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The Effect of Superabsorbent Polymer and Nano-Silica on the Properties of Blended Cement

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Abstract: Incorporating superabsorbent polymer (SAP), which has the abilities of absorption and desorption in cement mortar, can achieve the effect of internal curing. It is expected that the incorporation of nano-silica will improve the workability and strength in cement mortar/concrete. Hence, this study aims to examine the effect of SAP and nano-silica on the properties of blended cement paste. The experimental investigations via several tests such as consistency, setting time, compressive strength, UPV, and acid test were performed. Based on energy-dispersive X-ray analysis (EDX) test and scanning electron microscopy (SEM) test results, the morphology of hydration products and mineral compositions of cement paste were further analysed, and the mechanism of SAP with 0.2% and 0.3% and NS with lower percentages ranging from 0.5% to 2% on the performance of cement paste was studied. The results exhibited that incorporating SAP in various percentages from 0.5% to 2% prolonged the initial setting time, reduced the fluidity, and increased the water content and formation of pores. In addition, various percentages ranging from 0.5% to 2% of NS were added; thereby, an increase in the hydration process and refining the microstructure was found. The microscopic test results showed that the blended cement paste can effectively improve the denser microstructure and refine the pore structure.

Keywords: cement paste; superabsorbent polymer; nano-silica; setting time; microscopic studies; strength; durability

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Citation: Muthalvan, R.S.; Ravikumar, S.; Avudaiappan, S.; Amran, M.; Aepuru, R.; Vatin, N.; Fediuk, R. The Effect of Superabsorbent Polymer and Nano-Silica on the Properties of Blended Cement. *Crystals* **2021**, *11*, 1394. https://doi.org/10.3390/cryst11111394

Academic Editor: Yurii Barabanshchikov

Received: 21 October 2021
Accepted: 12 November 2021
Published: 15 November 2021

1. Introduction

Cement mortar is the basic constituent used in any form of construction throughout the world over the years. The evolution of infrastructure development demands the enhancement and incorporation of new properties for the cement mortar. The degree of hydration of cement plays a major role in the strength and durability of concrete, which can be obtained by optimising the pore structure, reducing porosity, and reducing macropores to produce high-performance concrete [1,2]. Previous research studies carried out on the hydration mechanism of cement showed that the inner structure of concrete will have a fine pore size and compact structure if the water–binder ratio is relatively low in concrete [3,4]. However, this creates a more unhydrated cement, resulting in a decrease in internal relative humidity and an increase in the autogenous shrinkage of concrete [5,6]. Effective water curing will ensure the hydration process of cementitious materials in concrete. If the moisture is not sufficient, it will lead to early shrinkage and cracking of the concrete, which will adversely affect the growth of mechanical and durability properties of concrete at a later age. In practice, external water curing is usually adopted, but it is very difficult to produce timely and adequate curing; therefore, it may lead to microcracks in concrete [7,8]. The internal curing method has been studied for many years, and it has been proved that it is an effective way to ensure the hydration process in concrete. This internal curing is adopted by introducing an absorbent material such as a superabsorbent polymer (SAP), which will be added in advance into the concrete. SAP is a type of synthetic polymer that has a unique capacity to absorb and retain water as much as possible. When it is added to the concrete, it acts as an internal curing agent in the hydration process of the cementitious material [9]. When the relative humidity inside the cement matrix gradually drops, the absorbent material supplies the necessary water for the hydration of the cement paste, which leads to the formation of calcium silicate hydrate (CSH) gel in a longtime process. This CSH gel formation seals the cracks; thus, SAP plays a major role in achieving self-healing cement mortar [10,11]. The introduction of superabsorbent polymer (SAP) induces the self-healing mechanism (however, only the microlevel cracks could have been healed in practice) in concrete/mortar specimens, while, on the other hand, it reduces the characteristic strength adversely [12]. Various researchers carried out concrete incorporating SAP research, and the influence of SAP on the construction performance, internal curing, shrinkage, strength, and durability of concrete has been studied [13–15]. It has been concluded that the addition of SAP has both positive and negative effects on the concrete properties. Added in concrete, SAP absorbed water in the mix, which reduces the slump of the concrete and thereby affects concrete workability [16–18]. If SAP has pre-absorbed water content, it can increase the porosity and total pore volume of concrete, affecting its strength. However, if the SAP without the pre-absorbed water was added to concrete, it could reduce the porosity and total pore volume of concrete [19,20]. The performance of concrete added with different kinds of SAP was compared. The research result concluded that if the SAP particle size was small, the water absorption rate and the volume of SAP had a negative impact on the performance of concrete [21]. The addition of SAP with a w/b ratio of 0.45 does not have any negative impacts. If w/b was more than 0.55, it affects the compressive strength at early ages. However, higher doses of SAP result in improved tensile strength of mortar [22]. Intrinsic capacity to absorb water molecules by SAP particles can be used as an alternative in mortars to adjust the moisture level in the building envelopes passively. Adjusting the dosages of SAP particles, and the water-to-binder ratio using a flow table will help achieve a desirable mechanical resistance [23]. It is proven that incorporating SAP particles in cement mortar/concrete decreases its characteristic strength [24–26]. The nano-silica particles were added to the mix, acting as another admixture in forming CSH gel to nullify the strength loss [27]. The review paper discussed the effect of nano-silica (NS) addition on cement concrete and concluded that NS increases the flexural, compressive, split tensile strength of the concrete and yields a denser matrix than plain concrete. The use of nanomaterials also enhanced the durability of concrete, thus providing a sustainable solution in the civil engineering field [28]. NS has a significant effect on the rheology of the blended mix but provides early ionic strength by accelerating the hydration kinetics. Incorporating NS particles in the cement paste does not significantly affect the relationship between the Young modulus and the compressive strength of the ternary-blend cement paste [29]. Ternary-blended cement mix containing nano-silica and natural pozzolans outperformed the binary ones containing Portland cement and nano-silica in terms of reducing costs and environmental effects and improving mechanical properties and durability. Ternary-blended mixtures incorporating 4% nano-silica with 15% zeolite performed well at 90 days with 51.9% strength improvement than control mix [30]. Various researchers performed mechanical and durability studies on cementitious composites added with different types of nanomaterials. In one of the studies, the effect of nanoparticles on microstructural surface characteristics, abrasion resistance, and skid resistance of concrete pavements were seen. Nano-modified concrete yielded the most considerable improvement in abrasion resistance (about 23%) [31]. Lefever et al. [32] reported that the addition of SAP lowers the compressive strength due to pore formation, and the nano-silica increases the property by the nucleation effect and early pozzolanic reaction, the combination of SAP and nano-silica showed an almost identical mechanical performance to the reference mixture. Baloch et al. [33] investigated the co-effects of super absorbent polymer (SAP) and nano-silica (NS) in superplasticised cement paste (SCP) systems. They concluded that SAP increased the SP demand of the SCP system even with the additional entrained water. The presence of SAP itself as a hydrogel can be responsible for decreased flow. The addition of nano-silica in concrete improved the mechanical and shrinkage properties of both SAP-modified conventional and SCP formulations. These improvements are mainly contributed to the physical packing effect and high pozzolanic activity of NS particles. It was concluded in the study that the consumption of $Ca(OH)_2$ and increased production of CSH contributed to improved properties. It was further inferred that a rational content of 2% NS for SCPs and 1% for conventional formulations corresponds to the best mechanical properties [34]. This paper suggested for future studies that the usage of high doses of nano-silica particles of about NS > 2% may lead to scenarios such as agglomeration, which have to be addressed. This SAP has a considerable ability to absorb water multiple times of its own weight. SAP has a continuous holding capacity of water utilised by the cement matrix to form the CSH gel [31,32]. Higher SAP dosage increases the swelling contrast and decreases the size of SAP voids in cement paste, thereby reducing its impact on strength. Previous papers have researched SAP dosages of 0.5%, 1%, and above, concluding that the higher the dosage of SAP is, the more the loss of strength in the concrete becomes [33,34]. Thus, it has been advised to investigate further studies of much lesser dosages of SAP (<0.5%). On the other hand, the addition of pozzolanic material such as fly ash, GGBS, and nanoparticles such as nano-silica and nano-lime were added to the mix, increasing the strength characteristics to compensate for strength loss caused by the addition of SAP [35–38]. It was recommended to use the SAP and NS particles because over-dosage or non-uniformity in the mixture leads to agglomeration of the particles, thus affecting the performance of the mix.

