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Evaluation of Combined Use of Waste Paper Sludge Ash and Nanomaterials on Mechanical Properties and Durability of High Strength Concretes

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ABSTRACT

The paper industry burns or buries a significant amount of waste paper sludge. This issue is not suitable from environmental and economic aspects. In this study, the mechanical and durability properties of high-strength concrete containing waste paper sludge ash (WPSA) were evaluated. The variables were WPSA (0, 5, 10, and 15% by weight of cement), silica nanoparticles (0 and 2.5 by weight of cement), and aluminum oxide nanoparticles (0 and 2.5 by weight of cement). Compressive strength, splitting tensile strength, flexural strength, and ultrasonic pulse velocity tests were conducted to evaluate the mechanical properties. The durability properties were also investigated using water penetration depth, water absorption, and electrical resistivity tests. The microstructure of the specimens was analyzed by preparing electron microscopic images. The combined effect of WPSA and nanoparticles on improving the mechanical and durability properties of high-strength concrete are better than using each of them alone. WPSA and nanoparticles react with calcium hydroxide formed due to cement's hydration, and silica produces hydrated calcium, which is the hard material that makes concrete strength. Consumption of calcium hydroxide and production of more hydrated calcium silicate in the presence of nanoparticles and WPSA are among the reasons for water absorption reduction, increased electrical resistance, and water penetration depth reduction in concrete specimens. By replacing part of the cement with WPSA, silica nanoparticles, and aluminum oxide nanoparticles, the transition zone between the aggregates strengthens, and the tensile and flexural strengths increased.

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1. INTRODUCTION

Extensive research showed that some paper industry waste can be used as raw materials in the construction industry [1-4]. Paper waste is obtained from an initial mechanical separation process and turns ash when burned [5-8]. In other words, waste paper sludge ash (WPSA) is a waste produced by the paper industry. These materials are produced when ink and pulp are burned to reduce their volume and produce energy [9]. According to EU standards, the combustion process usually occurs on a molten bed at 850 to 1100 degrees Celsius. The UK has 125,000 tonnes of waste paper ash per year, 70% is used for useless purposes such as spreading it on land to be recycled, and the remaining 30% is routinely transported to landfills [10]. Proper resource efficiency, reduced landfilling, and beneficial reuse are among the reasons why WPSA research has been promoted. WPSA has cement properties and reacts with water. However, due to its high porosity, it needs a lot of water. However, the combination of WPSA paper with pozzolanic materials can improve building materials' properties (including concrete). This combination can reduce harmful effects, such as reducing concrete strength [11, 12].

Past research on WPSA has focused on hydraulic

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properties, reactivity and their potential use as cementitious materials. For example, Pera and Amrouz [13] showed that WPSA could be converted to sticky calcareous at 700 to 750 ° C. Later, other studies confirmed such finding [11, 13]. Sutcu and Akkurt [14] investigated porous ceramics' production from a mixture of waste from the paper-making process and three different clays. The results showed that clay soils, aluminum silicate, alkaline clay, and refractory clay could produce refractory bricks [14]. Ismail et al. [15] investigated the manufacture of bricks from paper sludge and fuel oil ash. Although the strength of the prepared brick sample was slightly higher than the minimum required according to BS 6073, the brick sample density prepared from paper sludge and fuel oil ash showed a reduction of 26.10% compared to conventional bricks, which is an important issue [15].

Fava et al. [8] used WPSA as a supplement for cementitious materials and evaluated its application in concrete and building materials as adhesive materials along with cement. It was concluded that if WPSA is used in 10% of Portland cement, it will positively affect mortars' mechanical performance [8]. Martinez et al. [16] investigated bricks' production using two types of waste from the paper industry, including WPSA and waste from cleaning pulp. According to the results, increasing the porosity of the samples reduced the compressive strength [16]. Ahmad et al. [17] examined concrete containing WPSA as a substitute for cement. Mechanical properties, water absorption, and dry density at 28 days were examined. WPSA at a dose of 5% could be effective [17]. Wong et al. [9] examined hydrophobic concrete containing WPSA. The results showed that using 12% WPSA instead of cement without harmful effects on strength, hydration, and concrete density reduces water absorption, water absorption rate, and concrete electrical conductivity by 84, 86, and 85%, respectively [9].

Pourazar et al. [18] investigated concretes' properties containing waste paper ash on concretes containing silica fume. The results showed that the ratio of compressive strength to the tensile strength of concretes containing waste paper ash is 13.55 to 17.79, which is approximately ten times the ratio of ordinary concrete [18]. Marvroulidou and Shah [19] investigated the combined use of WPSA and active alkaline cement in concrete. The dose of activators, processing conditions, and processing time was studied. The results showed that mixing with WPSA in the activating system causes early concrete strength at ambient temperature and sufficient strength [19].

Chemicals penetrate the steel through the concrete's pores, causing corrosion, cracking, and eventually concrete failure. Chemical reactions of concrete take place at the nanoscale. The nanostructure of concrete deserves more attention to make concrete with dense nanostructure, minimum size, and pores, which affects concrete durability [20-22]. Recently, nanotechnology is used to reduce the permeability and concrete seepage [23-25]. In this research, the effect of WPSA in combination with silica nanoparticles and aluminum oxide nanoparticles is investigated.

Faez et al. [26] evaluated fresh and hardened self-compacting concrete properties containing aluminum oxide nanoparticles and silica fume. The results showed that using 2.5% aluminum oxide nanoparticles as a substitute for cement increased the compressive strength of 90 days by 86% [26]. Ghanbari et al. [27] evaluated the effect of nanosilica on the attributes of lightweight concrete containing zeolite and glass fibers. The results showed that the combined use of silica nanoparticles and glass fibers significantly affects self-compacting lightweight concrete containing zeolite and scoria aggregates; Depending on the dose of glass fibers and silica nanoparticles, the tensile strength has increased from about 3 to 56% [27]. Heidarzad et al. [28] evaluated the effects of aluminum oxide nanoparticles on the performance of concrete containing fibers. The results showed that the addition of aluminum oxide nanoparticles to specimens with and without glass fibers significantly increased the electrical resistance. Aluminum oxide nanoparticles prevented more ions in the concrete and reduced concrete corrosion by filling small cavities and increasing the concrete density [28]. Mostafa et al. [29] evaluated the effect of nano-silica and recycled nanomaterials (nano-glass waste, nano-rice husk ash, and nano-metakaolin) on the properties of high-performance concrete. According to the results, the microstructure of concrete and compressive strength is among the parameters that can affect these concretes' resistance against corrosion [29]. Fahmy et al. [30] investigated nanoparticles' effect on the behavior of high-strength concrete beams. Eight rectangular beams with the same geometric and steel specifications were made. The variables included type of nanomaterials (nanosilica, nano-titanium, and the combination of nanotitanium and nanosilica) and their doses. The dual-use of the hybrid mixture (nanosilica and nanotitanium) has significantly improved the compressive and tensile strength compared to using the same percentage of a single type of nanomaterials [30].

