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RESEARCH ARTICLE

Manufacturing nano novel composites using sugarcane and eggshell as an alternative for producing nano green mortar

Radwa Defalla Abdel Hafez[1] · Bassam A. Tayeh[2] · Khaled Abdelsamie[3]

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Abstract

The purpose of this study is to demonstrate the impact of incorporating two different types of green nanomaterials (sugarcane and eggshell) on destructive and non-destructive properties of mortar. Nano sugarcane (NSC) was manufactured by calcining sugarcane at temperatures of 600 °C for 3 h. On the other hand, nano eggshell (NES) was manufactured by calcining eggshell at temperatures of 600 °C for 6 h. The sugarcane ash and eggshell ash were then milled to nano size. The final nano-sized product replaced Portland cement with different dosages of 2, 4 and 6%. Sixteen mortar mixtures were designed and prepared using only NSC or only NES or a combined hybrid of NSC and NES. Consistency, compressive strength, flexural strength and microstructure analysis (scanning electron microscopy and energy-dispersive X-ray) tests were conducted to investigate the influence of replacement materials on the performance and properties of mortars. The results showed that the use of nano sugarcane and nano eggshell enhanced the compressive strength and reduced permeability of green mortar due to the micropore structure. At the ages of 7 and 28 days, mortars containing a replacement ratio of 2% (0.5% NSC + 1.5% NES) had remarkably improved mechanical properties, and the improvement in compressive strength reached 21.3% and flexural strength to 10.08% when compared to the control sample. The efficiency of NSC and NES in increasing mortar strength was also confirmed by microstructure analysis.

Keywords Nano sugarcane (NSC) · Nano eggshell (NES) · Compressive strength · Flexural strength · Non-destructive testing · Different burning time for NSC and NES · Mechanical grinding · Nano green mortar

Introduction

Reprocessing waste by-products is becoming increasingly important in today's world; much research is conducted on these materials, and they are used as additives in manufacture of special mortars in many countries (Shahidan et al., 2017, Alaloul et al., 2020, Amin and Tayeh 2020, Hamada et al., 2020, Hamada et al., 2020c). Sugarcane is one of the world's most widely grown crops with an annual production of above 2165 million tonnes (Joshaghani and Moeini

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Highlights
- The amount of amorphous silica produced is influenced by the NSC and NES burning hours and type of materials.
- Use of high temperatures for burning NSC and NES yielded better results.
- Better microstructure properties were obtained by replacing Portland cement with green nanomaterials.
- The mechanical method of preparing green nanomaterial is an effective nano-manufacturing approach.
- The combination of 2% (0.5% NES and 1.5% NSC) yields the best results.

✉ Bassam A. Tayeh
btayeh@iugaza.edu.ps

Radwa Defalla Abdel Hafez
Radwa_abdalhafz@techedu.sohag.edu.eg

Khaled Abdelsamie
Khalid_abdelsamee@yhoo.com

[1] Civil and Architectural Constructions Department, Faculty of Technology and Education, Sohag University, Sohag, Egypt

[2] Civil Engineering Department, Faculty of Engineering, Islamic University of Gaza, P.O. Box 108, Gaza Strip, Palestine

[3] Civil Department, Faculty of Engineering, Sohag University, Sohag, Egypt

2017). Sugarcane can be used as a mineral admixture in mortar, providing considerable cost and environmental benefits. When waste ashes are integrated into cement matrices, new applications can be destined and provided. Sugarcane bagasse ashes are often discarded when they could be used, for example in cement-based materials such as concrete and mortars, which emit less CO_2 into the atmosphere (Berenguer et al., 2020). Calcium carbonate is a natural calcium source that makes up 92–96% of the raw material in eggshells (ES). Overall ES quality can be affected by several factors, including hen strain/breed/genetics, age and nutritional diet (Baláž 2018). Selin (2007) and Rupasinghe et al. (2011) defined nanotechnology as 'in a matter, regulation of the structure based on molecule products and by-products'. On the one hand, several researchers defined nanotechnology as 'the understanding, manufacture, and control of matter down to the nanometre scale to produce materials with modern properties and functions' (Tayeh et al. 2020, Faried et al., 2021a, Zeyad et al., 2021a). On the other hand, nanotechnology has developed into a science and technology discipline that is focused on the future. Nanoengineered materials have been demonstrated to perform better than their larger counterparts (Mohammed et al. 2016, Faried et al., 2021a, Zeyad et al., 2021a, de Azevedo, Amin et al. 2022).

The word 'nanotechnology' is used in mortar science to explain how material properties at the nanometre scale can be used to alter the bulk properties of mortar (Amin, Zeyad et al. 2021a, b). Gaps between cement particles can be mechanically filled with nanoparticles (Tayeh et al., 2021, Amin et al., 2022). The best material composition would yield a high packing density due to its reduced porosity, resulting in low water demand and increased strength. Nano-silica chemically increases pozzolanic reactivity compared with silica fume. Furthermore, adding nano-silica to cement accelerates hydration. Nano-silica in the cementation matrix creates additional C–S–H gel, which spreads between the cement particles and improves their compaction, by creating a reaction between Sio_2 and Ca_2. The addition of C–S–H gel to cement accelerates hydration (Zapata et al. 2013; Zhang et al. 2018, Amin et al., 2022). Self-compacting mortar with 10% replacement (10% ES and 10% nano palm oil fuel ash) had the best flow capacity and workability, with the nano palm oil fuel ash samples benefitting the most. Partially replacing the cement improved flow capacity, and the self-compacting mortar with 20% partial replacements had the best workability amongst all the self-compacting mortars. Compressive strengths of 20% ES and 20% nano palm oil fuel ash after 28 days of curing were 41.34 and 42.4 kN/mm^2, respectively. Self-compacting mortar with 20 wt% partial replacements had a maximum flexural strength of 3.2 kN/mm^2 after 28 days of curing for ES 20 wt% and nano palm oil fuel ash 20 wt% (Ofuyatan et al., 2020). Nano eggshell (NES) had a low compressive strength at first, especially after 7 days of mortar curing. Compressive strength gradually increased after 14 and 28 days, particularly at replacement levels of 10 wt% and 20 wt% cement with nano palm oil fuel ash (NPOFA) contents. In mortar containing nano palm oil fuel ash and ES, water absorption was low, particularly at high nano palm oil fuel ash and ES contents. As a result, this mortar can withstand harsh environmental conditions including sulphate and acid attacks (Hamada, Tayeh et al. 2020, Hamada, Tayeh et al. 2020). Following chemical tests on sugarcane ash, substitution levels of 0%, 10%, 20% and 30% of total binder weight were selected. The compressive strength of mortar cubes increased with curing age for both mix ratios but decreased as sugarcane content increased. As 10%, 20% and 30% of cement were replaced with sugarcane, the compressive strength was reduced by 16.8%, 25% and 35.5%, respectively, compared with regulation (Joshaghani and Moeini 2018). The results showed that ash from sugarcane wastes calcined at 800 °C and 100 °C had pozzolanic activity. X-ray diffraction (XRD) patterns, transmission electron microscopy (TEM) images and pozzolanic activity tests show the effect of various factors on the activation of these ashes (Morales et al. 2009). It was close to fly ash, and previous research showed that using fly ash from sugar mills in Trangkil-Pati, Central Java, Indonesia, increased the compressive strength of lightweight mortar produced (Setyowati 2014). Compressive strength and corrosion resistance were comparable to control (OPC) samples when hardened cement paste samples were substituted with 10% sugarcane bagasse ash (Garrett et al. 2020). The results showed that 20% of optimum results in nano sugarcane (NSC) bagasse ash (5% NSC bagasse ash + 95% cement, 10% NSC bagasse ash + 90% cement, 15% NC bagasse ash + 85% cement and 20% NSC bagasse ash + 80% cement) were 34.25, 38.64, 43.42 and 44.84 N/mm^2, respectively. As a result, compared with conventional materials, those nanomaterials can have superior strength. Nano bagasse ash was used in cement mortar in this analysis, and it produced better results in sugarcane bagasse ash (Torres Agredo et al. 2014; Mamatha et al. 2017). The following are several advantages of the current work:

1) Waste disposal is made easier.
2) Abundant supply of starting materials reduces production costs.
3) Naturally produced starting powder can contain other elements (Zhou et al. 2015).

As a result, the goal of this research is to investigate the effects of employing nano sugarcane and nano eggshell on quality of green mortar. These nanomaterials are obtained by mechanically milling sugarcane ash and eggshell ash to obtain nano-sized particles. To obtain sugarcane ash and eggshell ash, following procedures were adopted:

To obtain eggshell ash, eggshells were burned for 6 h at 600 °C.

To obtain sugarcane ash, sugarcane ash bagasse was burned for 4 h at 600 °C.

