Synthesis and Characterization of Nanosilica from Rice Husk Ash Prepared by Precipitation Method

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ABSTRACT

Nanosilica was prepared by precipitation method and characterized by various analytical techniques. Transmission electron micrographs show nanosilica particles are in the agglomerate form with dimension of 50 nm. The particle shape was found to be uniform and agglomerated. The diffraction pattern of the particles shows a diffuse ring pattern with indicative of amorphous phase. X-ray diffractograms show that the obtained product is amorphous nanosilica and the specific surface area is 656 m^2g^{-1}. Subsequently, the infrared spectra data supports the presence of hydrogen bonded silanol group and siloxane groups in silica. In this study, nanosilica was introduced in cement paste. From the experimental results, it was found that the incorporation of the nanosilica in the cement paste increase the compressive strength when compared withthat of the portland cemen paste.

Key words: Nanosilica, Precipitation, Cement paste, Compressive strength

INTRODUCTION

Rice husk ash (RHA) is one of the most silica rich raw materials containing about 90-98% silica after a complete combustion. The selection of ash is important as the quality of ash determines the total amount as well as quality of silica recoverable. The initial step is extraction of silica from ash as sodium silicate using caustic soda (Kalapathy et al., 2000).

The reaction is as follows.

$SiO_2 + NaOH \rightarrow Na_2SiO_3 + H_2O$

(ash) (sodium hydroxide) (sodium silicate) (water)

In the second step of the process, silica is precipitated from sodium silicate using sulphuric acid

The reaction is as follows.

$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$

(sodium silicate) (sulphuric acid) (silica) (sodium sulphate) (water)

Since this technology is developed from the scale range of 10^{-9} m or 10 A° such as nanosilica has been added to polymer to increase strength, flexibility and aging resistance. Nanosilica which has amorphous structure also can improve the strength of the concrete (Jal et al., 2004). In this paper, preparation and characterization of pure silica from rice husk and nanosilica powder from pure silica were presented. The nanosilica powder which was introduced in cement paste was then investigated.

MATERIAL AND METHODS

Material

Rice husk ash was obtained from a brick factory and was washed by water and then burned at 700°C for 3 and 6 h. Quantitative chemical analyses of RHA were accomplished by X-ray fluorescence (Horiba Mesa-500w).

Synthesis pure silica powder from rice husk ash.

Ten grams of RHA samples were stirred in 80 ml of 2.0, 2.5 and 3.0 N sodium hydroxide solutions, respectively. RHA was heated in a covered 250 ml Erlenmeyer flask for 3 h. The solution was filtered and the residue was washed with 20 ml boiling water. The filtrate was allowed to cool down to room temperature and then added 5 N H_2SO_4 with constant stirring until pH 2 and then added NH_4OH until pH 8.5 left for 3.5 h. The filtrate was dried at 120°C for 12 h and pure silica powder was investigated by fourier transform infrared (FTIR- model tenser 27).

Preparation of nanosilica powder from pure silica powder.

Pure silica was extracted by refluxing with 6 N HCl for 4 h and then washed repeatedly using deionised water to make it acid free. It was then dissolved in 2 N NaOH by continuous stirring for 10 h on a magnetic stirrer and then concentrated H_2SO_4 was added to adjust pH in the range of 7.5-8.5. The precipitated silica was washed repeatedly with warm deionised water till the filtrate becomes completely alkali free. After the washing process silica powder was dried at 50°C for 48 h in the oven. Nanosilica powder was examined by TEM (Jeol, JEM2010), XRD (Megix-Pro Philips), specific surface area (Quanta Chrome Autosorp-1) and FTIR (Model tenser 27).

Preparation of paste specimen.

Nanosilica was used to replace Portland cement by direct weight 2, 4, 6 and 10 wt%. All the paste of 50×50×50 mm^3 cubes were cast using a water to binder ratio of 0.5 by weight. The samples were demoulded and cured in water at 20°C for specified period (7, 28 days). After curing, cement paste specimens were tested for compressive strength. The fracture surface (interface) of cement paste was examined using a SEM (Jeol JSM-5910).

RESULTS AND DISCUSSION

The rice husk ash (RHA) sample after completely burned at 700°C for 6 h presented the highest amount of silica content as shown in Table 1. The sample which was heated up to 700°C for 6 h generated the percentage yield up to 92.76 % due to some of organic matter was burnt out as shown in Table 2. The optimum concentration of sodium hydroxide is 2.5 N.