As mentioned above, WPSA and pozzolanic materials can protect the concrete against external degradation agents and prevent water penetration in concrete and reduce rebar corrosion. Water penetration prevention in concrete increases the efficiency of concrete structures. This prevents harmful substances from penetrating the concrete and steel. One of the benefits of hydrophobic concretes made from WPSA, and pozzolanic materials are filling voids and cavities in the cement matrix. However, the results of studies showed that although WPSA can improve the durability properties of concrete, but has little effect on improving the mechanical properties of concrete; Nanoparticles can overcome this weakness and improve the mechanical properties of concrete. The combined effect of WPSA and nanoparticles in high-strength concretes has not been evaluated in previous studies. Therefore, in the present study, the combined use of WPSA, silica nanoparticles, and aluminum oxide nanoparticles on high-strength concrete's mechanical and durability properties are investigated.

2. LABORATORY PROGRAM

Variables included WPSA (0, 5, 10, and 15% by weight of cement), type of nanoparticles (silica nanoparticles and aluminum oxide nanoparticles), and amount of nanoparticles (0 and 2.5% by weight of cement) respectively. The variables and the number of samples were selected according to the literature review experimental studies. The range of silica nanoparticles and aluminum oxide nanoparticles was considered based on studies about the use of nanoparticles. In most studies, the use of 2.5% nanoparticles was introduced as the optimal value [23-27]. The use of different amounts of nanoparticles can be considered as an important parameter. However, in this study, the main focus was on the mechanical properties, durability, and microstructure of high-strength concrete containing WPSA. The most important question that the authors sought to answer was whether the use of WPSA in combined with nanoparticles could improve engineering properties? Instead of using different amounts of nanoparticles, the authors increased the number of experiments to examine more specifications of these concretes. The experiments were conducted in four different sections. In the first part, the fresh properties of concrete were determined by slump test. In the second part, concrete's mechanical properties were evaluated using compressive strength, splitting tensile strength, flexural strength, and ultrasonic pulse velocity (UPV) tests. In the third part, the durability properties of concrete were assessed by performing water absorption, water penetration depth, and electrical resistivity. In the fourth section, the microstructure of concrete using scanning electron microscope (SEM) imaging was evaluated. According to the variables and experiments, 324 specimens were made according to Table 1. A flowchart of the study process is shown in Figure 1.

2. 1. Materials The used materials included coarse aggregates (gravel), fine aggregates (sand), cement, water, superplasticizer, WPSA, silica nanoparticles, and aluminum oxide nanoparticles.

2. 1. 1. Aggregates Aggregates in concrete make up about 75% of its volume, so their quality is important. In addition to being effective in concrete strength, aggregates are also effective in concrete durability and stability. Aggregates used in concrete include coarse aggregates about 63 to 73% of the total aggregates (maximum size is 23 mm), and fine aggregates are about 33 to 43% of the total aggregates (ranging in size from 3 to 76 mm). In this research, sand produced in Joban mine (Roudbar, Iran) has been used. The specific weight of sand and gravel is 2600 and 2650 kg/m^3. Grading of sand and gravel is in accordance with ASTM C330 [31] and is shown in Figure 2. The coarse and fine aggregates are in the range of ASTM C330. The dimensions of aggregates are selected in such a way that they are between the upper and lower boundaries of ASTM C330, so the standard requirements for the materials have been observed.

2. 1. 2. Cement Cement in concrete plays the role of bonding the components together. Portland type II cement was used as the primary adhesive in all samples and was prepared from the Hegmatan cement factory. This cement's chemical and physical characteristics based on ASTM C150 [32] are presented in Table 2. Specific gravity and pH of cement are 1.95 g/cm^3 and 9, respectively.

2. 1. 3. Silica Nanoparticles Silica nanoparticles are synthetic materials composed of very fine SiO_2 particles like cement and have high pozzolanic properties. The powder of this material is 99.9% silica, and its density is 0.2 g/cm^3. The specific surface area of silica nanoparticles is 50 to 100 m^2/g. Its solid particle diameter is about 50 to 100 nanometers. The

TABLE 1. Number of total specimens

Test	Number of mixture design	Number of specimens for each mixture	Age (days)	Number of total specimens
Compressive strength	12	3	7, 28 and, 90	108
Splitting tensile strength	12	3	28	36
Flexural strength	12	3	28	36
UPV	12	3	28	36
Water absorption	12	3	28	36
Water penetration depth	12	3	28	36
Electrical resistivity	12	3	28	36
Total				324

characteristics of silica nanoparticles are presented in Table 2.

2. 1. 4. Aluminum Oxide Nanoparticles These materials are white synthetic material composed of very small Al_2O_3 particles. They are used to improve ceramics' properties, solve their brittleness problem, increase the erosion resistance of coatings, and increase heat resistance [33]. The alumina in the nano-aluminum powder used in this research is more than 99%, its density is 3.89 g/cm^{3,} and the specific surface area of these nanoparticles is more than 138 m^2/g. Its solid particle diameter is about 20 nanometers.

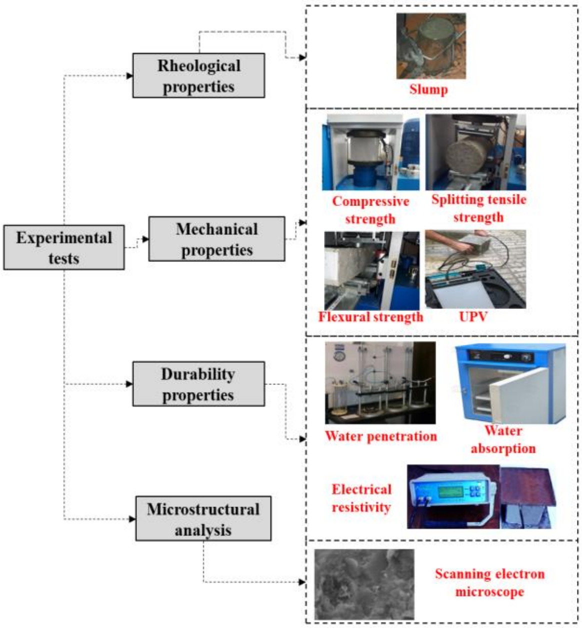


Figure 1. Geometric properties and steel reinforcement arrangement of original beams

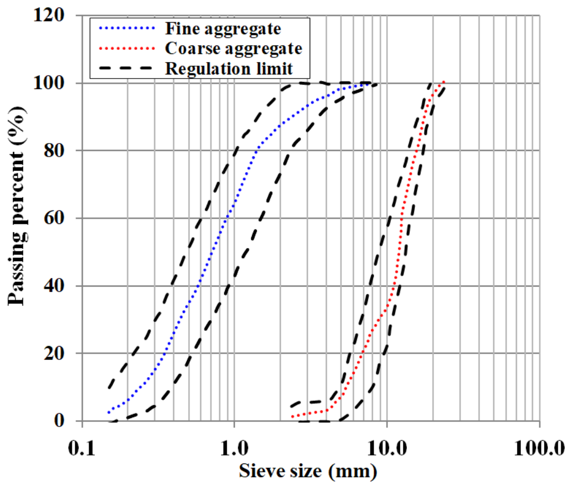


Figure 2. Garding curves of sand and gravel

2. 1. 5. WPSA The pulp production process begins with converting wood or lignocellulosic raw materials into flexible fibers suitable for making paper. During converting wood into pulp and paper, some wood particles and cellulose fibers enter the wastewater treatment system of factories. These cellulosic wastes, along with some minerals, are separated from the effluent treatment unit in the form of dry sludge and buried in the pits around the factories. One of the problems faced by the paper industry's production units is the significant volume of the waste remaining from the production process. These companies now bury all the sludge produced, and this is costly process.

