

## Acid Chemical Treatment for Creating Nano-Silica for Construction Applications

Dina E. Tobbala

Department of Civil and Architecture Constructions, Suez University, Egypt.

Department of Civil Engineering, Nile Higher Institute of Engineering & Technology, Mansoura, Egypt

### Abstract

Rice wastes are rich of silica content. It is subjected to an acid chemical treatment, followed by the sol-gel process, and then it is burned in an electric muffle furnace at a temperature of 600 degrees Celsius. This produced silica nanoparticles. The inorganic contaminants in the rice husk ash were purified by leaching them with Hydrochloric acid, boiling them between 80 and 85 degrees Celsius, and then washing them till the PH value reached the neutral limit of 7. The methods involved in preparing the nano-silica (NS) samples were carried out in somewhat different ways for each of the three samples. X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), and transmission electron microscopy (TEM) were used in order to evaluate each specimen in terms of its level of purity and the size of its individual particles. In addition, the three samples were added to concrete mix at 2% of the total binder content as an addition. The compressive strengths of these concretes were then assessed at 7 and 28 days of age and compared with the compressive strengths of normal and silica fume concretes. In addition, the performance of an NS sample with greater purity, smaller particle size, and better responsiveness in concrete was compared to that of commercial NS. It was discovered that the NS can be made at a price and with qualities comparable to those of commercially available NS, which were deemed to be fair. Additionally, the influence of locally manufactured NS on the strength of the concrete was shown to be more evident than the effect of commercially produced NS.

Key words: Acid Chemical Treatment; Sol-gel; Nano-Silica; Concrete.

### 1 INTRODUCTION

As a consequence of its high concentration of amorphous SiO<sub>2</sub> with spherical micro-particles, silica fume (SF) was formerly thought to be a highly reactive pozzolanic material. When combined with CH, it forms a gel composed of calcium silicate hydrate (CSH). When combined with free water, this gel may physically fill up micro and macro holes, lowering the matrix's porosity [1-3]. Because of the very high SiO<sub>2</sub> concentration and the extremely small amorphous particles, nano-silica (NS) is a more reactive pozzolanic than SF. Pastes made with NS cement have been shown to have extremely high strengths in previous research [4-9], as have mortars [10-12] and concrete [13-15]. The incorporation of NS has been shown to accelerate the pozzolanic reaction of cement, decrease pore size, and strengthen the interface connection between the cured cement paste and the aggregate [4, 10]. Chemicals were first used as a raw material in nano-silica production, with methods like hydrolyzing and condensing tetraethyl orthosilicate (TEOS) in ethanol with ammonia being among the first examples. Sequential implantation of Ag and Cu into silica for the formation of metal nano-silica [16], [17]. When making NS by chemical processes, controlling the size, form, and

purity of the final product is simple, but the high cost of raw ingredients makes this option unfeasible for most applications. Recently, efforts have been undertaken to make NS powder out of rice husk, which is a great source of SiO<sub>2</sub> for sheep. Burning husks with more than 95% SiO<sub>2</sub> is the simplest way to produce silica. There are three options for accomplishing the goals of improved purity and reduced particle size. Chemical precipitation, thermal leaching treatment, and any combination of the two are all valid categories for describing these procedures. Alkaline extraction (sodium silicate), acid precipitation (18 hours), and many washes and dryings yielded pure silica by chemical precipitation. [18-20]. There were a lot of stages involved, a lot of reagents needed, and it was quite expensive. Since the alkali-metals (Na, Ca, K, etc.) undergo a eutectic interaction with SiO<sub>2</sub>, the leaching treatment approach alone may yield extremely high purity NS with very tiny nano-size [21, 22]. While others have attempted to combine alkali extraction with heat treatment to get high purity NS, this approach is just as time-consuming and costly [23]. Citric acid leaching treatment and air combustion of rice husks were shown to be beneficial in the elimination of the metallic contaminants using easy and cheap techniques [24-27]. However, the concentration, temperature, and stirring duration of the citric acid solution had a significant impact on the interaction between the groups and the metallic impurities. Any substance added to concrete should either be readily accessible and inexpensive or provide significant enough improvement to justify the additional expense. Therefore, the purpose of this research is to synthesize high purity amorphous NS from Egyptian rice husks using thermal leaching treatment following sol-gel technique, which is more cost-effective, has higher purity, and produces smaller particles than commercial nano-silica (CNS). Three different rice husk NS samples were generated utilizing a leaching technique with very minor differences between them. Compressive strength at 7, 28, 56, 120, and 180 days was evaluated between the best-prepared NS concrete, CNS, SF, and plain concretes. Microstructure analysis of pastes at 7 and 28 days was performed using SEM.