The importance of research lies in the fact that no comprehensive study has been conducted so far to examine the effect of exposing sugarcane and eggshell to constant degrees of burning for two different durations. Also, its effect on the mechanical properties of nano green mortar has not been studied. In addition to that, the use of non-destructive test such as electron microscopy (SEM), energy dispersive X-ray (EDX) and transmission electron microscopy (TEM) is not employed before in any of the of studies to examine properties of nano green mortar.

Research plan

In this work, one was the control mix, and 16 mortar mixes were designed; three mixes contained NSC, three other mixes incorporated NES, and nine mixes contained (NES + NSC). The binder volume additions of NSC and NES were 2%, 4% and 6% for groups 1 and 2, and the last group was divided into three parts as follows: first part (0.5% NES + 1.5% NSC, 1% NES + 1% NSC and 1.5% NES + 0.5% NSC), second part (1% NES + 3% NSC, 2% NES + 2% NSC and 3% NES + 1% NSC) and third part (1.5% NES + 4.5% NSC, 3% NES + 3% NSC and 4.5% NES + 1.5% NSC). The effect of replacement materials on the performance properties of mortars was investigated using consistency, compressive strength, flexural strength and microstructure analysis (SEM, energy dispersive X-ray [EDX] and TEM) tests, see Fig. 1.

Research importance

- The relevance of this research lies in the study of sugarcane and eggshell, that is NSC and NES, which have not been extensively studied previously.
- NES and NSC were applied as partial replacements of cement mass to produce nano green mortar (NGM).

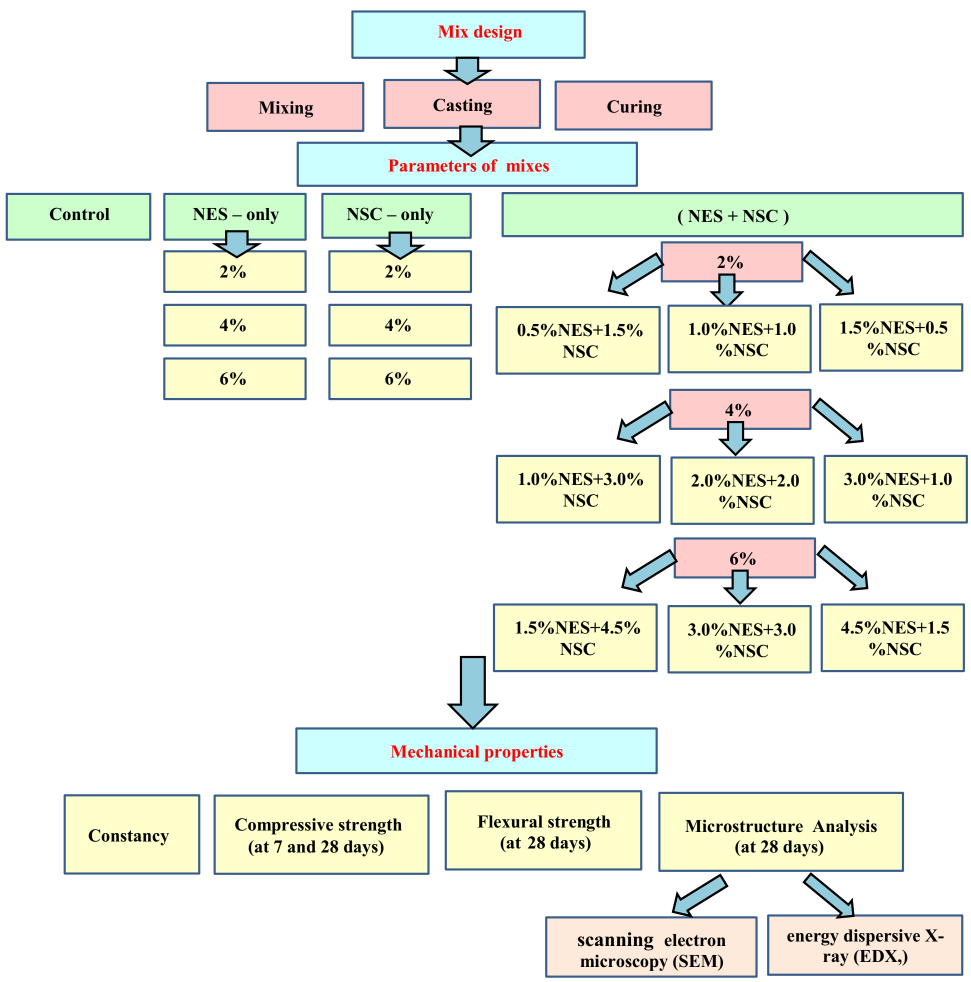


Fig. 1 Details of the carried out a research plan

- The mixture of different proportions of NSC with NES was used to produce NGM, wherein compressive strength 59.62 MPa was achieved at 28 days.
- The effects of NSC and NES on the microstructure and new and mechanical properties of NGM were investigated.
- The substance of this research lies in the study of sugarcane and eggshell, which has not been extensively studied previously.

Experimental work

Materials

Ordinary cement CEM I 42.5 N following BS EN 197–1 (EN, B. S., 2000) was used, and the chemical composition of the cement obtainable is shown in Table 1. Quartz sand < 2.00 mm with a specific gravity of 2.5 was used as a fine aggregate (see Fig. 2), and tab water was used; the specification of sand and water followed British standard, BS EN 196–1 (BSI 2005) and BS EN 1008 (EN, B., 2002), respectively. Furthermore, two types of nanomaterial, NSC and NES, were prepared and characterized.

Nanomaterial synthesis

Synthesis of NES

In Sohag, Egypt, raw eggshells were collected from restaurants and bakeries, as shown in Fig. 3. They were first washed with tap water to remove any dust or organic materials from the surface and then oven-dried for 1 day at 110 °C. To make NES, the dried ES were crushed and ground in a crushing machine to minimize particle size, then heated in an electric furnace to 600 °C for 3 h.

A 150-mm sieve was used to sieve ESP. To minimize the carbon content, passing powder with a particle size of 150 mm was collected and heated to 600 °C for 3 h in an electric furnace (Zeyad et al., 2018). The final product was NES, which had a particle size range of 31.09–43.83 and an average of approximately 38.98 nm. Figure 4 depicts the NES XRD test.

Table 1 Cement chemical and physical properties (CEM I 42.5 N)

Type	SiO_2	Al_2O_3	Fe_2O_3	Cao	MgO	SO_3	K_2 o	Cl^-	Loss on ignition	Density g/cm^3	Fineness
Cement	22	4.8	3.2	63.2	1.6	2.5	NA	0.05	2.2	3.18	3300 cm^2/g
Sugarcane	62.43	4.38	6.98	11.8	2.51	1.48	3.53	NA	4.73	1.05	44.75 nm
Eggshell	0.09	0.03	0.02	50.7	0.01	0.57	NA	0.219	0.56	1.23	38.98 nm

