



PERFORMANCE EVALUATION OF LIMESTONE CALCINED CLAY **BLENDED CEMENT-BASED MORTAR**

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ABSTRACT

This study investigates the effect of utilizing a combination of limestone and calcined clay as cement replacement on the physical and mechanical properties of both fresh and hardened mortar. Mortars are made of blends with different levels of cement replacement (%15, %30, %45) by substitution ratio of 2:1 (calcined clay: limestone), with adjusting gypsum content in order to prevent the system being under-sulfated for each systems composition. Furthermore, three type of calcined clays are used with various kaolinite contents at 86%, 55% and 48%, respectively in order to investigate the influence of the grade of calcined clays on the properties. The results showed a reduction in mortar workability with the increase in cement replacement. However, clays with 55 and 48% kaolinite were exhibited a better workability than fairly purer clays (86% kaolinite). Improvement in compressive strength compared to the OPC was noticed for LC³-50 containing fairly pure metakaolin (86% of kaolinite) content at early ages, whereas clays with 55 and 48% kaolinite reached a compressive strength nearly equal or slightly lower to the OPC in 28 days. However, all LC³ systems show a significantly improved results in terms of water absorption and pulse velocity compared to OPC at early ages onwards. Based on gained results, it can be said that there is a promising future for the using of a combination of limestone and calcined clay as cement replacement up to 45%, which can lead to more significant environmental and sustainable benefits.

Keywords: LC³, Calcined clay, Limestone, CO₂ reduction, Sustainability





تقييم أداء المونة الإسمنتية المحتوية على الحجر الجيري والطين المكلس

الملخص

تبحث هذه الدراسة في تأثير استخدام مزيج من الحجر الجبري والطين المكلس كبديل للأسمنت على الخواص الفيزيائية والميكانيكية للمونة في حالتيها الطازجة والمتصلبة. صنعت المونة من خلط ات بمستويات مختلفة من استبدال الأسمنت (٪ 15، ٪ 30، ٪ 45) بنسبة إحلال 1:2 (الطين المكلس: الحجر الجبري) ، مع تعديل محتوى الجبس في كل نظام من أجل منع انخفاض الكبريتات في الانظمة. علاوة على ذلك، تم استخدام ثلاثة أنواع من الطين المكلس بمحتويات مختلفة من الكبريتات في الانظمة. علاوة على ذلك، تم استخدام ثلاثة أنواع من الطين المكلس بمحتويات مختلفة من الكبريتات في الانظمة. علاوة و 88٪ على ذلك، تم استخدام ثلاثة أنواع من الطين المكلس بمحتويات مختلفة من الكاولينيت بنسبة 88٪ و 55٪ و 88٪ على ذلك، تم استخدام ثلاثة أنواع من الطين المكلس بمحتويات مختلفة من الكاولينيت بنسبة 68٪ و 55٪ و 88٪ على التوالي من أجل التحقق من تأثير درجة الطين المكلس على الخصائص. أظهرت النتائج انخفاضاً في قابلية تشغيل المونة مع زيادة استبدال الأسمنت. ومع ذلك، أظهرت الطينات التي تحتوي على و55 و 48٪ ملى الكاولينيت قابلية تشغيل أفضل من الطين الأكثر نقاء إلى حذ ما (86٪ كاولينيت). لوحظ انخفاضاً في مالكاولينيت المين المكلس على الخصائص. لوخل انخفاضاً في مالية تشغيل المونة مع زيادة استبدال الأسمنت. ومع ذلك، أظهرت الطينات التي تحتوي على 55 و 88٪ من الكاولينيت قابلية تشغيل أفضل من الطين الأكثر نقاء إلى حدٍ ما (86٪ كاولينيت). لوحظ انحسن في مقاومة الإنضغاط مقارنةً بـ OPC بالنسبة لـ 50-⁶ الذي يحتوي على 55 و 88٪ تحمن في مقاومة الانضغاط مقارنةً بـ OPC بالنسبة لـ 50-⁶ الذي يحتوي على محتوى كاولينيت إلى قوة ضغط مساوية تقريبًا أو أقل قليلاً من OPC في 88 يومًا. ومع ذلك، تظهر جميع أنظمة الى حد ما (68٪ من الكاو لينيت إلى قوة ضغط مساوية تقريبًا أو أقل قليلاً من OPC في 88 يومًا. ومع ذلك، من معار في 10 الحين الذي يحتوي على 55 و 88٪ تحما في مقاومة الأنينيت إلى قوة ضغط مساوية تقريبًا أو أقل قليلاً من OPC في 80 يومًا. ومع ذلك، تظهر جميع أنظمة الى حد ما (88٪ من الكار يحتوي على 55 و 88٪ حمار كاري ما معار في 20 و 65٪ ما 200 في 80 يومًا. ومع ذلك، تظهر معيو يا مام ولا مالي كار كام مور مع محتوي ما مام ومع دارة من معار في 20 ما معار كام كار كار ما معان من ما مام وما ميل مالحوا ما مام وما معار في يا مالي مام مور وم

