## Investigation of Compressive Strength of Cement/Silica Nanocomposite Using Synthesized Silica Nanoparticles from Sugarcane Bagasse Ash

Srijana Basnet, Sambridhi Shah, Rajendra Joshi and Rajesh Pandit<sup>\*</sup>

Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu, Nepal

(\*) Corresponding author: rajesh.pandit@trc.tu.edu.np (*Received: 30 June 2021 and Accepted: 3 January 2022*)

#### Abstract

The effect of silica nanoparticles used as a nanoporous additive in cement was studied with various wt-% ratios. Saccharum officinarum bagasse, an agricultural waste residue was used to synthesize silica nanoparticles as it contains a high amount of silica. The synthesized silica nanoparticles were characterized through XRD and FTIR spectroscopic analysis techniques. By the analysis of XRD, crystalline peaks were found to be particularly of quartz form of silica with an average size of 25.58 nm. The characteristic functional group of the extracted silica nanoparticle was observed at various absorption bands such as the peaks at 1056 cm<sup>-1</sup> and 794 cm<sup>-1</sup> correspond to Si-O-Si asymmetric and Si-O symmetric stretching modes respectively. The extracted silica nanoparticles were applied to form nanocomposites with cement to investigate their compressive strength and the silica nanoparticle was found to increase the compressive strength of cement due to the pozzolanic reaction of silica nanoparticles with Ca(OH)<sub>2</sub>.

Keywords: Compressive strength, FTIR, Silica nanoparticles, Sugarcane bagasse ash.

#### **1. INTRODUCTION**

Cement, a dominant binding material for concrete, is widely used in modern construction [1,2]. The major benefit of cement is its degree of strength that can be adapted to meet the needs of a specific construction by altering the water, cement, and the aggregate ratio [3]. Cement contains two major constituents i.e., tricalcium silicate and di-calcium silicate which gives calcium silicate hydrate  $(3CaO.2SiO_2.3H_2O)$  (C-S-H) and calcium hydroxide (Ca(OH)<sub>2</sub>) on hydration. The C-S-H is chiefly superintended for the improvement of the mechanical strength of concrete [4,5]. Concrete strengthens year by year due to the cement's ability to form bonds with surrounding moisture particles [6]. However, the quasi-brittle essence is the utmost hindrance to concrete and cement-based materials [7]. The chemomechanical are enhanced by influencing

the mechanism of early-stage hydration. Nano-additives allow the hybridization and maneuvering of the crystal structure at a discrete length scale at the early hydration and curing of cement paste [8]. Nano-silica [9], nano-alumina [10], nano-titania [11], nano-iron oxide [12], and nano-clay particles [13] are some of the additives used as supplements in cement paste. However, nano-silica is the one that remarkably influences the mechanical characteristics of cement-based composites attributed to elevated purity and high surface area. Moreover, silica nanoparticles act as nucleation sites for the extension of hydration products in cement due to their diminished size followed by their sizeable surface area, purity, and their amorphous nature [14]. Furthermore, silica nanoparticles prepared can be bv employing the green synthesis technique. For the green synthesis, various solid wastes can be used as the starting materials [15]. Some of the wastes containing silica are shown in Table 1.

Table 1.	Silica so	urces ar	nd its	composition	
in waste materials.					

Silica sources	Silica Content (%)		
Sugarcane bagasse ash	96.93 [15]		
Rice husk ash	93.20 [16]		
Sugarcane leaf ash	80.14 [17]		
Rice straw ash	75.00 [18]		
Wheat straw ash	55.00 [19]		
Corn cob ash	52.32 [20]		

Among the agricultural residue as presented in Table 1, silica content is maximum in sugarcane bagasse ash (SCBA), which is also highly porous with high surface accessibility and purity [15, 21]. The use of silica as a porous material is well established owing to its advantageous aspects such as low density, low toxicity with good biocompatibility, ease of substrate modification, stability, and cost-effectiveness [22, 23]. Hence, in this paper, nano-silica was extracted from sugarcane bagasse ash and used to prepare cement/silica nanocomposites varying in composition. Furthermore, it investigated the influence of nano-silica on the compressibility of strength the cement/silica nanocomposites.

## 2. EXPERIMENTAL

## 2.1. Materials

Sugarcane bagasse (SCB) was collected from local fruit shops in Kathmandu. The chemicals used in the experiment; hydrochloric acid (36.5 %), and sodium hvdroxide (98%)pellets were manufactured from Thermo Fischer Scientific India Pvt. Ltd. Mumbai and supplied from the local market in Kathmandu and used without further purification.