## **2. Experimental Program and Research Variables**

### **2.1 Synthesis of Nano-Silica**

At this stage, the objective is to study the difference between silica with or without leaching and the effect of using open air burning. A total of three distinct types of NS were made by altering the leaching and burning procedures. The NS<sub>w</sub> sample was manufactured without any leaching treatment and then burnt in a static air muffle furnace, whereas the NS<sub>L</sub> sample was treated with HCl and then burned under the same conditions as the NS<sub>w</sub> sample, and the NS<sub>O</sub> sample was leached with HCl and then burned in an open air melting furnace. Samples were analyzed using EDX, XRD, and TEM. A series of procedures were carried out on each NS sample in order to get it ready for testing.

Sample NS<sub>w</sub>: silica generated by burning husks directly without leaching

As a first stage of processing, we washed the rice husk three times with distilled water and then dried it at 110 degrees Celsius for two to three hours to remove any remaining impurities. The husk was cleansed, then 100 gm were burnt in an electric muffle furnace at a rate of 5 degrees Celsius per minute until the temperature reached 600 degrees Celsius, which took around 2 hours, and was then maintained at 600 degrees Celsius for another 4 hours. All of the aforementioned metrics are based on a series of trials. This process is helpful because it rids the rice of any organic matter that may otherwise contaminate it. To achieve fine white silica, the powders must be pulverized in an agate mortar after cooling.

Sample NS<sub>L</sub> : The inorganic contaminants K<sub>2</sub>O, CaO, and SO<sub>3</sub> were removed by leaching with Hydrochloric acid (HCl).

To do this, 50 gm of cleaned rice husk were soaked in a 1000 mL volume of 10% HCl solution. After being rinsed until the PH reached the neutral limit of 7, it was burnt in a reflux at 80-85°C for an hour while being stirred. The remaining husk was dried at 110 degrees Celsius for two to three hours. All three of the aforementioned procedures for making NS<sub>w</sub> were also employed to make NS<sub>L</sub>.

Sample NS<sub>O</sub>:

The same method as for NS<sub>L</sub> was used to create this sample, with the difference that the burning was performed in an open-air melting furnace rather than a static air muffle furnace, maintaining almost the same chamber size and temperature and heating rate. Burning explosions, especially CO<sub>2</sub>, may be reduced with the use of an open-air furnace.

## 2.2 Concrete Contaminants

All of the mixes relied on ordinary Portland cement (OPC) of Type I, the parameters of which are shown in Table 1. Natural sand (S) was used as the fine aggregate, and its fineness module was 2.6 and its specific gravity was 2.67. Dolomite (D) was used as the coarse aggregate, and its NMZ was 12 mm. Its specific weight was 2.7, and its water absorption was 3%. The SP used was 1.08 kg/m<sup>3</sup> dense Sika ViscoCrete 5-930, which was compliant with ASTM-C-494 Types G and F. It was used to improve the workability of concrete mixtures and to facilitate the distribution of NS throughout the mixture. Also used was silica fume (SF), the chemical and physical characteristics of which are mentioned below. Table 1 shows the purity and particle size distribution of a commercial nano-silica (CNS) utilized for comparison. At 1000 LE/Kg, the price of CNS was rather high.

**Table 1:** The chemical and physical properties

Items	OPC	SF	CNS
<b>Chemical composition (wt%)</b>			
SiO <sub>2</sub>	22.0	95.0	100
Al <sub>2</sub> O <sub>3</sub>	6.6	0.9	-
Fe <sub>2</sub> O <sub>3</sub>	2.8	0.6	-
CaO	60.1	0.3	-
MgO	3.3	0.9	-
SO <sub>3</sub>	2.1	0.5	-
LOI	2.6	2.1	-
<b>Physical properties</b>			
S.G	3.15	2.15	2.0
Avg. particle size (nm)	21000	1000	30

### 2.3 Mixing and Testing design

Table 2 shows the ingredients used to make the three different batches of concrete ( $M_{NSW}$ ,  $M_{NSL}$ , and  $M_{NSO}$ ). All batches had the same proportions of water to binder (W/B), supplementary powder to binder (SP/B), and sand to total aggregate (S/Agg), with binder made up of Cement (C), Sand (S), and nano silica (NS), in that order. Binder was limited to C in the inert control mixture.