كلمات مفتاحية: طين مكلس, حجر جيري, استدامة, صناعة الاسمنت

1 INTRODUCTION

Concrete is one of most important and it's extensively used construction materials in the world, concrete systems present cement as the most important component in the concrete system. the global demand for Portland Cement (PC) in the construction industry is projected to rise from 3.27 billion tonnes in 2010 to 4.83 billion tonnes in 2030 due to the rapid growth of population in recent years [1]. Indeed, with the manufacture of one tonne of cement approximately





0.8-0.9 tonnes of Carbon Dioxide (CO₂) are launched into the atmosphere in addition to sulfur dioxide (SO₂) or nitrogen oxides (NOx) which can cause the greenhouse effect and acid rain [2]. The raw meal used to produce cement clinker consist of about 75-79% of calcium carbonate, which is added in the form of limestone. therefore, 50-60% of the CO_2 emissions, originates from the decarbonation (calcination of calcium carbonate decomposing to calcium oxide). The other 40-50% of the total amount of CO2 originate from the fuel used in the firing process and the electricity used for grinding and transportation to a lesser extent [3]. A strategy for a high reduction of CO₂ emissions during cement manufacture may be to reduce the amount of clinker content in the cement by replacing it with supplementary cementitious materials (SCMs). The use of Supplementary Cementitious Materials (SCMs) has been adopted for the purpose of not only reducing the quantity of OPC in construction but also improving strength and enhancing durability of concrete [4]. However, most SCMs commonly used in construction such as slag, silica fume, and fly ash are industrial by-products, which its availability declines with a decrease in the production of steel, silicon metal, and coal, respectively [5, 6]. Furthermore, the growing worldwide demand for cement, however, and the declining availability of the aforementioned SCMs has driven the pursuit of alternative mineral additions [7]. In such a situation, the use of metakaolin in combination with limestone in OPC-based systems is reported to obtain increased substitution levels without impacting concrete characteristics [8, 9]. Recent studies showed that by combining limestone with calcined clay, can give synergetic benefits from both well-known systems [8]. When limestone and metakaolin are combined, in addition to the pozzolanic reaction of metakaolin and limestone reaction with C_3A , there is the synergetic effect between alumina in calcined clay and limestone producing carboaluminate phases. Monocarboaluminate hydrate are formed and fill the pore spaces which will increase the strength [8, 10]. The stoichiometric formation of monocarboaluminate hydrate (an AFm