Many of the previous studies dealt with mechanical and durability studies of SAP-blended cement mortar/concrete [39–42]. However, previous researchers have not studied the fresh properties of blended cement paste with lower dosages of SAP and NS. Hence, this study aims to address two issues: (i) the effect of SAP and nano-silica on the standard consistency and setting times of blended cement paste and (ii) the effect of SAP and nano-silica on the compressive strength and UPV test of blended cement mortar. In this study, the performance of the blended cement mix with 0.2% and 0.3% SAP and with 0.5%, 1%, 1.5%, and 2% NS was investigated. The sodium polyacrylate (a type of SAP) particles in the range of 65–100 microns and nano-silica of 12 nm were used in this study.

2. Materials and Mix Proportions

Experiments were conducted on cement paste with varying SAP and nano-silica percentages with and without superplasticisers. Microstructural studies for the blended cement paste such as scanning electron microscopy (SEM, Dextrose Technologies Private Limited, Bengaluru, India) and energy-dispersive X-ray analysis (EDX, Dextrose Technologies Private Limited, Bengaluru, India) were carried out, and the results are presented. Blended cement mortars incorporating SAP and nano-silica were prepared to investigate the compressive strength, UPV, and acid tests. Portland pozzolana cement (PPC) 53-grade was used to prepare testing samples in this study. M-sand is the most common alternative of river sand used in construction activities as fine aggregate. The M-sand used in this study was double washed to fulfil the quality requirements.

The M-sand used in this study had a specific gravity, determined by IS 2386 [43], as 2.64. The fineness modulus as per IS 383:2016 [44] was determined as 2.9. This modulus confirms that the fine aggregates' average size was between the 2nd and 3rd pan, i.e., between 300 and 600 micron sieve belongs to medium sand. Sodium polyacrylate (SAP) is a synthetic superabsorbent polymer that has the ability to absorb as much as 500 times its mass in water.

Figure 1b shows the SAP before water absorption. When dry, it is seen as a white powder but turns into a gel-like substance when wet after water absorption (Figure 1c) and is primarily used as a thickening agent. The SAP particle size was 70–120 microns. The SAP was added to the cement mix to absorb the water and retain it as long as possible to support the specimen to form calcium silicate hydrate (CSH) gel. SAP is the main admixture added to the cement mix in optimum dosage to obtain the sustainable cement mortar.

(**a**) (**b**) (**c**)

Figure 1. (**a**) Blended cement mix; (**b**) SAP before water absorption; (**c**) SAP after water absorption.

Additionally, nano-SiO_2 is a fluffy white powder composed of high purity amorphous silica powder. Due to its small particle size, nano-SiO_2 had the advantages of large specific surface area, strong surface adsorption, large surface energy, high chemical purity, and good dispersion. The particle size of nano-SiO_2 used in this experimental study was about 12 nm. The nano-silica was added at an optimum quantity in the cement mixture to improve the strength and durability characteristics of the blended cement mortar.

SEM analysis was performed using a VEGA3 TESCAN microscope for studying the microstructural properties of SAP, nano-silica, and setting time samples of the blended mix. Figure 2a,b present SAP and nano-silica SEM images, respectively. The SAP particles show a flaky shape with sharp-edged angularity, whereas the nano-silica particles show a rounded shape. EDX is an analytical technique used for studying the elemental properties/chemical characterisation of the samples. This technique was used in conjunction with SEM. The samples were examined by passing an electron beam with an energy of 10 keV strikes the sample's surface, causing X-rays to emit from the samples. The energy of the emitted X-rays varies depending upon the elemental properties of the sample. In this study, SAP, nano-silica, and setting time samples of the blended mix were tested, and the percentage weight and percentage atom elements present in the samples were determined.