**Table 2:** Mixture proportions of concrete mixes

	Mix no.	C (K/m <sup>3</sup> )	W(K/m <sup>3</sup> )	S(K/m <sup>3</sup> )	D (K/m <sup>3</sup> )	SF(K/m <sup>3</sup> )	NS(K/m <sup>3</sup> )	NS type	SP(K/m <sup>3</sup> )
<b>control</b>	$M_0$	400.0	120.0	792.3	1188.4	0.0	0.0	0.0	16.0
<b>SF mix</b>	$M_{SF}$	400.0	138.0	740.8	1111.2	60.0	0.0	0.0	18.4
<b>Part 1</b>	$M_{NSW}$	400.0	140.4	733.6	1100.5	60.0	8.0	NS <sub>W</sub>	18.7
	$M_{NSL}$	400.0	140.4	733.6	1100.5	60.0	8.0	NS <sub>L</sub>	18.7
	$M_{NSO}$	400.0	140.4	733.6	1100.5	60.0	8.0	NS <sub>O</sub>	18.7
<b>Part 2</b>	$M_{PNS}$	400.0	141.6	730.1	1095.1	60.0	12.0	NS <sub>O</sub>	18.9
	$M_{CNS}$	400.0	141.6	730.1	1095.1	60.0	12.0	CNS	18.9

The silica fume (SF) was added to the cement in the  $M_{SF}$  mix at a ratio of 15%. In addition, the appropriate NS was added to the cement ratio of 2% in mixes  $M_{NSW}$ ,  $M_{NSL}$ , and  $M_{NSO}$ . The other two mixtures,  $M_{PNS}$  and  $M_{CNS}$ , were made to evaluate how well commercially available nano-silica (CNS) compares to the open-air NS (NS<sub>O</sub>) in concrete. Out of the three NS samples created, the one with the best attributes and performance was used to create a mix ( $M_{PNS}$ ). It can be shown in Table 2 that the NS was included in both mixtures at a 3% cement percentage.

The following methods were used to create all of the NS-containing mixtures:

A small mixer was used to mix the NS with only the mixing water at a high speed of 120 rpm for 2 minutes. Second, for one minute, cement, silica fume, sand, and dolomite were dry mixed at a medium speed of 80 revolutions per minute. Third, for around 30 seconds with the mixer running at medium speed, two-thirds of water with NS was added gradually to the dry ingredients. In other words, SP and the rest The next 30 seconds were spent adding the remaining one-third of the agitated water. After a 30-second pause, the ingredients were combined again for one minute at high speed. Cubes of  $70.7 \times 70.7 \times 70.7$  mm concrete were poured, shook on a vibrating table for 24 hours, then demolded and cured by being submerged in water at room temperature until the testing date. Compression testing was performed at 7, 28, and 180 days of age for the  $M_{NSW}$ ,  $M_{NSL}$ , and  $M_{NSO}$  mixes, and at 7, 28, 56, 120, and 180 days of age for the  $M_{PNS}$  and  $M_{CNS}$  mixes, all in accordance with BS 1881: part 116: 1983.

Small cubes ( $50 \text{ mm} \times 50 \text{ mm} \times 50 \text{ mm}$ ) were made from cement pastes of mixes  $M_o$ ,  $M_{SF}$ , and  $M_{NSO}$  to examine the effect of NS inclusion on the microstructure of the specimens. Compression was applied to the point of failure on these cubes, and then tiny samples were obtained from the centers of each cube to use in the scanning electron microscope analysis (SEM). In this experiment, a Field Emission Scanning Electron Microscope was used (FE-SEM). Specimens were examined at 7 and 28 days post-fracture to examine the fracture surface.

### 3. Findings, Analysis and Discussion

X-ray diffraction (XRD) examination of the  $NS_w$ ,  $NS_L$ , and  $NS_O$  samples is shown in Figure 1. None of the two samples differ in any way from being amorphous in form. This finding agrees with those of prior research [29, 30]. According to previous reports, ash derived from burning untreated and varied HCl-treated husk samples all displayed amorphous shape. This finding is in line with the current investigation. Acid treatment of the husk was likewise observed to have no effect on the silica structure. The average particle size (PS) and the estimated oxides contained in the three samples are shown by TEM and EDX, respectively (Figures 2 and 3). As can be shown in Fig. 2-a, the purity of  $NS_w$  produced by directly burning cleansed husk was 97.15%, and it included small amounts of K, Ca, and S oxides. According to Fig. 3-a, the average particle size was 28.5 nm. It seems thus that  $NS_w$  portrayals fared better than SF ones. As far as food preparation goes,  $NS_w$  was fairly affordable at 124 L.E/Kg. Since most metallic impurities could be removed by hot HCl treatment, the purity of the  $SiO_2$  could be raised to 99.51%, as shown in Fig. 2-b, with a mean particle size (PS) of 8.51 nanometers (nm) in Fig. 3-b. Decreased impurities are mostly the consequence of a chemical interaction between acid and metals, with the metals being filtered out afterwards [29]. In contrast, the price of preparation rose to 550 L.E/Kg. The price went up because of the citric acid solution, the distilled water necessary to get the pH down to 7, and the electricity used to run the drying furnace.