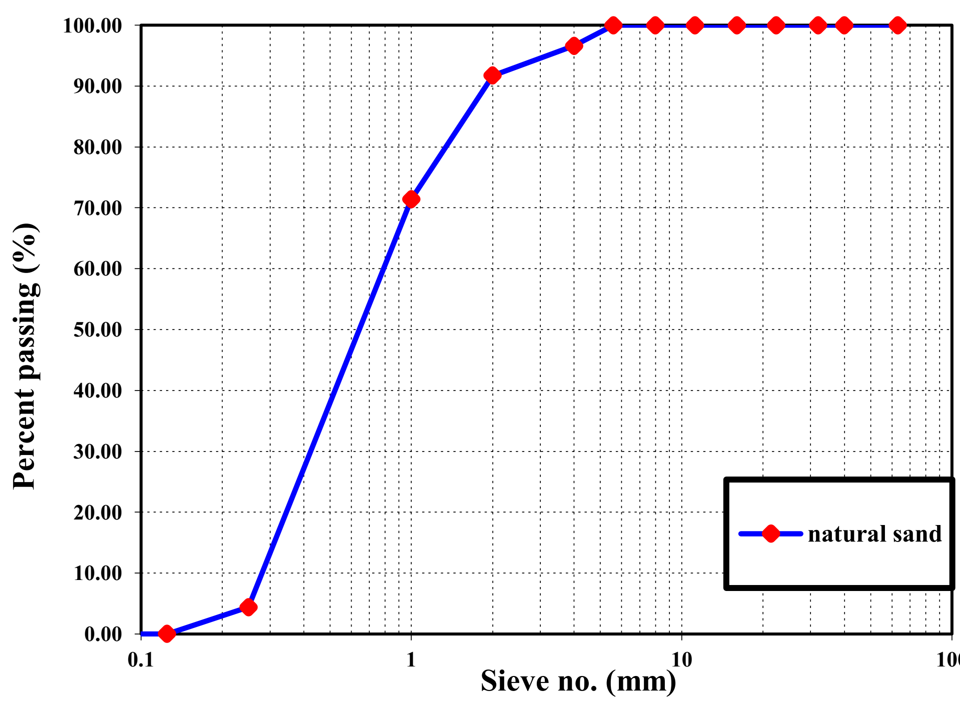
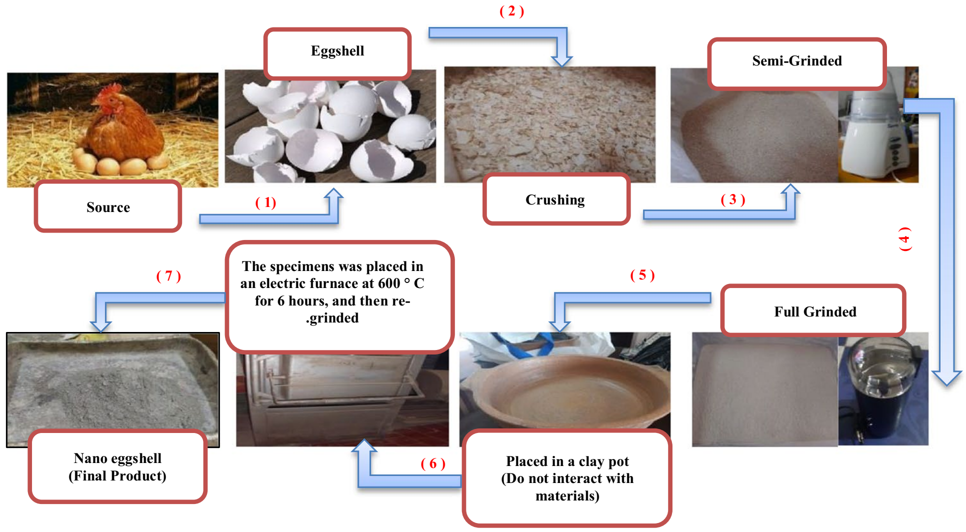


Fig. 2 Sieve analysis diagram of the used aggregate

Fig. 3 Scheme the steps of NES production



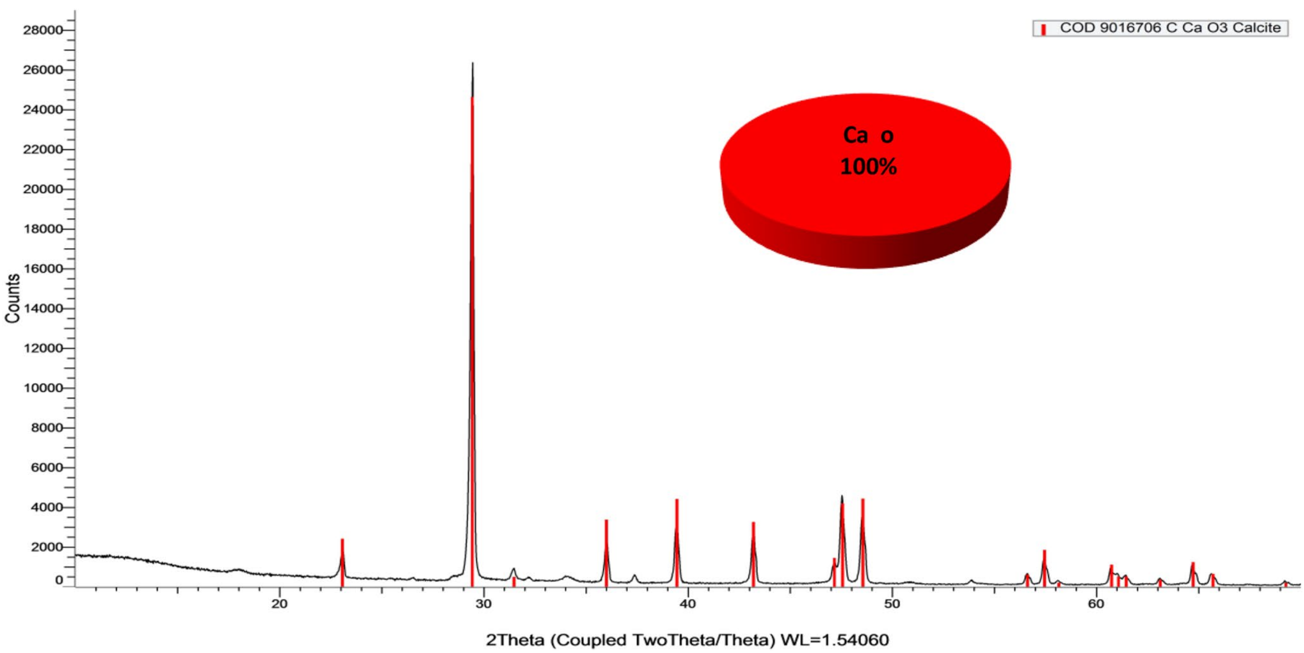


Fig. 4 XRD test of NES

Synthesis of NSC

NSC was collected in its raw form from a sugarcane factory in Qena, Egypt. Raw sugarcane was dried for 1 day in an electric oven at 110 °C and sieved through a 150-mm sieve to remove large and unusual particles including kernels and fibres. The sieved NSC was ground for about 30,000 cycles by the Faculty of Engineering, Civil Department, Sohag University, Egypt, to achieve a fine particle size. To increase sugarcane production by eliminating unburned fuel, the sugarcane was heated to 600 °C for about 4 h in an electric furnace (Johari et al. 2012).

Figure 5 shows that the resulting product, NSC, was subjected to the second stage of sieved using the same Los Angeles machine for another 30,000 cycles to manufacture NSC, and the same method was used in references (Chandara et al. 2010; Islam et al. 2016). The fine particle size of NSC was investigated at Egypt's Centre of Excellence for Advanced Research in Fluid Flow Lab, yielding a particle size range of 8.77–69.78 with an average of 44.75 nm. Figure 6 depicts the XRD test of NSC.

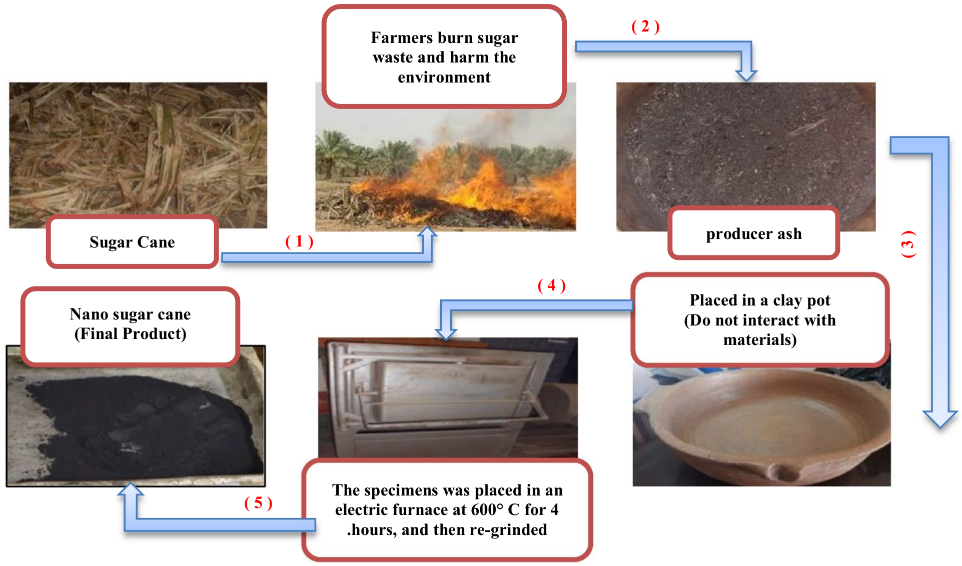


Fig. 5 Scheme the steps of NSC production

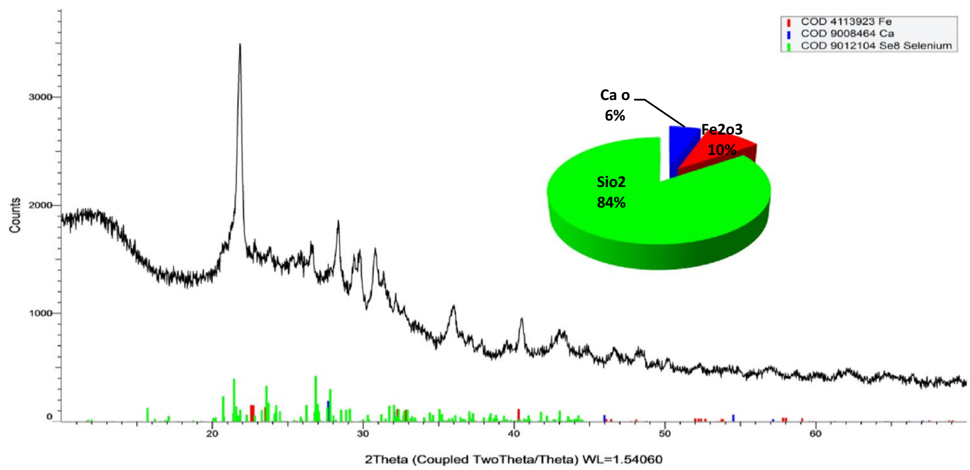


Fig. 6 XRD test of NSC

Characterization of nanomaterial

The quantity and homogeneity of the component as well as the material's performance can be used to judge the success of the chemical synthesis of nanoparticles. Characterization was carried out in the Nanotechnology Centre, National Research Centre, to confirm the development of NES and NSC without using any chemicals that harm the samples; provide some information about its shape, size, surface area, roughness profile and pore size; and confirm the composition of NES and NSC using TEM.

