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Improvements of mechanical and physical features of cement mortar by nano Al_2O_3 and $CaCO_3$ as additives

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Abstract. The current research aimed to investigate the impact of Al_2O_3 and $CaCO_3$ nanoparticles on the properties of cement mortar. The research methodology primarily focused on preparing mortars using Al_2O_3 nanoparticles with a mean diameter of ~50 nm and $CaCO_3$ nanoparticles with a particle size of 100 nm. These were utilized at three different substitution levels of 1, 3, and 5 % by weight of cement as binary blending materials. The mechanical and physical properties of the cement mortar (compressive strength, density) were tested after 7 and 28 days, while ultrasonic pulse velocity was tested after 28 days of water curing. The experimental results illustrated that utilizing Al_2O_3 nanoparticles improved the mortar's compressive strength at an early age (7 days of curing) more than at 28 days, with 3 % substitution being the optimal proportion. Similarly, the use of $CaCO_3$ nanoparticles as a binary blending mixture at substitution levels of 1, 3, and 5 % by cement weight improved the compressive strength at an early age (7 days of curing) more than at 28 days. The optimal proportion was again 3 %, with 87 and 40 % improvement for 7 and 28 days of curing, respectively. When comparing Al_2O_3 and $CaCO_3$ nanoparticles, the latter yielded better results than Al_2O_3 nanoparticles for both early and later ages. The combined effect of substituting 1 and 3 % of Al_2O_3 and $CaCO_3$ nanoparticles in cement mortar increased compressive strength by 28 and 74 % at 7 days of curing and by 30 and 42 % at 28 days of curing, respectively.

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1. *Introduction*

Ordinary Portland cement (OPC) is a type of cement that is commonly used in concrete and mortar. It is produced by heating limestone and clay at high temperatures and grinding the resulting material into a fine powder [1–3]. OPC is the most common type of cement utilized in various applications, including foundations, driveways, sidewalks, and walls. It is also used in the construction of structures, including bridges and dams. OPC is highly durable and has a long lifespan. It is also inexpensive and has high compressive strength (CS), meaning it can withstand substantial pressure. OPC concrete is made by mixing OPC with water and aggregate (sand, gravel, or crushed stone). This mixture is then poured into forms and allowed to harden. OPC concrete is strong, durable, and has a long lifespan, making it a popular choice for construction projects [4].

OPC is an essential component of concrete, but its manufacturing process produces significant amounts of CO_2. The primary sources of CO_2 emissions are the burning of fossil fuels (coal and natural gas) used to heat the rotary kiln and the calcination process itself [5–7]. Other sources of emissions include the combustion of fuels to operate grinding, as well as the transportation of materials throughout the cement manufacturing process [8–12]. However, there are several alternatives to OPC such as fly ash, slag cement, silica fume, and nanomaterials that have recently been incorporated into the cement matrix, which involved various types such as SiO_2, TiO_2, Al_2O_3, carbon nanofibers, Fe_2O_3, and $CaCO_3$ nanoparticles. It has been found that the nanomaterials utilized in cement and concrete improve their mechanical, physical, and other properties [13].

The addition of nanoparticles such as Al_2O_3 and $CaCO_3$ to cement mortar has been studied to understand their effects on mechanical and physical features. Research has shown that including 1 and 3 % Al_2O_3 nanoparticles in cement mortar improves its mechanical properties, including compressive, tensile, and flexural strength [17]. Similarly, adding SiO_2 and $CaCO_3$ nanoparticles to cement mortars increases their flexural and CS, with optimal replacement levels of 2 and 4 %, respectively [18].

Several researchers investigated the influence of adding Al_2O_3 to concrete to improve workability and CS. In [14, 15], the influence of Al_2O_3 nanoparticles with an average diameter of 15 nm on the CS and workability of blended concrete was investigated. The nanoparticles were incorporated into the system at four distinct weight percentages: 0.5, 0.1, 1.5, and 2.0 %. The findings indicate that incorporating Al_2O_3 nanoparticles at an optimal substitution level of 2 % yields concrete with enhanced strength features. Nevertheless, the optimal structural integrity of the concrete was achieved when a cement replacement of 1.0 wt% was employed. The increase in Al_2O_3 nanoparticle amount resulted in a reduction in the workability of fresh concrete. Based on the findings, it can be inferred that incorporating Al_2O_3 nanoparticles as a partial substitute for cement enhances CS of concrete, albeit at the expense of reduced workability.