In this study investigates the effect of addition of paper WPSA in the production of high-strength concretes. Waste paper sludge of Chouka Plulp & Paper factory (Rezvan-Shar, Iran) was used to make the specimens. A lot of waste paper sludge from the paper production process is accumulated in this factory in recent decades, the ashes of which can be used to produce concrete. The waste received from the factory was stored in the open air for seven days and then burned at 700 ° C. The chemical composition and mineral oxides in WPSA were evaluated using X-ray fluorescence (XRF) Spectrometer (Table 2). The paper sludge of Chuka factory was a solid waste consisting of fiber and ash residues produced by the paper-making and pulping process. This sludge was prepared, and its ash was used as a substitute for a part of cement. The potential of this

TABLE 2. Chemical characteristics of the binder materials

Components	Cement	AlNPs	SNPs	WPSA
Al_2O_3	4.95٪	99≥٪	-	6.83
CaO	62.95٪	25ppm ≤	-	25.43
SiO_2	21.27٪	-	99.98≥	10.79
Fe_2O_3	4.03٪	80ppm ≤	-	0.46
MgO	1.55٪	-	-	0.87
SiO_3	2.26٪	-	-	0.32
K_2O	0.65٪	-	-	0.22
Na_2O	0.49٪	70ppm ≤	-	0.15
MnO_2	-	3ppm ≤	-	-
Cr	-	4ppm ≤	-	-
Co	-	2ppm ≤	-	-
LOI	-	-	1.00 ≤	54.34
Specific gravity (g/cm^3)	0.75	3.89	0.5	2.81
Specific surface area (m^2/g)	326	138	200	168

recycled material for concrete production was evaluated based on the concept of biorefining. The used binder materials are presented in Figure 3.

2. 1. 6. Superplasticizer Excellent concrete water reducer, suitable for producing super-smooth concrete and self-compacting without additional water and aggregates separation and compatible with various types of cement are the main features of this superplasticizer. This product is manufactured in accordance with ASTM C494 [34]. The specific weight of this superplasticizer is 1.1 g/cm^2.

2. 1. 7. Water Tap water was used to make concrete specimens. According to ASTM C1602 [35], drinking water is suitable for concrete and does not need to be tested [36]. The water's pH was 7.5, the total hardness is 241 mg/l, the amount of chloride is 21.6 mg/l, and the amount of sulfate is 84.75 mg/l, which are within the permissible values mentioned.

2. 2. Experimental Tests Slump test was conducted to measure the workability of concrete in accordance with ASTM C143 [37].

The compressive strength test was performed according to ASTM C39 [38]. This test is one of the most important factors in the quality control of concrete and expresses the sample's tolerance in compressive strength. Cube molds with dimensions of 150×150×150 mm were used to make the specimens. The molds were kept in the open air for 24 hours after filling. After this period, the molds were opened, and the specimens were placed in the water tank and tested at the desired ages (7, 28, and 90 days).

The splitting tensile strength test was performed according to ASTM C496 [39]. In this experiment, standard cylinders of 150×300 mm were used. The conditions for preparing and storing the samples were the same as for compressive strength specimens. Specimens were placed horizontally in the machine. The load gradually increased, and due to the pressure in the direction perpendicular to the pressure, tension was created, and the specimen was broken. The load increase was uniform and loaded at a rate of 0.8 to 1.2 MPa per minute until the specimen was broken. The maximum load was read and recorded by the device. Splitting tensile strength was obtained by Equation (1). In this regard, T is the splitting tensile strength, P is the fracture load, L is the sample length, and D is the sample diameter. Failure of specimens in splitting tensile strength test was performed at 28 days of age.

Figure 3. Photo images of binder materials used

$$T = \frac{2p}{\pi L d} \tag{1}$$

The flexural strength test was performed according to ASTM C293 [40]. The specimens had dimensions of 100×100×500 mm and were placed symmetrically on two simple supports. They were then loaded evenly and without sudden changes. Flexural strength was calculated by Equation (2). In this experiment, specimens were broken at 28 days of age.

$$S_f = \frac{3pl}{2bh^2} \tag{2}$$

In this equation, S_f is the flexural strength, p is the applied force in the middle of the span, b is the width of the section, h is the section's height, and l is the distance between the two supports.

The ultrasonic pulse velocity test was performed according to ASTM C597 [41]. The ultrasonic pulse velocity method involves measuring the travel time of an ultrasonic pulse passing through a concrete specimen. An ultrasonic device consists of an electronic circuit to generate a pulse and a transmitter to transmit these electronic pulses to mechanical energy, which have vibrational frequencies in the range of 15 to 50 kHz. The travel time between pulses is measured electronically. The pulse propagation's average velocity is obtained by dividing the path length between the transmitters by travel time. There are three methods for measuring pulse velocity in concrete: direct transfer, indirect transfer, and surface transfer. In the present study, the direct method is used by placing the transmitters between two surfaces in front of concrete samples. Pulse velocity is affected by several factors without taking into account the properties of concrete, including the smoothness of the contact surface of the specimen under test, the effect of path length on pulse velocity, test temperature, concrete moisture conditions, the presence of steel rebars, pulse velocity methods for evaluating concrete structures. Attempts have been made to correlate pulse velocity with strength and other properties of concrete. UPV test was performed at 28 days of age.

The initial (30 minutes) and final (72 hours) volumetric water absorption test was performed according to ASTM C642 [42]. Equation (3) was used to determine the water absorption percentage.

$$\text{Water absorption percentage} = \frac{m - m_0}{m_0} \times 100 \tag{3}$$

m is the wet weight of the specimen, and m_0 is the dry weight of the specimen.

The test for determining the water penetration depth in concrete was performed according to DIN1048 [43]. This experiment aims to assess the penetration of pressurized water in hardened concrete that has been treated in water. In this way, water is applied by pressure to the surface of hardened concrete, and then the specimen is divided into two halves, and the penetration depth related to water advancement is measured. 150×150×150 mm cube specimens were used. The sample was placed in the machine, and water pressure of 450 to 550 kPa was applied for 72 hours. During the test, the surfaces of the specimens that are not exposed to water pressure are continuously checked so that water does not leak. After applying pressure for a specified period of time, the sample is removed from the device, and excess surface water droplets are removed from the sample on which water pressure is applied. The sample is then split in half perpendicular to the surface exposed to water pressure. When the sample was divided into halves, the halved sample's surface exposed to water pressure was placed down, and the leading water sample was marked on it as soon as it was broken. The progress of water penetration is clearly visible on the surface. The maximum penetration depth was measured and recorded in millimeters.