phase) is by reaction of one mole of metakaolin with one mole of calcium carbonate CC in the presence of water and calcium hydroxide CH to give 1 mol of monocarboaluminate. This corresponds to an addition with a weight ratio of 2:1 metakaolin:limestone [8]. Based on this principle, it is possible to replace a mass of clinker by a similar mass of Metakaolin and calcium carbonate mixed in a 2:1 ratio, respectively, to form hydration products which are able to fill the pore system of the cement matrix and contribute to the strength [8, 10, 11]. This ternary blend of limestone, calcined clay and clinker is called LC^3 which is on its way to prove its potential in the cement industry by addressing the two issues of CO₂ emissions and saving natural resources. Moreover, In Europe, India and South America, Limestone calcined clay cement (LC^3) has been studied for several years, but the exploration of LC^3 in Libya is still in its infancy. Furthermore, South Libya contains clay of different qualities and composites based on grade of kaolinite content. Therefore, in this study, performance-based approach is adopted in order to evaluate the physical and mechanical properties of limestone calcined clay cement and compare it to OPC. Mortars are made of cements with different levels of cement replacement (%15, %30, %45) by substitution ratio of 2:1 (calcined clay: limestone). Moreover, three types of calcined clays are used to investigate the influence of the grade of calcined clays on the properties of LC³-blends.

2 EXPERIMENTAL WORK

2.1 MATERIALS PROPERTIES

2.1.1 CEMENT

The cement used in this study is Portland cement (CEM I 42.5N), manufactured by Al-Fattaih Cement Factory and conforming to the Libyan standard which mainly based on the European EN 197-1. The physical and chemical properties and phase composition of the cement used are given in Table 1.



2.1.2 LIMESTONE

Limestone used in this study was supplied by Al-Fattaih Cement Factory. limestone is ground by using Los-Angeles aberration machine, this process converts lumps Limestone to powder form. after grinding, Limestone powder is collected from passing 90-micron sieve. Knowing, limestone powder is used in mixtures without thermal treatment. The physical and chemical properties of the limestone used are given in Table 1.

2.1.3 GYPSUM

The gypsum (>85% purity) used in this study It was supplied by Al-Fattaih Cement Factory. The addition of gypsum was chosen (to obtain around 6% overall gypsum for each system) in order to prevent the system being undersulfated for the blends and to optimize early-age strengths.

2.1.4 CALCINED CLAY

three different clays of varying kaolinite contents sourced from south region of Libya were used in this study. The clay produce from these places were crushed and calcined at 800 °C for 2 h. After the calcination process, the calcined clay was cooled then milled to pass on 90-micron sieve. The details of mineral clay are shown in Table 2, it was taken from a previous study by a research group from Sebha University [12]. The physical and chemical properties of calcined clays used are given in Table 1. The chemical composition of the calcined clays was compared with ASTM C 618 for its suitability to use it as a pozzolan.

Chemical composition %	OPC	LS	MKA	MKB	МКС	Phase composition % - cement
CaO	63.09	54.53	0.117	0.102	0.172	C ₃ S 58.14
SiO ₂	19.88	2.729	54.775	71.557	74.33	C ₂ S 13.22
AI_2O_3	5.37	1.641	41.033	26.025	21.589	C ₃ A 9.4

Table 1: The physical and chemical properties of calcined clays

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Fe ₂ O ₃	2.86	0.064	1.48	1.255	1.009	C ₄ AF	8.7		
MgO	1.52	0.598	0.349	0.108	0.244				
Na_2O	0.01	-	-	-	-	ASTM C618 require	ements		
K ₂ O	0.95	-	-	-	-	Total of SiO_2 ,	MKA	MKB	МКС
Cl	0.018	0.092	-	-	-	70%	97.29	98.84	96.93
SO ₃	2.59	0.026	0.033	0.013	0.017	SO ₃ < 4%	0.033	0.013	0.017
TiO ₂	0.31	0.004	-	0.679	0.956	LOI < 10%	0.91	0.84	0.96
MnO	0.041	0.008	0.021	0.025	0.025	•			
P_2O_5	0.18	0.065	0.053	0.052	0.06				
Loss on ignition	2.54	41.26	0.91	0.84	0.96	Gypsum	> 85% j	purity	
Physical property									
Specific gravity	3.158	2.690	2.510	2.570	2.606				
Blaine's (m ² /kg)	364	581	1318	962	994				