On the other hand, the use of $CaCO_3$ nanoparticles as an additive material to improve concrete properties has become widespread in recent times. In [16], the impact of $CaCO_3$ nanoparticles on cement paste was investigated. Nanoparticles were added at three various substitution levels (1, 2, and 3 %) of cement weight, with a mean particle size of 15–50 nm. The water-to-cement ratio was 0.45. CS testing was conducted at 7 and 28 curing days. Samples were cubic bars with dimensions of 20×20×80 mm. The result illustrated that CS at both ages increased with the content of $CaCO_3$ until it reached an optimal content of 2 % and then it decreased. When the content of $CaCO_3$ was 2 %, the CS was 111.2 and 108.6 % of blank sample at the age of 7 and 28 days, respectively. The enhancement of flexural and CS of hardened cement paste was due to the consuming and refinement of $Ca(OH)_2$ grain, which occurred during the hydration of cement especially at early ages.

Numerous studies have focused on improving the mechanical properties of cement mortar by utilizing nanoparticles, including Al_2O_3 and $CaCO_3$ nanoparticles. Most researchers have observed that substituting cement with Al_2O_3 nanoparticles at levels of 4–5 % by cement weight reduces the CS of cement mortar. At the same time, some authors observed that a 5 % substitution of Al_2O_3 nanoparticles increases CS, while others have reported no significant effect. Regarding $CaCO_3$ nanoparticles, most researchers have noted that substitution levels of 3, 4, and 7 % by cement weight increase CS at 7 and 28 curing days, whereas some have observed a decrease in CS at both ages. Based on previous research, inconsistent findings have been reported when using different proportions of Al_2O_3 and $CaCO_3$ nanoparticles. Therefore, this research aims to study the impact of adding nanomaterials, including Al_2O_3 and $CaCO_3$ nanoparticles, to cement mortar and to determine the optimal percentage of nanoparticle addition to improve the physical and mechanical properties of cement mortar.

2. Material and Methods

2.1. Materials

OPC was used; the physical and chemical properties are illustrated in Table 1. This work used locally available natural sand as fine aggregate with a fineness modulus of 2.17. The grading of this aggregate was zone 3 as limits of Iraqi specification, No. 45/1984.

Table 1. The physical and chemical properties of cement.

Chemical composition	Amounts (by weight), %	Limits of Iraqi Requirement No. 5/2019 (42.5 R)
Cao	60.60	–
SiO_2	19.80	–
Al_2O_3	4.80	–
Fe_2O_3	3.00	–
MgO	3.50	≤ 5.0 %
SO_3	2.22	≤ 2.8 % if C3A > 3.5 %
Loss on Ignition	3.10	≤ 4.0 %
Insoluble residue	0.70	≤ 1.5 %
OPC main compounds (Bogue's Eq.)		
C_3S		59.63
C_2S		11.78
C_3A		7.64
C_4AF		9.12
Test name	Findings	Limits of Iraqi Requirement No. 5/2019 (42.5 R)
Fineness (Blaine method), m^2/kg	320	≥ 280
Setting time (Vicat's method),		
Initial setting time (min)	90	≥ 45
Final setting time (h)	5	≤ 10
CS (MPa),		
Early strength (2 days)	24	≥ 20
Standard strength (28 curing days)	43	≥ 42.5

Al_2O_3 nanoparticles with a mean particle size of ~50 nm were used. The physical and chemical properties of Al_2O_3 nanoparticles are given in Table 2.

Table 2. Physical and chemical properties of Al_2O_3 nanoparticles.

Purity, %	Mean particle size, nm	Typical impurities	Ca	K	Cu	Mg	Fe	Mn	Zn	Si
99.9+ (trace metal basis)	50	–	< 100	< 100	< 10	< 50	< 100	< 50	< 50	< 100

$CaCO_3$ nanoparticles with a mean particle size of less than 100 nm were used. The properties of $CaCO_3$ nanoparticles are illustrated in Table 3.

