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Effect of Calcite on Fresh and Hardened Properties of Expanded Perlite Blended Cement Mortars

Mustafa ENSARİOĞLU[1], Serhat DEMİRHAN[1*]

ABSTRACT: Expanded perlite, which is mostly used for purposes such as lightweight concrete and insulation, contains a high percentage of silica and alumina. Problems in early-term strength development occur with the high-volume substitution of normal weight and lightweight pozzolanic materials in the production of cement. It was thought that it would be important to examine the early period fresh and hardened properties of expanded perlite blended cement including nano and micronized calcite minerals with high reactivity and high specific surface area/volume ratio. For this purpose, a total of nine different mortar mixtures containing 0%, 6% and 18% expanded perlite and 5% nano and micronized calcite were designed for replacing by cement. For mortar samples modified with nano and micronized calcite and containing expanded perlite at different rates; mini slump flow test, standard consistency, setting times and soundness (Le Chatelier Method) tests/analyses were performed as fresh and early period properties. In addition to this, compressive strength and ultrasound pulse velocity tests were performed for the curing ages of 7, 28 and 120 days as hardened properties. Experimental results showed that expanded perlite negatively affects the early and hardened properties of mortars, and in general, improvements are achieved with nano/micronized calcite substitution. 23.2% and 45.4% of strength development has been achieved in the mixture including both calcite and expanded perlite within the curing ages of 7-28 days and 7-120 days, respectively. Also, a maximum of 5.7% of reduction was observed in EP blended cement mortars, including 18% of EP. Since nano-sized calcite has a higher surface area compared to micronized calcite, better contributions to the fresh and hardened properties were observed in the utilisation of nano-sized calcite.

Keywords: Expanded perlite, Nano and micronized calcite, Setting time, Unit weight, Ultrasonic pulse velocity

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INTRODUCTION

Activities in mankind's life results in a worldwide CO_2 emission which is approximately 3.6 billion tons of CO_2 for each year (Long et al., 2019). A remarkable quantity of which (5-10%) belongs to the production process of cement clinker, namely ordinary Portland cement (Thwe et al., 2021). It is well-known that the utilization of mineral admixtures such as silica fume, fly ash, and blast furnace slag in the production of cement-based materials makes the hardened properties of cementitious material stronger and denser, namely better performance properties (Noaman et al., 2019). Performance contributions of pozzolans depends on its type and replacement fraction; but in general, pozzolans enhance fresh and hardened properties, i.e., strength and durability, and also decrease the fee of the cement-based material (Soydan et al., 2018). In terms of sustainability, they are great of importance and make some contributions such as enhanced durability which decreases repair charge and materials consumption, reduced energy and also cement production which lessens environmental impact of clinker production via decreased CO_2 emission (Erdem et al., 2007). Perlite is a naturally available glassy pozzolanic material widely available in lots of countries such as Turkey, Greece, Hungary and Japan (Rashad, 2016). Particularly this reserve is more abundant in Turkey, in which two-third of the world perlite reserve is available (El Mir et al., 2020). Owing to its glassy structure including high amount of silica and alumina, Perlite is categorized as a natural pozzolanic material. When Perlite is undergone to high amount of heat, it expands five to twenty times its raw volume and having too low specific gravity makes it more available for applications such as lightweight cementitious systems, both thermal and acoustic insulations and gardening (El Mir and Nehme, 2017; Detphan et al., 2018; Saraçoğlu et al., 2020). Expanded perlite is a useful puzzolanic material being utilized in lots of areas, mostly as a light filler or an insulation material (Pichór et al., 2015). In the case of expandation of perlite material, as well as processing of expanded perlite, a remarkable amount of waste perlite is obtained. Residual perlite a material having a finer particle of intensely low bulk density (generally between 60 kg/m^3 and 120 kg/m^3) (Erdem et al., 2007), which makes the utilization of the expanded perlite too hard in terms of storing and transporting. Thus, utilization of residual expanded perlite is an issue either. This issue is widespread for all the manufacturers and users of expanded perlite, resulting in an increment in operational charges. Hitherto, a lot of methods such as an insulation material, a lightweight aggregate and so on for the utilization of residual expanded perlite has been followed. Since the utilization of expanded perlite as a partially replaced mineral by cement results in a reduction in the compressive strength of the cementitious materials (Abed and Nemes, 2019), therefore, activating the expanded perlite by utilization of nano-sized and micronized calcite minerals is another method in order to balance the reduction in strength or to enhance fundamental mechanical properties of cementitious material. Of which, the synergy between aluminate phase of the expanded perlite and calcite minerals will result in additional hydration products (Demirhan, 2020; Demirhan, 2022; Çalışkan et al., 2022), since expanded perlite has a high amount of aluminate phase. Nanomaterials are new generation particles that have a particle size of one billionth of a meter and have a high reactivity due to their high surface area to volume ratio. Nowadays, many nanomaterials such as Nano Al_2O_3 (NA), Nano TiO_2 (NT), Nano SiO_2 (NS) and Nano $CaCO_3$ (NCC) are used to improve the microstructural properties of cement-based materials and also to increase their performance. The action mechanisms of nansized materials can be generally evaluated under three main headings as nucleation effect, chemical effect and dilution effect. While nucleation and filler effects are dominant in the hydration mechanism with nanoparticles such as NS and NA (Guo and Li, 2021), chemical effect is more dominant (in addition to the nucleation and filler effect) in nano-sized limestone formations such as NCC and nano-sized calcite (NC) (Cao et al., 2019; Wu et al., 2021; Wang et l., 2018). In the production of nanomaterials, two types of production approaches are followed: Top-Down and Bottom-Up. In general, nanoparticles produced by the top-down approach are obtained with a well-graded grain distribution and relatively inexpensively. Nanoparticles produced by the bottom-up approach have a uniform particle size and are also relatively more expensive because a special application and production technique is required (Demirhan, 2020). Although general engineering properties of expanded perlite have been reported by lots of experimental studies (Khanna et al., 2018), no researches have been reported in the literature about the utilization of expanded perlite modified by nano-sized and micronized calcite minerals in manufacturing cements. Therefore, the main goal of the study was intended to examine the combination of expanded perlite and calcite minerals on the fundamental performance properties of cementitious mortar. Test results showed that the residual expanded perlite activated by calcite minerals could be used as a mineral admixture in the production of cementitious materials.