With an open-air furnace used in place of a static air muffle, the average PS was reduced by 6.5 nm, bringing the overall purity to 100%. More importantly, the cost of NS was reduced to 400 L.E/Kg because to the fact that an open-air furnace could burn 225% of the quantity burnt by muffles while using the same amount of time and electricity. This might be because combustion gases are expelled, and new oxygen is introduced into an open furnace. Therefore, the greatest cost-effectiveness is found at NS<sub>O</sub>. Experiments conducted previously found that heating the husk at a rate of 5°C / min till reaching to 600°C and fixed for 4hr was adequate to remove all organic components and convert them to gases, leaving just the inorganic metals as in sample NS<sub>w</sub>. After 4 hours of direct burning at 600 C, there was still 20% of the original weight of the husk in 100 gm. This demonstrated that the cleaned husk has around 80% organics and 20% inorganic metals, with the latter having been oxidized to amorphous oxides.

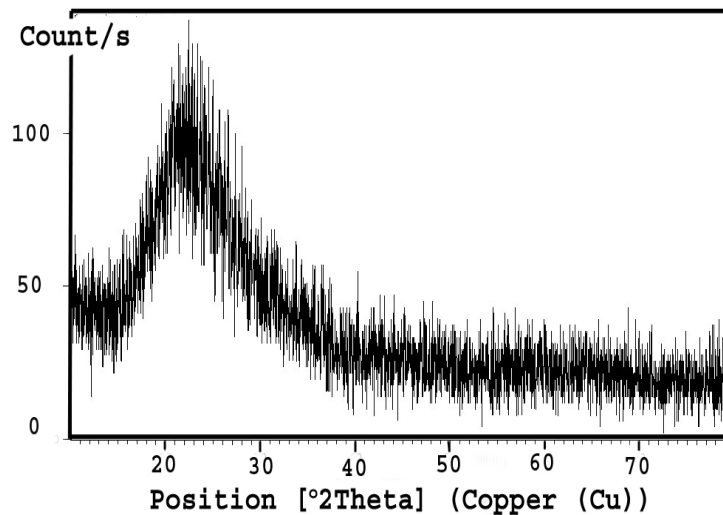
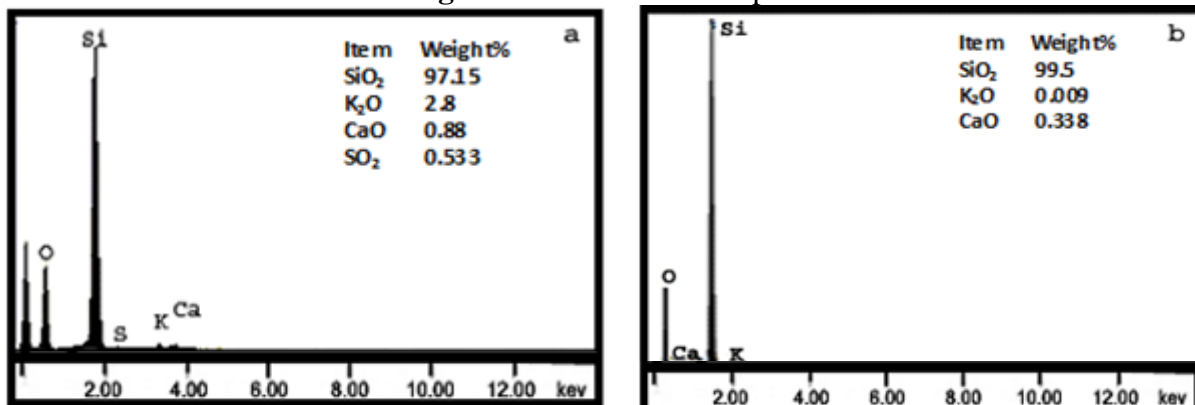
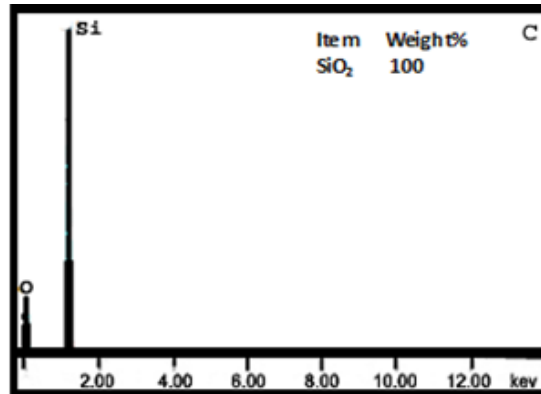


