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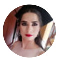

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Chemical shrinkage of paste and mortar containing limestone fines

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ABSTRACT

The effect of including limestone fine (LF) on the chemical shrinkage of cement paste and mortar is investigated. The cement was partially replaced with 0 to 20% (by weight) with LF. The water to cementitious material (CM) ratio was maintained at 0.45. In the mortar mixes, the proportion of sand (S) to CM was 1:2. In addition to the chemical shrinkage test, compressive strength and density of specimens were determined. The chemical shrinkage data were collected up to 28 days. However, for compressive strength and density, testing continued up to 90 days of water curing. There appears to be an increase in chemical shrinkage between 10 and 15% limestone for both paste and mortar mixes. The trend appears to be similar for the compressive and density results.

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1. Introduction

Climate change is real as demonstrated by the high frequencies of flood events, drought and storms in many parts of the world. This could be due to the high emission of harmful gases into the atmosphere including Carbon Dioxide (CO_2). Human activities are mainly to blame. Excessive use of natural resources and deforestations are examples. The United Nations (UN) are urging countries to take action to mitigate these effects through various actions including the 17 sustainable development goals [1] and conference of the parties which is known as COP26 [2]. The construction industry generates large amount of CO_2 partly due to the manufacturing of building materials. Therefore, using waste materials in construction applications is advantageous in that the amount of virgin materials and CO_2 are reduced [3–9].

Concrete is the only essential building material that cost-effectively delivers (strength, durability, security, safety…). Its main problem is the early-age cracking due to shrinkage [10]. Chemical shrinkage is mainly concerned with the internal volume change that results in the creation of pores which is a function of the degree of hydration. Chemical shrinkage is based on the difference between the initial reactants and the final hydration products [11,12]. Conventionally, chemical shrinkage was tested by three essential measurement methods: dilatometry, pycnometry and gravimetry. Dilatometry method is commonly used by many authors [13,14].

Over the last decades, incorporating supplementary cementitious materials SCMs have been widely used in concrete construction [3,7]. It was explored that they have many characteristics which authorize them to overcome concrete problems. Accordingly, the use of limestone as partial or total replacement of cement has been reported [15,16]. Limestone is a common sort of carbonate sedimentary rock. It is widely used in concrete production [17–22]. It is frequently considered as an inert filler material which plays the role of nucleation sites for hydrates and accelerates the chemical shrinkage [23]. Besides, LF could encourage the early age cement hydration and consequently improves the early strength. It also may drop the total porosity and may delay the initial and final setting time of concrete as well [24]. LF in concrete increases the chloride ion diffusion [25] and reduces the peak rate of heat evolution [26]. Several studies investigated the impact of substituting limestone fines (LF) as a partial replacement of cement on the chemical shrinkage of concrete. The chemical shrinkage of paste, mortar and concrete is affected by the addition of LF. The total chemical shrinkage for normal Portland cement was

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about 4.6% (by weight of cement) as reported by Le chatelier [27]. Khatib et al. [28] proved that combining LF with cement increased the chemical shrinkage. Besides, the addition of LF with calcined clay (CC) as a partial replacement of cement increased quickly the chemical shrinkage during 7 days [29].

2. Research significance

The chemical shrinkage test is mainly conducted on paste and there does not appear to be any research on mortar. Moreover, partial replacement of cement with non-calcined LF will reduce the amount of cement used; thereby, reducing the amount of CO_2 emitted to the atmosphere [30]. Therefore, this paper examines and compares the volume stability of both paste and mortar containing LF. The amount of LF ranged from 0 to 20% (by weight of cement) for both paste and mortar. The compressive strength and density tests were also evaluated.

3. Experimental work

3.1. Materials

Portland cement type I conforming to the Lebanese norms PA-L 42.5 N was used. The limestone fine was obtained from a quarry in Mount-Lebanon and was passed though the 300 μm sieve. The density, specific gravity and Blaine surface area of cement were 1440 kg/m^3, 3.15 and 399.8 m^2/kg respectively whereas the density, specific gravity and Blaine surface of LF were 2700 kg/m^3, 2.74 and 394 m^2/kg. The chemical and phase composition for cement and LF is presented in Table 1. The siliceous sand used had a fineness modulus of 2.8 and a maximum grain size of 2.3 mm.