MATERIALS AND METHODS

Materials

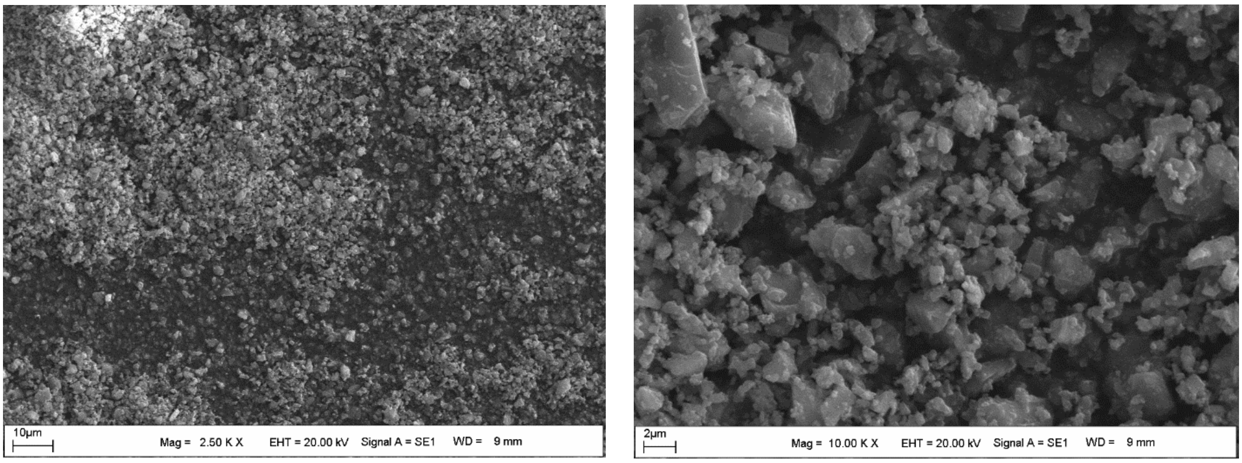
CEM I 52.5R white cement (PC) satisfying minimum requirements of TS EN 197-1 (2012), expanded perlite (EP), nano-sized calcite (NC), micronized calcite (MC), standard CEN Reference Sand with a maximum grain size of 2 mm satisfying requirements of TS EN 196-1 (2016) and also drinkable water were used in the production of mortar samples. Chemical compositions and physical properties of PC, EP, NC and MC are presented in Table 1. SEM images of NC and MC are given in Figure1.

Experimental procedure

Since nano-sized minerals have a high “specific surface area to volume ratio”, in general, there is a tendency of agglomeration in the utilisation of them. Therefore, even though both ultrasonication and surfactant were also widely preferred to overcome this issue, the most commonly used method of mechanical intergrinding was followed for the blending process of each mixture since mechanical intergrinding is widely preferred and more appropriate in the field applications (Demirhan, 2020). Nine mixtures were tailored in accordance with TS EN 197-1 and each mixture was produced depending on the amount of the ingredients (kindly, see Table 1). The data for mini slump flow test conducted in accordance with ASTM C 1437-15 (2015), standard consistency and setting times were obtained as a fresh property. In addition, unit weight and soundness (Le Chatelier) properties for 1-day curing age and both compressive strength and ultrasonic pulse velocity for the curing ages of 7, 28 and 120 days were also determined as a hardened property. For this purpose, in order to determine standard consistency and setting times, Vicat test were used in accordance with TS EN 196-3 (2017). Also, standard mortar prisms with dimensions of 4 cm × 4 cm × 16 cm were cast and a compression machine with a capacity of 2000 kN was utilized with respect to TS EN 196-1 for determining compressive strength of prism samples. Finally, UPV values of specimens were determined in accordance with TS EN 12504-4 (2021) just before applying compressive strength test to the prism samples, herein, on the same sample both of UPV and compressive strength test were applied. In the designed nine mixtures, replacement levels of 6% and 18% for expanded perlite and replacement levels of 0.0% and 5.0% were selected for minerals (NC and MC). Ingredients for all mixtures are given in Table 2. Every mixture has an abbreviation including both numbers and letters. An example of definition for mixture #5 is given in Figure 2.