Table 2: Location of clays and clays mineral

Symbol of site	Location	Kaolinite content (%)
MKA	10 km from Sebha	86
МКВ	10 km from Temenhint_Sebha	55
МКС	Tarout_Wadi Shatti	48

2.1.5 AGGREGATES

Fine aggregates used to produce the mortar cement in this study. The fine aggregate was a Natural sand, sand gradient falls outside the gradient limits specified in ASTM C778, but has been modified to conform to the standard sand specified in ASTM C778. The Particle size distributions of fine aggregates measured by sieving test is given in Figure 1. The physical property of Fine aggregates is given in Table 3

Table 3: physical property of Fine aggregates

Aggregate type	Specific Gravity	Absorption (%)	Moisture Content (%)	Fineness Modulus
Fine aggregates	2.73	2	0.56	2.07







Figure 1: Particle size distribution of Fine aggregate

2.1.6 MIX PROPORTIONS

The Mix design for the blended cement samples consisted of looking at changes in two parameters in the sample. The first parameter is the percentage of calcined clay and limestone (with fixed ratio 2:1) replacement, with calcined clay and limestone replacement the clinker at 15, 30 and 45%. Consequently, LC^3 -80, LC^3 -65 and LC^3 -50 correspond to blended cements with approximately 80%, 65% and 50% of clinker content. In addition, the cement in this study was directly used as a part of clinker, therefore, the addition of gypsum was chosen in order to prevent the system being under-sulfated for each systems composition. The second variable is the type of calcined clays used depending on the kaolinite contents in each clay type. Three type of calcined clays are used with various kaolinite contents at 86%, 55% and 48%, respectively. Therefore, ten standard mortar mixtures were prepared and used in this study according to ASTM C109, the proportions of materials for the standard mortar, one part of cement to 2.75 parts of graded standard sand by weight with a water-binder ratio of 0.485 as shown in Table 4.

Mixture ID		Cement		Coloined elem	T :	Gypsum	W/C····*	S/C**
		Clinker	Anhydrite	Calcilled clay	Linestone	addition	w/Cm ⁺	S/CIII**
OPC		94	6	-	-	-	0.485	2.75
LC ³ -80 (86)	$\mathbf{A}_{\mathbf{I}}$	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -80 (55)	BI	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -80 (48)	CI	80	5.11	9.33	4.67	0.89	0.485	2.75
LC ³ -65 (86)	$\mathbf{A}_{\mathbf{II}}$	65	4.15	19.33	9.67	1.85	0.485	2.75

Table 4: The proportions of materials for the standard mortars

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LC ³ -65 (55)	B _{II}	65	4.15	19.33	9.67	1.85	0.485	2.75
LC ³ -65 (48)	CII	65	4.15	19.33	9.67	1.85	0.485	2.75
LC ³ -50 (86)	A _{III}	50	3.19	29.33	14.67	2.81	0.485	2.75
LC ³ -50 (55)	B _{III}	50	3.19	29.33	14.67	2.81	0.485	2.75
LC ³ -50 (48)	CIII	50	3.19	29.33	14.67	2.81	0.485	2.75

W/Cm* - Water to cementitious ratio S/Cm** - Sand to cementitious ratio

2.1.7 **TEST METHODS**

The details of specimens and test methods used to determine fresh and hardened state mortar properties are presented in Table 5.

Table 5: test methods used to evaluate fresh and hardened state properties of mortar

State of Mortar	Property	Method	Specimen Dimensions	Testing period (days)
Forder encounting	Workability	(ASTM C1437)	-	-
Early properties	Fresh State Density	(ASTM C138)	75D, 50L mm	-
	Hardened State Density	(ASTM C830)	50 mm cubic	28
Hardened properties	Compressive Strength	(ASTM C109)	50 mm cubic	1, 3, 7, 14, 28, 90
	Flexural Strength	(ASTM C1018)	40X40X160mm	7, 28, 90
	water absorption	(ASTM C830)	50 mm cubic	7, 28, 90
	Ultrasound pulse velocity	(ASTM C597)	40X40X160mm	3, 7, 28, 90

