A FACILE SYNTHESIS AND CHARACTERIZATION OF AMORPHOUS SILICA (SiO_2) BY THERMAL TREATMENT ROUTE

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Abstract- A facile thermal treatment route was for the first time used to successfully synthesizeamorphous SiO2 nanoparticles. Various techniques were employed to study the structural, phase and elemental composition of the material at different calcination temperature of 500 and 550^oC. The XRD analysis confirms the formation of SiO_2 to be in an amorphous state and further revealed that the sample remained in amorphous state even at 550^oC. While the FTIR shows that the calcination has enable the removal organic source from PVP and formation of amorphous SiO_2 nanoparticles. The average particle size of the material estimated from the TEM images shows that the particle were <10nm. The EDX analysis has confirm that there was no loss of element in the process therefore thermal treatment method is effect for the synthesis of amorphous silica nanoparticles.

Keywords- Amorphous Silica, Calcination,

I. INTRODUCTION

Nanoscience and technology has tremendously contributed to emergence of smart functional materials, devices, as well as systems through nanometre scale manipulation of material. At present, there is much attention on nanomaterial dues their surface effect (that is large surface-to- volume ratio) and quantum confinement effect (size dependent properties). The physical and chemical properties of nanomaterial are affected by these factors, which makes it to differ from those of their molecular and bulk properties. In general terms, nanoparticles with zero dimensional nanostructures are classified based on their based on their compositions, such as metal oxide, transition metals, noble metal, magnetic metals, semiconductors or quantum dots. In the past decade, silicon oxides material, commonly refers to as silica, in their nano form have become vital materials in various field and specialization due the new physicochemical attributes which do not appear in the corresponding bulk materials. SiO_2 nanoparticles are have been studied as good host matrix for dopant such as transition metals ions and rare earth (Koao, Swart, Obed, & Dejene, 2011). SiO_2 also have wide applications and uses such as dielectric materials, elastomeric materials (such as in rubber industry), in flat panel displays, sensors, filters for exhaust gases, adsorbents, separations, biomedicine, sensors, drug delivery systems, oil-spill clean-up, heterogeneous catalysts in various chemical reactions and food materials.(Jesionowski, 2008; Pajonk, 1991; Naik & Sokolov, 2007; Yang, Wang, & Yang, 2008; Rao, Hegde, & Hirashima, 2007;Tang, Xu, Wu, & Sun, 2006; BernArdos, Kourimska, & others, 2013)

So many procedures and methods have been reported on the synthesis of nanoparticles of Silica with the aim of improving the chemical and physical features. Such methods include sol-gel technique(Nandanwar et al., 2015), carbothermal (Du and Zheng., 2011), hydrolysis reaction method(Luo et al.,2012), micro emulsion method (Finnie et al., 2007) combustion method (Hong et al., 2009)etc. Despite the fact that all effort has been put in place to prepare SiO_2 by the previous researchers, however, it is difficult to apply on large scale production due to the complicated procedure, high temperature involved, and longer period of reaction, toxic chemical reagent and by product release to the environment at the end of the experiments.

The present study synthesized amorphous SiO_2 nanoparticles by thermal treatment method from a solution containing silicon tetraacetate, PVP Poly (vinyl pyrrolidone) and deionizes water only (Al-Hada, et al., 2014; Goodarz et al., 2012). The influence of calcination temperature on the formation of SiO_2 Nanoparticles have been studied and discussed in details.

II. EXPERIMENTAL

MATERIALS

PVP Poly (vinyl pyrrolidone) 2900 molecular weight was used as capping agent which reduce agglomeration and stabilize the nanoparticle. Silicon tetraacete reagent 99% purity was purchased from sigma Aldrich and was used without further purification. Deionize water was used as a solvent.

PROCEDURE

A solution of PVP was made by dissolving 3g of PVP in 100ml and continuously stirred using magnetic stirrer for 2 hours, until no precipitate formed. Later 0.2mmol of silicon tetraacetate was added and stirred continuously for 2 hours, little particles were

observed in the solution the PH of the solution measured to be 3.4. The solution was poured into a clean petri dish and dried in an oven for 24 hour at 80°C. The resulting solid gel was grinded in a sterilized mortal into powder form. The powder was placed into crucible boat and calcinated in a box furnace at 500and550°C, with constant holding time of 3hours to decompose PVP and produce amorphous SiO_2.

