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Effect of Nano Silica Waste as Supplementary Additive Cementitious Materials in Cement Mortar Industry in Jordan

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Abstract

This present work investigates the possible effect of adding the recycled nano silica waste (NS) on the workability, density, heat of hydration, and mechanical behavior and properties of industrial cement mortar. The nano silica was collected from the aluminum fluoride plant of Jordan Phosphate Mine Company. Ten mixtures of cement mortar were prepared with nano silica to cement ratios of 0%, 0.5%, 1.5%, 2%, 2.5%, 4%, 6%, 8%, and 10%. The cement mortar and NS chemical compositions are analyzed using the X-ray Fluorescence Spectrometer technique. Flow table test is adopted to examine the workability of fresh mortar specimens after mixing. The average dry and bulk densities, in addition to compressive and flexural strengths, for different concentrations of nano silica mortar samples, particularly at a duration of 28 days, have recorded a significant improvement. The results indicate an improvement in the compressive and flexural strengths as nano silica increased in the mortar mixture.

Keywords:

Cement, Compressive and flexural strength, Mortar, Nano silica waste, Workability.

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1. Introduction

Solid waste materials represent a challenging environmental concern, particularly with the increasing demand for various production materials. The unplanned management of waste material escalating the environmental risk and hazards sharply. Dumping solid waste in authorized dumpsites such as landfills should be subjected to more control and examination. Many unauthorized dump sites, such as side drains, roadsides, and land spaces within the inhabited areas, lead to severe effects in the long and short term due to the gathering of solid waste in the surrounding environment, affecting ambient air, groundwater, soil, and vegetation.

Clearance and treatment of solid wastes conserve the environment and thereby human life and health. Unlimited serious issues may arise due to accumulated solid waste materials; therefore, it is of great concern worldwide. Several efforts have been reported by many researchers to recycle solid waste materials into useful materials, as input in a wide-ranging manufacturing process, as a method of decreasing the solid waste accumulation (Hou et al., 2013; Abd El-Aleem et al., 2014; Zhang et al., 2024). Pozzolanic materials are widely used as supplementary cementitious materials in mortar. Silicon dioxide is one of the most pozzolanic materials, which can be found as natural or waste material in many countries. Consequently, nano silica waste, which contains a large amount of silicon dioxide, is chosen to be used as a supplementary cement material during this study. NS waste replacement as a cementitious material can improve the mechanical properties of cement and mortar composites.

Durability, effectiveness, and cost reduction are important factors that control the suitability of any construction material. Hou et al. (013) investigated the potential of modifying mortars with NS material replacement at levels of 0%, 2.25%, and 5%, in order to measure the compressive strength. The achieved results indicated that the compressive strength increased for 2.25% and 5% NS-added material mortars, showing higher compressive strength than that of the control mortar samples at all periods up to 28 days. Therefore, it indicates a significant improvement in the mechanical properties with additional NS compared to the plain cement mortar. After 28 days, the 0% and 5% NS mortars showed a similar compressive strength (Hou et al., 2013).

The possible effect of adding NS on the microstructure of cement mortars was subjected to study. Ordinary Portland cement was replaced with 6.0% NS with an increment of 1.0%. The NS average particle size used is 15 nm at a water-binder ratio of 0.25 with 1.0% of superplasticizer. The results of thermal expansion, bulk density, and compressive strength indicated improvement of the various mechanical properties of cement mortars by adding NS up to 5.0% by mass (Abd El-Aleem et al., 2014; Haruehansapong et al., 2014; ASTM C1437, 1999; EN 1015-11, 1999).

Adding an appropriate amount of NS to the concrete can enhance the interface strength and refine the pores, which can effectively reduce the water permeability of concrete (Liu et al., 2019). Nano-silica can also optimize the microstructure of recycled concrete.

Nano-silica particles interact with freshly mixed concrete to alter its workability, setting time, consistency, and other properties (Makarova et al., 2017). The latent phase of setting time is reduced by around 1-2% with the introduction of NS; however, the actual setting time would still be between 90 and 100 min (Erdem et al., 2018).