Table 1. Chemical composition and physical properties of mortar ingredients

Chemical Composition, %	**PC**	**EP**	**NC**	**MC**
SiO_2	21.6	75.93	0.4	0.28
Al_2O_3	4.05	9.67	0.03	0.19
Fe_2O_3	0.26	1.16	0.05	0.02
MgO	1.3	0.04	0.5	0.75
CaO	65.7	0.55	55.4	55.98
SO_3	3.3	0.05	0.04	0.03
Na_2O	0.77	3.3	0.03	-
K_2O	0.19	5.2	0.01	-
Loss on ignition	3.2	3.1	43.5	42.75
Physical Properties				
Specific gravity, gr/cm^3	3.09	0.7	2.69	2.69
Blaine, cm^2/gr	4412	4600	-	-
BET surface area, m^2/kg	-	-	7.4	3.45



NC MC

Figure 1. SEM images of NC and MC

Table 2. Mixture proportions of standard mortars

Mix #	**Mix ID**	**PC, gr**	**EP, gr**	**Water/Binder**	**Sand, gr**	**NC, gr**	**MC, gr**	**Superplasticizer, gr**
1	C100_PER0	450	0			-		-
2	C100_PER0_MC	427.5	0			-	22.5	-
3	C100_PER0_NC	427.5	0			22.5	-	-
4	C94_PER6	423	27			-		0.16
5	C94_PER6_MC	400.5	27	0.5	1350	-	22.5	0.19
6	C94_PER6_NC	400.5	27			22.5	-	0.23
7	C82_PER18	369	81			-		1.37
8	C82_PER18_MC	346.5	81			-	22.5	1.55
9	C82_PER18_NC	346.5	81			22.5	-	1.55

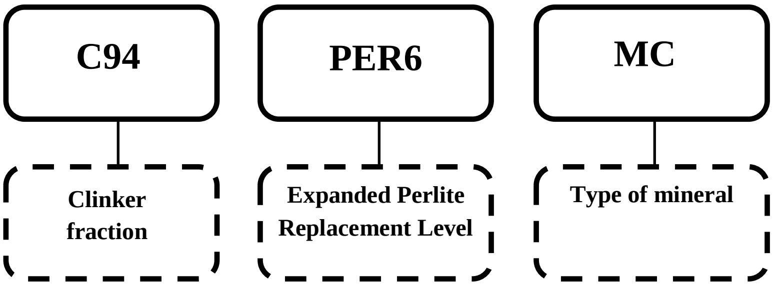


Figure 2. Representative labelling of the mixtures

RESULTS AND DISCUSSION

Soundness (Le Chatelier Method)

Measurements of soundness (Le Chatelier Method), consistency, initial and final setting times were conducted in accordance with TS EN 196-3 (2017), and the test results are given in Table 3, Figure 3, Figure 4 and Figure 5, respectively. As seen in Table 3, no expansion was observed in the mixtures including expanded perlite (EP) (Erdem et al., 2007), while an acceptable expansion within the standard range (maximum 1 cm) was observed in the control mixtures without EP. Therefore, it was revealed that the volume expansion in the mixtures produced with the EP replacement was within an allowable standard range, namely within 10 mm.

Table 3. Le Chatelier Measurements of the mixtures

Mixture ID	A (Initial, cm)	B (Final, cm)	B-A (Difference, cm)
C100_PER0	4.5	4.7	0.2
C100_PER0_MC	2.6	2.7	0.1
C100_PER0_NC	2.1	2.1	-
C94_PER6	2.5	2.5	-
C94_PER6_MC	2.6	2.6	-
C94_PER6_NC	1.1	1.1	-
C82_PER18	2.4	2.4	-
C82_PER18_MC	1.1	1.1	-
C82_PER18_NC	1.9	1.9	-

Consistency

The consistency (%) of the mixtures are given in Figure 3. As seen from the figure, the amount of water of the consistency was increased as the EP replacement level increased. This is a result of the high porosity of EP and the high specific surface area in the voids (Esfandiari and Loghmani, 2019). The consistency of the mixtures in which EP is substituted by 6% and the control mixtures without EP are very close to each other, and an increase in the water requirement was observed when the replacement ratio increased to 18%. Since calcite has a hydrophobic characteristic (Turgut, 2018; Turgut and Ogretmen, 2019; Demirhan, 2020), a partial decrease in consistency has been observed with the use of both nano and micronized calcite.