## 2.2. Methods

# A. Extraction of Silica Nanoparticles from Sugarcane Bagasse ash (SCBA)

Sugarcane bagasse (SCB) was washed and sun-dried for 4 days. The dried SCB was burnt for 5 hours to form white ash [24]. 20 g of SCBA was dispersed in 120 mL of distilled water and was neutralized using 1 N HCl with continuous stirring for 2 hrs. The ash residue was filtered, and washed and 1 N NaOH was added with constant stirring to produce a sodium silicate solution [25]. The solution was filtered and the residue was washed with boiling water to remove the carbon residue. The filtrate was again neutralized by 1 N HCl to get silica gel. After aging the silica gel for 18 hours, water was added to it to make a slurry which was then centrifuged for 15 minutes. The gel was then dried in a hot air oven for 24 hours.

## B. Preparation of Cement/Silica Nanocomposite

The cement/silica nanocomposites were prepared in 90/10, 85/15, and 80/20 weight ratios of cement and nano-silica by adding 25% distilled water to the total weight of mixtures. Four different cubes  $(2\times2\times2 \text{ cm})$ were prepared in the wooden mold and allowed to be set for 24 hours. Further, the prepared cubes were cured by immersing them in distilled water for 7 days.

## 2.3. Characterization Techniques

The XRD analysis technique was used to determine the crystalline structure and size of synthesized silica nanoparticles. The crystalline size of silica nanoparticles was calculated by using Debye-Scherer's equation [26].

Crystallite size (D) =  $(K\lambda)/\beta\cos\theta$ )

where, K = Scherer's constant a having value 0.9,

 $\lambda$  = wavelength of X-ray.

 $\beta$  = Full width of half maximum intensity (FWHM) whose value must be in radian.

The instrument used for XRD analysis of the synthesized silica nanoparticles was Bruker D2 Phaser Diffractometer (USA) with a monochromatic CuK $\alpha$  radiation source ( $\lambda = 0.15418$  nm) at angle 2 $\theta$ ranging from 10° - 80°. Likewise, FTIR spectroscopic technique was used to obtain the infrared absorption spectrum to identify the functional groups present in the synthesized silica nanoparticles. The instrument used for FTIR analysis of extracted silica was the Affinity-1S FT-IR IR Spectrometer (SHIMADZU, Japan) where spectra were analyzed using the KBr pellet method in the spectral range of  $4000 - 400 \text{ cm}^{-1}$ . Furthermore, the compressive strength test was done to measure the maximum amount of compressive load that a material can breaking. Compressive bear before strength was calculated using equation (1) [27]:

 $Compressive strength = \frac{Breaking load (N)}{Cross-sectional area (m^2)} \quad (1)$ 

The compressive strength of the prepared sample block was tested using Compression Testing Machine (C.T.M.), Harrish and Terrish, India.

#### **3. RESULTS AND DISCUSSIONS**

## 3.1. XRD Analysis of Silica Nanoparticles

The extracted white powder was subjected to determination of the crystallite size via X-ray diffraction (XRD) analysis. Vindication of the effectiveness and the size of silica nanoparticles were provided by XRD analysis. The X-ray diffractogram of the sample is shown in Figure 1:



Figure 1. XRD pattern of extracted silica nanoparticle.

From Figure 1, the diffraction peaks were assigned at 28.68°, 31.96°, 40.86°, 45.67°, 50.86°, 58.72°, and 67.98° of 20 values which were particularly of quartz form of silica and have been keenly indexed as trigonal crystalline structure [28]. The crystalline phase of silica in sugarcane ash was related to the condition of combustion, as the temperature was increased the amorphous silica present in sugarcane was transformed into crystalline silica polymorphs, like quartz [29,30]. The average crystallite size of synthesized nanoparticles was found to be 25.58 nm using Debye Scherer's equation.

### **3.2. FTIR Analysis of Extracted Silica** Nanoparticles

FTIR analysis was carried out for the identification of unknown samples through absorption of the functional group. The FTIR spectra of synthesized silica are shown in Figure 2.