Table 3. The physical and chemical properties of $CaCO_3$ nanoparticles.

Appearance	Density	Size	Specific area	MgO	Moisture amount	Loss on ignition	Residue on Sieve	$CaCO_3$ amount
White powder	2.5	> 100	≥ 20	≤ 0.8	≤ 0.9	44 ± 1	≤ 0.02	≥ 96

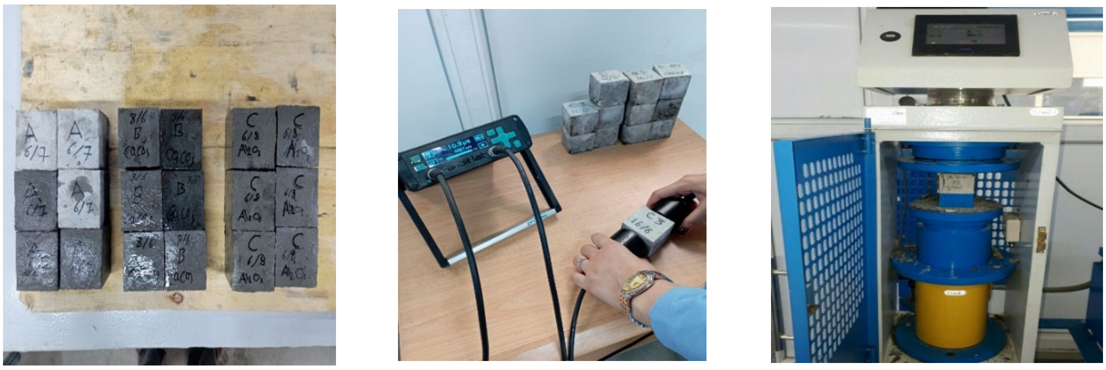
Alumina + Iron oxide	PH	Insoluble matter with acid	Activation rate	DOP absorbed does
≤ 0.3	8.5–9.7	≤ 0.3	≥ 95	35–55

Finally, tap water was used throughout this work for mixing and curing.

2.2. Mixing Procedure and Samples Preparing

In this study, nine types of mixes were produced to investigate the impact of Al_2O_3 and $CaCO_3$ nanoparticles on the CS of cement mortar. These mixes were designated as A, B_1, B_2, B_3, C_1, C_2, C_3, B_1+C_1, and B_2+C_2. Type A mixture was produced of fine natural aggregate, cement, and water. Types B_1, B_2, B_3, C_1, C_2, and C_3 were produced with various content of Al_2O_3 and $CaCO_3$ nanoparticles. These mixes were prepared with cement substitution levels of 1, 3, and 5 % by weight. The B_1+C_1 and B_2+C_2 mixtures were produced with Al_2O_3 and $CaCO_3$ nanoparticles at cement substitution levels of 1 and 3 % by weight.

In the initial step of mixing the nanomaterials, the binder-to-sand ratio was 1:2 and the water-to-cement ratio was 0.46. The materials were mixed in a dry condition for 1 min and another 2 min after adding the water. Cubes with dimensions of 50×50×50 mm were cast for CS testing and compacted in two layers on a vibrating table, with each layer vibrated for 15 sec. The mold was covered for 24 hours. Then, the samples were demolded and cured in water for the 24 hours. The CS tests were conducted at 7 and 28 curing days. The mixtures with Al_2O_3 and $CaCO_3$ nanoparticles are presented in Fig. 1 and Table 4.



a) b) c)

Figure 1. a) various mortar cubes, b) test of ultrasonic pulse velocity (UPV), c) CS test machine.

Table 4. Mixtures of Al_2O_3 and $CaCO_3$ nanoparticles.