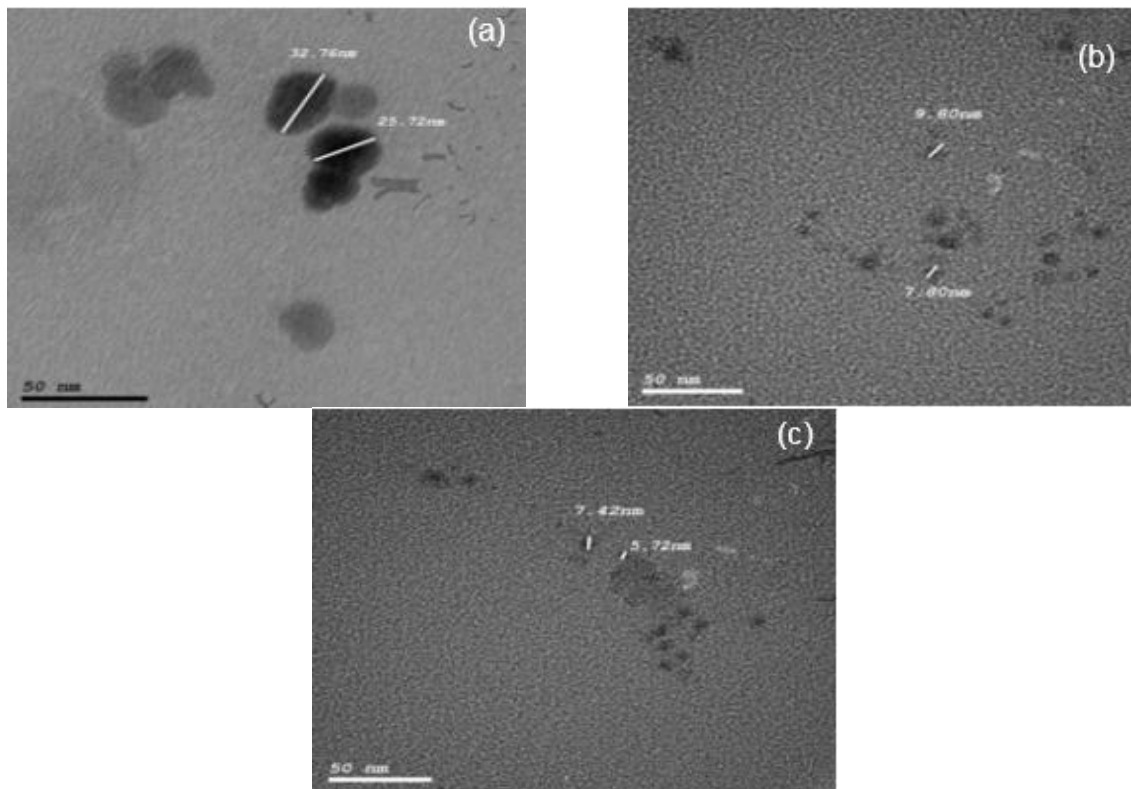
Figure 1: XRD of NS samples







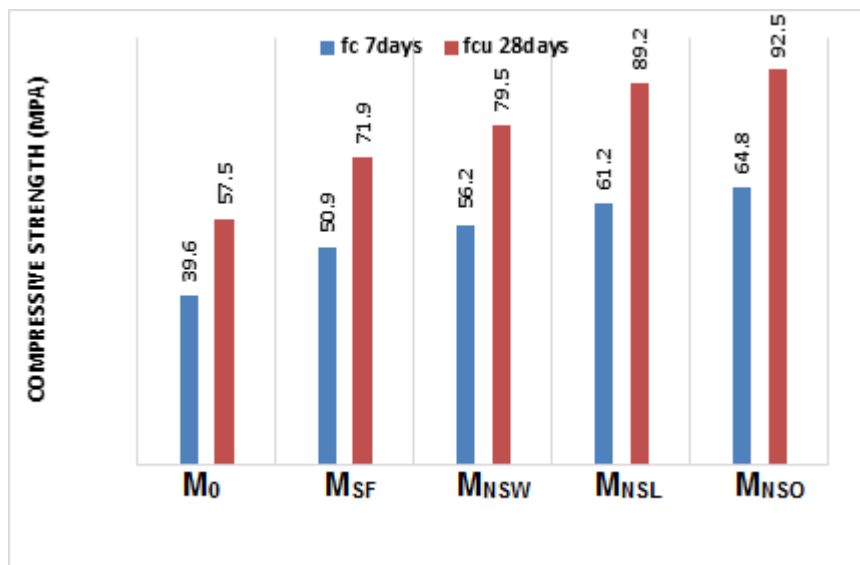
**Figure 2:** EDX of (a) NS<sub>w</sub>, (b) NS<sub>L</sub> and (c) NS<sub>O</sub>.



**Figure 3:** TEM of (a) NS<sub>w</sub>, (b) NS<sub>L</sub> and (c) NS<sub>O</sub>.

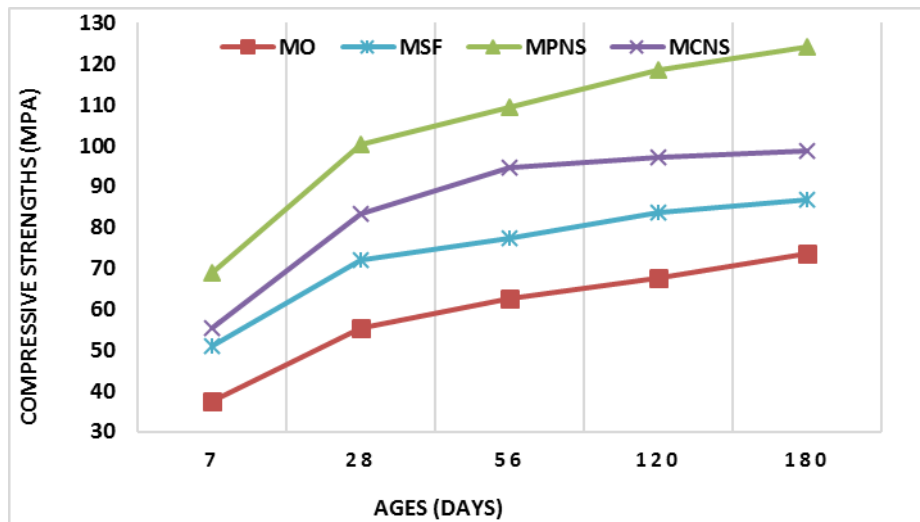
Figure 4 displays a comparison between the control and SF mixes and the compressive strength of the nano-sample mixes at 7 and 28 years. Compressive strength rose from 37.6 to 55.5 MPa at 7 and 28 days of maturity for the control mix, and from 50.9 to 71.9 MPa when 15% SF was added. The insertion of mixtures led to further increases in compressive strength in the NS samples. Mixes containing 2% NS<sub>w</sub>, NS<sub>L</sub>, or NS<sub>O</sub>, for instance, had compressive strengths of 79.5, 89.2, and 92.5 MPa, respectively, after 28 days of curing.

While increasing the compressive strength of the control mix by around 30% with 15% SF, an additional increase of about 37% was achieved by adding 2% NS<sub>0</sub>. Since higher levels of silica purity result in a greater pozzolanic reaction and smaller particle sizes improve the powder's surface area in concrete, making NS<sub>0</sub> the optimal sample. When comparing NS<sub>0</sub> with CNS, the findings showed that the former was the superior prepared sample (PNS). NS<sub>0</sub> preparation costs are estimated to be 40% of CNS costs. PNS also has a lower PS than CNS. Figure 5 displays the results of a comparison between the compressive strength values of 3% PNS and 3% CNS mixes at 7, 28, 56, 120, and 180 days of age, as well as the values for the control and SF mixes. From days 7 to 180, the PNS mix had a compressive strength of 68.8 MPa, 100 MPa, 109 MPa, 118 MPa, and 124.2 MPa, whereas the CNS mix had a strength of 55.5 MPa, 83.6 MPa, 94.5 MPa, 97.2 MPa, and 98.8 MPa. Specifically, PNS increased SF strength after 28 days by 39.6 percent whereas CNS increased it by just 16.2 percent. Thus, PNS outperformed CNS throughout the board. Because PNC particles are smaller than CNS ones, this may be the case.