Table 1. Chemical composition of RHA before after burning out at 700°C for 3 and 6 h.

Components expressed as oxides	RHA as-received	RHA after burning at 700°C for 3 h.	RHA after burning at 700°C for 6 h.
SiO_2	96.51	97.86	98.14
Al_2O_3	0.15	-	-
Fe_2O_3	0.17	0.07	0.07
CaO	0.66	0.52	0.46
ZrO	0.05	0.01	0.03
MgO	0.77	0.29	-
P_2O_3	0.21	-	-
Mn_2O_3	0.21	0.16	0.16
SO_3	0.04	0.07	0.07
LOI	-	0.01	0.02
SUMMATION	100	100	100

Table 2. Effect of concentration of sodium hydroxide on the percentage yield of pure silica.

Concentration of sodium hydroxide (Normal)	Percentage yield of pure silica (%)
2.0	64.22
2.5	90.34
3.0	91.11

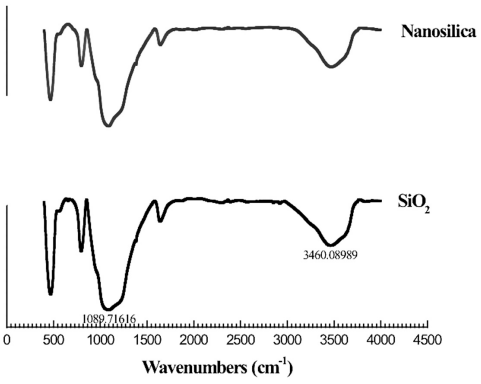


Figure 1. Fourier transform infrared spectra of pure silica product.

The major chemical components in pure silica are shown in Figure 1. The broad band between 2800 and 3750 cm^{-1} is due to silinol OH groups and adsorbed water. The predominant absorbance peak at 1320 cm^{-1} is due to siloxane bonds (Si-O-Si). The peaks between 1200 and 700 cm^{-1} are attributed to vibration modes of the gel net work.(Jal et al., 2004) TEM micrographs of nanosilica after extracted from pure silica are shown in Figure 2. Micrograph of the nanosilica particles show agglomerate form of dimension about 50 nm and specific surface area is 656 m^2g^{-1}. The particle shape distribution is found to be uniform. The diffraction pattern of a main phase particle (Figure 3) shows a diffuse ring pattern which is indicative of an amorphous phase.

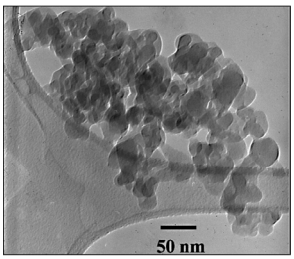


Figure 2. TEM micrograph of nanosilica powder.

Figure 3. Diffraction pattern of powder.

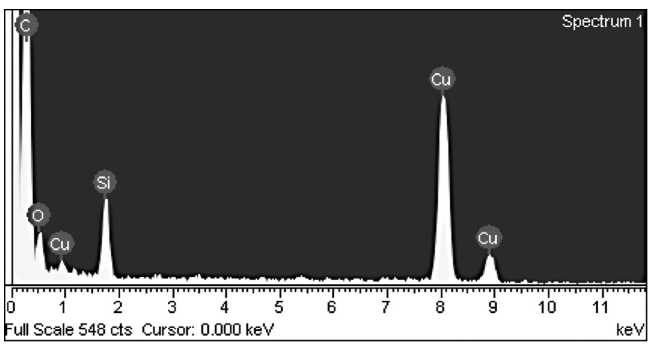


Figure 4. EDS of nanosilica particles.

EDS profile of nanosilica particles contain predominantly the elements of Si, O, C and Cu. Both Si and O peaks correspond to the silica (Figure 4). The dominant signals originate from copper and carbon due to TEM copper grid and carbon coating.

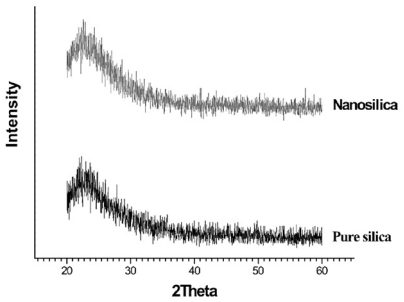


Figure 5. Diffractograms of pure silica and nanosilica.