3.2. Mix proportions

Ten mix designs were evaluated in this study. Of the ten mixes, five were mortar mixes and the remaining five are paste mixes. In all mixes, the cement in the paste and mortar are partially substituted by weight with 0, 5, 10, 15 and 20% LF. To facilitate comparison between mortar and paste mixes and to yield reasonable consistency without the need of any chemical admixtures, the water to binder ratio (w/b) was kept constant at 0.45 for all mixes and the sand to binder ratio was taken equal to 2 for mortar mixes. The binder comprises cement and LF. For each mix, two replicate specimens were prepared. The total number of specimens tested was 100. The key design information of paste and mortar mixes area presented in Table 2.

3.3. Specimen preparation and testing

The materials for each mix were weighed. For the paste and mortar mixes, the dry materials were thoroughly mixed by hand and placed in the mixer. Then the mixer was turned on and the water was slowly added to the dry materials until homogeneous mix is achieved. This took roughly 3 min.

For the chemical shrinkage test, a glass bottle of 250 ml in size, a plastic funnel, a spatula, a rubber stopper and a glass pipette were prepared. The paste or mortar sample was placed at the bottom of the bottle using a spatula and funnel. The weight of the sample was approximately 30 g. The sample was slightly shaken to level it off. The water was gently added on the top of the sample until the bottle is filled. Then, the rubber stopper including the pipette was inserted at the top of the bottle. Water was then added into the pipette until it was completely filled. In order to prevent the evaporation of water from the pipette, a drop of oil was added at the top of the water in the pipette. The whole setup was placed in a room at 25 ± 2 °C. The reduction in water level in the pipette with time was monitored every hour for the first 24 h, then every two days until the age of 28 days. The chemical shrinkage (*CS*) was then calculated using Equation (1):

$$CS = \frac{\Delta V}{W} \tag{1}$$

where ΔV is the volume change of the water in the pipette in ml and W is the weight of the paste or mortar sample in g. For the compressive strength test, standard steel cubic molds of 50 mm in size were employed and the testing conforms to ASTM C109 [31]. For the density test, it was conducted according to ASTM C188-14 [32]. The chemical shrinkage test procedure is presented in Fig. 1.

4. Results and discussion

4.1. Chemical shrinkage

The values of the chemical shrinkage test for paste and mortar samples during the first 28 days are displayed in Figs. 2 and 3 respectively. Chemical shrinkage values in paste and mortar increased with the increase of LF content up to a maximum value then decreased. For example, the chemical shrinkage for control mix in paste samples achieved a value of 0.036 ml/g of binder at 14 days. This value went up to a maximum value of 0.084 ml/g at 15% LF then dropped. Same trend was shown for mortars. At the age of 28 days, the incorporation of 10 and 15% LF in mortar and paste samples respectively attained the highest shrinkage values (0.076 and 0.115 ml/g of binder). The sharp drop that happens after the inclusion of 10 and 15% of LF in mortar and paste specimens respectively is mainly because of the stabilization of the ettringite due to the chemical effect of LF. This stabilization could increase the solid volume resulting from the hydration mechanism (external expansion) which consequently decline the total system volume [33]. It is well observed that the incorporation of LF increased the chemical shrinkage values in both paste and mortar samples. This can be explained by the fact that chemical shrinkage is the result of cement hydration mechanism [23]. Actually, LF plays the role of a filler due to its fine particles size and also provides nucleation sites for hydrations products to precipitate [33]. Hence, with the presence of LF, the hydration mechanism is more progressed and leads to an increase in chemical shrinkage.