Corrosion is an electrochemical phenomenon in which rebars act as anodes and concrete as cathodes, creating an electric current between the rebars and the concrete surface. In this case, the mobility of ions is visible. The more and easier this movement is, the lower the resistance to ion mobility and the greater the concrete's electrical conductivity. Therefore, one of the simplest ways to test concrete durability is to determine its electrical resistance. Moisture reduces electrical resistance. The presence of small cracks that fill with water also reduces electrical resistance. A concrete electrical resistance measuring device was used to determining the electrical resistivity. Specimens were tested at 28 days of age. An average of three readings was reported for each mixture. Two copper plates were placed in complete contact with two concrete surfaces. Equation (4) was used for determining the electrical resistivity.

$$\rho = \frac{R.L}{A} \tag{4}$$

where ρ is the specific electrical resistance of concrete in ohm-meters, R is the electrical resistance read by the device in ohms-meters. A is the area on concrete in square meters, and L is the distance between the positive and negative poles in meters. The estimation of corrosion probability of reinforcements buried in concrete based on electrical resistance is presented in Table 3.

2. 3. Mixture Design

The ratios of the concrete mix components were determined using the absolute volume method in accordance with ACI 211 [47]. The water to cement ratio was considered 0.45 after reducing the aggregate moisture. The materials for one cubic meter of concrete are presented in Table 4. WPSA in the amount of 0, 5, 10, and 15% by weight of cement, silica nanoparticles at 0 and 2.5% by weight of cement, and aluminum oxide nanoparticles at 0 and 2.5% by weight of cement were used. The reason for choosing 2.5% nanoparticles is that this value has been introduced as the optimal value in many studies [26-28]. On the other hand, the main variable studied in the present study is WPSA, and four different values were considered for it.

The combined use of WPSA and two different types of nanoparticles in high-strength concrete is one of the

TABLE 3. Estimation of corrosion probability of buried rebars in concrete based on electrical resistivity [45, 46]

Electrical resistivity (kΩ-cm)	Estimation of corrosion probability
Less than 50	Very high
Between 50 and 100	High
Between 100 and 200	Low
More than 200	Very low

TABLE 4. Mixture design

Mix code	Cement	WPSA[a]	SN[b]	AlN[c]	Sand	Geavel	SP[d]
P0	550	0	0	0	705	1075	3.85
P0S	536.25	0	13.75	0	705	1075	3.98
P0AL	536.25	0	0	13.75	705	1075	4.10
P5	522.5	27.5	0	0	705	1075	4.85
P5S	508.75	27.5	13.75	0	705	1075	5.10
P5AL	508.75	27.5	0	13.75	705	1075	5.21
P10	495	55	0	0	705	1075	5.36
P10S	481.25	55	13.75	0	705	1075	5.46
P10AL	481.25	55	0	13.75	705	1075	589
P15	467.5	82.5	0	0	705	1075	6.21
P15S	453.75	82.5	13.75	0	705	1075	6.43
P15AL	453.75	82.5	0	13.75	705	1075	6.81

WPSA: Waste paper slag ash

SN: Silica nanoparticles

AlN: Aluminium oxide nanoparticles

SP: Superplasticizer

most important objectives of the present study. Concrete mixing using a mixer was initially done dry; The aggregates were mixed dry with cement and stirred in a mixer for 60 seconds. Then WPSA and nanoparticles were added to them, and the stirring operation was continued for another 45 seconds. Finally, water and superplasticizer were added to the mixture, and the mixing process was performed for another three minutes.

2. 4. Curing The samples' storage and curing conditions according to each series of specimens' condition were as follows: in the first 24 hours in the mold and then kept wet and transferred to the water tank, and stored in accordance with ASTM C192 [48].

3. RESULTS AND DISCUSSION

3. 1. Slump The workability depends on the slump and consistency of the concrete. The specimens' workability was evaluated using the slump test, and the results are presented in Figure 1. The slump of specimens containing 0, 5, 10, and 15% WPSA was 46, 51, 68, and 76 mm, respectively. The slump of specimens containing 0, 5, 10, and 15% WPSA in which 2.5% silica nanoparticles were used was 47, 54, 73, and 79 mm, respectively. The slump of specimens containing 0, 5, 10, and 15% WPSA in which 2.5% aluminum oxide nanoparticles were used was 56, 65, 78, and 81 mm, respectively. ACI 318-99 [47] proposed an allowable slump range for different RC building members (25 to 100 mm). All slump values obtained are in the mentioned range. Also, according to Figure 4, addition of 5 to 15% WPSA to the specimens with and without nanoparticles reduced the concrete slump from 11 to 76%, depending on the amount of nanoparticles compared to the control sample. The addition of nanoparticles and WPSA reduces slump and increases the possibility of locking. By adding superplasticizer to concrete specimens containing WPSA and nanoparticles, good workability can be achieved. By using appropriate amounts of WPSA, silica nanoparticles, and aluminum oxide nanoparticles along with suitable superplasticizer, operational mixing schemes used in projects can be achieved.

3. 2. Compressive Strength The mean compressive strength of the specimens at the ages of 7, 28, and 90 days is shown in Figure 5. The compressive strength of 7 days of specimens containing 0, 5, 10, and 15% WPSA were 38.9, 39.1, 40.3, and 37.1 MPa. The compressive strength of 7 days of specimens containing 0, 5, 10, and 15% WPSA in which 2.5% silica nanoparticles were used were 46.3, 47.6, 49.3, and 46.3 MPa, respectively. Also, the compressive strength of 7 days of specimens containing 0, 5, 10, and 15% WPSA in which 2.5% aluminum oxide nanoparticles were used were 44.6, 46.3, 48.7, and 45.2 MPa, respectively. According to the mentioned values, it is observed that adding WPSA to concrete samples without nanoparticles increased the 7-day compressive strength by about 1 to 4%, and addition of WPSA to concrete specimens containing nanoparticles increased the 7-day compressive strength about 14 to 27%.

Also, the 28-day compressive strength of P0, P5, P10, and P15 specimens were 57.1, 58.9, 60.3, and 55.6 MPa, respectively. By addition of 2.5% silica nanoparticles to the mentioned specimens caused their 28-day compressive strength to be 66.1, 67.2, 72.1, and 65.7 MPa, respectively. Also, the 28-day compressive strength of P0Al, P5Al, P10Al, and P15Al specimens were 65.3, 66.5, 70.2, and 65.5 MPa, respectively. Addition of 5 to 15% WPSA to specimens containing nanoparticles increased the 28-day compressive strength of the specimens by about 15 to 26%, depending on the amount of nanoparticles.