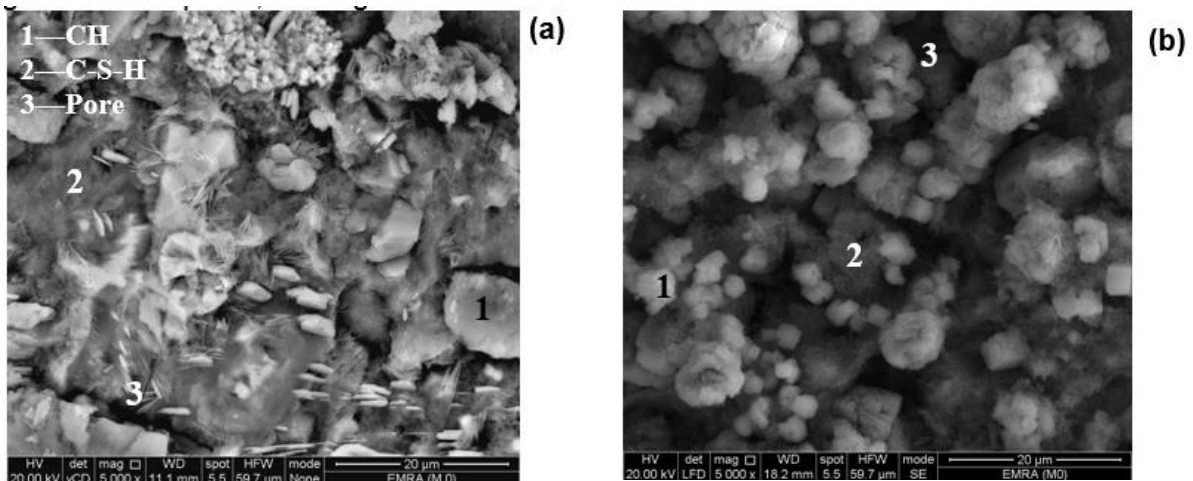
**Figure 4:** Compressive strengths of NS samples at 7 and 28 days