*Figure 2. FTIR spectrum of extracted silica nanoparticle.* 

Figure 2 showed a significant spectrum of various characteristics absorption bands identifying the major functional group as Si-O-Si asymmetric and Si-O symmetric stretching. The peaks at 1049 cm<sup>-1</sup> and 794 cm<sup>-1</sup> were due to the Si-O-Si asymmetric bond of siloxane groups and Si-O symmetric stretching modes respectively. The band centered at 447 cm<sup>-1</sup> was assigned to the bending frequency of Si-O-Si stretching [31]. The band around 1629 cm<sup>-1</sup> was usually because of O-H bending vibration from the Si-OH silanol group [32]. No peaks were detectable between  $2800 \text{ cm}^{-1}$  and  $3000 \text{ cm}^{-1}$ . It means there were no original organic compounds on the surface in silica after controlled combustion and extraction which was engrossing as organic molecules. especially with carboxylates were tough to eliminate from metallic oxides [33].

# **3.3.** Compressive Strength Test of Cement/Silica Nanocomposites

The compressive strength of pure cement and cement/silica nanocomposites (90/10, 85/15, and 80/20 of weight ratios) block was calculated using equation (1). The calculated value was plotted in a bar

Hydration of cement component

$$2 \text{ Ca}_3 \text{SiO}_5 + 7 \text{ H}_2 \text{O}$$
  
(Tricalcium silicate)

 $\rightarrow 3 \text{ CaO} \cdot 2 \text{ SiO}_2 \cdot 4 \text{ H}_2\text{O} + 3 \text{ Ca(OH)}_2$ Calcium silicate hydrate

The reaction of silica nanoparticles with calcium hydroxide

+

SiO<sub>2</sub>

 $Ca(OH)_2$ 

*Figure 3.* Compressive strength test of extracted silica nanoparticle.

Furthermore, the compressive strength of cement/silica nanocomposite was improved due to the packing effect of silica nanoparticles. The nanoparticle is used as a filler substance that elevates not only the strength but also density as well, mainly by filling the interstitial voids and pores inside the matrix [35]. Several kinds of research on various days strength tests C-S-H

(calcium silicate hydrate)

indicated the enhancement of compressive strength to 20 - 30% from 7 days to 28 days [36, 37]. Hence, the strength can be further increased with the reduction of pore size by silica nanoparticles [38].

## **4. CONCLUSION**

The silica nanoparticles were extracted from sugarcane bagasse ash by the green synthesis method. The extracted silica nanoparticles were characterized by XRD and FTIR spectroscopic techniques. The extracted silica nanoparticles were blended with cement with varying composition such as 10 %, 15 %, and 20 % to form nanocomposites. The compressive strength of nanocomposites was enhanced with the increase in the number of silica nanoparticles as they improved the pozzolanic reaction and the formation of additional C-S-H was accomplished by cement hydration.

## ACKNOWLEDGEMENTS

The authors would like to acknowledge the National Academy of Science and Technology (NAST) Khumaltar, Lalitpur for XRD analysis and the Department of Plant Resources, Thapathali, Kathmandu, Nepal for FTIR analysis. Also, grateful to the Central Material Testing Laboratory, Institute of Engineering, Pulchowk Campus, Lalitpur, Nepal for the compressive strength test.

#### **CONFLICT OF INTEREST**

The authors declare that they have no conflict of interest.