It can be observed from Figs. 2 and 3 that chemical shrinkage for mortar specimens are lower than those of paste. For example, at 5% LF content, the drop in chemical shrinkage value between mortar and paste is 26.6% at 7 days curing. This is likely to be due to the higher cement content in the paste than that of mortar which would increase the hydration reaction resulting in more chemical shrinkage.

Table 1
Composition of cement and LF.

Oxide	SiO_2	AL_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	L.O.I	Residues	C_3S	C_2S	C_3A	C_4AF
Cement %	18.53	3.93	3.06	61.78	1.74	2.92	0.18	0.47	6.30	1.00	71.50	7.11	5.24	9.31
LF %	5.17	1.65	0.77	50.98	0	0.21	0.18	0.12	39.64	1.28	NA	NA	NA	NA

Table 2
Details of paste and mortar mixes.

	Amount (kg/m^3)							
Paste code	Cement	LF	Water	Mortar code	Cement	LF	Sand	Water
P-0	1303	0	586	M−0	657	0	1314	296
P-5	1237	66	586	M−5	625	32	1314	296
P-10	1177	126	586	M−10	595	62	1314	296
P-15	1123	180	586	M−15	569	88	1314	296
P-20	1074	229	586	M−20	544	113	1314	296

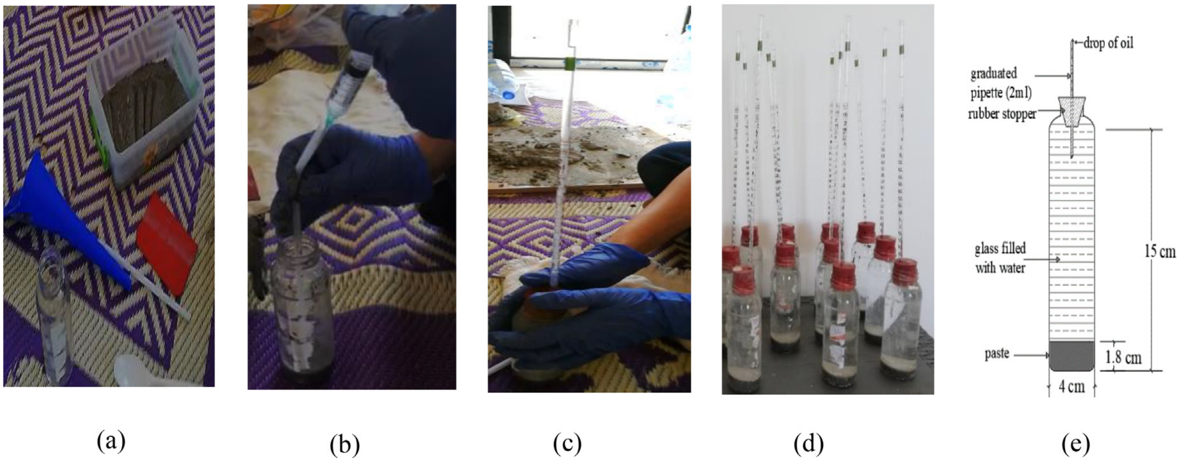

Fig. 1. Chemical shrinkage test procedure: (a) mixing of materials, (b) addition of water on paste surface by using a syringe, (c) fixing of rubber stopper and pipette, (d) chemical shrinkage specimens, (e) chemical shrinkage test set-up.