Sample	Cement, gm	Sand, g	Water, g	Nano-Al_2O_3, g, %	Nano-$CaCO_3$, g, %	W/C
A (control)	650	1300	300	–	–	0.46
B_1	643	1300	300	7, 1 %		0.46
B_2	630	1300	300	20, 3 %		0.46
B_3	620	1300	300	30, 5 %		0.46
C_1	643	1300	300		7, 1 %	0.46
C_2	630	1300	300		20, 3 %	0.46
C_3	620	1300	300		30, 5 %	0.46
B_1+C_1	643	1300	300	3.5, 1 %	3.5, 1 %	0.46
B_2+C_2	630	1300	300	10, 3 %	10, 3 %	0.46

3. Result and Discussion

3.1. Mechanical and Physical Properties

Fig. 2 demonstrates the CS findings of the selected mixtures before and after replacing OPC with different proportions of Al_2O_3 and $CaCO_3$ nanoparticles. The use of nanomaterials (Al_2O_3, $CaCO_3$, and Al_2O_3+$CaCO_3$) at substitution levels of 1 and 3 % increases the CS at early ages, whereas the substitution level of 5 % leads to significant decrease in CS due to the formation of ettringite [23]. The 3 % substitution level is considered the optimal replacement ratio at early ages.

The use of nanomaterials (Al_2O_3, $CaCO_3$, and Al_2O_3+$CaCO_3$) at substitution levels of 1 and 3 % increases the CS at later ages, whereas the substitution level of 5 % leads to significant decrease in CS due to the conversion of ettringite to cement gel (C–H, C–S–H). The 3 % substitution level is considered the optimal replacement ratio at later ages.

When the content of Al_2O_3 nanoparticles is increased to 3 %, the mechanical properties are severely reduced. The researchers attribute this to a reduction in the distance between nanoparticles and the limited space for $Ca(OH)_2$ crystals to grow to an appropriate size, along with nanoparticle agglomeration, which together cause a reduction in CS. The results demonstrate that 1 % of $CaCO_3$ nanoparticles yields the highest CS at all ages. Due to their high surface energy during the hydration process, these nanoparticles grow and clusters, acting as nuclei that accelerate and enhance the hydration process.

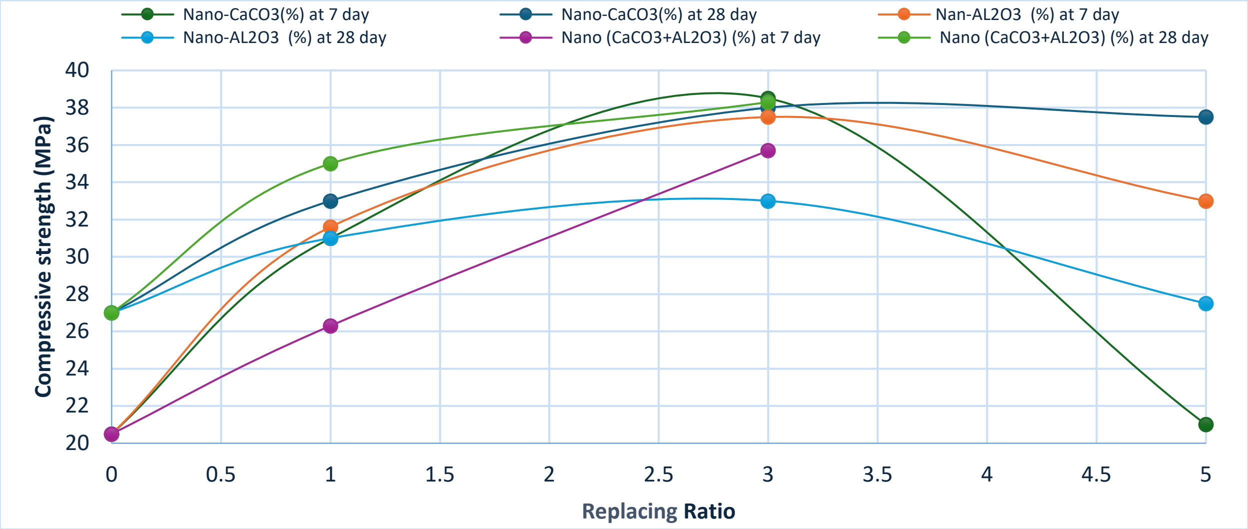


Figure 2. Impact of adding $CaCO_3$, Al_2O_3, and $CaCO_3$+Al_2O_3 nanoparticles to OPC on CS at 7 and 28 curing days.