The SAP and nano-silica were mixed and added to cement mix in this study in various percentages such as (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2: 2), (0.2:1.5), (0.2:1), (0.2:0.5), and it is used as blended cement mix (Figure 1a) to produce sustainable mix. The 53 grade PPC, conforming to the standards suggested by IS 1489 (Part I; 1991) [45], and the M-sand, conforming to the standards suggested by IS 383 (2016) [44], were used to cast blended cement mortar samples with 0.5% superplasticisers and 0% superplasticisers.

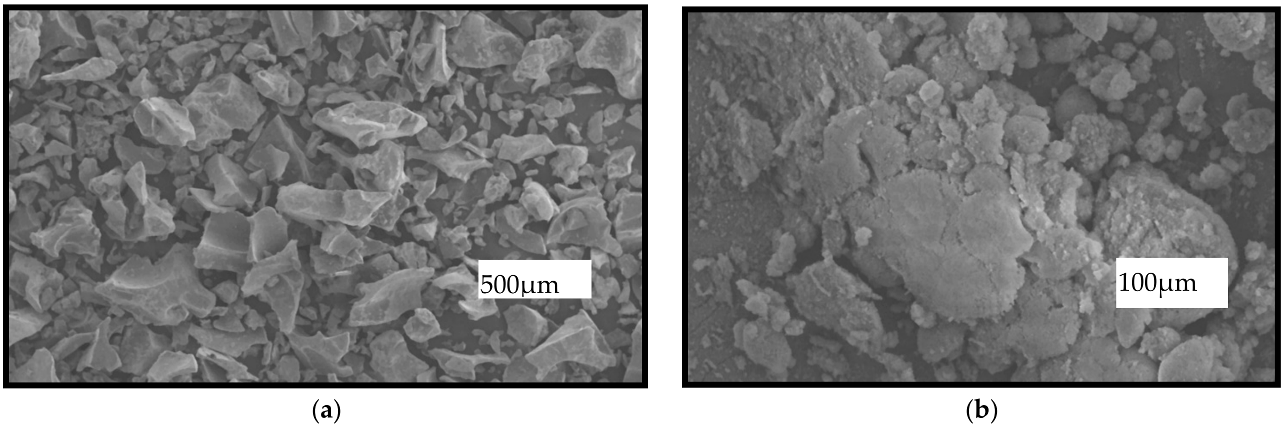


Figure 2. (**a**) SEM image of SAP particles; (**b**) SEM image of nano-silica.

3. Preparation of Specimens

3.1. Consistency Test

Consistency is the ability of a freshly mixed cement paste or mortar or binder mix to flow. Briefly, 400 g of the dry mix was prepared by mixing cement with each percentage combination of SAP and NS—namely, (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2:2), (0.2:1.5), (0.2:1), (0.2:0.5), and was weighted, and water was added as 28% of the mix to prepare the blended cement paste as a trial basis. The standard consistency test was carried out using the Vicat apparatus (Figure 3) in the laboratory as per the IS 4031 (Part 4; 1988) [46]. The Vicat plunger would stop penetrating around 5–7 mm before it reached the bottom surface. The standard or normal consistency test was performed to find the amount of water content required to produce blended cement paste. Trials were conducted with a 0.5% increment of water until the standard consistency (p) was reached. Based on the trials, with varying percentages of water, pastes were prepared to find the required amount of water for achieving the standard consistency (p). The paste was mixed thoroughly within 3 to 5 min and filled in Vicat mould using a trowel. The plunger having a 5 mm round needle was released and allowed to sink into the test mould. The plunger penetration was observed, and the procedure was repeated for various trials until obtaining the required reading of penetration of plunger about 33 to 35 mm from the top of the mould. The corresponding water content added was noted as the standard consistency of the mix. The standard consistency test was conducted for all the 8 blended mixed, and the corresponding water content added was noted as the standard consistency of the mix.

Figure 3. Vicat's mould apparatus setup on consistency test of cement paste.

3.2. Test Samples' Setting Times

Determination of initial and final setting times was performed according to IS:4031 (Part 5; 1988) [47], which requires preparation of cement paste by adding 0.85 times of water of standard consistency. The initial setting time is the time when the cement paste starts losing its plasticity from adding water to it. The time from adding water to when the mix began hardened and attained certain structural strength is regarded as the final setting time.

The blended mix was gently mixed for the initial setting time (Figure 4) to obtain a uniform mix and filled in the Vicat mould within 3 to 5 min (gauging time). A 1 mm square needle is used to penetrate into paste at every 2 min intervals until the index scale shows 5 mm to 7 mm from the bottom of the mould. The needle was replaced with an annular attachment to determine the final setting time (Figure 4). Readings were noted at every 30 min interval; the attachment was dropped on the paste surface till the annular attachment failed to make an impression. A total of 400 g of the dry mix was prepared by mixing cement with each percentage combination of SAP and NS—namely, (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2:2), (0.2:1.5), (0.2:1), (0.2:0.5), and was weighted, and 0.85 P of water by weight of the mix was added to it. Since the water to be added for attaining the consistency of the cement paste is so high, a superplasticiser may be added to reduce the required water content. None of the previous experimental studies dealt with the influence on setting time after adding a superplasticiser. In this study, the poly-carboxy-based superplasticiser of 0.5% was added for the 8 mixes considered. The variation between their setting time was blended cement mix with SP and without SP was observed. These test procedures were repeated for determining the initial and final setting time for the considered 8 mixes.