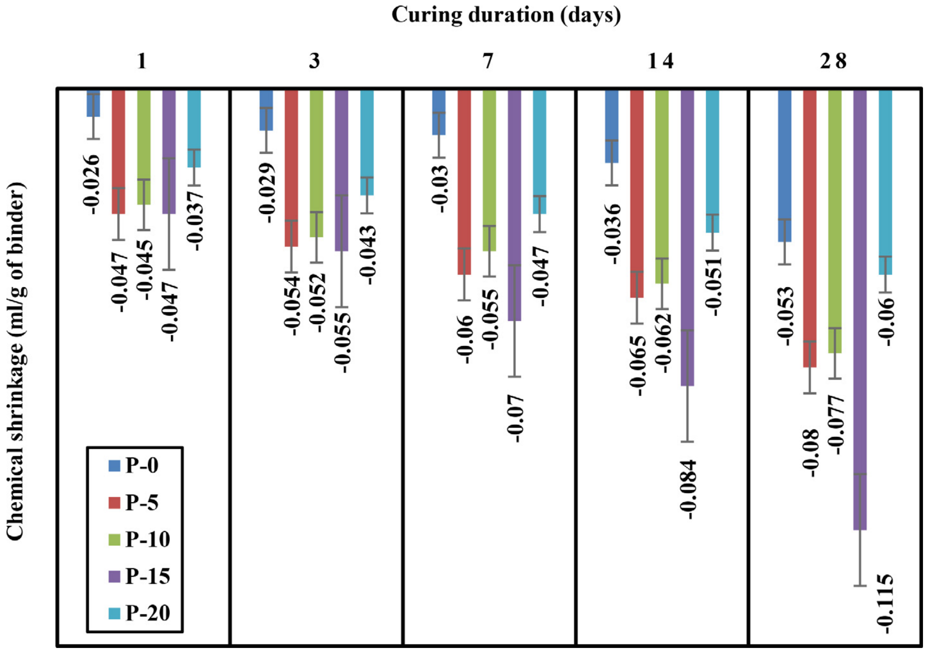

Fig. 2. Chemical shrinkage of pastes up to 28 days.

4.2. Compressive strength and density

The compressive strength development of paste and mortar mixes containing various amounts of LF at different curing times (1 to 90 days) are shown in Figs. 4 and 5 respectively. The strength of paste and mortars samples develops quickly with the presence of LF. As displayed in these plots, the strength of 15% LF in pastes and 10% LF in mortars realized the highest value among all other percentages. Two explanations can be made: a) LF can fill the pores between cement particles due to its fine particles size. This is known as the filling effect [33]. Hence, paste and mortar samples become more compacted resulting in an increase in compressive strength [33]. Besides, in the occurrence of this filler, the hard skeleton may be strengthened leading to a more uniform distribu-

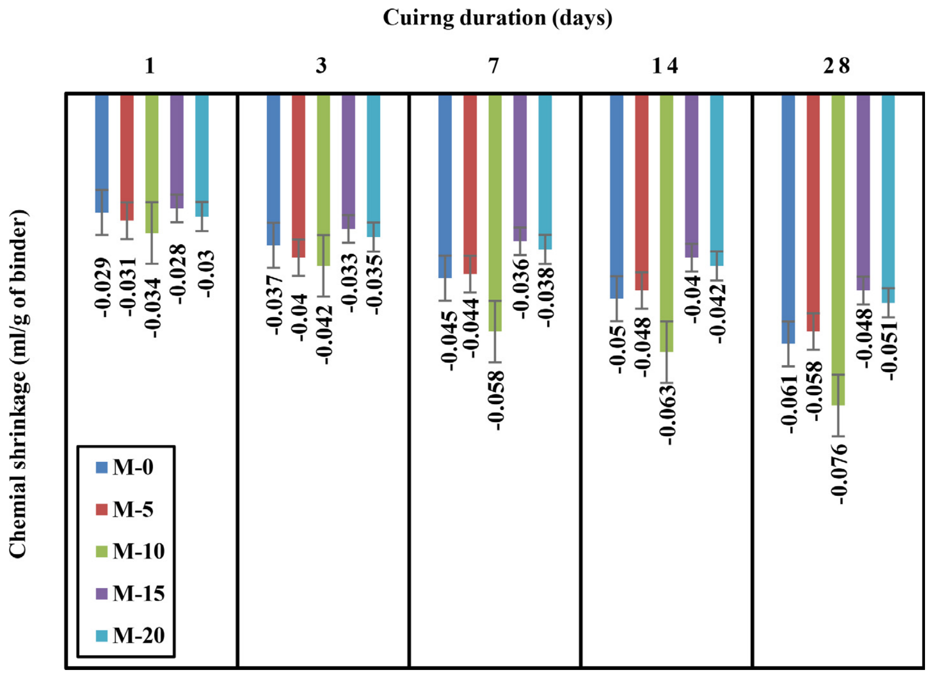

Fig. 3. Chemical shrinkage of mortars up to 28 days.