Using a combination of WPSA and nanoparticles was much better than using each one individually. For example, the 28-day compressive strength of a specimen using 10% WPSA and 2.5% silica nanoparticles was 26% higher than that of the control sample. The compressive

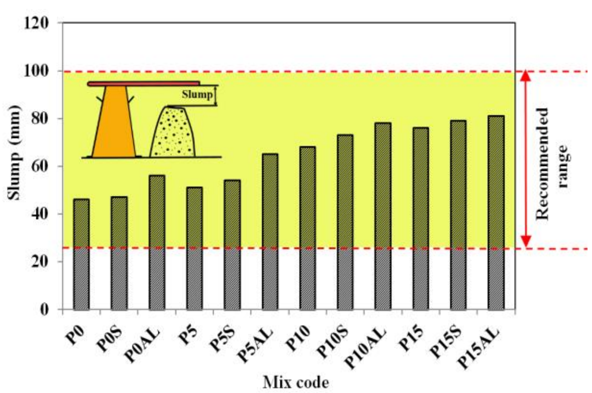


Figure 4. Slump value of concrete specimens containing different amounts of WPSA and nanoparticles

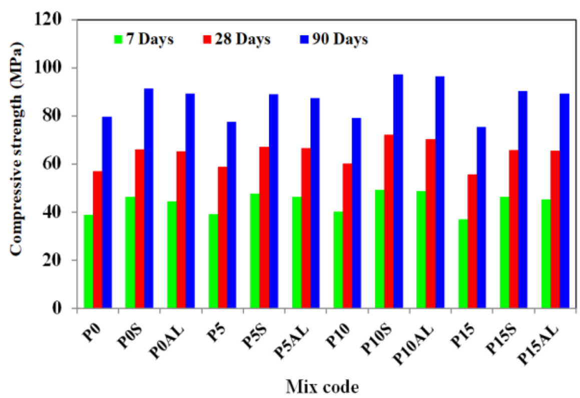


Figure 5. Compressive strength of specimens at 7, 28, and 90 days

strength of a specimen using 10% WPSA increased by only 4%, and the compressive strength of a specimen using only 2.5% silica nanoparticles was 16% higher than the control sample.

As the age of concrete increases, the compressive strength of the specimen increases. The 90-day compressive strength of concrete specimens containing nanoparticles and WPSA increased by about 10 to 22% compared to the control specimen, depending on the amount of nanoparticles and WPSA. Also, the highest 90-day compressive strength was obtained in the samples in which 10% WPSA and 2.5% silica nanoparticles were used, and its value was 97.3 MPa.

The use of silica nanoparticles and aluminum oxide nanoparticles in combination of WPSA has increased the compressive strength compared to the control sample. Excess silica is mixed with cement compounds to form C-S-H gels, and compressive strength was achieved by filling pores in concrete.

As the WPSA increased to more than 10%, the compressive strength decreased with a greater slope. The reason for this is that when the amount of WPSA in concrete exceeds a certain limit, WPSA is dehydrated due to the high intensity of the reaction. The use of superplasticizers up to a certain percentage of nanoparticles compensates for this deficiency, but due to the limited use of superplasticizers in higher percentages of WPSA, this problem persists, resulting in some porosity of the concrete, which results in decreases of the compressive strength.

Another point is that WPSA increases the compressive strength in a short time due to its specific surface area and high reaction intensity. WPSA reacts with calcium hydroxide formed due to cement curing and hydrates the production of calcium silica, the hard material that makes concrete strength. As shown in Figure 1, in the 7-day specimen, the upward trend and in the 28- and 90-day specimen, the growth has decreased. The reason for this behavior is probably that the 7-day samples did not suffer from water deficiency due to the intensity of the reaction at the time of the test. As a result, internal porosity has not yet formed in the concrete, but this has happened in the case of 28 and 90-days concrete.

3. 3. Splitting Tensile Strength The 28-day splitting tensile strength of specimens and the percentage of changes compared to the control specimen are shown in Figure 6. Single-use of WPSA in specimens does not play a significant role in splitting tensile strength of specimens and, in some cases, has even reduced the splitting tensile strength of specimens (P10 and P15). The combined use of 2.5% silica nanoparticles and 0, 5, 10, and 15% WPSA increased the splitting tensile strength by 14.8, 14.6, 15.6, and 12.8%, respectively. Also, the combined use of 2.5% aluminum oxide nanoparticles and 0, 5, 10, and 15% WPSA increased the splitting tensile strength by 7.7, 10.5, 9.2, and 6.9%, respectively. As with compressive strength results, the combined use of nanoparticles and WPSA has a more effective role in increasing splitting tensile strength than their single-use. The splitting tensile strength largely depends on the area of transition between the aggregate and the cement paste. By replacing part of the cement with WPSA, silica nanoparticles, and aluminum oxide nanoparticles, the transition zone between the aggregates is strengthened, and thus the tensile strength is increased.

Figure 7 shows the relationship between splitting tensile strength and compressive strength of specimens containing WPSA and nanoparticles at 28 days of age. The compressive strengths presented in this figure are the compressive strengths of cylindrical specimens. For this purpose, all 28-day compressive strengths of concrete cubic specimens were converted to cylindrical equivalent compressive strength using conversion coefficients. This diagram also presents the relationships between tensile strength and compressive strength proposed by ACI 318R-95 [49] and CEB-FIP [50].

The range of compressive and tensile strengths obtained is within the range of values of the mentioned

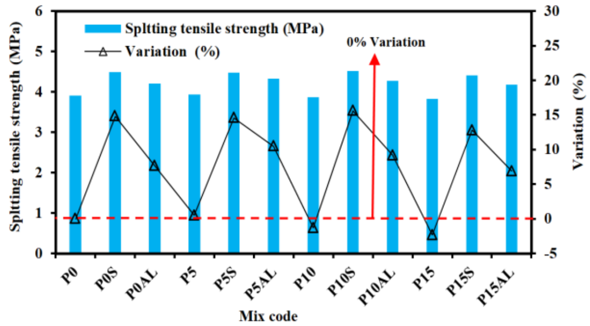


Figure 6. Splitting tensile strength and the percentage of changes compared to the control sample

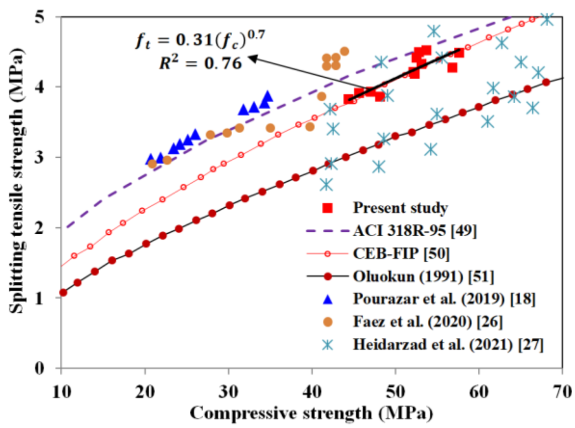


Figure 7. Relationship between splitting tensile strength and compressive strength and comparison with regulations and other studies

standards. Figure 7 shows the range of compressive and tensile strengths of some similar studies. Pourazar et al. [18] investigated the effect of WPSA and silica fume on concrete specimens. As can be seen, the range of compressive and tensile strengths obtained in the present study is in a higher range than concretes containing WPSA and silica fume. This result showed that the use of nanosilica in combination with WPSA has a much better performance compared to the use of silica fume. Nanoparticles have a more effective role in improving the mechanical properties of concrete due to higher filling. Also, the results reported by Faez et al. [26] and Heidarzad et al. [28] are shown in Figure 7, which used silica nanoparticles and aluminum nanoparticles to make concrete samples, respectively. The purpose of this chart is to ensure the accuracy of the results. The difference in the present study's values can be due to the water to cement ratio, quality of materials, type of additives, and the curing type.