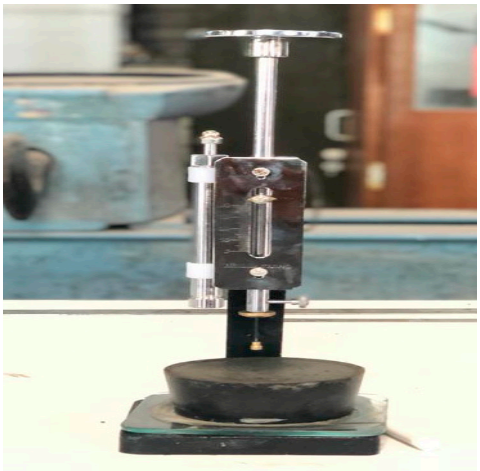


Figure 4. Vicat's mould apparatus setup on setting test of cement paste.

3.3. Samples to Find the Compressive Strength of Concrete

To determine the compressive strength of the blended cement mortar, the blended cement mortar samples were prepared according to IS 4031 (Part VI; 1988) [48]. The mortar cube moulds of 70.6 mm $\times$ 70.6 mm size, porking rod, and a vibrating machine were used. The room temperature was recorded as 27 $\pm$ 2 °C, based on the recommendations. The PPC conformed to the standards suggested in IS 1489 (Part I; 1991) [45], and the M-sand conformed to the standards of IS 383 (2016). The test setup for compressive strength is shown in Figure 5a. The dry mix was prepared by mixing cement with each percentage combination of SAP and NS—namely, (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2:2), (0.2:1.5), (0.2:1), (0.2:0.5), and M- sand in the ratio of 1:3. The dry mix was mixed gently at least for a minute, and the water was added to the mix as per the IS standards (P/4 + 3) percentage of the total weight of the blended cement and M-sand for obtaining a uniform mixture, where P is the standard consistency of the corresponding mix determined earlier. Figure 5b depicts the prepared blended mix samples of size 70.6 mm × 70.6 mm.

(**a**) (**b**)

Figure 5. (**a**) Test setup of compressive test; (**b**) preparation of blended mix samples.

3.4. Samples to Find the Quality of Concrete by UPV Test

The compressive strength specimens were used to test the ultrasonic pulse velocity test, which is an in situ non-destructive test to check the quality of concrete. In this test, the strength and quality of concrete were assessed as per IS 13311 (Part I; 1992) [49] by measuring the velocity of an ultrasonic pulse passing through a concrete structure. Here, the ultrasonic pulse was generated by an electroacoustic transducer. Figure 6 depicts the test setup of the UPV test. After the pulse was induced into the cement mortar (or any structural/concrete specimen) from a transducer, it multiple reflected off the boundaries of the different material phases within the specimen. A complex system of stress waves was developed in the specimen, including longitudinal, shear, and surface waves. The receiving transducer was used to detect the onset of the longitudinal waves, which is the fastest. Since the length of the mortar specimen was 70.6 mm (<500 mm), the natural frequency of the transducer used here was 150 kHz as per the IS standards. Additionally, Figure 7 shows more deterioration and weight loss while exposed to the acidic medium. Exposure of mortar specimens to various acidic media generally results in degradation, which was most obvious on the exposed surface. It was proven that reaction products include, in addition to silica hydrogels, aluminium and iron oxides, and acid calcium salts, which, if soluble, were removed from the solution, resulting in a decalcification process reflected in weight and losses in physical–mechanical properties.

Figure 6. UPV testing setup to test blended cement mix mortar samples.

Figure 7. Deterioration of blended cement mortar when it is exposed to acid medium.

4. Results and Discussion

4.1. Setting Time

The samples were prepared for various blended mix proportions. Various testings were conducted to study the effect of SAP and NS on the properties of blended cement paste. The results are discussed below.

The standard consistency of the PPC without adding SAP and NS used was found to be 34%. The standard consistency test was conducted for all the eight blended mixes, and the corresponding water content added was noted (Table 1) as the standard consistency of the mix. It was generally observed that the addition of SAP and NS requires more water for obtaining a uniform mix, to gently bind while attaining complete hydration with the cement.

Table 1. Percentage of consistency and setting times of blended cement paste.

MIX ID	Mix Details	Consistency (%)	Initial Setting Time (min)	Initial Setting Time (SP-0.5%) (min)	Final Setting Time (min)	Final Setting Time (SP-0.5%) (min)
M1	SAP-0.3%-NS-2.0%	42	265	315	725	650
M2	SAP-0.3%-NS-1.5%	40	235	275	775	675
M3	SAP-0.3%-NS-1.0%	38	205	245	820	740
M4	SAP-0.3%-NS-0.5%	36	190	230	860	775
M5	SAP-0.2%-NS-2.0%	39	250	290	700	615
M6	SAP-0.2%-NS-1.5%	38	215	260	750	665
M7	SAP-0.2%-NS-1.0%	37	200	250	795	725
M8	SAP-0.2%-NS-0.5%	36	190	250	835	745

The initial and final setting times for the eight mixes considered in this study were determined using the Vicat apparatus. In these mix proportions, the poly-carboxy-based superplasticiser of 0.5% was added for the eight mixes considered. Table 1 presents the observed variation between their setting time with SP and without SP. It was found that the addition of a constant quantity of superplasticiser (0.5%) further prolonged the initial setting time and shortened the final setting time.

Strength specimens were tested corresponding at 3, 7, 14, and 28 days from the time of adding water to the mix (age of the specimens). The compressive strength of the specimens was recorded by applying load uniformly, and the rate of loading increased gradually, starting from zero to 35 $N/mm^2/min$. The mixes M3 and M7 (Figure 8) having the dosage of NS as 1% were determined to be the optimum, as they showed the maximum compressive strength.