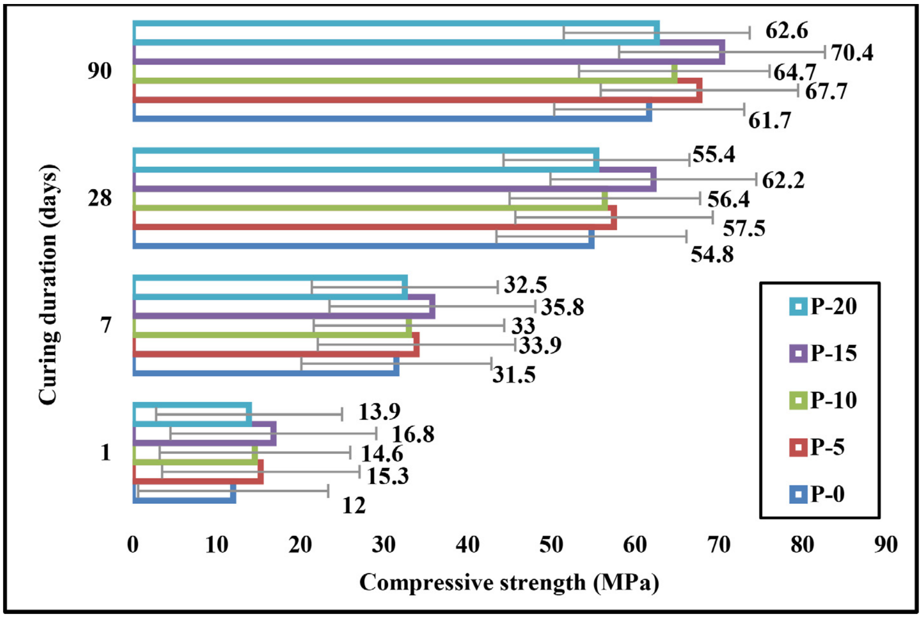

Fig. 4. Variation of compressive strength of paste containing varying amounts of limestone fines.

tion of small C-S-H crystals and fine pore structure which accelerate the cement hydration [34,35]. It was also noticed that beyond the optimal value of LF addition (at 15% and 10% LF in paste and mortar mixes respectively), the compressive strength decreased. This drop is mainly due to the lower cement content in the mix and the dilution effect as the LF increases [36]. This is in agreement with the results reported elsewhere [37,38]. Same trend occurs for other curing durations. The maximum compressive strength was 70.4 and 31.6 MPa for paste and mortar mixes respectively when cured at 90 days.

Similarly, as observed in chemical shrinkage results, the compressive strength values in mortar are lower than those in paste for the same LF content. For example, the mortar mix containing 5% LF showed a 57.8% decrease compared to the pastes at the same LF replacement at 28 days of curing. As indicated previously, the paste contains higher amount of cement compared to mortar for the same LF content.

Table 3 reports the density values for paste and mortar mixes cured for different durations. The density increases with an increase in curing age for both paste and mortar. For example, for mortar containing 5% LF, the density varied from 2280 kg/m^3 at 1 day of curing to 2440 kg/m^3 at 90 days curing. Also, the density of the pastes is lower than that of mortars for all mixes regardless of LF content. At the age of 28 days, the paste containing 10% LF the density is 2060 kg/m^3 whereas the equivalent mortar specimen is 2360 kg/m^3 which is an increase of 14.5%. Figs. 6 and 7 show the