3. 4. Flexural Strength The flexural strength of the specimens at the age of 28 days and their percentage change compared to the control specimen are shown in Figure 8. The use of 5 and 10% WPSA has increased the flexural strength by about 1.1 and 2%, respectively. But the use of 15% WPSA has reduced the flexural strength by about 1.5%. The use of nanoparticles in combination with WPSA has led to an increase in the specimens' flexural strength. The highest increase in flexural strength was obtained in the P10S specimen and the flexural strength of this specimen increased by 14.3% compared to the control sample. Comparing flexural strengths of specimens containing silica nanoparticles and aluminum oxide nanoparticles showed that the combined use of silica and WPSA nanoparticles has a more influential role in increasing the flexural strength of concrete specimens than specimens containing aluminum oxide and WPSA.

3. 5. Ultrasonic Pulse Velocity (UPV) Among the non-destructive methods available, the use of UPV, in addition to making it possible to measure hardness on the actual structure, can also reveal some defects of concrete [52]. UPV test can be used for determining the concrete uniformity, concrete strength, the modulus of elasticity, properties of concrete over time, the degree of hydration of cement, the durability of concrete, damaged layer action in concrete, and crack depth [53]. Figure 9 shows the UPV of the specimens in different states. This diagram also provides details on the classification of concrete types in terms of quality. This classification was proposed by Whitehurst [54], and concrete is divided into four categories in terms of quality: excellent, good, medium, and doubtful. The results showed that the specimens' UPV is in the range of 3.5 to 4.5 km/h and has good quality. Adding WPSA to the specimen increased the UPV. The highest increase of UPV was obtained in samples in which 10% WPSA was used in combination with 2.5% nanoparticles (silica and aluminum oxide).

3. 6. Water Absorption CEB divides concrete [55] into three categories based on initial water absorption: poor quality (5 to 6%), medium (between 3% and 5%), and good (less than 3%). According to the CEB classification, the control sample is in the middle category. Figure 10 shows the initial and final water absorption of the specimens in different states. Single-use of WPSA and nanoparticles and their combined use have reduced the water absorption percentage of the specimens. The initial water absorption percentage of concrete specimens containing WPSA is in the range of 2.25 to 3.67%. According to the mentioned classification, it can be stated that all the concrete specimens examined, in which WPSA, silica nanoparticles, and aluminum nanoparticles were used, are in the good quality category.

The combined use of nanoparticles and WPSA in all mixing designs have reduced the water absorption percentage of concrete. For example, the use of 10% WPSA and 2.5% silica nanoparticles reduced the initial and final water absorption of concrete by 28% and 29%, respectively. The combined use of nanoparticles and WPSA causes the concrete pores to fill more and decrease water absorption. Consumption of calcium

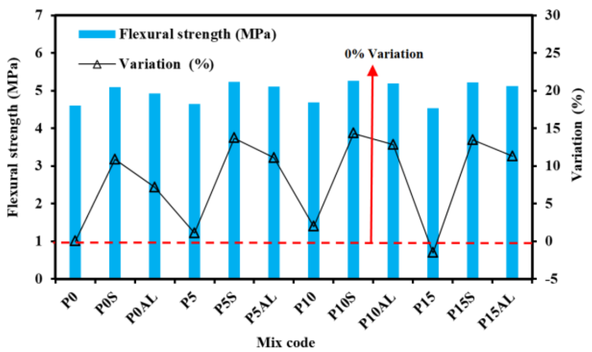


Figure 8. Flexural strength and percentage of changes compared to the control specimen

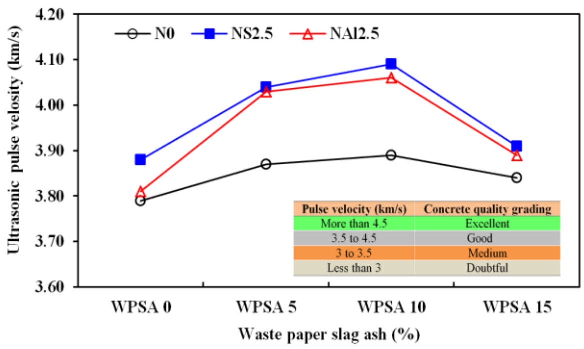


Figure 9. UPV of specimens

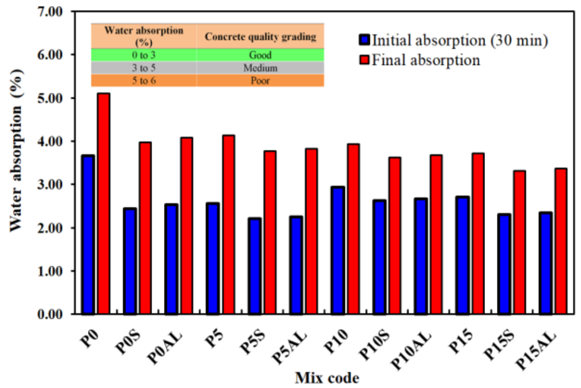


Figure 10. Percentage of initial and final water absorption of specimens in different states

hydroxide and production of more hydrated calcium silicate in the presence of nanoparticles and WPSA are other reasons for the decrease in concrete water absorption.

3. 7. Water Penetration Depth Figure 11 shows a graph of water penetration versus WPSA for specimens with and without nanoparticles. By replacing WPSA or silica nanoparticles and aluminum oxide nanoparticles, the water penetration depth index is improved. The P15S mixture, in which 15% WPSA and 2.5% silica nanoparticles were substituted for Portland cement, showed the lowest penetration depth, which is 3.76 mm. The use of WPSA alone in the specimens showed less effectiveness in improving the water penetration depth than the mixture containing WPSA and nanoparticles, which in this case can also be attributed to the lower pozzolanic activity of WPSA compared to the combination of WPSA and nanoparticles.