2.1.8 **Results and discussion**

2.1.9 WORKABILITY

The flow test was carried out following ASTM C1437-15. The flow of the fresh mortars measured after tapping 25 time in 15 seconds on a flow table. Figure 2 shows the flow values obtained for all mixes. The flow values for all the ternary blends containing limestone and calcined clay were lower than the OPC, and decreased with increasing replacement levels. The addition of limestone and calcined clay adversely affects the rheology of the mix. The decrease in flow may be due to the high specific surface area of the MK leading to high water demand [13]. It can be seen that, OPC obtained 75.1% of flow whereas flow values reduced remarkably to 62.2% for A_{III} and decreases slightly





to 71.6% and 71.2% for B_{III} and C_{III} respectively. However, it is worth mentioning that the cements with MKA needed more water in relation to the cements with MKB and MKC, is attributed to the results of granulometry in which the MKA had a higher surface area than the MKB and MKC, both have slightly similar surface area. Furthermore, the kaolinite content of MKA (86%) more than that of MKB (55%) and MKC (48%). This implies that kaolinite content in MK may reduce the workability. These results are in agreement with the findings by Avet [14].



2.1.10 DENSITY OF FRESH AND HARDENED MORTAR

The fresh density of mortar was slightly influenced by different blends cement type. It can be observed that the fresh density of mortar decreases slightly when the replacement levels increases compared to OPC mortar as shown in Figure 3. However, the fresh density fluctuated between 2150 kg/m³ and 2087 kg/m³. It can be seen that, there was no notable change in mortar fresh density for all mixes.

Regarding the dry bulk density, the highest and lowest bulk density were 1740 kg/m³ and 1706 kg/m³ obtained for OPC and C_{III} mortars respectively. This result indicates that using limestone and calcined clay does not impact the dry bulk density of mortar. These results are in agreement with the findings by Nguyen [15].







Figure 3: The density in fresh and hardened state of the mixes mortars

2.1.11 COMPRESSIVE STRENGTH

Figure 4 depicts the development of compressive strength of LC³ blends and OPC cement mortars up to 90-day period. At early ages (1d and 3d), all the LC^3 blends exhibited lower compressive strength than the OPC. The strength reduction became more pronounced as the calcined clay and limestone content increased. At 7 days, improvement in compressive strength compared to the OPC was noticed for all mixes containing MKA, whereas the LC^{3} -80 and LC^{3} -65 blends containing MKB and MKC reached a compressive strength similar or slightly higher to the OPC in 14 days, while the LC^{3} -50 blends had nearly equal or slightly lower compressive strength than the OPC in 28 days. The excellent performance of LC³-MKA mixes could be related to its high kaolinite content compared to LC³-MKB and LC³-MKC mixes. Previous studies have shown that LC^3 mortars can achieve a compressive strength higher or similar to OPC mortar after 7 and 28 days, with replacement rates of up to 45% [8]. However, the clay utilized was a highly pure calcined clay with a high kaolinite content. Other researchers [16] showed that utilizing low-grade calcined clay (44% kaolinite content), mortars with 20% Portland cement substitution could develop a similar compressive strength to OPC mortar at 28 days but lower at 7 days. Moreover, compressive strength decreased noticeably in comparison with OPC mortar at all ages for mortars with 40% Portland cement substitution. Those results are consistent with the results presented in Figure 4. However, the LC³ exhibited





strength development up to 28 days and the development of strength between 28 to 90 days was lower in comparison with OPC, especially in mixtures with a high substitution level. The lack of strength development at late ages can be due to the lack of portlandite and the decrease of the clinker hydration due to the high dosage of limestone and calcined clay. This is consistent with a previous study [17], where a loss of linearity in the strength-kaolinite content relationship was observed at later ages.