#### REFERENCES

- Biernacki, J. J., Bullard, J. W., Sant, G., Brown, K., Glasser, F. P., Jones, S., Ley, T., Livingston, R., Nicoleau, L., Olek, J., Sanchez, F., Shahsavari, R., Stutzman, P. E., Sobolev, K. P., Prater, T., "Cements in the 21st Century: Challenges, Perspectives, and Opportunities", *J. Am. Ceram. Soc.*, 100(7) (2017) 2746-2773.
- 2. Maghsoudi, A. A., Soheil, M. J., Darbhenz A., "Effect of the Nanoparticles in the New Generation of Concretes, SCC", *Int. J. Nanosci. Nanotechnol.*, 6(3) (2010) 137-143.
- Chandwani, V., Agrawal, V., Nagar, R., "Applications of Artificial Neural Networks in Modeling Compressive Strength of Concrete : A State of the Art Review", *Int. J. Curr. Eng. Technol.*, 4(4) (2014) 2949–2956.
- Papatzani, S., Paine, K., Calabria, H. J., "The Effect of the Addition of Nanoparticles of Silica on the Strength and Microstructure of Blended Portland Cement Pastes", *Boston, May 12-15, International Concrete* Sustainability Conference, (2014) [Online]. Available: http://www.nrmcaevents.org/?nav=display&file=648.
- 5. Jafarbeglou, M., Abdouss, M., Ramezanianpour, A. A., "Nanoscience and Nano Engineering in Concrete Advances A Review", *Int. J. Nanosci. Nanotechnol.*, 11(4) (2015) 263-273.
- 6. Gupta, S., "Application of Silica Fume and Nanosilica in Cement and Concrete A Review", J. Today's Ideas Tomorrow's Technol., 1(2) (2013) 85–98.
- Li, X., Liu, Y. M., Liw, G., Li, C. Y., Sanjayan, J. G., Duan, W. H., Li, Z., "Effects of Graphene Oxide Agglomerates on Workability, Hydration, Microstructure and Compressive Strength of Cement Paste", *Constr Build Mater.*, 145 (2017) 402–410.
- Jamsheer, A. F., Kupwade, P. K., Büyüköztürk, O., Bumajdad, A., "Analysis of Engineered Cement Paste using Silica Nanoparticles and Metakaolin using <sup>29</sup>Si NMR, Water Adsorption and Synchrotron X-ray Diffraction", *Constr Build Mater*, 180 (2018) 698–709.
- Senff, L., Hotza, D., Repette, W. L., Ferreira, V. M., Labrincha, J. A., "Effect of Nanosilica and Microsilica on Microstructure and Hardened Properties of Cement Pastes and Mortars", *Adv. Appl. Ceram.*, 109(2) (2010) 104–110.
- El-Gamal, S. M. A., Abo-El-Enein, S. A., El-Hosiny, F. I., Amin, M. S., Ramadan, M., "Thermal Resistance, Microstructure and Mechanical Properties of Type I Portland Cement Pastes Containing Low-Cost Nanoparticles", *J. Therm. Anal. Calorim.*, 131,(2) (2018) 949–968.
- 11. Pérez-Nicolás, M., Navarro-Blasco, I., Fernández, J. M., Alvarez, J. I., "The Effect of TiO<sub>2</sub> Doped Photocatalytic Nano-additives on the Hydration and Microstructure of Portland and High Alumina Cements", *Nanomaterials*, 7(10) (2017) 329-350.
- 12. Amer, A. A., El-Sokkary, T. M., Abdullah, N. I., "Thermal Durability of OPC Pastes Admixed with Nano Iron Oxide", *HBRC J.*, 11(2) (2015) 299–305.
- 13. Shakrani, S. A., Ayob, A., Rahim, M. A. A., Alias, S., "Performance of Nano Materials in Pervious Concrete Pavement: A Review", *AIP Conf. Proc.*, 2030(November) (2018) doi: 10.1063/1.5066649.
- 14. Gaitero, J. J., Zhu, W., Campillo, I., "Multi-Scale Study of Calcium Leaching in Cement Pastes with Silica Nanoparticles", *Nanotechnology in Construction*, 3 (2009) 193-198.
- 15. Norsuraya, S., Fazlena, H., Norhasyimi, R., "Sugarcane Bagasse as a Renewable Source of Silica to Synthesize Santa Barbara Amorphous-15 (SBA-15)", *Procedia Eng.*, 148 (2016) 839–846.
- Bhagiyalakshmi, M., Yun, L. J., Anuradha, R., Jang, H. T., "Utilization of Rice Husk Ash as Silica Source for the Synthesis of Mesoporous Silicas and their Application to CO<sub>2</sub> Adsorption through TREN/TEPA Grafting", J. Hazard. Mater., 171(1–3) (2010) 928–938.
- 17. Arumugam, A., Ponnusami, V., "Modified SBA-15 Synthesized using Sugarcane Leaf Ash for Nickel Adsorption", *Indian J. Chem. Technol.*, 20(2) (2013) 101–105.
- Binod, P., Sindhu, R., Singhania, R. R., Vikram, S., Devi, L., Nagalakshmi, S., Kurien, N., Sukumaran, R. K., Pandey, A., "Bioethanol Production from Rice Straw: An Overview", *Bioresour. Technol.*, 101(13) (2010) 4767–4774.
- Roselló, J., Soriano, L., Santamarina, M. P., Akasaki, J. L., Melges, J. L., Payá, J., "Microscopy Characterization of Silica-Rich Agrowastes to be used in Cement Binders: Bamboo and Sugarcane Leaves", *Microsc. and Microanal.*, 21(5) (2015) 1314–1326.