Characterization of NES and NSC by TEM

In electron microscopy, TEM is the most widely used method for characterizing nanomaterials. TEM provides chemical knowledge and photographs of nanomaterials with a spatial resolution similar to atomic dimensions. When an electron beam interacts with a thin foil specimen, the incident light is converted into elastically or inelastically dispersed electrons. The distance between the objective lens, the specimen and the image plane is multiplied by the lens. TEM has more benefits in terms of spatial precision as well as analytical and efficiency measurements. TEM provides knowledge about nanoparticles by using energetic electrons to provide information about morphologic, compositional and crystallographic information in bright-field and dark-field photos. TEM is based on optical microscopy theory. Electrons are replaced by electrons, optical lenses are replaced by electromagnetic lenses, and images are displayed on a screen rather than through an eyepiece. Strong magnification and provision of knowledge about compound and element structures are two advantages of TEM according to the references (Anjum 2016; Kumar et al. 2019). NES and NSC are made up of clusters of nano-sized crystals of particles, as shown in Fig. 7 (sizes are approximately 38.98 and 44.75 nm for NES and NSC, respectively).

Mix proportions

With a proportion of 1:3:0.5 cement to sand to water, the control mix was designed to achieve a fair cement mortar consistency. However, three mixes of group I (2% NSC, 4% NSC and 6% NSC) were prepared with partial replacement ratios of 2%, 4% and 6% of cement by NSC. Another three mixes of group II (2% NES, 4% NES and 6% NES) were prepared to compare the effect of using NES containing 2%, 4% and 6% of NES as a replacement ratio of cement, and group III was divided into three parts as [(25%, 75%), (50%, 50%) and (75%, 25%) of 2%], [(25%, 75%), (50%, 50%) and (75%, 25%) of 4%] and [(25%, 75%), (50%, 50%) and (75%, 25%) of 6%], as shown in Table 2. Sixteen different cement mortars containing different replacement ratios of cement by NSC only, NES only and (NSC + NES) were designed.

Testing procedure

To determine and compare the mechanical properties of cement mortar, the mortars were divided into three series (control, group I with NSC replacement ratios, group II with a replacement ratio of NES and group III with a replacement ratio of NES with NSC).

The ASTM C192-02 standard was followed for the mixed design and curing conditions (ASTM, A., 2013). The consistency of the fresh mortars was determined by conducting 48 tests with the flow table process, which was used to calculate the flow of mortar using the ASTM C1437-15 standard (Standard 2007), and the tests were carried out on three samples, with the average values being calculated. Then, ninety-six cube specimens with dimensions of 100 × 100 × 100 mm were prepared and cured to perform compression tests after 7 and 28 days, and the tests were conducted on three samples, whereby the average compressive strength values were measured, whereas flexural strength tests were performed using 48 prismatic specimens of 40 × 40 × 160 mm using a three-point testing system and tested at a curing age of 28 days according to the BS EN 196–1 standard (BSI 2005), and the tests were carried out on three samples, with the average flexural strength values being calculated, see Fig. 8. The effect of replacement materials on the performance properties of mortars was investigated using microstructure analysis (SEM, EDX and TEM).

Results and discussions

The physical and mechanical properties of the NES and NSC findings were investigated. The experiment's findings, such as accuracy, compressive strengths at 7 and 28 days, flexural power and microstructural analysis, are shown in Table 3.

Fresh properties

Consistency

To reduce permeability, nano sugarcane and nano eggshell modified cement mortar were employed as a surface protection material. This is done by filling the pores between the aggregate grains, and this makes it an impermeable mortar (Supit et al., 2013, Nivethitha et al. 2016). In this study, mixtures were produced having constant water to cementation materials ratio (w/cm) of 0.5 and replacement ratios of cementation materials with NSC and NES of 2%, 4% and 6%. Moreover, the NSC and NES had a lower specific gravity (2.2) than cement (3.2), which resulted in increased powder volume relative to the control sample and low workability for the same amount of water, as shown by the

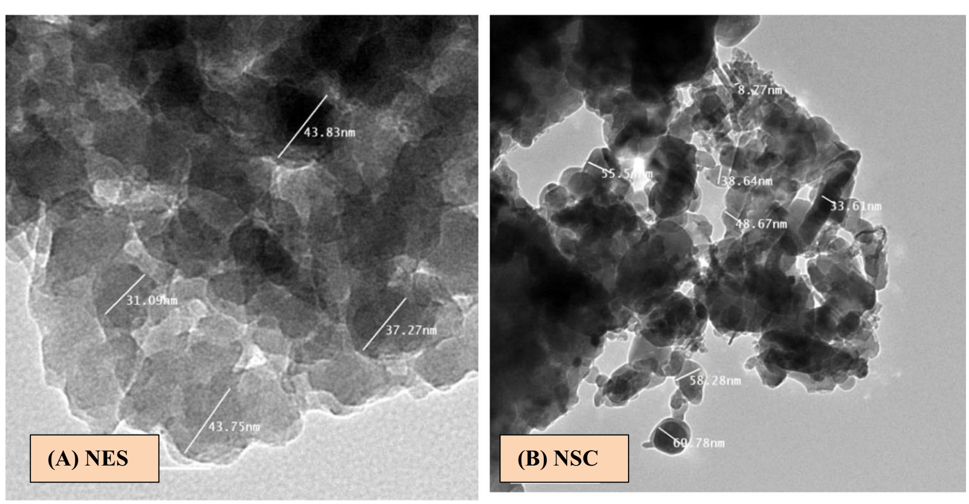


Fig. 7 TEM test for **A** NES and **B** NSC

Table 2 Cement mortar composition (kg/m^3)

Sample				Cement	Sand	Water	NSC	NES
C—0.0%				496.4	1489.2	248.2	0.00	0.00
Group I	NSC—2%			486.47	1489.2	248.2	9.93	0.00
	NSC—4%			476.55	1489.2	248.2	19.85	0.00
	NSC—6%			466.62	1489.2	248.2	29.78	0.00
Group II	NES—2%			486.47	1489.2	248.2	0.00	9.93
	NES—4%			476.55	1489.2	248.2	0.00	19.85
	NES—6%			466.62	1489.2	248.2	0.00	29.78
Group III	[25% of 2% + 75% of 2%]	Eq	2%	486.47	1489.2	248.2	2.48	7.44
	0.5% NES + 1.5% NSC = 2%	Ratio						
	[50% of 2% + 50% of 2%]	Eq		486.47	1489.2	248.2	4.965	4.965
	1% NES + 1% NSC = 2%	Ratio						
	[75% of 2% + 25% of 2%]	Eq		486.47	1489.2	248.2	7.44	2.48
	1.5% NES + 0.5% NSC = 2%	Ratio						
	[25% of 4% + 75% of 4%]	Eq	4%	476.55	1489.2	248.2	4.96	14.89
	1% NES + 3% NSC = 4%	Ratio						
	[50% of 4% + 50% of 4%]	Eq		476.55	1489.2	248.2	9.925	9.925
	2% NES + 2% NSC = 4%	Ratio						
	[25% of 4% + 75% of 4%]	Eq		476.55	1489.2	248.2	14.89	4.96
	3% NES + 1% NSC = 4%	Ratio						
	[25% of 6% + 75% of 6%]	Eq	6%	466.62	1489.2	248.2	7.445	22.335
	1.5% NES + 4.5% NSC = 6%	Ratio						
	[50% of 6% + 50% of 6%]	Eq		466.62	1489.2	248.2	14.89	14.89
	3% NES + 3% NSC = 6%	Ratio						
	[75% of 6% + 25% of 6%]	Eq		466.62	1489.2	248.2	22.335	7.445
	4.5% NES + 1.5% NSC = 6%	Ratio						

