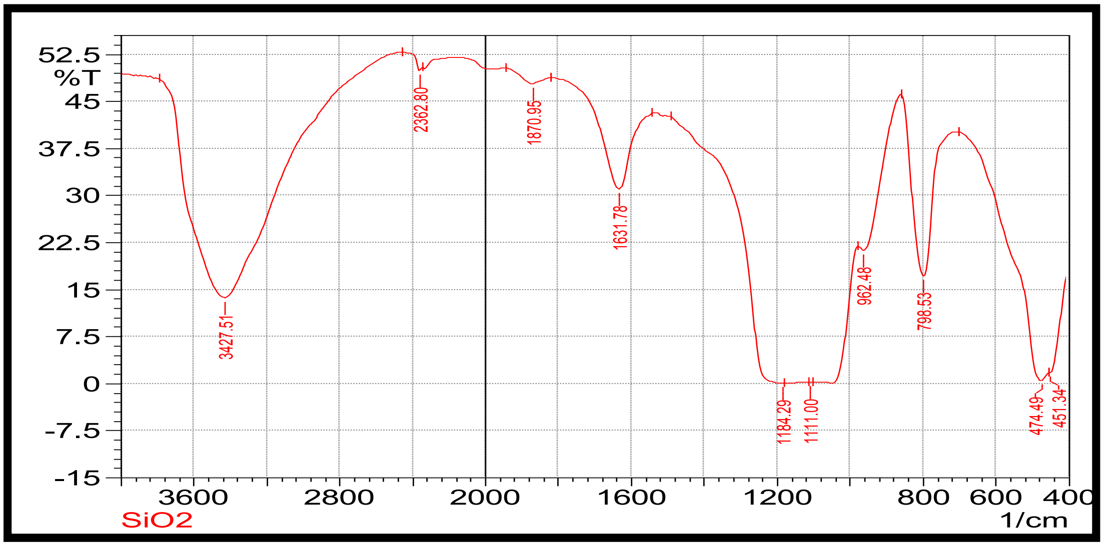

Fig.6a. FTIR spectrum of precipitated SiO_2

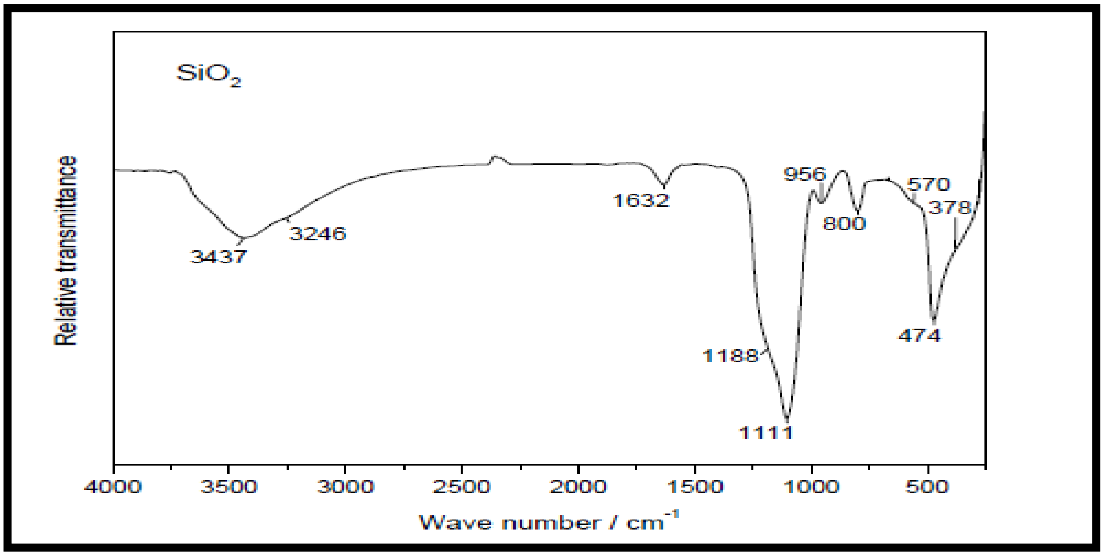

Fig. 6b. FTIR spectrum of precipitated SiO_2 [13]

CONCLUSION

To improve the physical properties and optimize the cost of the compound. a nanoparticle silica is prepared from water glass for using as a filler in nanocomposite. The results show that irregular spherical shapes of silica with particle size ranges less than 100 nm ~ 25-43 nm investigated by TEM and SEM. semi-amorphous structure at ($2\theta = 23^\circ$) characterized by X-ray diffraction. The FTIR spectra shows several characteristic peaks similar to that of amorphous silica. A nano-silica powder of 0.2817g/ml density with a purity of 100% were obtained from low-cost raw materials.

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