Hydration properties comprise different properties such as water-holding capacity (WHC), water mobility, or osmotic pressure. WHC is defined as the ability of food ingredients or foods to hold water during the application of osmotic pressure or centrifugation (Robertson et al., 1981).

Silica nanoparticles are also called silicon dioxide particles, and they could be used as additives to enhance concrete's mechanical and durability characteristics (Kumar and Komarasamy, 2016; Fallah and Nematzadeh, 2017; Rashad, 2019). The influence of nano-silica on the nanostructure of binder pastes also enhanced the durability of concrete (Smirnova, 2018a; Smirnova, 2018b; Saidova et al., 2021).

The microstructure photographs and the compressive strengths of NS-containing cement mortars with variable sizes of 12- 40 nm were presented. Cement was partially replaced with different ratios of NS ranging from 3% to 12% by weight. The sand/binder and water-binder ratios were fixed at 2.75 and 0.65 for all the mixtures, respectively. Flexural strength increased by 28% and 19.2–27% at 7 and 28 days, respectively. SEM images showed that the nanoparticles acted as a filler and activator to enhance the hydration process and to develop the microstructure of the cement mortar if the nanoparticles were dispersed uniformly.

Test results indicated that NS addition significantly enhanced the compressive strength of the examined cement mortar, taking into consideration that the strength improvement was NS particle size-dependent. The cement mortar containing 40 nm size NS gave a higher compressive strength compared with 12 nm and 20 nm NS, probably due to their agglomeration and ineffective dispersion (ASTM C348-1997; Haruehansapong et al., 2014; EN 16-1, 2016; Al-Zboon, 2018; Al-Mousa and Al-Zboon, 2022).

This work aims to study the physical and mechanical performance of ten mortar mixtures with NS waste replacement ratios designated as 0%, 0.5%, 1%, 1.5%, 2%, 2.5%, 4%, 6%, 8%, and 10% of cement weight.

2. Experimental Work

A. Chemicals and types of instruments used

A Philips 3040/60 X-ray fluorescence spectrometer (XRF) is used with automated sample operation at 40 kV and 20 mA, with an X-ray wavelength of 1.54056 Ao, scanning for 2Ө from 3° to 83° at a speed of 2°/min and a step size of 0.05°. Scanning Electron Microscopy (SEM) is used with a Dual Beam combines versa 3D microscope at an operating voltage of 1 kV and a resolution of 7 nm at 30 kV at beam coincident point. Compression and Flexural Testing are conducted using the MATEST hydraulic machine C108N with a capacity of 3000 kN, ± 0.5% load accuracy, starting loading of 0.54 kN/sec until failure, for hydration test. TAM AIR isothermal calorimetry instrument with accuracy limit of ± 0.15 ºC, operating range of 5 ºC to 90 ºC and detection limit of 8 µW is used. All cement mortar experiments are conducted in the concrete lab at Yarmouk University of Jordan. Portland cement (type I 42.5N), collected from different cement companies in Jordan, recorded 3.26 g/cm^3 specific gravity and 2.03 fineness modulus, complying with Jordanian Standard Specifications (JSS 30-1/2007) and European Standard (EN 19-1/2000), is used as a binder material. The analysis results of the cement oxides using XRF techniques for cement are given in Table (1).

Table 1: XRF results of cement compositions and percentages.

Parameter	Chemical Name	Percentage (%)
CaO	Calcium oxide	65.10
SiO_2	Silicon dioxide	16.20
Al_2O_3	Aluminum oxide	9.60
Fe_2O_3	Ferric oxide	9.10
LOI	Loss on ignition	1.0

To prepare the mortar mixes, locally available natural sand was collected from a quarry located at the Jerash Governorate, north of Jordan; the collected sand materials show a fineness modulus of 3.94, water absorption of 0.71, and a specific gravity of 2.65g/cm^3, which complies with Jordanian Standards (JSS 96/1987).