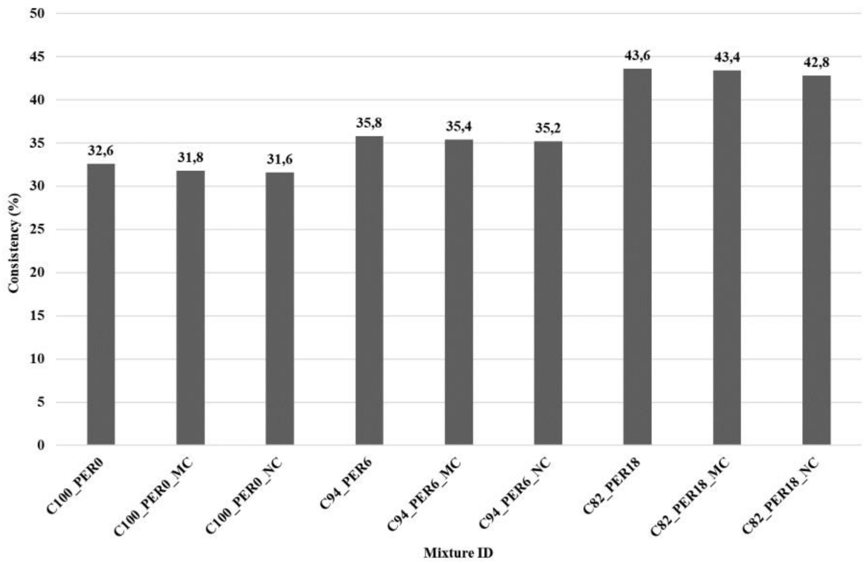


Figure 3. Consistency of mixtures (%)

Setting Times

Initial and final setting times of mixtures are given in Figure 4 and 5, respectively. As seen in the figures, since the clinker utilization rate decreased with the replacement EP, in general, delay in initial and final setting times was observed (Erdem et al., 2007). A partial decrease was observed in the setting times of the mixtures including nano and micronized calcite. This is because the promoted chemical reaction between calcite and aluminate phase of the binder, additional hydration products produced and therefore, a reduction in the setting times observed (Demirhan, 2020). Due to the low hydration mechanism of EP, which is explained in detail in the compressive strength section, an ignorable improvement was observed in both setting times with the use of calcite.

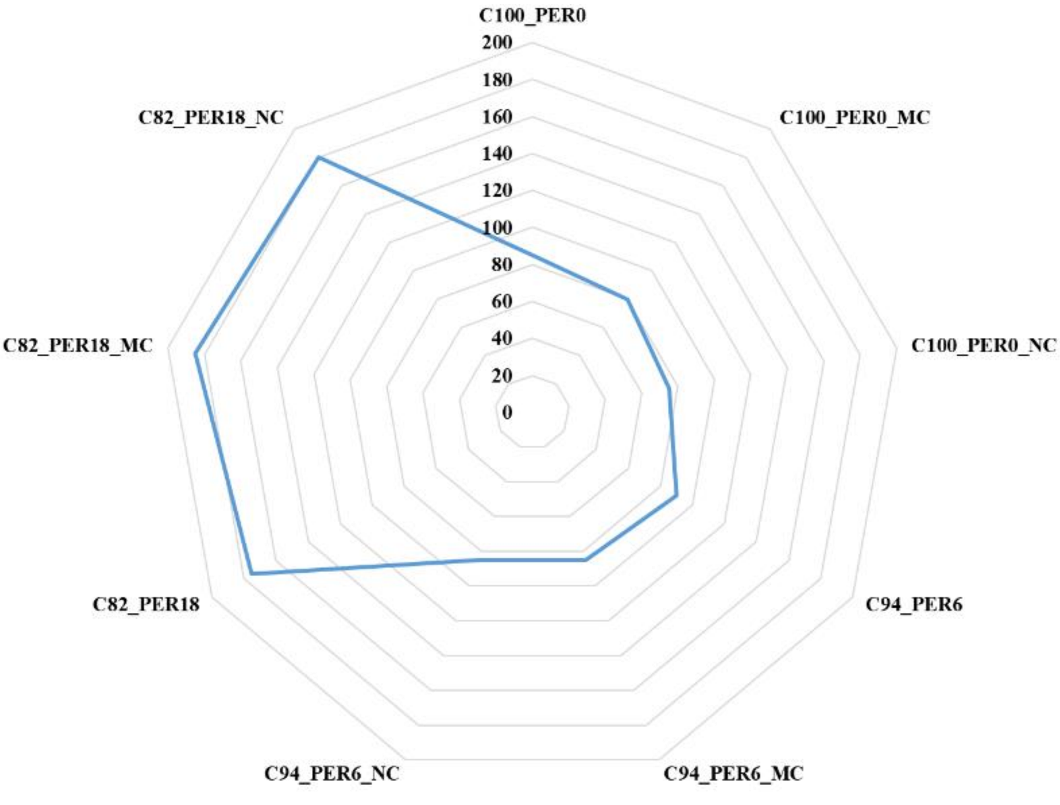


Figure 4. Initial setting times (min.)