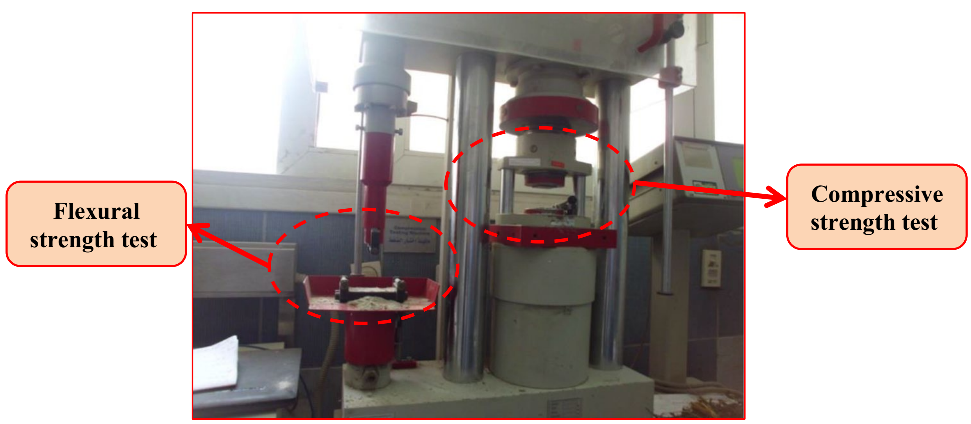


Fig. 8 Compressive and flexural strength machine for mortar tests only

consistency value of the blended cement paste. In this study, lower substitution ratios were used, resulting in no need for additional water, as shown in Fig. 9. The result agreed with Bahurudeen et al.'s findings (Bahurudeen et al. 2015). The consistencies of fresh mortars decreased with using nano-silica (NSC) and nano calcium (NES) when using the same water ratios and increasing the replacement ratios. The consistencies of 2% NSC, 4% NSC and 6% NSC were lower than those of the control mix by approximately 2.70%, 6.76% and 9.46%, respectively. However, the consistencies of 2% NSC, 4% NSC and 6% NSC were lower than those of the control mix by approximately 1.35%, 4.73% and 7.43%, respectively. The shape, surface area, ratios of replacement by nanomaterials and aggregation of the nanoparticles as a direct cause of the decrease in consistency (Meng et al. 2012, Roychand et al., 2016, Nguyen et al. 2017).

The explanation for this is that nano-silica and nano calcium (NES) had a low water demand due to the small size of nanoparticles compared with cement, which reduced the amount of free water available and densified the

Table 3 Characteristics of NSC and NES mortar

Sample				Consistency	Compressive strength at 7 days	Compressive strength at 28 days	Flexural strength at 28 days
				mm	N/mm^2	N/mm^2	N/mm^2
C—0.0%				148	37.50	49.15	8.93
Group I	NSC—2%			144	44.88	58.61	9.52
	NSC—4%			138	42.20	58.25	9.32
	NSC—6%			134	39.32	52.40	9.06
Group II	NES—2%			146	39.25	52.02	8.85
	NES—4%			141	38.86	50.53	9.00
	NES—6%			137	37.6	48.66	8.92
Group III	[25% of 2% + 75% of 2%] 0.5% NES + 1.5% NSC = 2%	Eq Ratio	2%	147	45.2	59.62	9.83
	[50% of 2% + 50% of 2%] 1% NES + 1% NSC = 2%	Eq Ratio		145	43.8	58.35	9.72
	[75% of 2% + 25% of 2%] 1.5% NES + 0.5% NSC = 2%	Eq Ratio		144	43.25	58.1	9.64
	[25% of 4% + 75% of 4%] 1% NES + 3% NSC = 4%	Eq Ratio	4%	140	43.1	58.23	9.45
	[50% of 4% + 50% of 4%] 2% NES + 2% NSC = 4%	Eq Ratio		139	42.5	57.84	9.38
	[25% of 4% + 75% of 4%] 3% NES + 1% NSC = 4%	Eq Ratio		140	41.3	56.8	9.27
	[25% of 6% + 75% of 6%] 1.5% NES + 4.5% NSC = 6%	Eq Ratio	6%	135	39.85	49.8	9.12
	[50% of 6% + 50% of 6%] 3% NES + 3% NSC = 6%	Eq Ratio		133	39.14	48.62	9.06
	[75% of 6% + 25% of 6%] 4.5% NES + 1.5% NSC = 6%	Eq Ratio		130	38.34	48.21	8.93

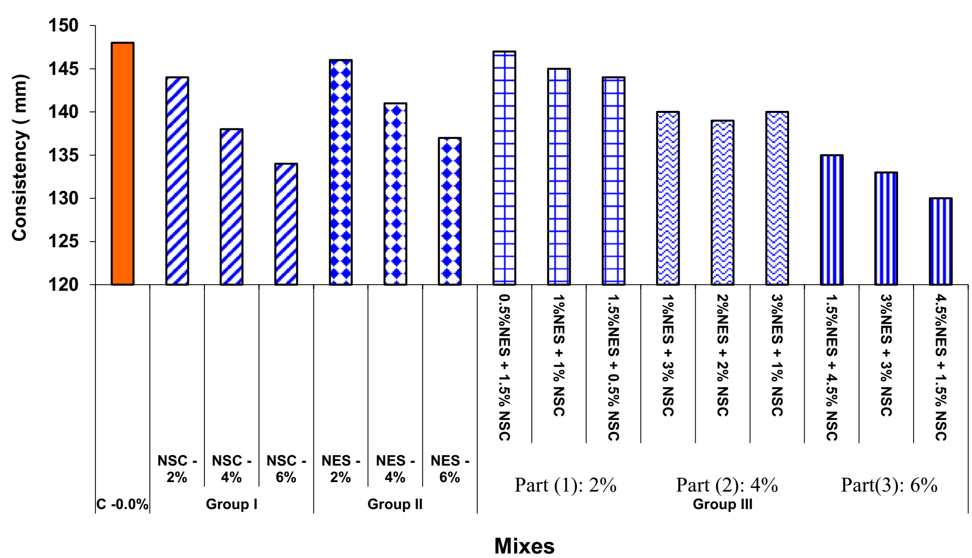


Fig. 9 Constancy values for all mixtures

microstructure of the substituted mixtures (Horszczaruk et al. 2014). NSC required less water than NES in mixtures of NSC and NES. The explanation for this is due to NSC's large surface area, which contributed to increased water demand. More water was needed to maintain that consistency because a binder with a larger surface area needed more water to achieve a specified consistency. Therefore, small replacement ratios were ineffective at this stage, see Fig. 10. This result was in line with the results of other studies (Martins and Bombard 2012; Yu et al. 2014). For group III, the addition of NSC with NES simultaneously in mortar decreased the workability by 12.16% because a portion of the mixing water was absorbed by NSC and NES particles. Water molecules were attracted towards the particles due to the high reactivity. As a result, a higher amount of SP was needed to compensate for the reduction in free water to improve workability at high replacement ratios of more than 10%. Many studies have found that adding high-surface-area mineral particles to cement mixtures necessitates a higher amount of SP to preserve workability (Barkoula et al. 2016; Chithra et al. 2016; Li et al. 2017; Andrei et al. 2021).

Hardened properties

Compressive strength

Three 100 × 100 × 100 mm cubes were tested at curing ages of 7 and 28 days for each mix in a compressive strength test. The addition of NSC to the mortar mix increased the compressive strength of mortar at an early age and strengthened it at a later age, as shown in Fig. 11. The results can be clarified by the high pozzolanic reaction of NSC compared