Waste nano silica, with a density of 1.38 g/cm^3 and an absorption ratio of 65%, was collected from the aluminum fluoride plant of Jordan Phosphate Mine Company (Aqaba-Jordan) and used as the pozzolanic material during the current study. NS oxides and compounds were analyzed using an X-ray fluorescence spectrometer (XRF). The results are shown in Table 2, indicating the predominance of SiO_2. The water used is clean and fresh potable water.

Table 2: XRF results of NS waste compositions and percentages.

Composition	Chemical Name	Percentage (%)
SiO_2	Silicon dioxide	78.10
Al_2O_3	Aluminum oxide	4.40
Fe_2O_3	Ferric oxide	4.05
LOI	Loss on ignition	13.45

B. Preparation of Mortar Specimens

Mortar samples were prepared based on cement/sand and cement/water ratios of 1:2.75 and 1:2 by weight, respectively (EN 1015-11). NS waste with 0%, 0.5%, 1%, 1.5%, 2%, 2.5%, 4%, 6%, 8%, and 10% proportions by weight of cement replacement was used for preparing the cement mortar cubes and prisms as given in Table (3).

Table 3: Proportions of the prepared mixtures.

Mortar Designation		Mix proportion				
		Cement (gm)	Sand (gm)	Water (gm)	NS Designation	
					NS/C (%)	Weight (gm)
Cubes	S 0	600	1650	300	0	0
	S 0.5	600	1650	300	0.50	3
	S 1	600	1650	300	1	6
	S 1.5	600	1650	300	1.5	9
	S 2	600	1650	300	2	12
	S 2.5	600	1650	300	2.5	15
	S 4	600	1650	300	4	24
	S 6	600	1650	300	6	36
	S 8	600	1650	300	8	48
	S 10	600	1650	300	10	60
Prisms	S 0	1230	3382.5	615	0	0
	S 0.5	1230	3382.5	615	0.50	6.15
	S 1	1230	3382.5	615	1	12.3
	S 1.5	1230	3382.5	615	1.5	18.45
	S 2	1230	3382.5	615	2	24.6
	S 2.5	1230	3382.5	615	2.5	30.75
	S 4	1230	3382.5	615	4	49.2
	S 6	1230	3382.5	615	6	73.8
	S 8	1230	3382.5	615	8	98.4
	S 10	1230	3382.5	615	10	123

All the mortar samples were mixed using a rotary mixer with a beater to achieve homogeneity all. Initially, the cement and sand were mixed in dry form thoroughly for 2-3 minutes; the NS was added to the mixture and mixed again for 3 minutes. Flow table measurement was performed immediately after mixing was completed (ASTM C1437-1999; EN 1015-11, 1999). Thereafter, the mortars were cast in 5 x 5 x 5 cm cubes for all mixtures to test the density and the compressive strength (EN 1015-11, 1999; Li et al., 2004; Meral and Remzi, 2004; Gopinath et al., 2012; EN 16-1, 2016; How-Ji et al., 2019). Nine Prisms specimens have been prepared (4 x 4 x 16 cm) for each mixture to conduct the flexural testing (modulus of rupture) at the same ages as the compression test (ASTM C348, 1997). A vibrator table was utilized to achieve the desired compaction. The samples were demolded after 24 hours and cured at 23 ± 2 °C in fresh water until reaching the required testing age. Triplicate sample techniques were implemented in all tests to achieve the maximum precision and accuracy (±1 σ).

Hydration heat every 15 minutes for the first 24 hours after casting was determined for mortar samples, as shown in Fig. 1. The mortars were cast in 50-mm test cubes. To measure the hydration heat, DM 6801A and digital thermometers connected to thermocouples were used. The instruments were inserted inside the mortar cube in the middle at a depth of about 2.5cm. The time elapsed after the first contact between the cement and water was measured. Software was implemented on a camera to record the measurements during 24 hours after casting.