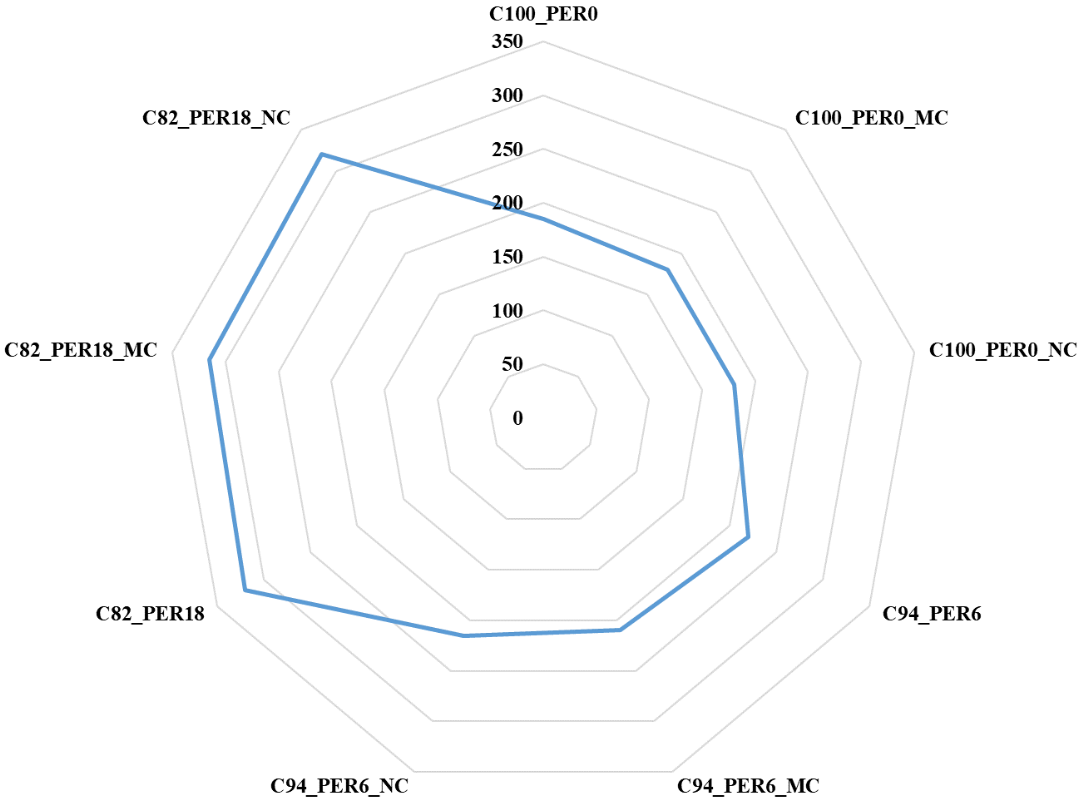


Figure 5. Final setting times (min.)

Mini Slump Flow and Amount of Superplasticizer

In the current experimental study, mini-slump flow diameters of fresh mortars were determined in accordance with ASTM C1437 (2013). In ASTM C1437, ±10% difference in the mini-slump flow diameter of the reference and modified mixtures is considered as the same workability. In this way, superplasticizer was used until the workability loss due to the utilization of EP was stayed within the limit values of ±10%. Therefore, the mini-slump flow diameters of the mixtures and the amount of plasticizer used until ±10% limit values are reached are given in Table 4. As seen in Table 4, a decrease in flow diameters was obtained with the use of EP (Bageri et al., 2021; Lanzón and García-Ruiz, 2008), and thus an increase in water demand was observed with the use of EP (El Mir and Nehme, 2017). This is a result of the large surface area and porous structure of EP. In addition, the workability was got worse with the substitution of EP with higher rates (i.e., 18%), and therefore the need for water in mixtures including EP increased to achieve sufficient workability (Sabet et al., 2013; Karein et al., 2018). In other words, its water absorption capacity due to its porous structure and its water absorption ability similar to fine porous aggregate resulted in higher water requirement in the mixtures (Sabet et al., 2013; Esfandiari and Loghmani, 2019).