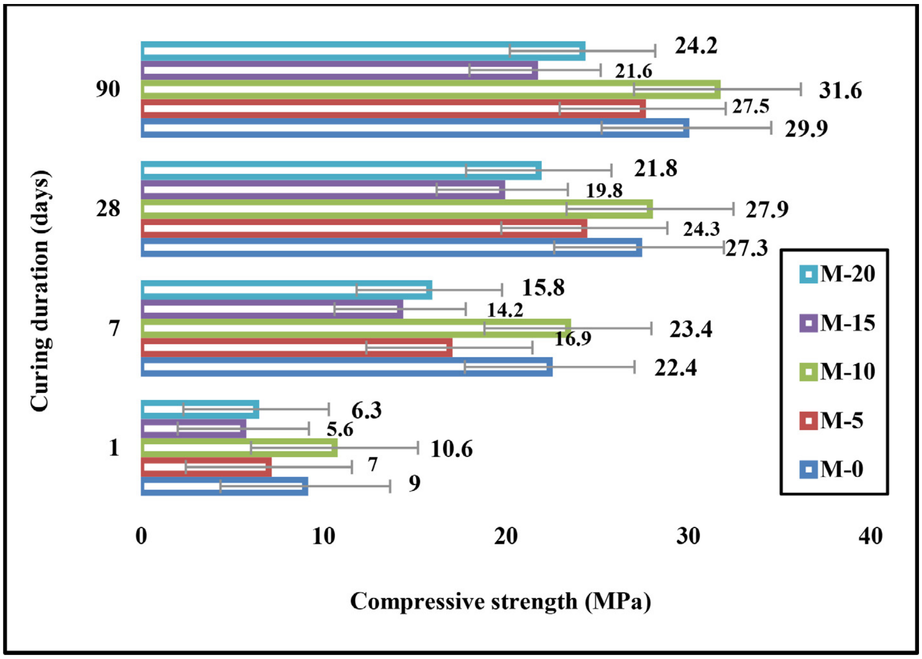

Fig. 5. Variation of compressive strength of mortar containing varying amounts of limestone fines.

Table 3
Density (kg/m^3) of paste and mortar mixes at different curing times.

Code		Age (days)			
		1	7	28	90
Paste	P-0	2080	2090	2120	2130
	P-5	2060	2080	2090	2110
	P-10	2030	2050	2060	2070
	P-15	2090	2110	2130	2140
	P-20	2050	2060	2070	2080
Mortar	M–0	2260	2350	2400	2430
	M–5	2280	2360	2410	2440
	M–10	2250	2310	2360	2420
	M–15	2300	2390	2420	2470
	M–20	2240	2300	2350	2400

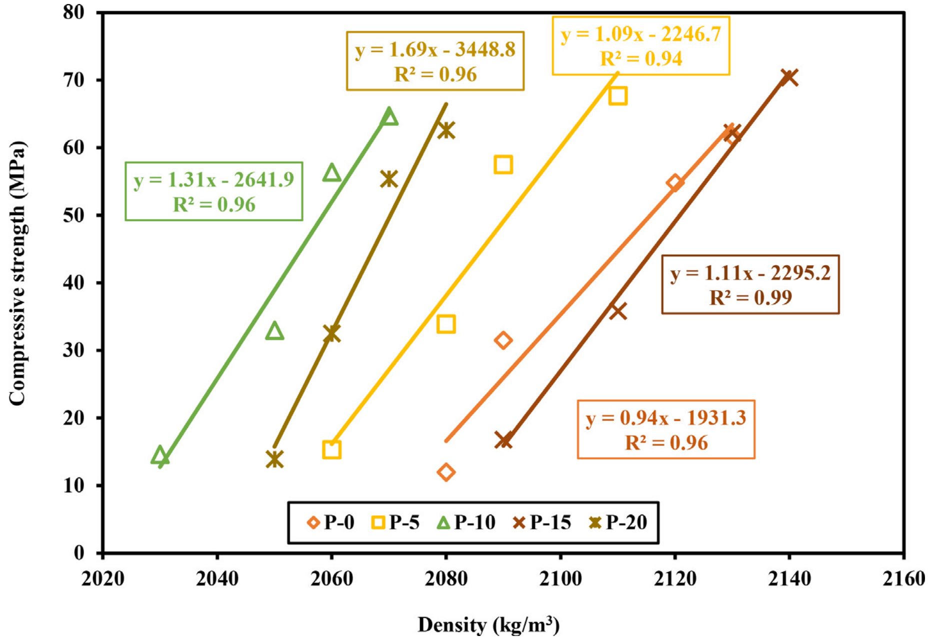

Fig. 6. Relationship between compressive strength and density for paste mixes.