Fig. 10 Reduction in constancy (%) for all mixtures

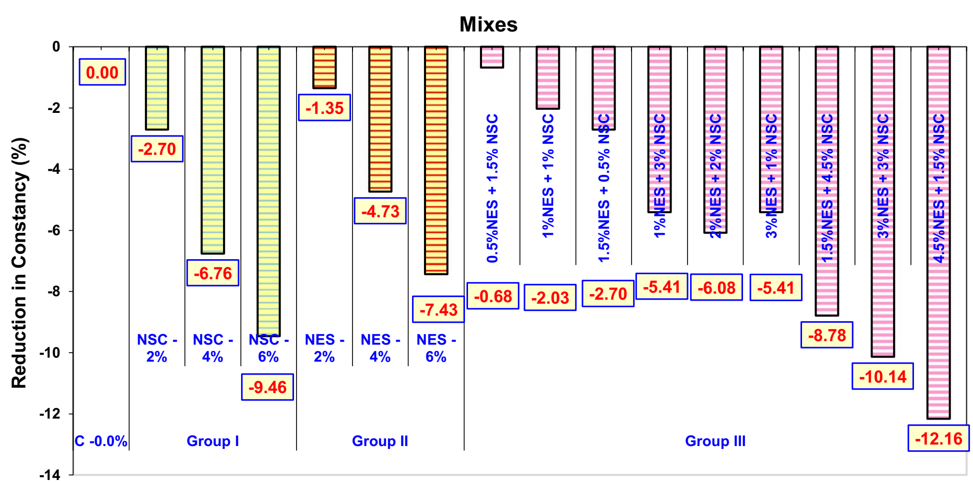
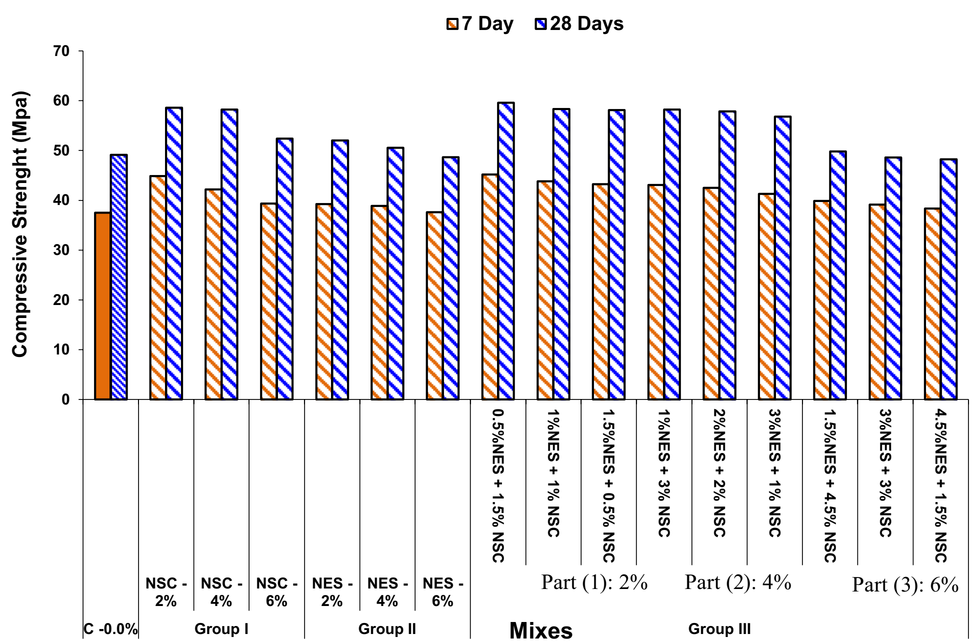


Fig. 11 Compressive strength of mortar containing NSC and NES at 7 and 28 days



with that of NES, which lacked silica and alumina required to form C–S–H gels (Chandara 2011; Nadziri et al. 2018). By contrast, NES had a high content of CaO, which was required to prepare extra C–S–H gels (Pliya and Cree 2015; Jhatial et al. 2018). The 7-day compressive strengths of 2% NSC, 4% NSC and 6% NSC were higher than those of the control mixture by approximately 19.68%, 12.53% and 4.85%, respectively, and compressive strengths at 28 days for the same mixtures were increased by 19.25, 18.51 and 6.61 MPa, respectively, as presented in Fig. 2. Consequently, all NSC mortar mixes improved at 7 and 28 days compared with 0.0% C. Zhang et al. (2020) proved that compressive strength decreases due to an increase in the cement replacement by NSC, in particular after the addition of 2%. Compressive strength for NSC mix increases after 7 days by ratios up to 38.03% at an age of 28 days. Previous studies (Lim et al. 2015; Zeyad et al. 2017, Hamada et al., 2020) concluded that the decrease in compressive strength values at an early age becomes noticeable with a rising degree of cement replacement by NSC because the pozzolanic material of NSC can be highly active at a late age. After all, the pozzolanic material of NSC can be highly active at a late age. Nonetheless, the majority of previous research has shown that integrating NSC with small particle size into mortar results in high strength at a late age.

The 7-day compressive strengths of 2% NES, 4% NES and 6% NES were higher than those of the control mix by approximately 4.67%, 3.63% and 0.27%, respectively. On the 28th day, the compressive strengths of 2% NES and 4% NES were increased by 5.84% and 2.81%, respectively; as an exception, that of 6% NES was lower than those of the control mix by 0.99%, as presented in Fig. 12. Compressive strength was reduced because NES had a high quantity of $CaCO_3$ (more than 90% of the total content) (Murakami, Rodrigues et al. 2007; Hernández-Hernández et al. 2008). Binici et al. (2014) discovered that compressive strength reduction at all ages, especially in the first days, occurs when NES is integrated into fine aggregate in cement mortar compared with NSC mixtures. They used NES as a cement replacement by 2%, 4% and 6% in cement mortar and found that compressive strength slightly increases when ESPs are added to the mortar at all levels compared with control or NES mixture. Yerramala (2014) used NES due to its high content of CaO_3 to improve the compressive strength of cement mortar. They found that CaO_3 enhances the mechanical properties of cement mortar at a late age. Because of the quick creation of the C–S–H gel, which aided in cement hydration, mixtures having nanomaterial replacement have decreased porosity. Overall, nanoparticles lowered the amount of voids dramatically (Salman et al., 2020, Gopalakrishnan and Kaveri 2021, Khalil et al., 2021, Tosee et al. 2021).

Finally, when using NSC and NES together, the pozzolanic reaction for SiO_2 and CaO3 caused the compressive strength of the mortar containing NSC and NES to increase over time. The 7-day compressive strengths of group III mixtures were higher than those of the control mix by approximately 20.53%, 16.80%, 15.33%, 14.93%, 13.33%, 10.13%, 6.27%, 4.37% and 2.24%. However, the 28-day compressive strengths of the same mixtures were increased by 21.30%, 18.71%, 18.20%, 18.47%, 17.68%, 15.56% and 1.32%, except that those of 3% NES + 3% NSC and 4.5% NES + 1.5% NSC mixes were lower than those of the control mix by 1.07% and 1.91%, as presented in Fig. 12. According to these studies, the compressive strength of mortar mixes containing NSC with NES together increases at the early age and then increases gradually in the late age. In general, this study showed that

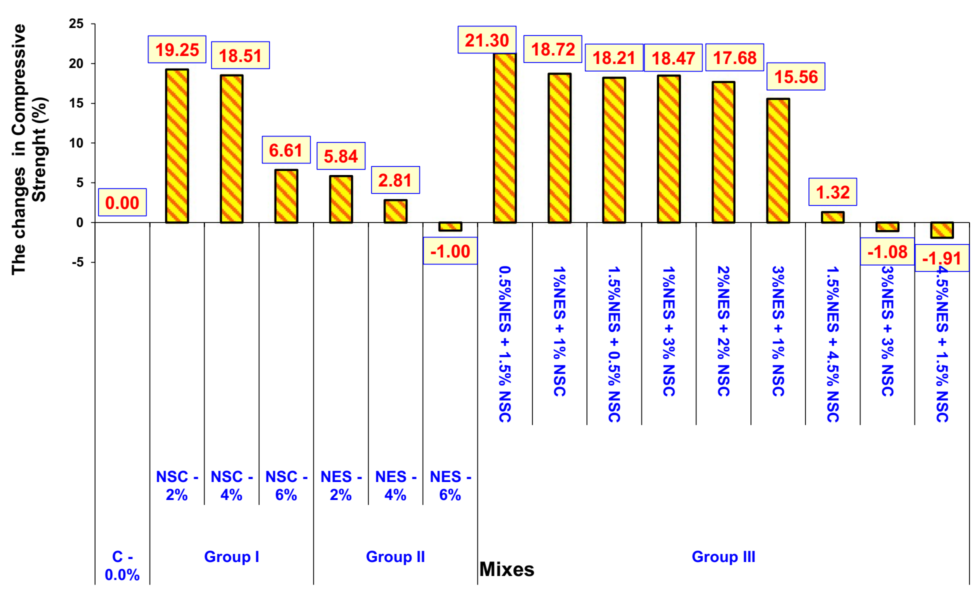


Fig. 12 The changes in compressive strength (%) at 28 days

NSC with NES can be used as reinforcement in mortar composites with an optimum dose of 2%.