Table 4. Mini slump flow diameters and the amount of the superplasticizer (SP)

Mixture ID	Mini slump flow diameters, cm	SP, gr
C100_PER0*	11.94	0
C100_PER0_MC*	11.86	0
C100_PER0_NC*	11.79	0
C94_PER6	10.82	0.16
C94_PER6_MC	10.79	0.19
C94_PER6_NC	10.93	0.23
C82_PER18	10.75	1.37
C82_PER18_MC	10.78	1.55
C82_PER18_NC	10.74	1.55

*Control mixtures

Sample Unit Weights

Unit weight is a property that is directly dependent on the ingredients of cement-based materials, and it varies depending on whether the specific gravity of the ingredients is relatively low or high. If the cementitious material replaced by cement has a relatively lower specific gravity, a decrease in unit weight is observed, and vise versa. Relative (comparing to the control mixture) unit weight values of one-day prismatic mortar samples are given in Figure 6. As seen in the figure, a decrease in unit weight values was observed as EP replacement level was increased for all mixture groups, regardless of nano or micronized calcite contents. This is a result of relatively lower specific gravity value of EP (Esfandiari and Loghmani, 2019; Demirboğa et al., 2001). Thus, with the increase in the replacement level of EP substituted for cement, a decrease in unit weight was observed (Demir and Baspinar, 2008). In the blended mixtures, the lowest unit weight values were observed in the mixtures containing 18% of EP, while the highest unit weight values were observed in the mixtures containing 6% of EP substituted for cement. In addition, no significant change in unit weight was observed when nano and micronized calcite were compared with each other.

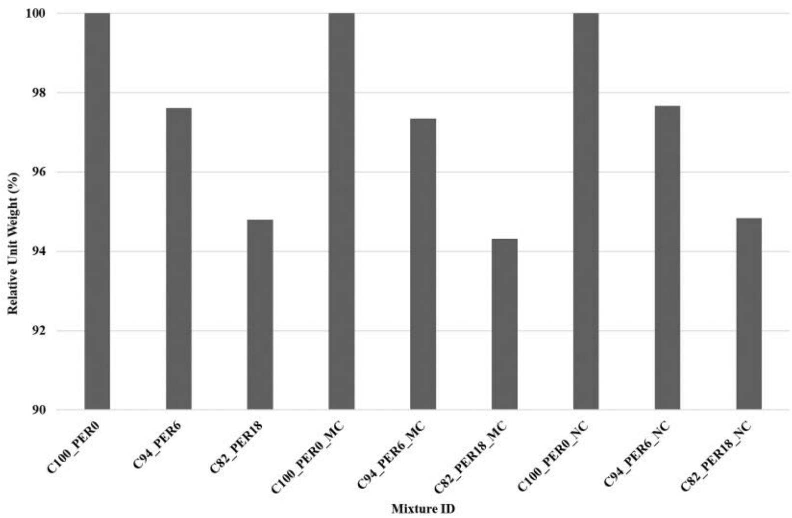


Figure 6. Relative unit weight values (comparing to the control mixture)

Compressive Strength

The average compressive strength values for the curing ages of 7, 28 and 120 days of the mixtures are given in Figure 7. A representative figure of the compressive strength development of the 120-day samples compared to the 7-day curing age is also given in Figure 8. Although significant increases in compressive strength were obtained with the use of EP in some studies (Kotwica et al., 2017), this was not valid for the current study. Thus, compressive strength of the mixtures including EP was decreased up to curing age of the 28 days. The reason for this decrease in compressive strength with the increase in the replacement level of EP is due to the very low pozzolanic activity of EP in the early age (Torres and García-Ruiz, 2009; Demir and Baspinar, 2008). The 28-day compressive strengths of the mixtures containing EP varied in the range of 50.10 and 56.80 MPa, while the 120-day compressive strengths were varied in the range of 57.30 and 63.40 MPa. The lowest compressive strength values for the 28-day curing age were observed in the mixtures including 18% of EP. However, this decrease in compressive strength is not very high. For the 28-day cure age, a 7.4% reduction in compressive strength was observed compared to the control mixture. On the contrary, an increase in compressive strength of the mixtures at curing ages of 120 days was observed (Karein et al., 2018). El Mir and Nehme (2017) were reported that the contribution of EP to strength development was significant even at the 400th day of the curing age. In the mixtures without nano and micronized calcite, the reduction rate in the compressive strength at the curing ages of 7 and 28 days were determined as 3.0% and 2.6% for the replacement level of 6%, respectively. Also, these values were 14.8% and 7.4% for the replacement level of 18%, respectively. On the other hand, in the curing age of 120 day, an increment of 0.9% and 5.1% was observed for the replacement levels of 6% and 18%, respectively, in the mixtures without nano and micronized calcite. Mineral admixtures affect the compressive strength of cement-based materials with three basic mechanisms of action: filling effect, dilution effect and pozzolanic reactions. The results given in Figure 7 show that EP shows dilution effect at different levels until the 28-day cure age and contributes to the microstructural development with both filling effect and pozzolanic reactions after the 28-day cure age. On the other hand, based on the results given in the figure, it was revealed that the dilution effect was more dominant at curing age of 28-day regardless of replacement levels. As a result of this, the mixtures including EP could not reach and/or exceed the compressive strength of the control mixtures (Ramezanianpour et al., 2014). In addition, as seen in Figure 7, the compressive strengths of the mortars including 6% of EP reach the control mixtures for the curing age of 28 day. This shows that the 6% substitution rate is the most suitable replacement level for the current designed mixtures. In a previously conducted study by Esfandiari and Loghmani (2019), the most appropriate replacement level was found to be 6%. This also confirms the results of the current experimental study. Both nano and micronized calcite increase the hydration mechanism of C_3S, known as nucleation effect/seeding effect. Nano-sized calcite is known for the accelerating the formation of hydration products as hydration reactions start (Sato and Beaudoin, 2011) primarily by promotion of C–S–H gels owing to nucleation effect. It has been stated that reaction between $CaCO_3$ and C_3S results in the production of C–S–H gels, CH, and calcium carbosilicate hydrates (Demirhan, 2020). In addition, by reacting with the aluminate phase of the binder material known as chemical effect, it also produces additional hydration products and therefore contributes to the strength development of the cementitious materials (Demirhan, 2020). This situation, namely the synergy between the calcite and the aluminate phases of the binder material, is directly dependent on the crystal structure of the pozzolanic material in particular. Compressive strength development of curing age of 120 day compared to 7-day compressive strength is illustrated in Figure 8. In general, a contribution was made to the strength development in the mixtures including nano and micronized calcite, and the highest contribution was obtained with nanocalcite. Although the aluminate phase of EP is high, a low chemical effect, namely lower strength development, was detected in mixtures containing nano and micronized calcite. This may be due to the exposure of EP to high temperature during the production of perlite and as a result, reaction capability of EP has been affected. In addition, compared to micronized calcite, surface area/volume ratio of nanocalcite, namely its specific surface area, is much higher. Therefore, its contribution to the hydration mechanism was relatively higher. The slight increase observed in the compressive strength of the control mixtures including perlite with nano and micronized calcite is most probably a result of the chemical effect arising from the presence of a low amount of aluminate phase in white normal portland cement utilized in the production of mixtures (See Table 1) (Çiftçi and Demirhan, 2021; Demirhan, 2020).