III. RESULT AND DISCUSSION

THERMOGRAVIMETRIC ANALYSIS (TGA-DTG)

The appropriate temperature to start the calcination process was determined by thermogravimetric analysis measurement and its derivative (TGA-DTA). Figure 1 indicates the weight loss percentage as a function of temperature of the dried sample before the calcination. The sample shows two distinct decomposition stages. The first was weight loss at 80°C which was attributed to the moisture already contained in the sample. The second stage of weight loss was observed at a temperature of 433°C which indicates that most PVP has been decomposed. There is no significant weight loss the moment the temperature reaches 485°C; this is due to complete decomposition of PVP there by turning into carbonaceous product such that only amorphous silica (SiO_2) remains as final residue (Al-Hada, et al.,2014).

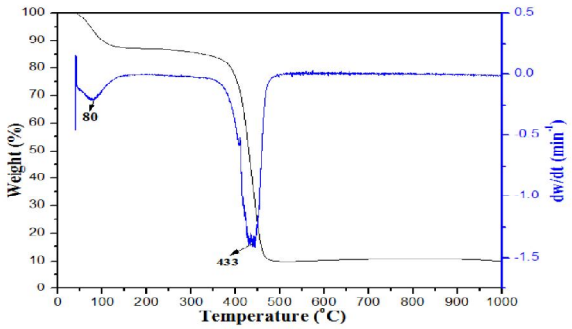

Figure 1: The thermogravimetry (TG) and thermogravimetry derivative (DTG) curves for PVP used to determine the minimum calcination temperature at heating rate of 10°C/min

Phase and elemental analysis.

FTIR Spectroscopy aid in analyzing multi component and gives the related information of the material phase composition and the nature of interaction existing in various kinds of polymers. In this study, FTIR analysis was used to determine the calcination temperature at which the formation of Silica (SiO_2) occurred without any trace of organic agent been detected. The organic and inorganic behaviour of the sample before and after calcination within the range of 280-4000cm^{-1} has been shown by the FTIR spectra in figure 2. The PVP spectra presents the band 1431cm^{-1} C-H bending vibrations originated from methylene group is observed. The band at 1649.cm^{-1} was due to C=C stretching and while the one at 689 was due C-N bending. The two different bands at 2951cm^{-1} and 3457cm^{-1} observed is attributed to C-H stretching and N-H stretching vibration respectively (Gene et al., 2014). While the sample was calcinated at 500°C for 3hrs the broads bands due to the organic material (PVP) has been totally decomposed and disappeared; only vibrational spectra of pure Silica (SiO_2) observed. This is evident in (Figure 2) the spectra at 445 cm^{-1} band correspond to O-Si-O bending vibration. Si-O-Si symmetric stretching vibration were observed at 798$cm^{1}$and O-Si-O asymmetric stretching was observed at 1062cm^{-1}(Akl et al., 2013).

The elemental analysis of SiO_2 nanoparticles was tested by Energy-dispersive X-ray spectroscopy (EDX). The fundamental principle is such that each particular element possesses a unique atomic structure allowing unique set of peaks on its X-ray spectrum. The purpose of carrying out the EDX analysis here also was to enable us confirm Elemental composition of the constituent atoms and figure out if there are any foreign impurity atoms. Figure 3 shows the EDX spectrum and SiO_2 calcined at 500°C. The corresponding peaks of Si, and O_2 were observed in the sample which confirms the formation of pure SiO_2. In conclusion, the EDX analysis confirms the formation of SiO_2 and also proves that the thermal treatment technique is effective, as there were no loss of element was observed in the process.