Flexural strength

Flexural strength acts in the same manner as compressive strength. Compared with the control specimen, Fig. 13 shows that incorporating NSC, NES and NES + NSC increases flexural strength. Compared with NES mortars, the addition of NSC mortar improved flexural strength even more. Figure 13 exhibits the increase in flexural strength for 2% NSC, 4% NSC and 6% NSC by 6.61%, 4.37% and 1.46%, respectively, compared with the control specimen. However, a decrease in flexural strength was observed for 2% NES and 6% NES by 0.89% and 0.11%, respectively, compared with the control specimen, except for 2% NES whose flexural strength slightly increased by 0.78%. Mixing NSC with NES in the mortar recorded higher results than the control mixture, and blending 0.5% NES + 1.5% NSC revealed the highest flexural strength of value 9.83 MPa and increased by 10.08% of the control mixture, which confirmed the similarity between compressive strength and flexural strength. The findings were consistent with those of many previous studies (Dhengare et al. 2015, Parthasarathi et al. 2017, Gabol et al., 2019, Khalafalla 2019, Abo-alnour et al., 2020, Owuamanam and Cree 2020, Sun et al. 2020). Flexural strength is attributed to fast consumption of the $Ca(OH)_2$ that is produced during Portland cement hydration, especially at early ages. This fast consumption is due to strong reactivity of the nanoparticles (Amin and K. Abu el-Hassan, , 2015; Wu et al. 2020; Gamal et al. 2021).

Figure 14 shows the flexural strengths of mortar mixes with and without nano and the flexural/compressive (F/C) ratios. The F/C ratios were 16.24%, 16.0% and 17.29% for 2% NSC, 4% NSC and 6% NSC, respectively, and 17.01%, 17.81% and 18.33% for 2% NES, 4% NES and 6% NES.

NES with NCS mixtures.

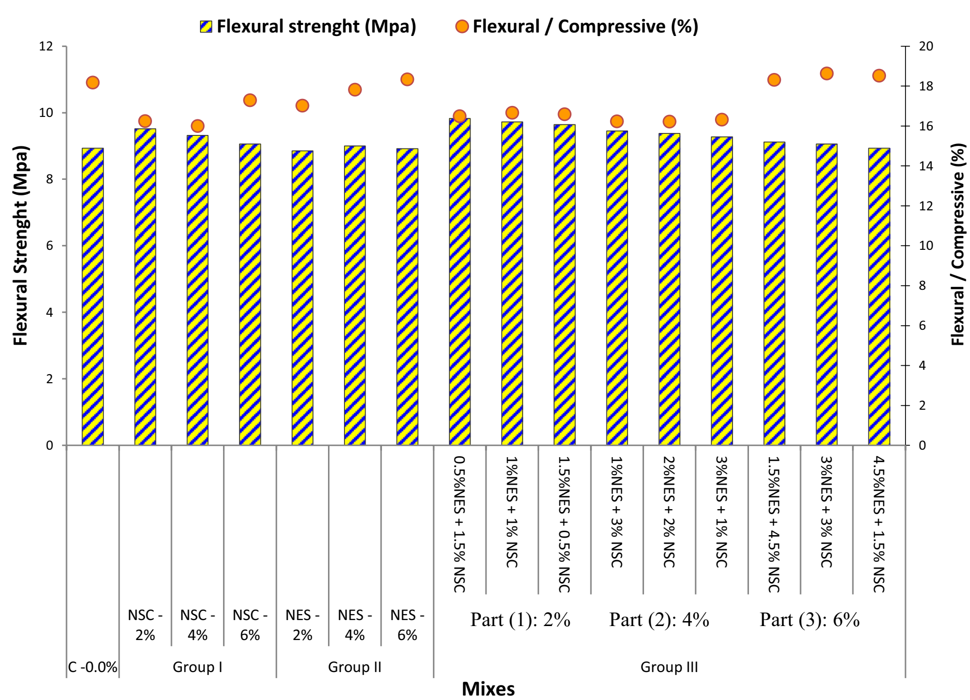
The F/C ratios for 1.5% NES + 4.5% NSC, 3% NES + 3% NSC and 4.5% NES + 1.5% NSC were increased by 18.31%, 18.63% and 18.52% compared with the control, and the rest of the mixtures for group III decreased by 16.49%, 16.66%, 16.59%, 16.23%, 16.22% and 16.32%, for the control mixture. However, the majority of previous studies (kumar Jha, Sanodiya et al., Sundaravadivel and Mohana, Al-Mansour et al. 2019, Cordeiro et al., 2019, Xu et al. 2019, Indrasti et al., 2020) showed that integrating NSC and NES with small particle sizes into mortar results in high flexural strength at 28 days of age.

Analysis of microstructure

SEM and energy dispersive X-ray test of nano mortar

The SEM/EDX results of NES and NSC specimens are shown in Fig. 15. SEM is a critical test for determining the nanomaterial's final form and morphology. In this analysis, NSC and NES were improved in several stages, including grinding sieved SC and ES using a machine to obtain SCP and ESP, heating SCP and ESP in an electrical furnace to

Fig. 13 Flexural strength of all mixtures at 28 days



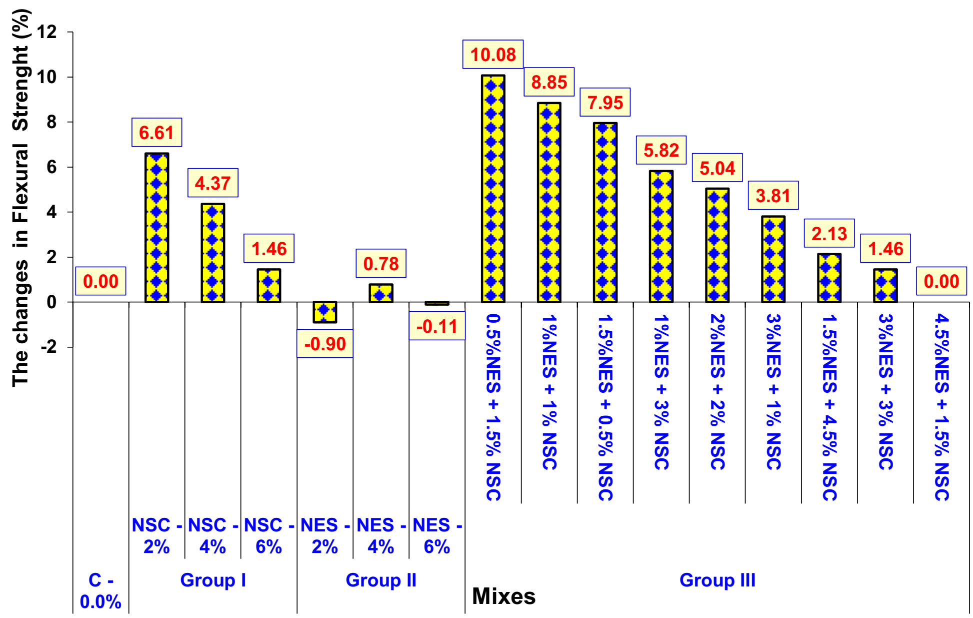


Fig. 14 The changes in flexural strength (%) at 28 days

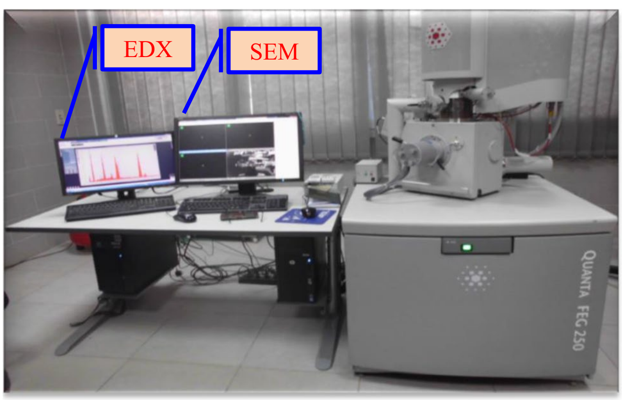


Fig. 15 SEM and energy dispersive X-ray device

obtain more fineness for SCP and ESP, and other grinding SCP and ESP to obtain NSC and NES. The results of SEM/EDX indicated that the particles had a porous texture, and the shape ranged between semicircular and angular. The particle size of NES was smaller than that of NSC by 38.98 and 44.75 nm for NES and NSC, respectively. The microstructure and morphology of interfacial transition zones of cement paste (control, 2% NES, 2% NSC and 0.5% NES + 1.5% NSC) were investigated. The microstructure of the 0.5% NES + 1.5% NSC specimen was less porous and had fewer cracks compared with that of control, 2% NES and 2% NSC specimens. By using NES, microvoids were decreased due to the high percentage of SiO_2 in the NSC and the low addition percentage of $CACO_3$. The key binding step of Portland cement-based materials is the C–S–H gel. However, due to the presence of crystal-rich layers of C–H gel, the contact point between the C–S–H gel NES and NSC increased, positively affecting the latter's strengthening and crack resistance effect, as shown in Figs. 16 and 17. Non-evident cracks between the nano and cement paste indicated the presence of a chemical reaction between them. This finding was more evident in 0.5% NES + 1.5% NSC. An EDX test was performed to determine the quantitative composition of the materials. The physical properties and the chemical compositions of NES and NSC changed owing to the treatment method. The results of the EDX test showed that the chemical compositions of NES had different concentrations compared with those of NSC. SiO_2 and CaO_3 had the highest concentrations amongst the oxides, and this finding agrees with the reference (Hernández-Hernández et al. 2008). Nanomaterials help to eliminate small pores and reduce large pores to a large extent and open the new field for the use of nanomaterials in the restoration and reinforcement of concrete structures (Behnia et al., 2021, El-Midany et al. 2021; Castro-Rojas et al. 2021, Cuenca et al., 2021). This depends on the amount of nanomaterials added or replaced to cement.