Figure 4: Compressive strength development of OPC and LC³ blends mortars at different ages

2.1.12 FLEXURAL STRENGTH

Results of the flexural strength of LC^3 blends and control cement mortars at various curing periods up to 90-day are presented in figure 5. At 7 days, flexural strength for all LC^3 mixes containing MKA globally follow the same trends as compressive strengths. whereas, improvement in flexural strength compared to the OPC was noticed for LC^3 -80 mixes containing MKB and MKC. Beyond the LC^3 -80 level, sharp reduction in strength was observed. However, at the later ages (28 and 90 days), improvement in flexural strengths for LC^3 -65 blends containing MKB and MKC compared to OPC was noticed. Moreover, all the





 LC^3 blends show higher or nearly equal bending strength values with respect to the OPC, especially at 90 days.



Figure 5: Flexural strength of OPC and LC³ blends mortars at different ages

2.1.13 WATER ABSORPTION

The results of water absorption of LC^3 blends and OPC cement mortars cured at three distinct ages are shown in Figure 6. It is clear that the water absorption values for all the LC^3 blends were lower than those of the OPC at all ages. It can also be observed that the higher the replacement level of cement with limestone and calcined clay, the lower is the water-permeable absorption especially at later ages. The decrease in the water absorption value of LC^3 blends could be attributed to a reduction in the total volume of capillary pores as well as increased tortuosity. The hydration products formed by the pozzolanic reaction of metakaolin in LC^3 blends are deposited in the pores and contribute to minimize the capillary porosity of the system. The refinement of pore structure caused by pozzolanic reaction of metakaolin has been widely reported in the literature.







Figure 6: Water-absorption of OPC and LC³ blends mortars at different ages

2.1.14 ULTRASOUND PULSE VELOCITY (UPV)

The results of ultrasound pulse velocity (UPV) of LC3 blends and OPC cement mortars cured at various periods up to 90-day are presented in figure 7. At early ages (3d), all the LC³ blends mortars with the exception of LC³ blends containing MKC showed higher ultrasound pulse velocity values compared to the OPC. As expected, improvement in pore structure occurred with the increase in hydration period. At 7 days onwards, all the LC³ blends exhibited higher pulse velocity values than the OPC. The correlation between compressive strength and pulse velocity of LC³ blends mortar are shown in Figure 6. It can be seen that with the correlation coefficient of $R^2 = 0.89$, the LC³ blends mortars followed a trend similar to that of OPC mortar.







Figure 7: Ultrasound pulse velocity of OPC and LC³ blends mortars at different ages



Figure 8: Relationship between the compressive strength and pulse velocity of OPC and LC³ blends mortars

2.1.15 CONCLUSIONS

In this study, the feasibility of using a combination of limestone and calcined clay as cement replacement was investigated. The effect of different cement replacement level with limestone and calcined clay on various engineering properties of both fresh and hardened mortar was investigated. Furthermore, the influence of the grade of calcined clays on the properties was also studied. Based on the results of this study, the following conclusions were drawn:



- 1. A reduction in mortar workability was observed with the increase in cement substitution by limestone and calcined clay. It can also be observed that the clays with 55 and 48% kaolinite are better than fairly purer clays (86% kaolinite) in terms of workability. It can be due to the MKA had a higher surface area than the MKB and MKC. However, the use of superplasticizers is a strategy that can contribute to overcome the workability reduction observed when using calcined clay.
- 2. Similar or slightly higher compressive strength to OPC is obtained already at 7 days for LC^3 -50 containing fairly pure metakaolin 86% of kaolinite content. However, at 28 days, the use of calcined clays with 48% of kaolinite or more reach strengths similar or closely to OPC, demonstrating the potential of using these widely-available clays as cement substitute.
- 3. all LC³ systems show a significantly improved results in terms of water absorption and pulse velocity compared to OPC at early ages onwards. The progressive replacement of cement with limestone and calcined clay promotes the porosity refinement, which is beneficial for the restricting the movement or transport of fluids/ions in the mortar.
- 4. In conclusion, it can be said that there is a promising future for the using of a combination of limestone and calcined clay as cement replacement up to 45%, which can lead to more significant environmental and sustainable benefits.

2.1.16 ACKNOWLEDGMENT

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