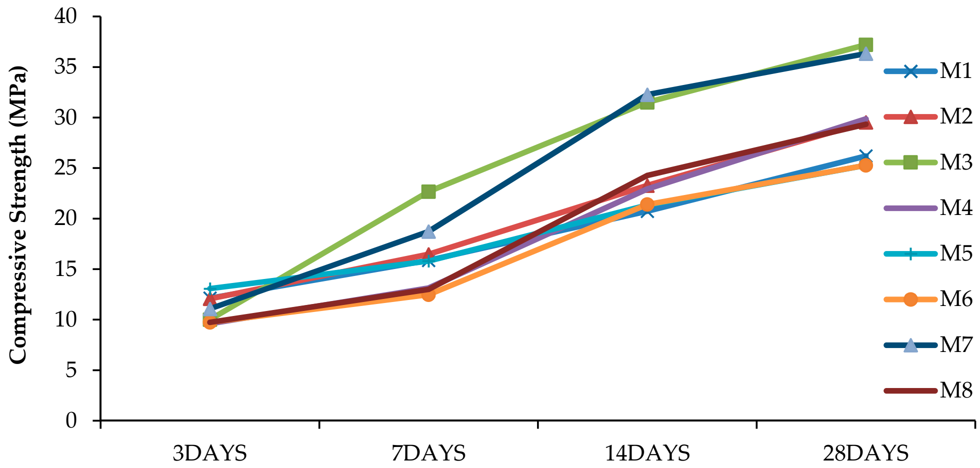


Figure 8. Results of compressive strength of blended cement mortar.

The specimens of different mix combinations were tested on the 28th day for the velocity of the ultrasonic pulse transmitting through them. The average values of the velocity of the transmitting pulse (Table 2) corresponding to three specimens from each mix seemed to be 'good' specimens based on the suggestions listed in Table 2, adopted from IS 13311 (Part I; 1992) [45].

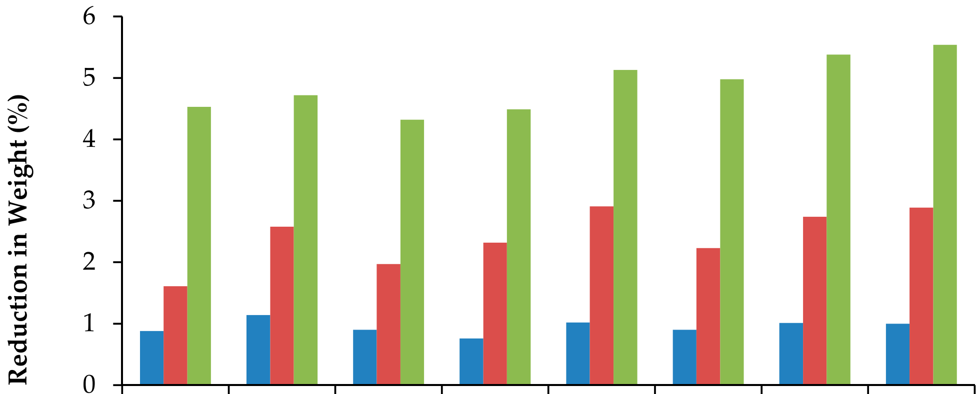
Table 2. Results of UPV test of blended cement mortar.

MIX ID	Mix Details	Ultrasonic Pulse Velocity Test (UPV) @ 28 Days		
		Velocity (m/s)	Time Taken (Micro Seconds)	Quality of the Specimen
M1	SAP-0.3%-NS-2.0%	4.10	17.07	GOOD
M2	SAP-0.3%-NS-1.5%	4.02	17.40	GOOD
M3	SAP-0.3%-NS-1.0%	3.91	17.90	GOOD
M4	SAP-0.3%-NS-0.5%	3.88	18.07	GOOD
M5	SAP-0.2%-NS-2.0%	3.95	17.73	GOOD
M6	SAP-0.2%-NS-1.5%	3.94	17.77	GOOD
M7	SAP-0.2%-NS-1.0%	3.94	17.80	GOOD
M8	SAP-0.2%-NS-0.5%	3.88	18.07	GOOD

The compressive specimens were prepared to determine the durability properties of the blended cement mix when it is exposed to acid attack for 7, 14, and 28 days. Blended cement mortar cubes were prepared similar to compressive strength specimens for various percentage combination of SAP and NS—namely, (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2:2), (0.2:1.5), (0.2:1), (0.2:0.5). The prepared samples were cured for 28 days. Then, the specimens were taken out from curing and kept at room temperature for 2 days to attain a constant weight. Afterward, those specimens were weighed and immersed in a 5% H_2SO_4 solution. The specimens were taken out from acid solution in 7, 14, and 28 days, kept at room temperature for 2 days to attain a constant weight, and were weighed to determine weight loss.

Figure 7 depicts the deterioration of blended cement mortar specimens taken out after the acid test. This weight loss is because the sulphuric acid reacts with CSH gel to form silicate oxide in aqueous state and calcium salts of soluble nature. Scaling and softening of cement paste occurs due to the decomposition of $Ca(OH)_2$, and the formation of a large amount of gypsum, which is washed off. The microstructure becomes weak, and cement paste is lost, thus justifying the weight loss of the specimen.

From the observed results (Figure 9), it is found to be that the mortar sample of mixes having 0.2% of SAP content (M5, M6, M7, and M8) showed more deterioration and weight loss while exposed to the acidic medium (Figure 7).



	M1	M2	M3	M4	M5	M6	M7	M8
7 Days	0.88	1.14	0.9	0.76	1.02	0.9	1.011	1
14 Days	1.61	2.58	1.97	2.32	2.91	2.23	2.74	2.89
28 Days	4.53	4.72	4.32	4.49	5.13	4.98	5.38	5.54

Figure 9. Acid test results of blended cement paste.

4.2. Microstructural Characteristics

A scanning electron microscope (SEM) was used to analyse the internal microstructural characteristics. The textural and compositional interrelationships, as well as the internal bonding of the hydrated cement paste, were studied for various mixes of varying dosages of SAP and nano-silica—namely, (0.3:2), (0.3:1.5), (0.3:1), (0.3:0.5), (0.2:2), (0.2:1.5), (0.2:1), (0.2:0.5), using SEM. The samples taken from the test block used to determine the setting time using Vicat's mould were tested (Figure 10) for their microstructural properties using SEM. Figure 10A–H show the set of SEM images of test block samples without superplasticiser, while Figure 10I–P depict the set of SEM images of the test block samples with an added superplasticiser. The ettringite formation (needle-like structure) was observed in the SEM images of the test block samples, confirming the cement paste's setting. These ettringites (chemically hexa-calcium aluminate tri sulphate hydrate) were responsible for cracks in hardened cement paste/cement mortar/cement concrete (shrinkage effect) due to its expansion nature during the hydration of cement. However, these ettringites were unstable and gradually converted into monosulphate as the final hydration product. The specimens containing superplasticiser showed better internal bonding and compositional placement compared with the samples without superplasticiser. These specimens were free from the agglomeration of nano-silica particles; thus, the superplasticiser plays a significant role in forming a fine binding matrix for the formation of CSH gel during the hydration process.