Fig. 3 demonstrates the density findings of the selected mixtures before and after replacing OPC with different proportions of Al_2O_3 and $CaCO_3$ nanoparticles. The use of 1 % of Al_2O_3 nanoparticles increases the density at 7 and 28 days, whereas the use of 3 % of Al_2O_3 nanoparticles significantly decreases the density at early ages. The use of 1 % of $CaCO_3$ nanoparticles decreases the density at 7 and 28 days, whereas the use of 3 % of $CaCO_3$ nanoparticles slightly increases the density at the early ages and significantly increases it at later ages. The combination of 1 and 3 % of Al_2O_3+$CaCO_3$ nanoparticles reduces density at both 7 and 28 curing days, with better results observed at later ages compared to earlier ones.

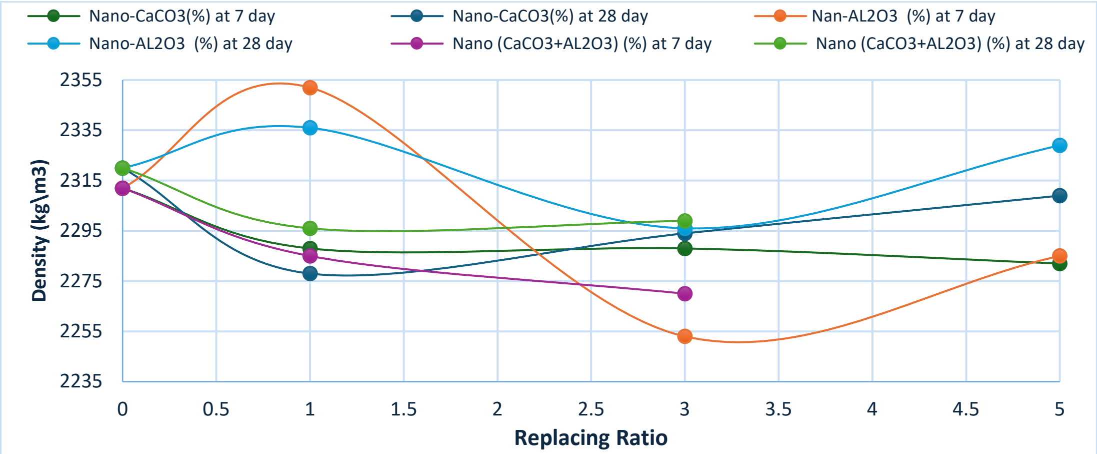


Figure 3. Impact of adding $CaCO_3$, Al_2O_3, and $CaCO_3$+Al_2O_3 nanoparticles to OPC on density at 7 and 28 curing days.

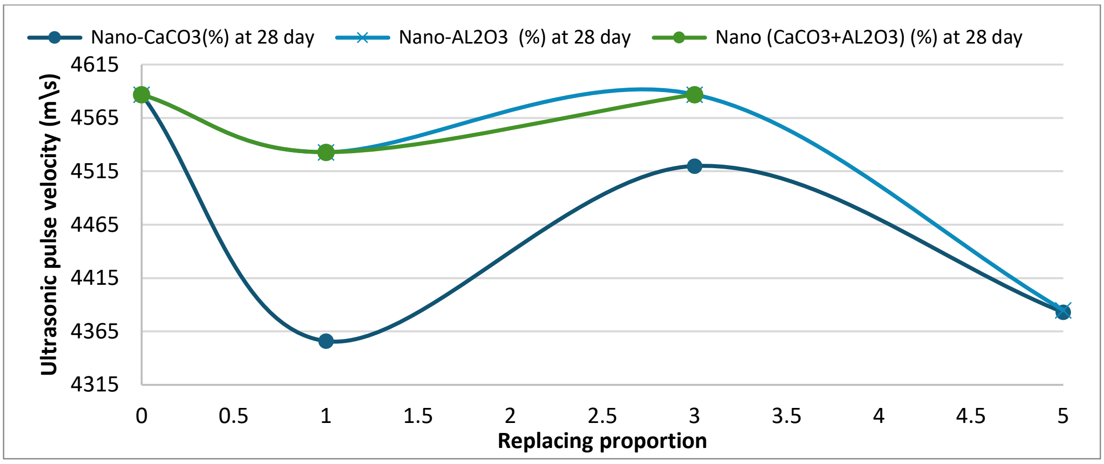


Figure 4. Impact of adding $CaCO_3$, Al_2O_3, and $CaCO_3$+Al_2O_3 nanoparticles to OPC on UPV at 7 and 28 curing days.