The use of 5, 10, and 15% WPSA reduced the water penetration depth by 16, 22, and 25%, respectively, compared to the control sample. Replacement of WPSA with cement has a greater contribution and effect on improving the absorption properties and permeability of concrete than the strength properties of concrete. The use of 5, 10, and 15% WPSA in specimens containing 2.5% silica nanoparticles reduced the water penetration depth by 22, 26, and 30%, respectively, compared to the control sample. Also, the use of 5, 10, and 15% WPSA in specimens containing 2.5% aluminum oxide nanoparticles reduced the water penetration depth by 21, 25, and 29%, respectively. The combined use of silica nanoparticles, aluminum oxide nanoparticles, and WPSA in high-strength concrete improved the microstructure and reduces the pores' size, leading to reduced permeability and increased resistance to aggressive materials as chlorine ions and the risk of carbonation.

3. 8. Electrical Resistivity Due to the fact that the corrosion process of steel rebars in concrete is an electrochemical process, so the electrical resistivity of concrete has a significant role in corrosion, and it can be expected that concretes with higher electrical resistivity showed better performance in reducing corrosion of steel rebars. Figure 12 shows the electrical resistivity of the specimens in different states. The use of WPSA has increased the electrical resistivity of concrete. With increasing WPSA value, the electrical resistivity of concrete specimens has increased more. P5, P10, P15 mixtures' electrical resistivity were 10, 19, and 29% higher than the control specimen (P0), respectively. Elkey and Sellevold [45] and Ong and Saraswathy [46] divided concrete specimens into four categories based on electrical resistivity and corrosion probability (Figure 12). Accordingly, specimens containing WPSA and nanoparticles are less likely to corrosion. The combined use of nanoparticles and WPSA has increased the electrical resistance by about 18 to 67 percent. A significant increase in electrical resistivity of specimens containing WPSA, silica nanoparticles, and aluminum oxide nanoparticles was due to pozzolanic reactions and reduced porosity of concrete pores. Examining these results, it is clear that the degree of permeability for all projects containing WPSA has been associated with a decrease.

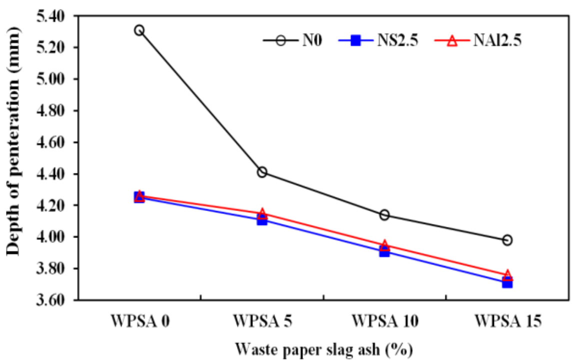


Figure 11. The water penetration depth of specimens in different conditions

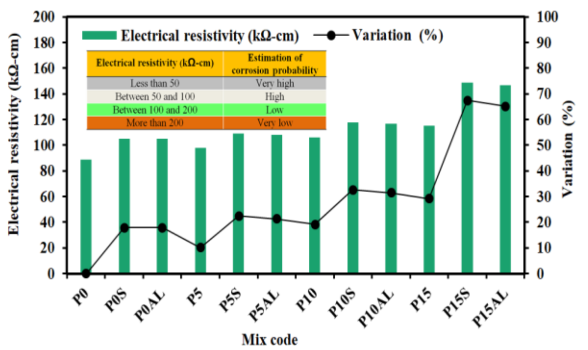


Figure 12. The electrical resistivity of specimens in different states

3. 9. Scanning Electron Microscope (SEM) Imaging

The results of experiments performed on concrete specimens containing nanoparticles and WPSA showed that 10% WPSA combined with 2.5 nanoparticles (silica and aluminum oxide) has an effective role in improving the mechanical properties and durability of concrete. Therefore, specimens P0, P10, P10S, and P10Al were selected to study the microstructure, and their SEM images were prepared. Figure 13 shows the SEM images of P0, P10, P10S, and P10Al. In the control sample (P0), there is no continuous continuity between the concrete components, and the components do not have an acceptable consistency. By creating C-H- S gel, nano-silica increases the compressive strength and adhesion of concrete components.

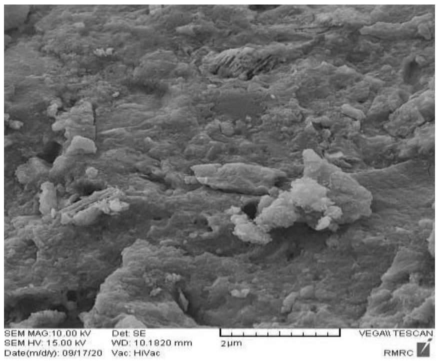
WPSA increases the durability of concrete by closing cavities. Comparison of SEM images of the control sample (without nanoparticles and WPSA) and the rest of the images containing nanoparticles and WPSA shows that the control sample has more component rupture and has more cavities. Nanoparticles reduce these cavities, resulting in increased compressive and tensile strengths and reduced water permeability.

In the microstructure of ordinary cement paste, it is observed that a large amount of needle-shaped hydrates surrounds the C-S-H gel. Comparison of this image and the image of cement paste made with 10% WPSA and 2.5% nanoparticles (silica and aluminum oxide) showed the better effect of this proposed compound on the microscopic structure of cement paste. However, in some parts of the figure, local growth of hydrated crystals was observed, which added to their non-uniformity.

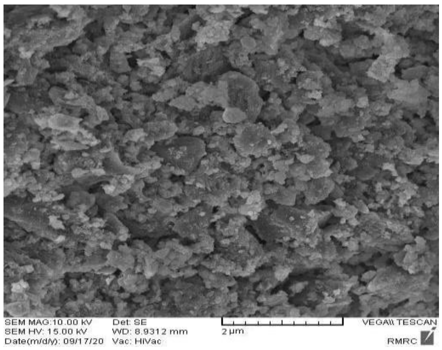
Also, microscopic pores on a more or less uneven surface can be seen in these images. This image shows the effect of nanosilica on reducing the size of large crystals. It is also observed that WPSA and nanoparticles by filling the gel's pores reduce the size and number of microstructural cavities of the samples compared to samples without nanomaterials, resulting in greater coherence and integration for these specimens. As can be seen from the pictures, the results of the experiments are consistent with the photographs taken of the microstructure of the cement paste.