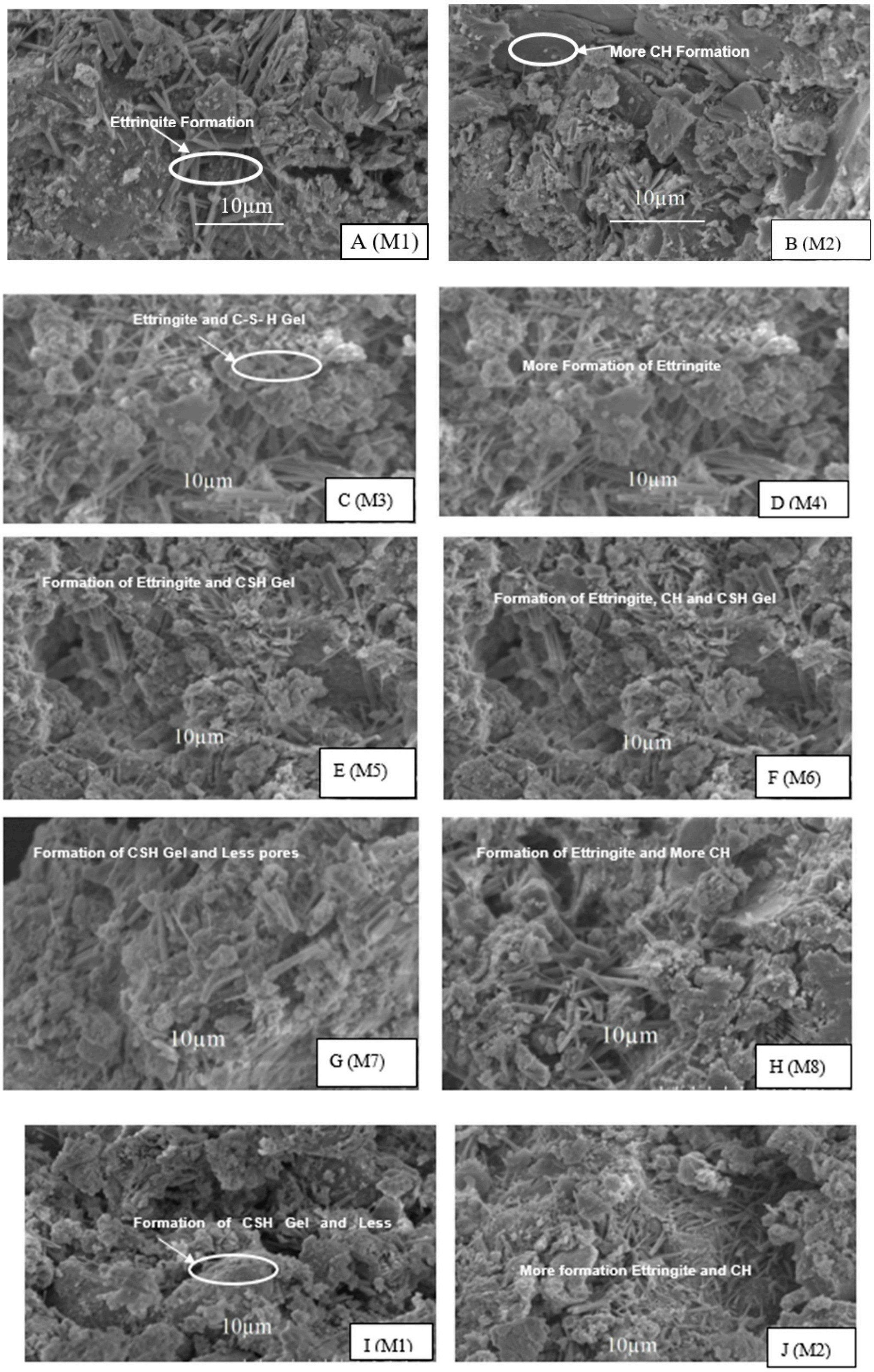


Figure 10. *Cont.*

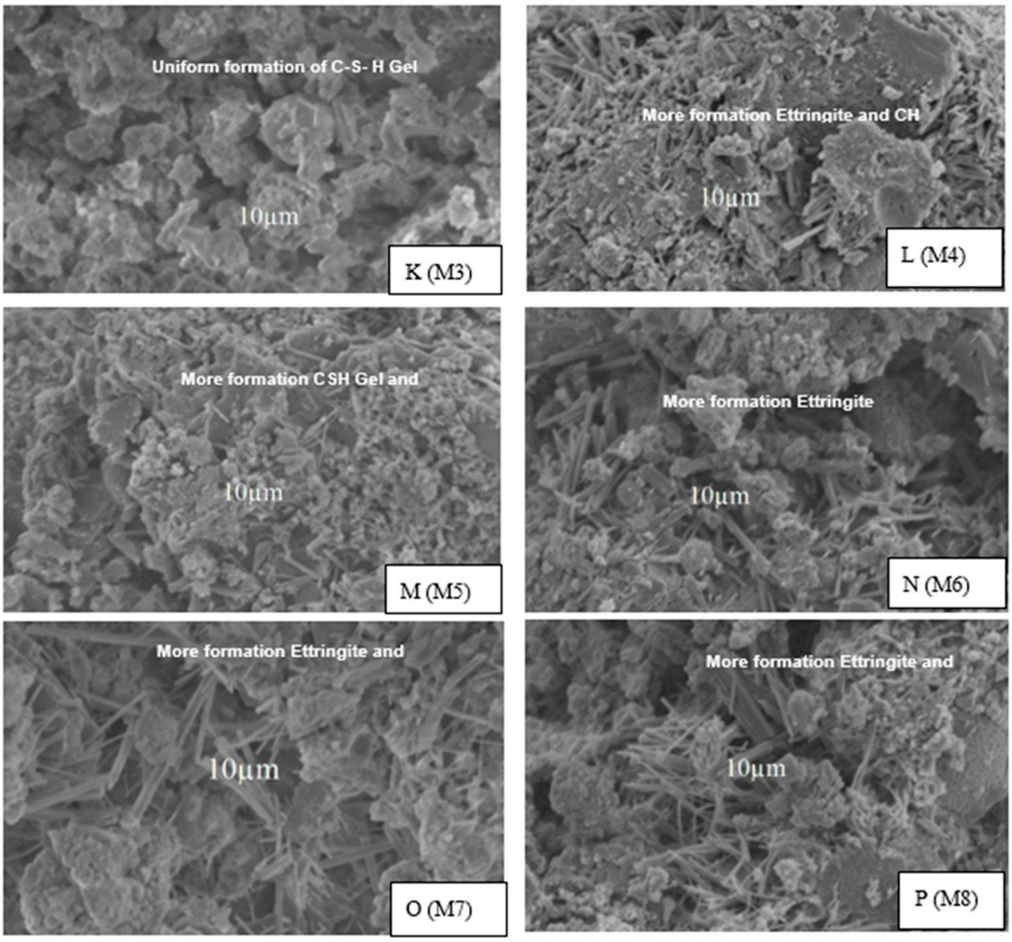


Figure 10. (**A**–**H**) SEM images of test block samples of different mixes containing different dosages of SAP and nano-silica without superplasticiser; (**I**–**P**) SEM images of test block samples of different mixes containing different dosages of SAP and nano-silica with a constant dosage of superplasticiser (0.5%).

The EDX analysis (energy-dispersive X-ray analysis), referred to as EDS or EDX, is an X-ray technique used to identify the elemental composition of materials. The data generated by EDX analysis consist of spectra showing peaks that correspond to the elements making up the true composition of the sample being analysed. Elemental mapping of a sample and image analysis are also possible. The technique can be qualitative, semi-quantitative, and quantitative and also provide the spatial distribution of elements through mapping. The EDX technique is non-destructive and specimens of interest