- 20. Okoronkwo, E. A., Imoisili, P. E., Olusunle, S. O. O., "Extraction and Characterization of Amorphous Silica from Corn Cob Ash by Sol-Gel Method", *Chemistry and Materials Research*, 3(4) (2013) 68–72.
- 21. Faizul, C. P., Abdullah, C., Fazlul, B., "Review of Extraction of Silica from Agricultural Wastes using Acid Leaching Treatment", *Adv. Mat. Res.*, 626 (2013) 997–1000.
- 22. Maity A., Polshettiwar, V., "Dendritic Fibrous Nanosilica for Catalysis, Energy Harvesting, Carbon Dioxide Mitigation, Drug Delivery, and Sensing", *Chem Sus Chem*, 10(20) (2017) 3866–3913.
- 23. Ghasemi, E., Ghahari, M., "Synthesis of Silica Coated Magnetic Nanoparticles", Int. J. Nanosci. Nanotechnol., 12(2) (2015) 133-137.
- 24. Kalapathy, U., Proctor, A., Shultz, J., "A Simple Method for Production of Pure Silica from Rice Hull Ash", *Bioresour. Technol.*, 73(3) (2000) 257–262.
- 25. Rafiee, E., Shahebrahimi, S., Feyzi, M., Shaterzadeh, M., "Optimization of Synthesis and Characterization of Nanosilica Produced from Rice Husk (a Common Waste Material)", *Int. Nano Lett.*, 2(1) (2012) 1–8.
- 26. Gorji, B., Fazaeli, R., Niksirat, N., "Synthesis and Characterizations of Silica Nanoparticles by a New Sol-Gel Method", J. Appl. Chem. Res., 6(3) (2012) 22–26.
- 27. Assaedi, H., Alomayri, T., Kaze, C. R., Jindal, B. B., Subaer, S., Shaikh, F., Alradddai, S., "Characterization and Properties of Geopolymer Nanocomposites with Different Contents of Nano-CaCO<sub>3</sub>", *Constr. Build. Mater.*, 252, (2020) 119137.
- Pabst W., Gregorová, E., "Elastic Properties of Silica Polymorphs-a Review", Ceram. Silik., 57(3) (2013) 167–184.
- 29. Alves, R. H., Da Silva Reis, T.V., Rovani, S., Fungaro, D. A., "Green Synthesis and Characterization of Biosilica Produced from Sugarcane Waste Ash", *JChem.*, (2017) 1-10.
- 30. Ferreira, C. S., Santos, P. L., Bonacin, J. A., Passos, R. R., Pocrifka, L.A., "Rice Husk Reuse in the Preparation of SnO<sub>2</sub>/SiO<sub>2</sub> Nanocomposite", *Mater. Res.*, 18(3) (2015) 639–643.
- Santana Costa J. A., Paranhos, C. M., "Systematic Evaluation of Amorphous Silica Production from Rice Husk Ashes", J. Clean. Prod., 192 (2018) 688–697.
- 32. Imoisili, P. E., Ukoba, K. O., Jen, T. C., "Green Technology Extraction and Characterisation of Silica Nanoparticles from Palm Kernel Shell ash via Sol-gel", *J Mater. Res. Technol.*, 9(1) (2020) 307–313.
- 33. Rovani, S., Santos, J. J., Corio, P., Fungaro, D. A., "An Alternative and Simple Method for the Preparation of Bare Silica Nanoparticles using Sugarcane Waste Ash, an Abundant and Despised Residue in the Brazilian Industry", J. Braz. Chem. Soc., 30(7) (2019) 1524–1533.
- Montgomery, J., Abu-Lebdeh, T. M., Hamoush, S. A., Picornell, M., "Effect of Nano Silica on the Compressive Strength of Harden Cement Paste at Different Stages of Hydration", Am. J. Eng. App. Sci., 9(1) (2016) 166–177.
- 35. Singh, L. P., Goel, A., Bhattachharyya, S. K., Ahalawat, S., Sharma, U., Mishra, G., "Effect of Morphology and Dispersibility of Silica Nanoparticles on the Mechanical Behaviour of Cement Mortar", *Int. J. Concr. Struct. Mater.*, 9(2) (2015) 207–217.
- 36. Tobon J. I., Paya J., Restrepo O. J., "Study of Durability of Portland Cement Mortars Blended with Silica Nanoparticles", *Constr. Build. Mater.*, 80(1) (2015) 92-97.
- 37. Singh L. P., Ali D., Sharma U., "Studies on Optimization of Silica Nanoparticles Dosage in Cementitious Systems", *Cem. Concr. Compos.*, 70 (2016) 60-68.
- 38. Gaitero, J. J., Campillo, I., Guerrero, A., "Reduction of the Calcium Leaching Rate of Cement Paste by Addition of Silica Nanoparticles", *Cem. Concr. Res.*, 38(8–9) (2008) 1112–1118.