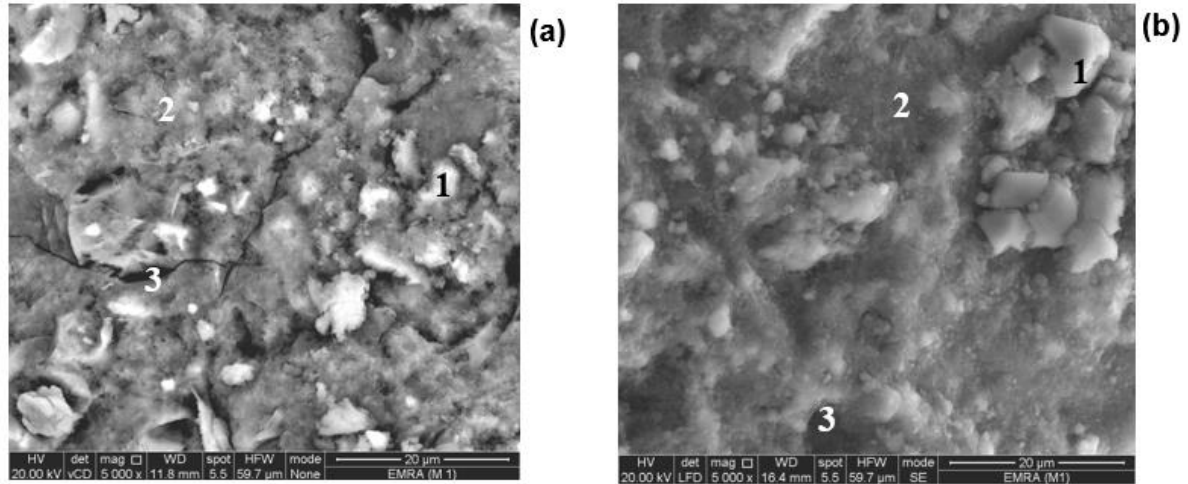


**Figure 5:** Compressive strengths of PNS vs.CNS

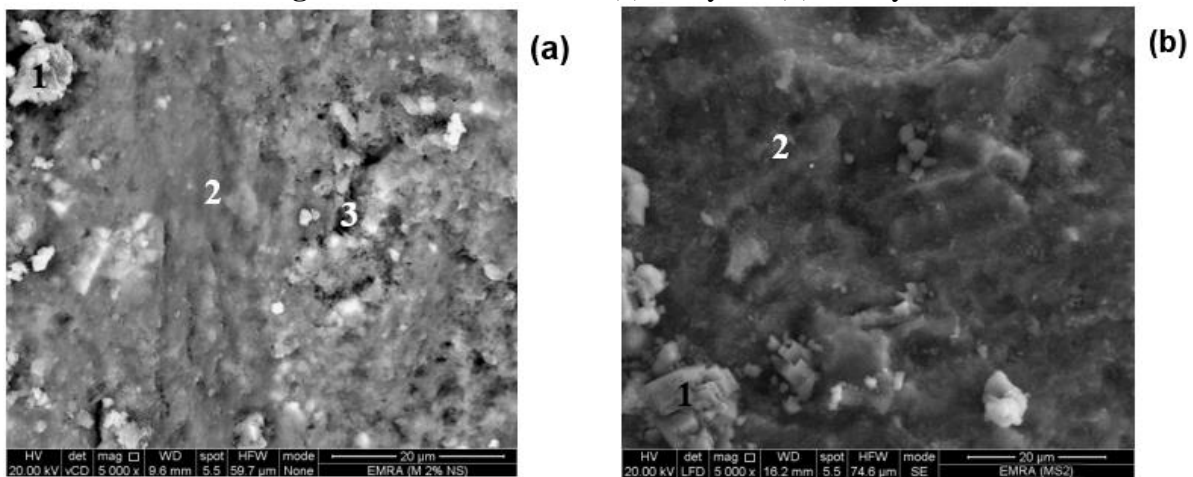
Microstructure (SEM) images of 7 and 28 day old control, SF, and NS<sub>0</sub> cement pastes are shown in Figures.6-8. Observe Fig. 6 to see how the CSH gel of MO paste occurs as discrete clusters that are lapped and joined with needle hydrates and large crystals of CH.



**Figure 6:** SEM of Mo at (a) 7 days & (b) 28 days



**Figure 7:** SEM of MSF at (a) 7 days & (b) 28 days.



**Figure 8:** SEM of  $M_{NSO}$  at (a) 7 days & (b) 28 days

Observe Fig.7 to see how the SF mixture increases the CSH gel while decreasing the CH and eliminating the needle hydrates. This occurred because a reaction between CH and SF produced CSH, which caused this effect. CSH molecules, in general, are larger than CH ones. Adding 2%  $NSO$ , which has a very high surface area, had a vital role in speeding up the reaction, resulting in more dense CSH clusters with very tiny separated CH crystals and a substantial reduction in the average size of the pores due to the nano-filler effect of the NS (see Fig. 8).



#### 4. Conclusions

1. The amorphous  $\text{SiO}_2$  with 97.15% purity, 28.5 nm PS, and a cost of 124 L.E./Kg was created by direct burning of cleaned husk. However, the price climbed to 550 L.E./Kg as a result of the treatment's higher PS of 8.5 nm and purity of 99.5%. Burning in an open-air furnace also reduced the PS to 6.5 nm while increasing the purity to 100 percent. The price of  $\text{NS}_L$  was cut in half to 400 L.E./Kg while time and electricity were saved thanks to a 225% increase in muffles' burning capacity.
2. Because of the high purity and nano-PS in all three samples,  $\text{NS}_W$ ,  $\text{NS}_L$ , and  $\text{NS}_O$ , they exhibited greater pozzolanic reactivity and a larger surface area than SF.
3. Their mixtures had higher compressive strengths compared to the control and SF mixtures. At 28 days old, the addition of SF increased the strength of the control mix by 29.5%, whereas the samples increased it by 43.2, 60.7, and 66.7%, respectively.
4. Since  $\text{NS}_O$  is composed of 100%  $\text{SiO}_2$  and 6.5nm PS, and since its mix exhibited the highest compressive strength across all NS sample mixes across all ages, it was selected as the best PNS.
5. Concrete reinforced with PNS had higher compressive strengths than concrete reinforced with CNS at all ages. For instance, compared to CNS, PNS increased SF strength by 39.6% at 28 days of life.
6. Microstructure SEM images reveal that the addition of 2%  $\text{NS}_O$  enhanced the density of the matrix by reacting with CH and reducing the pores, and that the elimination of most of the CH resulted in a more impermeable paste matrix.

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