3. Results and Discussion

A. Workability

Flow diameter measurements are used to measure the workability of all mortar specimens (ASTM C1437-1999; Said et al., 2012; Danute et al., 2012; Chenglong and Yu, 2019). Figure (1) shows the average diameter variation of the flow table test for mortars containing NS waste.

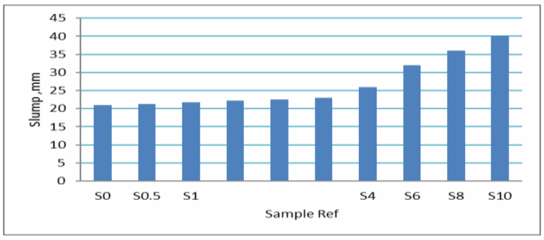

Fig. 1: Mortar average flow diameters with NS waste contents.

It is worth mentioning that NS addition increased the average spread diameter on the flow table of the mortars. An increase in workability was observed from 0.5% NS waste substitution to 10% NS waste, which confirmed the maximum workability among all mixtures. NS waste contents behaved as a cementitious material, which reduced the cement content in the mortar mixture and led to an increase in the amount of lubricating water available in the mixture with the increase in NS waste ratio. The higher workability with a higher NS content could be explained by the following: the effect of a high NS content on workability is greater than the effect of using mixing water to coat the higher NS content, which results in higher workability. This occurs when there is a high content of NS because the particles repel one another, which increases the amount of water between them and improves workability (Ammar, 2012; Al-Mousa and Al-Zboon, 2022).

B. Heat of hydration

Figure (2) represents the influence of NS waste contents on the hydration heat of fresh mortars. The heat of hydration curve is found to have gone through four stages: first increasing, then decreasing, then increasing once more, and lastly decreasing. This result is in line with the research conducted by Al-Mousa and Alzboon (2022), who attributed this behavior to the production of distinct products at each phase. After 5.5 hours, the hydration rate dropped by 0.2, 0.6, and 1.1 mW/g for S2, S6, and S10, respectively, compared to the control sample. After 24 hours, the drop was 0.3, 0.5, and 0.6 mW/g, respectively.

The results indicate that the control specimens gave the optimum peak of heat value. After 24 hours, with increasing NS content, the heat value decreased to the minimum among all specimens due to its agglomeration and ineffective dispersion and decrease of cement contents (Al-

Mousa and Alzboon, 2022). Numerous factors, including cement composition, temperature, NS size, chemical admixtures, cement type, physical properties, curing conditions, and w/c ratio, could be responsible for this decrease in heat of hydration as NS contents increase (Mehta and Monteiro, 1993).

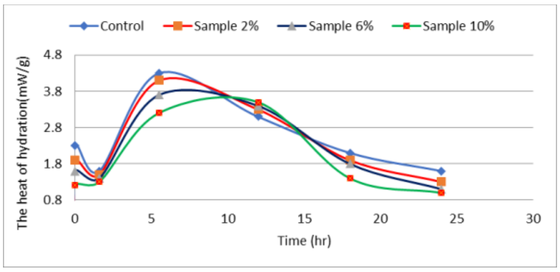

Fig. 2: Heat of hydration variations with NS waste contents.

C. Density

Figure (3) shows the bulk density and average dry density for different NS waste ratios after 28 days of curing. The average densities declined as the NS waste content increased. The fresh density decreased by 10, 10.8, and 13.8% with NS contents of 2, 6, and 13%, respectively. Similarly, the dry density decreased by 13.4, 13.4, and 17.5% for the NS contents, respectively. This is probably because the NS has a lower density than other constituents. Additionally, the addition of NS reduces the microstructure's density, which lowers the mixtures' fresh and dry unit weights (Ji, 2005).