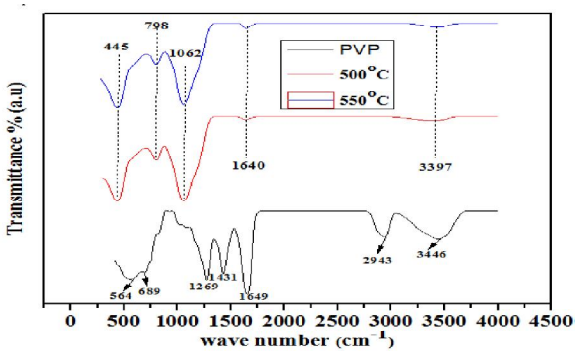

Fig 2:FTIR spectra of PVP and silica (a) PVP before calcination, 500 and 55°C

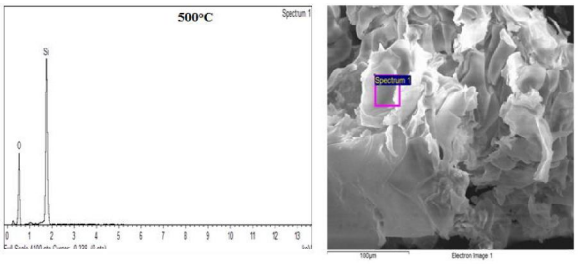

Figure 3: EDX spectra of SiO_2 calcined at 500°C

IV. STRUCTURAL ANALYSIS

Figure 4 presents the XRD patterns of the sample before and after the calcination. The broad spectrum shownby the precursor before calcination implies that the sample was in amorphousstate even though it

seems to be dried crystal (Naseri et al., 2011)While for the sample calcined at 500 and 550°C, the spectrum yet exhibits no sharper or narrower diffraction, which implies that the material were all in amorphous state (Rida and Harb 2014), which corresponds to Cristobalite beta high with reference code number 98-006-1840

TEM Images Silica (SiO_2) nanoparticles

Transmission electron microscopy (TEM) analysis was used to study the material calcined at 500°C and 550°C on the shape, size and particles distribution of the prepared SiO_2 nanoparticles. The TEM images were obtained using Hitachi H-7100 at accelerating voltage of 50kV. The distribution of the particles and average size of the nanoparticles determined using image J software. In this work, the particles size SiO_2 found to be 6.28nm - 6.51nm as the temperature varies from 500 to 550°C figure 5. The images seems to be quantum dot (<10nm) at all calcination temperatures and the smallest particle size was obtained at the temperature of 500°C and the particles size reluctantly increases with the increase in temperature. This implies that thermal treatment method was for the first time used to prepare amorphous silica nanoparticles with less than 10nm particle size.

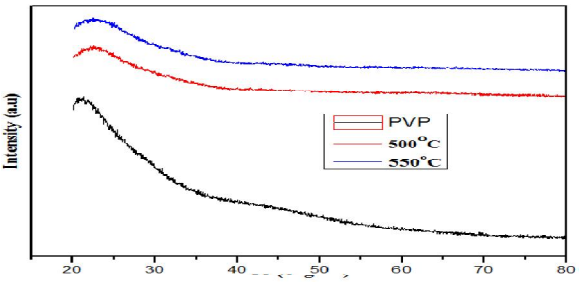

Fig 4: XRD patterns of silica with different calcination temperature PVP before calcination,500 and 550°C

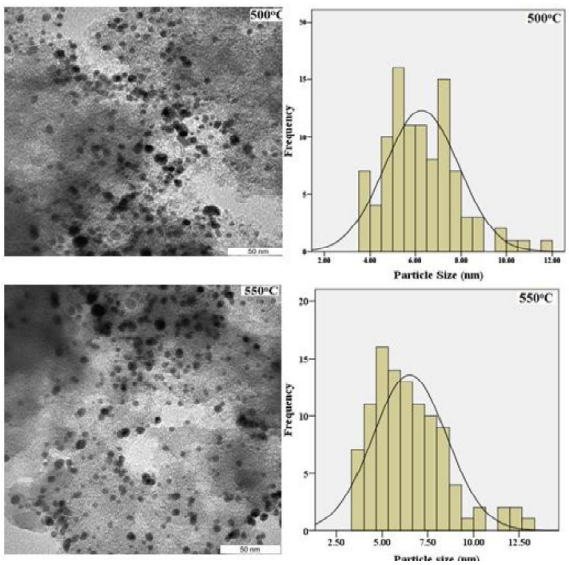

Figure 5: TEM Images Silica (SiO_2) nanoparticles calcined at 500 and 500°C.

CONCLUSION

SiO_2 nanoparticles have been synthesized successfully for the first time by thermal treatment method using only silicon tetraacetate as Si precursor, PVP as capping and deionized water as solvent. The calcination has help in successful removal of organic material, thus leaving a residue of pure SiO_2 nanoparticles.

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