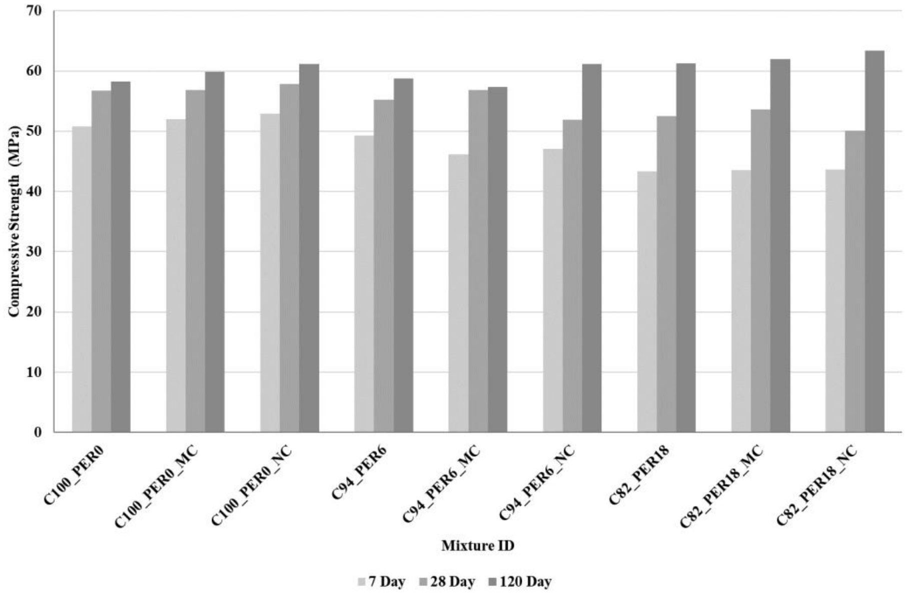


Figure 7. Compressive strength values for the curing ages of 7, 28 and 120 days

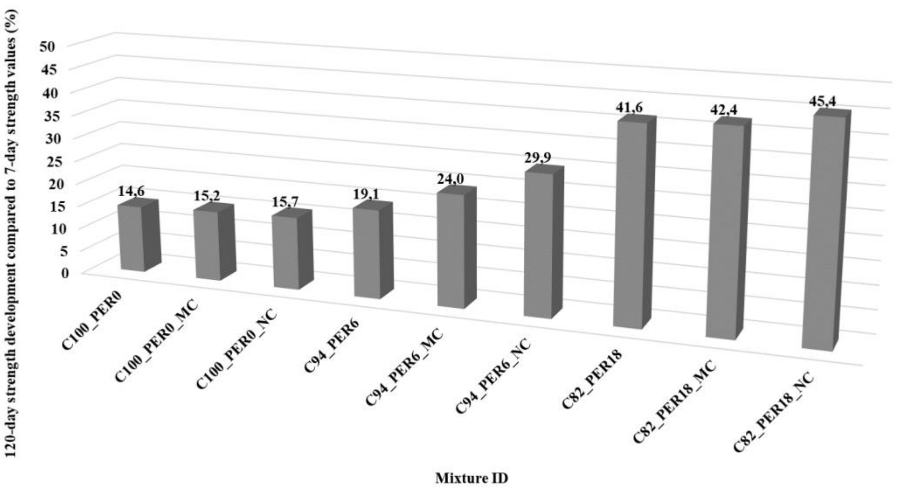


Figure 8. Compressive strength development in 120-day curing age compared to 7-day curing age