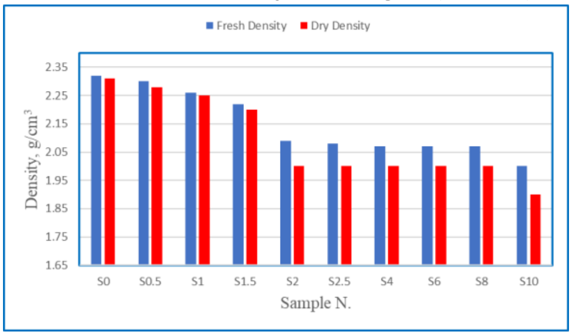

Fig. 3: Mortar average dry and fresh densities with NS waste contents.

D. Compressive and flexural strengths

Fig. 4 represents the average compressive strength results for different NS ratios as a function of curing time. It highlights that the compressive strength is improving due to additions of NS waste at the ages of 7 and 28 days. Both ratios of 8% and 10% enhanced the compressive strength by 15% at all curing ages. Li et al. (2004) found that adding 10% NS to cement mortar increased its compressive strength by up to 20%. They suggested that the increase in compressive strength is ascribed to NS's function in packing and filling the mixes' pores, which produces a strong resistance to the compressive force. Stefanidou et al. (2012) found that at low NS contents, cement paste's compressive strength could be improved by up to 25% after 28 days, and the optimum result is obtained at 0.5% NS. Additionally, Berra et al. (2012) demonstrated a 7-11% increase in compressive strength after seven days, and a replacement ratio of 0.8% yielded the optimum compressive strength. Jo et al. (2007) found that adding 6% NS to cement mortar resulted in a greater increase in compressive strength (about 64%) after 28 days.

The slight decrease in the compressive strength for S6 (0.18 MPa) could be attributed to the small difference in silica content between S4 and S6 (9.0 grams).

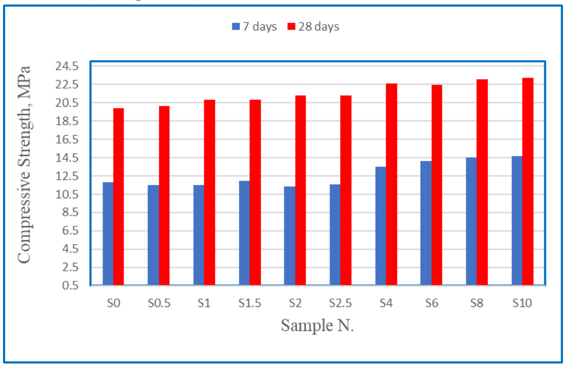

Fig. 4: Average compressive strength test results for different NS waste contents.

Fig. 5 illustrates flexural strength test results and the behavior for different contents of NS waste mortar prisms. The flexural strength shows an insignificant trend with a mostly positive impact. The flexural strength increase (+)/decrease (-) is: 29%, 11.8%, -6%, 30%, 19% for NS ratios of 2%, 4%, 6%, 8%, and 10%, respectively. Except for S56, it is noticed that the flexural strength is improved with additional NS waste contents at the duration of 28 days. The results show that NS waste acted as an activator to enhance the hydration process and to develop the microstructure of the cement mortar. The replacement of NS waste slightly promoted the strength of cement mortar, especially at early ages, mainly because of the packing effect. At later curing ages, the strength improvement was attributed to the higher content of calcium silicate hydrate (C-S-H) product microstructure. In a similar line, Li et al. (2004) found that using NS at a rate of 3-10% enhanced the flexural strength by as much as 28% and 27% at seven and 28 days, respectively. According to Stefanidou et al. (2012), a low NS concentration of 1% to 2% can maximize flexural strength after three days of casting.

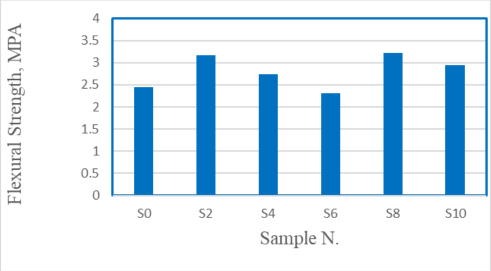

Fig. 5: Effect of NS waste content on flexural strength of cement mortars.