4. SUMMARY AND CONCLUSION

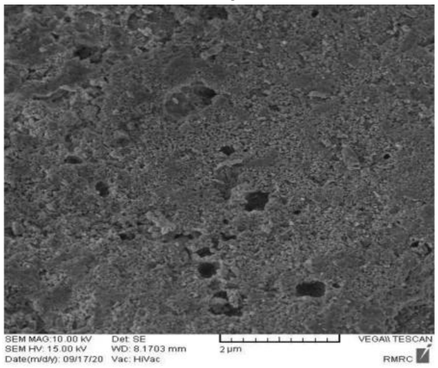
In this paper, the mechanical properties, durability, and microstructure of high-strength concrete containing WPSA and nanoparticles were investigated. The effect of WPSA on improving the mentioned characteristics was one of the main objectives of the present study. For this purpose, compressive strength, splitting tensile strength, flexural strength, UPV, water absorption, water penetration depth, and Electrical resistivity tests were performed. Also, the microstructure of concrete specimens containing WPSA and nanoparticles was evaluated using SEM images. This section summarizes the most important results:



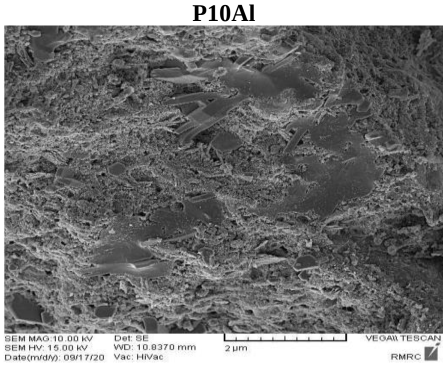
P0



P10



P10Al



P10S

Figure 13. SEM images of specimens

- The combined use of WPSA and nanoparticles is better than the single-use of each. For example, the 28-day compressive strength of the specimen containing 10% WPSA and 2.5% silica nanoparticles was 26% higher than that of the control specimen. The compressive strength of the specimen containing 10% WPSA increased by only 4%, and the compressive strength of the specimen containing only 2.5% silica nanoparticles was 16% higher than that of the sample.
- The use of silica nanoparticles and aluminum oxide nanoparticles in combination with WPSA has increased the compressive strength compared to the control specimen. This is because of the excess silica mixes with the cement compounds to form the C-S-H gel. Increasing the strength is done by filling the pores of the concrete.
- WPSA increases compressive strength in a short time due to its specific surface area and high reaction intensity. WPSA reacts with calcium hydroxide formed due to cement curing and hydrates the production of calcium silica, which is the hard material that makes concrete strength.
- In 7-days specimens, an increase in compressive strength growth was observed, and, in 28 and 90-day specimens, a decrease in compressive strength growth was observed. The reason for this behavior is probably that the 7-day specimens did not suffer from water deficiency due to the intensity of the reaction at the time of the test. As a result, internal porosity has not yet formed in the concrete, but this has happened with 28 and 90-day-old concretes.
- Single-use of WPSA in specimens does not play a significant role in the splitting tensile strength of specimens and, in some cases, has even reduced the tensile strength of specimens. The combined use of nanoparticles and WPSA has a more effective role in increasing splitting tensile strength than their single-use. The amount of tensile strength of concrete depends to a large extent on the transition area between the aggregate and the cement paste. By replacing part of the cement with WPSA, silica nanoparticles, and aluminum oxide nanoparticles, the transition zone between the aggregates is strengthened, and thus the tensile strength has increased.
- The highest increase in flexural strength was obtained in the P10S specimen and the flexural strength of this specimen increased by 14.3% compared to the control specimen. Comparison of flexural strengths of specimen containing silica and aluminum oxide nanoparticles shows that the combined use of silica and WPSA nanoparticles has a more effective role in increasing the flexural strength of concrete specimens than specimen containing aluminum oxide and WPSA.
- The combined use of silica nanoparticles, aluminum oxide nanoparticles, and WPSA in high-strength concretes improves the microstructure and reduces porosity, leading to reduced permeability and increased resistance to aggressive materials such as chlorine ions and the risk of carbonation.
- The combined use of nanoparticles and WPSA has increased the electrical resistance by about 18 to 67 percent. A significant increase in electrical resistance of specimens containing WPSA, silica nanoparticles, and aluminum oxide nanoparticles was due to pozzolanic reactions and reduced porosity of concrete pores. Examining these results, it is clear that the degree of permeability for all projects containing WPSA has been associated with a decrease.
- WPSA and nanoparticles by filling the gel's pores reduce the size and number of microstructural cavities of the specimens compared to samples without nanomaterials, resulted in greater coherence and integration for these samples.

The paper industries use two common incineration and landfill methods to dispose of their waste, which is a matter of exorbitant costs. Hazardous compounds in this type of waste cause environmental pollution and endanger human health. According to the results, the use of WPSA in the production of concrete is more economical and environmental due to less use of cement. The use of WPSA from paper mills in the production of concrete leads to economic and ecological benefits. The present study results that were about the management of WPSA and their use in the concrete showed that these materials can be used as a substitute for a part of cement (up to 10%) and reduce the percentage of water absorption and strength of concrete. In this study, an attempt was made to overcome the strength reduction weakness by using silica nanoparticles and aluminum oxide nanoparticles. In these cases, higher percentages of waste paper waste can be used. In addition, the proposed concrete can be used to repair the piers of bridges and hydraulic structures.

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Persian Abstract

چکیده

واحد های تولیدی صنایع کاغذسازی حجم قابل توجه‌ای از پسماند باقیمانده کاغذ را سوزانده و یا دفن می کنند. این موضوع از جنبه‌های زیست محیطی و اقتصادی مناسب نمی‌باشد. در مطالعه حاضر اثر استفاده ترکیبی از خاکستر لجن ضایعات کاغذ (WPSA) و نانو مواد بر خصوصیات مکانیکی و دوام بتن‌های با مقاومت بالا ارزیابی شده است. WPSA (۰، ۵، ۱۰ و ۱۵ درصد وزنی سیمان)، نانو ذرات سیلیس (۰ و ۲.۵ درصد وزنی سیمان) و نانودرات اکسید آلومینیوم (۰ و ۲.۵ درصد وزنی سیمان) متغیرهای مورد بررسی بودند. خصوصیات مکانیکی با آزمایش‌های مقاومت‌های فشاری، کششی شکافت، خمشی و سرعت امواج فراصوت ارزیابی شد. همچنین دوام بتن با انجام آزمایش‌هایی نظیر عمق نفوذ آب، جذب آب و مقاومت الکتریکی انجام گردید. ریزساختار نمونه‌ها با تهیه تصاویر میکروسکپی الکترونی تحلیل شد. اثر ترکیبی WPSA و نانوذرات بر بهبود خواص مکانیکی (مقاومت‌های فشاری، کششی و خمشی) و دوام بتن‌های با مقاومت بالا، بهتر از استفاده تکیِ از هر کدام از آنها می‌باشد. WPSA و نانو ذرات با کلسیم هیدروکسیدی که به دلیل هیدراتاسیون سیمان تشکیل میشود، واکنش می‌دهد و سیلیکا کلسیم هیدراته تولید میکند که همان ماده‌ی سخت عامل مقاومت بتن است. مصرف شدن هیدروکسید کلسیم و تولید سیلیکات کلسیم هیدراته‌ی بیشتر در حضور نانو ذرات و WPSA از جمله دلایل کاهش جذب آب، افزایش مقاومت الکتریکی و کاهش عمق نفوذ آب در نمونه‌های بتنی می‌باشد. با جایگزینی بخشی از سیمان با WPSA، نانوذرات سیلیس و نانوذرات اکسید آلومینیوم، ناحیه‌ی انتقال بین دانه‌های سنگی تقویت می‌شود و بدین ترتیب مقاومت‌های کششی و خمشی افزایش می‌یابد.