Strong broad peaks of nanosilica and pure silica are centered range on ≈ 22-23° (2θ), which are in keeping with the strong broad peak of a characteristic of amorphous SiO_2 (Byung et al., 2007). The results show that both nanosilica and pure silica are in an amorphous state (Figure 5).

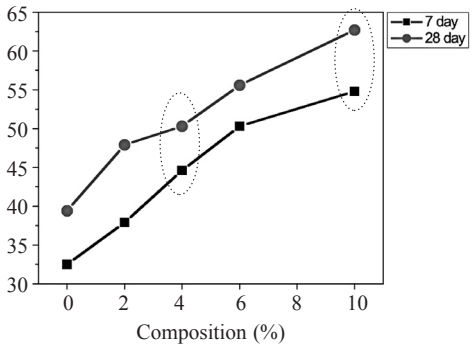
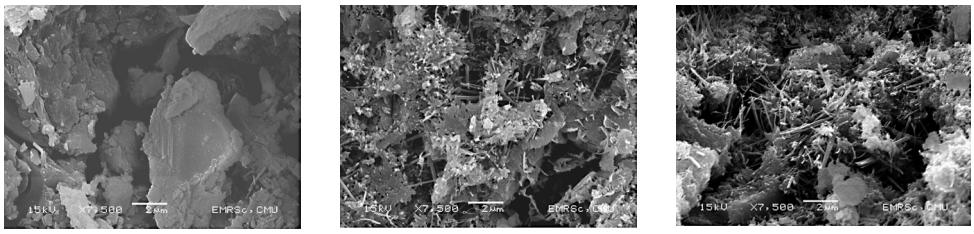


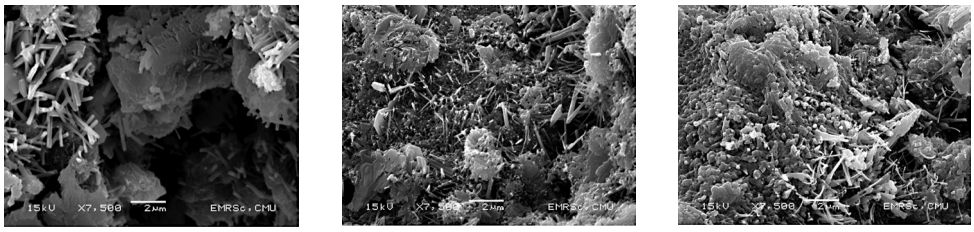
Figure 6. Compressive strength of nanosilica.

It can be seen in Figure 6 that the compressive strength was developed in cement paste containing nanosilica particles in every case and higher than that of control cement paste. The difference in the strength development of the cement paste can be attributed to pozzolanic reaction. The strength of the cement paste was found to increase as the nanosilica content increased from 2 to 10 wt%.



(a) (b) (c)

Figure 7. SEM micrograph of portland cement pastes at 7 days of (a) portland cement paste, (b) paste with additive NS 4%wt, (c) paste with NS 10%wt.



(a) (b) (c)

Figure 8. SEM micrograph of portland cement paste at 28 days (a) portland cement paste, (b) paste with additive NS 4%wt (c) paste with NS 10 %wt.

The mechanism predicted by the compressive strength test and SEM examinations were performed. Addition of nanosilica in cement paste led to differences in the microstructure of the hardened pastes. SEM micrographs of cement pastes with added nanosilica show C-S-H gel in isolation surrounded and connected with many needle-hydrates in the SEM micrograph of portland cement paste (Figure 7 and 8). On the other hand, dense and compact phases reveal that a formation of hydration products and reduction in the number of $Ca(OH)_2$ crystals (Hui et al., 2003).

CONCLUSION

Nanosilica particles which obtained from the rice husk ash are in the agglomerate form which dimension of 50 nm and specific surface area 656 m^2g^{-1}. The particle shape distribution is found to be uniform. The diffraction pattern of the particles show a diffuse pattern which indicative of amorphous phase and supported by XRD patterns. The infrared spectral data also supports the presence of hydrogen bonded silanol group and the siloxane groups in the silica. Based on the results of compressive strength test, it is expected that nano scale silica behaves not only as filler but also improve the strength of cement paste.

Therefore, the addition of nanosilica particles in cement paste, introduce high performance to concrete.

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