Conclusion

The mechanical properties of mortar containing NSC and NES were evaluated in this report, and the following conclusions were reached.

The TEM results of NES and NSC showed that their particle sizes were 38.98 and 44.75 nm, respectively, and they could be used to improve the internal structure and durability of mortar.

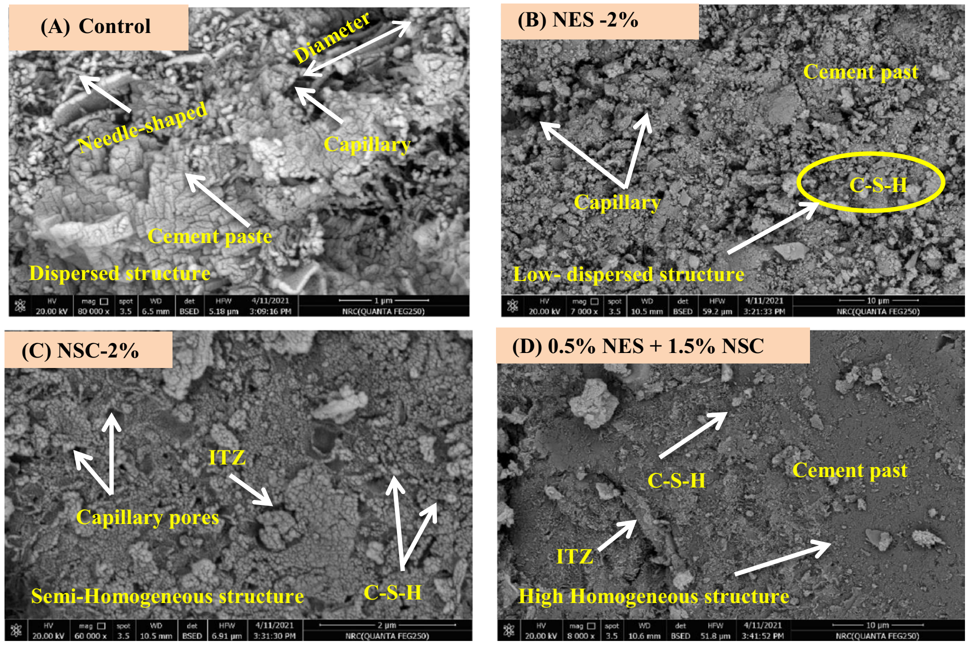


Fig. 16 SEM test for mixes: **A** control, **B** 2% NES, **C** 2% NSC and **D** 0.5% NES + 1.5% NSC at 28 days

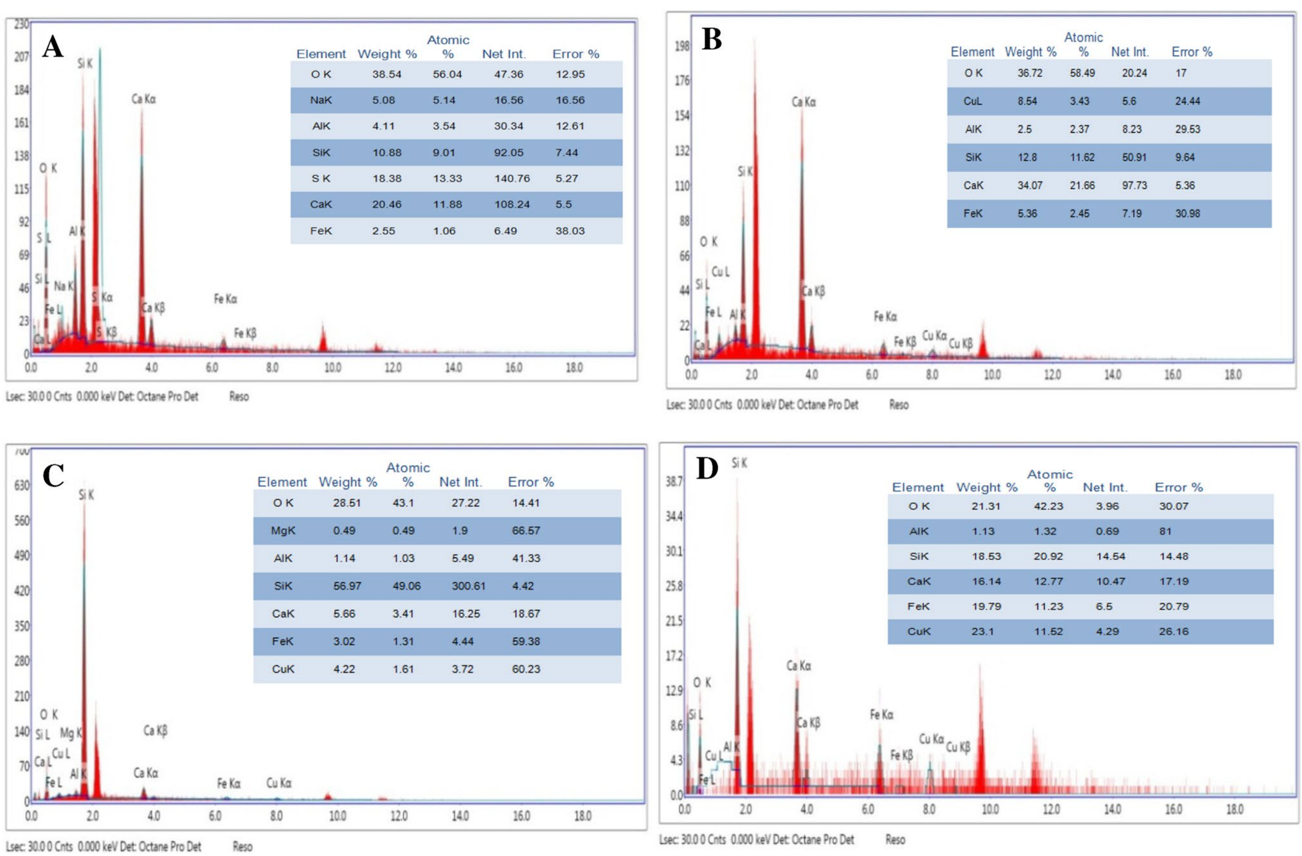


Fig. 17 EDX test for mixes: **A** control, **B** 2% NES, **C** 2% NSC and **D** 0.5% NES + 1.5% NSC at 28 days

Using nano sugarcane (NSC) and nano eggshell (NES) reduced the consistency of fresh mortars and NSC needed more water than NES by 9.46% and 7.43%, respectively.

The compressive strengths of NSC and NES were high at an early age, particularly after 7 days of mortar curing. After 28 days, compressive strength gradually increased, especially at replacement levels of 2% cement with NSC and NES contents.

NSC with NES was effective in the case of compressive strength. However, the addition of 2% (0.5% NES + 1.5% NSC) positively affected the strength of specimens due to the high reactivity for both, and the improvement in compressive strength reached 21.3%.

A high flexural strength value was achieved due to the replacement of cement by 2% (0.5% NES + 1.5% NSC) by 10.08% when compared to the control sample. Flexural strength behaved similarly to compressive strength.

The SEM/EDX results of NSC and NES showed that their particle size was smaller than that of cement. The main components were SiO_2 and $CACO_3$.

The microstructure (SEM/EDX) of the 0.5% NES + 1.5% NSC was less porous and had fewer cracks compared with that of control, 2% NES and 2% NSC, and microvoids were reduced due to the high percentage of SiO_2 in NSC with low addition percentage of $CACO_3$ by using NES.

Nanomaterials help to eliminate small pores and reduce large pores to a large extent, and this depends on the amount of nanomaterials added or replaced with cement.

In summary, NSC had substantial effects on mortar properties, especially at a late age. NES increased the mechanical properties but within acceptable limits. Thus, these materials can be used to improve the sustainability of mortar and materials by using renewable by-product materials.

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Author contribution Radwa Defalla Abdel Hafez: conceptualization, methodology, investigation, formal analysis, visualization and writing—original draft.

Bassam A. Tayeh: writing—review and editing.

Khaled Abdelsamie: writing—original draft.

Data availability Not applicable.

Declarations

Ethics approval and consent to participate. Not applicable.

Consent for publication. Not applicable.

Competing interests The authors declare no competing interests.

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