4. Conclusions

In the current investigation, NS waste materials were added at 0%, 0.5%, 1%, 1.5%, 2%, 2.5%, 4%, 6%, 8% and 10% to the cement mortar. NA material was collected from the aluminum fluoride plant of Jordan Phosphate Mine Company. The results show that with the increase of NS waste contents, a decrease in the heat of hydration and density is marked. The results indicate compliance with the Jordanian Standard Specifications (JSS 30-1/2007), as the study shows an improvement in workability and compressive strength with the increasing of NS waste, with insignificant impact on the flexural strength, which makes NS a suitable additive to mortar cement.

However, incorporating NS into cement mortar mixtures generally enhances their mechanical properties. At a low addition ratio (0.5%) of NS, a slight decrease (2.8%) in the compressive strength occurred at seven days, and about 2.7% at an addition ratio of 1%, which proved the feasibility of the addition process. Adding NS to the cement mortar mixture resulted in an increase in the compressive strength up to 16.75% at 28 days. The workability of cement mortar increased with increasing the number of NS in the mixture. Mixtures of Cement mortar containing NS have more homogeneous binders and fewer pores than conventional mixtures. In terms of mixing techniques, better strength was achieved by adding NS solution with water first before adding dry materials, giving better results for compressive and flexural strength. Incorporation of NS waste in cement mortar can reduce the dry weight by up to 17.7%, 13.4%, and 2.6% for adding NS by 10%, 4%, and 2.6%, respectively, provided light weight mortar and lower weight of the structure. The use of NS in cement mortar mixtures provided an attractive option for recycling NS waste. Increasing the percentage of NS, the rate of heat hydration decreases, so it can be used to reduce and adjust the heat hydration. It is recommended to add NS to the mixture in the form of an aqueous solution rather than as a solid; use NS with high purity to avoid the adverse effects of the impurities, and add dispersant to NS to avoid the dispersion problem, especially in the case of agglomerated NS. Further studies should be carried out regarding the long-term effects of NS on concrete and cement mortar. It is necessary to conduct a feasibility study to determine the benefit /cost analyses of using NS in cement mortar mixtures.

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6. Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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تأثير مخلفات النانو سيليكا كمواد اسمنتية مضافة لصناعة الملاط الاسمنتي في الأردن

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الملخص

يبحث هذا العمل في التأثير المحتمل لإعادة تدوير نفايات النانو سيليكا من خلال دراسة وتقييم قابلية التشغيل والكثافة وحرارة الماء والخواص الميكانيكية لملاط الاسمنت. تم جمع عينات النانو سيليكا من مصنع فلوريد الألومنيوم التابع لشركة منجم الفوسفات في الأردن. تم تحضير عشرة مخاليط من الملاط الإسمنتي مع النانو سيليكا إلى الاسمنت بنسب 0%، 0.5%، 1%، 1.5%، 2%، 2.5%، 4%، 6%، 8%، و10%. تم تحليل التركيبات الكيميائية لملاط الاسمنت والنانو سيليكا باستخدام تقنية مطياف الأشعة السينية المفلورة. يتم اعتماد اختبار جدول التدفق لفحص قابلية التشغيل لعينات الملاط الطازجة بعد الخلط. سجل متوسط الكثافات الجافة والتشكيلية للتراكيز المختلفة لمونة النانو سيليكا بعد 28 يوماً تحسناً واضحاً، بالإضافة إلى مقاومة الانضغاط والانثناء. أشارت النتائج إلى تحسن في مقاومة الانضغاط والانحناء مع زيادة النانو سيليكا في خليط الملاط.

الكلمات المفتاحية:

الاسمنت، مقاومة الانضغاط والانثناء، الملاط، نفايات النانو سيليكا، قابلية التشغيل.

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