It was found that the CS of cement mortar with Al_2O_3 nanoparticles (1, 3, and 5 %) increased by 54, 82, and 61 % at 7 curing days and 14, 22, and 1 % at 28 curing days, respectively. It was observed that the use of 5 % of Al_2O_3 nanoparticles reduced the CS to a level close to that of the control sample A. It might be due to an excess of Al_2O_3 nanoparticles beyond what is necessary to react with the free lime during the hydration process, resulting in excess silica leaching out and weakening the concrete, as it only replaces a portion of the cementitious material. Additionally, weak areas might result from defects created during nanoparticle dispersion. The best CS among all cement mortar mixtures was observed with substitution levels of 1 and 3 % of Al_2O_3 nanoparticles, attributed to the high activity of Al_2O_3. The presence of nanoparticles in the cementitious system leads to the consumption of portlandite $Ca(OH)_2$ through a pozzolanic reaction, filling capillaries, reducing porosity, and increasing strength.

Furthermore, no clear effects were observed when replacing with 1, 3, and 5 % of Al_2O_3 and $CaCO_3$ nanoparticles on the density and UPV of cement mortar at 7 and 28 curing days, respectively. This is probably attributed to the small size of cubes with dimensions of 50×50×50 mm.

For $CaCO_3$ nanoparticles replacing cement at 1, 3, and 5 % by weight, the findings illustrated that the CS increased by 51, 87, and 2.4 % at 7 curing days and by 22, 40, and 38 % at 28 curing days, respectively. It was noted that the increase in CS was observed at 1 and 3 % substitution levels, because the $CaCO_3$ nanoparticles are chemically stable, fill the pores, and increase surface activity.

The substitution of 1 and 3 % of combined nanoparticles in cement mortar increased CS by 28 and 74 % at 7 curing days and by 30 and 42 % at 28 curing days, respectively. This is due to the high activity of the Al_2O_3 nanoparticles, whose presence in the cementitious system leads to the consumption of portlandite $Ca(OH)_2$ through a pozzolanic reaction that fills capillaries, reduces porosity, and increases strength, while the $CaCO_3$ nanoparticles fill the pores and increase surface activity.

4. Conclusion

The mechanical properties of cement mortar affected by the substitution of Al_2O_3 and $CaCO_3$ nanoparticles were investigated in the current research. Based on the replacement of Al_2O_3 and $CaCO_3$ nanoparticles in cement mortar, the following was observed:

1. The CS of cement mortar with 1 and 3 % Al_2O_3 nanoparticles substitution by cement weight increased by 54 and 82 % at 7 curing days, and by 14 and 22 % at 28 curing days, respectively.
2. The use of 5 % Al_2O_3 nanoparticles reduced the CS at 28 curing days to a value close to that of the control sample A. The best CS among all cement mortar mixtures was observed with 1 and 3 % substitution of Al_2O_3 nanoparticles.
3. The substitution of 1 and 3 % of combined nanoparticles in cement mortar increased the CS by 28 and 74 % at 7 curing days, and by 30 and 42 % at 28 curing days, respectively.
4. Al_2O_3 nanoparticles increased CS at 7 days but reduced it at 28 curing days. With 3 % substitution of $CaCO_3$ nanoparticles, the CS at 7 and 28 curing days was higher than that of the control sample A, whereas with 5 % substitution of $CaCO_3$ nanoparticles, the CS decreased at 7 curing days and increased at 28 curing days.
5. No significant influence was observed when Al_2O_3 and $CaCO_3$ nanoparticles were substituted at 1, 3, and 5 % by cement weight on the density and UPV of cement mortar at 7 and 28 curing days.

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