can be examined in situ with little or no sample preparation. The EDX analysis was performed for the samples (test block samples obtained from setting time test) of M3 (Figure 11A), and M7 (Figure 11B) mixes without the superplasticiser, and also of M3 (Figure 11C) and M7 (Figure 11D) mixes with superplasticiser. Tables 3 and 4 present their element compositions. The element analysis results showed that there was no significant difference in their composition in terms of% of atom and% of weight, and only their structural placement and bonding nature differed, thus justifying their compressive strength characteristics in cement mortars.

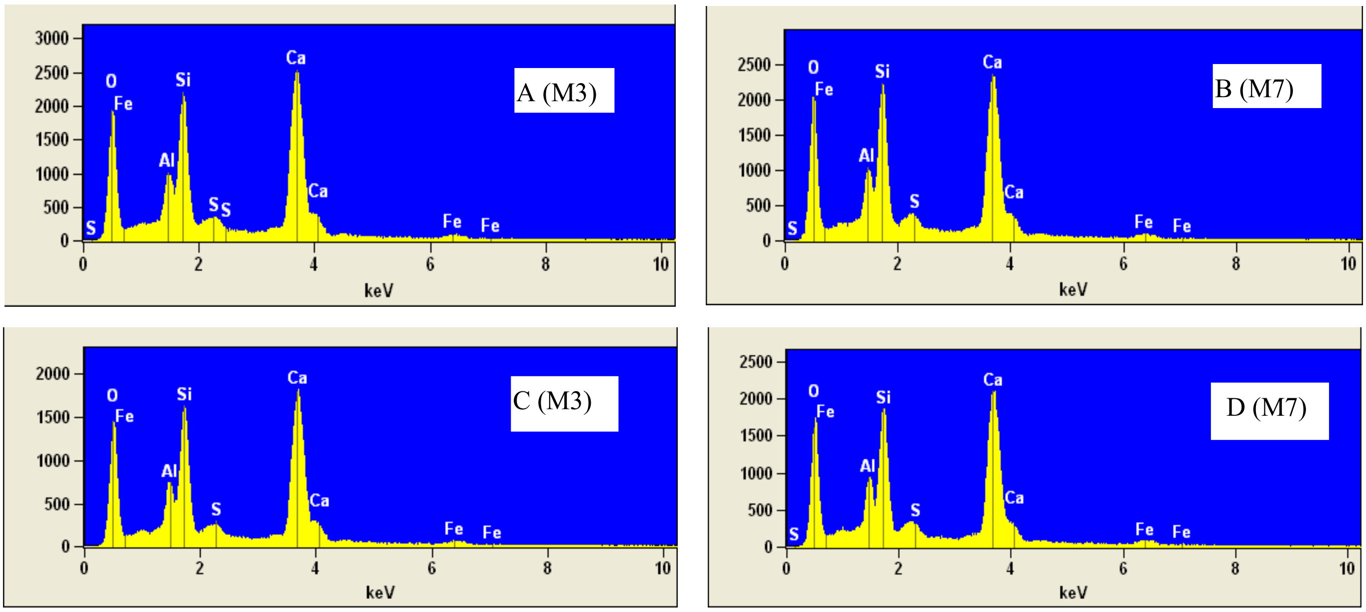


Figure 11. (**A**,**B**) EDX output of setting time samples of blended mixes M3 and M7 without superplasticiser; (**C**,**D**) EDX output of setting time samples of blended mixes M3 and M7 with superplasticiser.

Table 3. The element compositions for the setting time samples of. blended mixes M3 and M7 without superplasticiser.