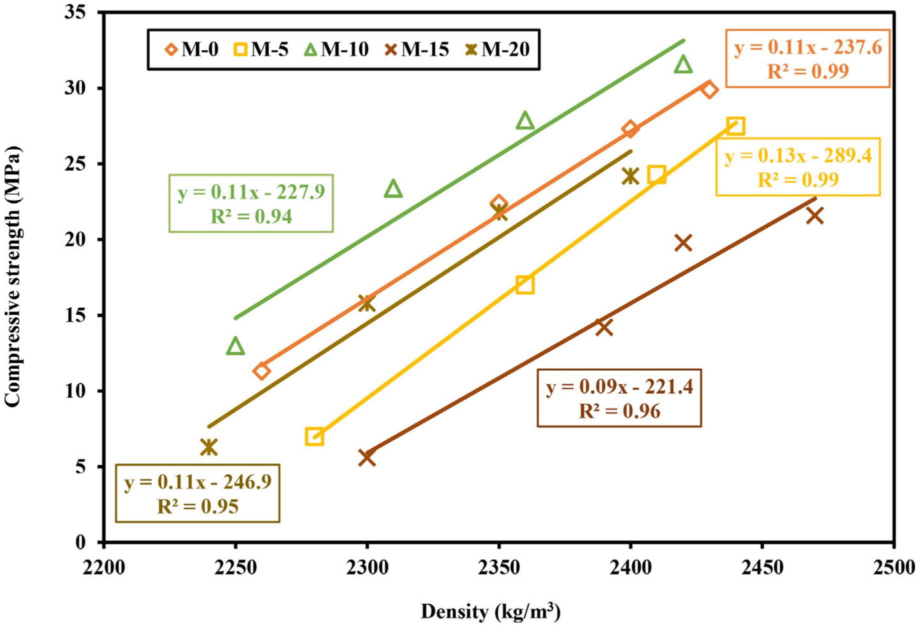

Fig. 7. Relationship between compressive strength and density for mortar mixes.

correlation between the compressive strength and density for mortar and paste respectively. The density data seems to be positively correlated with those of compressive strength. An increase in density resulted in an increase in compressive strength regardless of the LF content and the curing age. If a linear best-fit is plotted for each mix, an R^2 above 0.94 is obtained indicating strong correlation.

5. Conclusions and recommendations

The following conclusions are based on the results of the experimental work of this investigation.

- There is a maximum increase in chemical shrinkage for both paste and mortar containing limestone fine between 10% and 15%. The maximum values of chemical shrinkage obtained were 0.115 ml/g and 0.076 ml/g of cementitious materials for paste and mortar respectively.
- The paste samples yielded higher values of chemical shrinkage than mortar samples. This is likely to be due to the higher cement content in the paste which would increase the rate of hydration.
- The compressive strength results seem to be associated with LF content. The maximum compressive strength values in paste and mortar samples were achieved at 10 and 15% LF respectively (31.6 and 70.4 MPa).
- The compressive strengths of mortar are lower than those of paste. This is partly due to lower cement content in the mortar compared to those of the paste.
- The compressive strength seems to be positively correlated with chemical shrinkage for both pastes and mortars. An increase in chemical shrinkage is associated with an increase in the compressive strength.
- The density of paste and mortar increases with the age of curing regardless of the LF content. The presence of LF in paste and mortar leads to a slight reduction in density.
- The effect of surface area of limestone on the chemical shrinkage and other properties requires further investigation. Also, the effect of curing temperature on the volume stability of paste and mortar needs to be examined. It would be useful to design a chemical shrinkage test suitable for concrete and compare it with mortar and paste.

Declaration of Competing Interest

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

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