Ultrasonic Pulse Velocity (UPV)

UPV results of all the mixtures for the curing ages of 7, 28 and 120-day are given in Figure 9. As seen in the figure, as EP replacement level increased, a decrease in UPV values was observed. This was a probable result of the increased porosity and void structure. Regardless of EP and nano/micro calcite replacement levels, the UPV results of all mixtures were determined between 4.08 km/h and 4.45 km/h. According to Malhotra (1976) (Malhotra, 1976), the durability of cement-based materials with UPV results between 3.66-4.58 km/h is classified as "good" (Demirhan, 2020). Therefore, depending on the UPV results, it could be concluded that all mixtures were "good" regardless of the perlite substitution ratio. UPV test results showed that in addition to the additional hydration products formed as a result of chemical action, nano and micro-sized calcite minerals also improve the microstructure of the voids. This is directly related to the filler effect because relatively higher UPV values are the result of a denser and more interlocked microstructure (El Mir and Nehme, 2017). In addition, higher UPV results were obtained in mixtures including nano and micro-calcite compared to the control mixtures (mixtures without calcite). Relatively higher values were obtained in mixtures including nano-calcite. This result could also be attributed to the reasons previously stated in the compressive strength section. Also, UPV values increased with increase in curing age. This can be explained by the filling of microstructural cracks and voids of the matrix due to (i) hydration products formed by the ongoing hydration development, (ii) filler effect of minerals, and (iii) additional hydration products (Demirhan, 2020). In addition, as seen in Figure 9, the UPV results of the 120-day mixtures were close to each other, while the UPV results of the mixtures including both 6% of EP and nano/micronized calcite were the highest. This result supports that 6% replacement level is the most appropriate substitution rate for microstructural development, as stated in the compressive strength section.

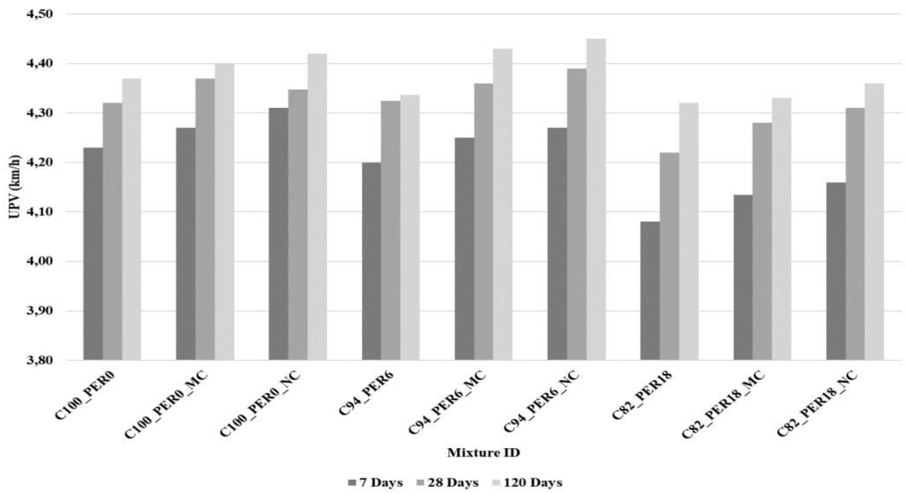


Figure 9. UPV values for the curing ages of 7, 28 and 120 days

CONCLUSION

Based on the results of the current experimental study, the following conclusions could be deduced: Regardless of replacement level, an allowable volume expansion up to 2 mm was determined in the mixtures including EP. Due to the high porosity and void ratio of EP, its specific surface area increases. As a result, decrease in workability was observed as the EP replacement level increased. A slight improvement (0.67% for micronized calcite and 1.27% for nanocalcite) were detected in the workability of the mixtures, including calcite. In general, regardless of calcite type of, a slight shortening in setting times was observed in mixtures including calcite while it was a delay in mixtures including EP. Higher replacement levels of EP resulted in lower unit weight. Regardless of calcite minerals, in general, the increase in replacement level of EP resulted in a decrease in compressive strength for the 7 and 28 cure ages. At the curing age of 120-day, this was resulted in an increase of 0.9% and 5.1% for the 6% and 18% replacement levels, respectively. A decrease was observed in the UPV values as a result of the increased porosity and void structure when replacement level of EP was increased. Regardless of the curing age, since the UPV values obtained in all mixtures were higher than 3.66 km/h, it could reveal that UPV results obtained from all the mixtures could be titled as "good".

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Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author's Contributions

The authors declare that their contribution is the same and equal.

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