Element	M3 without SP		M7 without SP	
	Weight%	Atom%	Weight%	Atom%
O	48.05	66.89	48.74	67.45
Al	4.26	3.51	4.35	3.57
Si	12.72	10.08	13.21	10.41
S	0.85	0.59	0.82	0.56
Ca	33.88	18.83	31.89	17.62
Fe	0.224	0.09	0.99	0.39

Table 4. The element compositions for the setting time samples of blended mixes M3 and M7 with superplasticiser.

Element	M3 with SP		M7 with SP	
	Weight%	Atom%	Weight%	Atom%
O	48.26	67.12	47.42	66.49
Al	4.31	3.55	4.61	3.83
Si	12.81	10.15	13.10	10.46
S	0.59	0.41	0.37	0.26
Ca	33.25	18.46	32.24	18.05
Fe	0.77	0.31	2.27	0.91

Furthermore, the cementitious composites and concrete research [35–38] were carried out with the SAP dosages of around 1%, 2%, 5%, 10%, and above to achieve the self-healing effect. However, research results showed very poor strength characteristics, leading to examining much lower SAP dosages such as 0.2% and 0.3% in this experimental study. The nano-silica was chosen to add as an admixture to compensate for the strength loss due to the addition of SAP. This study dealt with the various combinations of dosages of NS, along with the SAP particles. The addition of SAP and nano-silica particles seemed to require more water for its binding, along with the hydration of cement, and an increase in the dosages of SAP and NS increased the water requirement, which is similar to the results of other NS studies reported by many researchers worldwide [50–53]. The mix M3 and M7 (having 1% NS significantly) seemed to have more compressive strength comparatively. The cement mortar samples of much higher 0.3% SAP dosage showed better results than the 0.2% SAP dosage samples. Thus, the SAP dosage plays a significant role in characterising its durability resistance against acid exposure. An in-depth investigation is needed in future studies for determining its underlying mechanism. This paper dealt with the detailed study on the microstructural characteristics of SAP, along with nano-silica, for compensating the effect of strength loss by forming CSH gel on blended cement mortars and their enhancement.

5. Conclusions

The effect of SAP and nano-silica on the properties of blended cement paste was studied based on experimental investigations of its consistency, setting time, compressive strength, UPV, and acid test. Based on EDX and SEM test results, the morphology of hydration products and mineral compositions of blended cement paste were further analysed, and the mechanism of SAP and NS on the performance of cement paste was studied. Based on the analyses, the following conclusions were made:

- The standard consistency of the PPC without adding SAP and NS was found to be 34%. It is generally observed that the addition of SAP and NS requires more water for obtaining a uniform mix, to gently bind while attaining complete hydration with the cement.
- The initial and final setting times for the eight mixes considered in this study were determined. It was found that the addition of a constant quantity of superplasticiser (0.5%) further prolonged the initial setting time and shortened the final setting time.
- The compressive strength specimens were tested for eight blended mixes corresponding at 3, 7, 14, and 28 days, and it was found that M3 and M7 mixes, having the dosage of NS as 1%, were the optimum, as they showed the maximum compressive strength.
- The specimens of eight blended mix combinations were tested on the 28th day for the UPV test. It was found that the average values of the velocity of the transmitting pulse in all the blended mix specimens exhibited better enhancement in the level of hardening with the addition of an accelerator.
- The results revealed that the blended mix mortar samples having 0.2% of SAP dosage (M5, M6, M7, and M8) showed more deterioration and weight loss while exposed to the acidic medium.
- The ettringite formations (needle-like structure) were observed in the SEM images of the blended mix samples, which confirmed the setting of the cement paste.
- The SEM images of blended mix containing SP indicated a better CSH gel formation than blended mix without SP. The specimens containing superplasticiser showed a better internal bonding and compositional placement, compared with the samples without superplasticiser. These specimens were free from the agglomeration of nano-silica particles; thus, the superplasticiser plays a significant role in forming a fine binding matrix for the formation of CSH gel during the hydration process.
- The EDX analysis was performed for the samples of M3 and M7 blended mixes without SP, and M3 and M7 blended mixes with SP; it was found that there was not a significant difference in their composition in terms of % of atom and % of the weight, and only their structural placement and bonding nature differed.

From these conclusions, it is revealed that the addition of 0.2% and 0.3% SAP and 1% NS to Portland cement using 0.5% superplasticiser positively affects pore structure refinement, strength, and durability of the blended cement mortar. Hence, this mix has a greater potential to be used as repairing mortar in structural elements.

Incorporating SAP in concrete has several advantages, but it will increase the consistency and initial setting time. Future research could include reducing the initial setting time of blended cement by adding the accelerating agents.

Author Contributions: Conceptualization, R.S.M.; data curation, S.R., S.A., M.A., R.A., N.V. and R.F.; formal analysis, R.S.M., S.R., S.A., M.A., R.A., N.V. and R.F.; funding acquisition, M.A., N.V. and R.F.; investigation, R.S.M. and S.R.; methodology, R.S.M. and S.R.; project administration, S.A. and R.A.; resources, M.A., R.A., N.V. and R.F.; software, S.A.; supervision, S.R.; validation, S.A., M.A., R.A., N.V. and R.F.; visualization, S.A., M.A., R.A., N.V. and R.F.; writing—original draft, R.S.M. and S.R.; writing—review & editing, S.A., M.A., R.A., N.V. and R.F. All authors have read and agreed to the published version of the manuscript.

Funding: The research was partially funded by the Ministry of Science and Higher Education of the Russian Federation as the grant Self-Healing Construction Materials (Contract No. 075-15-2021-590 dated 4 June 2021).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data sharing is not applicable.

Acknowledgments: The experiments in this study were performed at the Structural Engineering Laboratory, Department of Civil Engineering, CEG, Anna University, Chennai, India. The authors wish to thank the Head of the department and non-teaching staff for providing support and access to a laboratory and procuring materials used in the study.

Conflicts of Interest: The authors declare no conflict of interest.

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