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PERFORMANCE OF MODIFIED LIME MORTAR FOR CONSERVATION OF ANCIENT BUILDINGS

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ABSTRACT

In this paper the efficiency of using different clay based binders was explored to modify the main properties of air lime mortar which is commonly used as binding materials for conservation of ancient buildings. Two different types of Kaolinite and Metakaolin were used for the proposed modification in addition to Hib (Heeba in Arabic), Burned Hib and fine crushed clay bricks (Homra in arabic). The Kaolinite, Hib and Homra were selected as they are usually mixed –locally- with lime mortar to provide suitable binding properties. The influence of burning Kaolinite and Hib on different properties of the binding mortar mixes was explored. On the other hand, different lime replacement ratios were also explored in the proposed mixes. Fresh properties of the proposed mortar mixes were evaluated such as setting time and flowability. On the other hand, main physical and mechanical properties of the hardened mixes were evaluated such as density, water absorption, porosity and strengths (e.g. compressive strength) at different ages. Chemical analysis was performed for the raw material used to make the different mixes. During this study, mainly natural materials were used to produce binding mixes having wide range of strengths. Such mixes can be applied for the compatible conservation cases depending on the original ancient material's strength. On the other hand, all the proposed mixes showed a remarkable reduction of setting time, porosity and water absorption-compared to the control lime mix- providing faster hardening and more durable conservation applications.

Key Words

Lime, Workability, lime mortar, Pozzalanic activity, Porosity, Conservation

INTRODUCTION

Mortars used for bedding and jointing masonry units and rendering masonry surfaces are composed of binders and aggregates [1, 2]. Historically, different types of binder. Materials have been used in the construction of masonry buildings, mud mortars are the oldest documented mortars and were used in the construction of the first collective settlements in Mesopotamia 10,000 years ago [2]. Gypsum mortar is a binder long used in the brick vaults and arches due to its quick setting and high mechanical strength [2, 3]. Lime mortars have been the most widely used in the construction of the buildings since their first known use in Egypt in 4000 BC [2, 4].



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Lime mortars can be classified as air lime (i.e. non-hydraulic) and hydraulic lime. Non-hydraulic lime mortars are produced by mixing slaked lime with aggregates and harden by evaporation and carbonation of lime due to carbon dioxide in the air.

Hydraulic lime mortars are produced either by mixing lime with pozzolans containing amorphous active silicates and aluminates or by developing hydraulic phases through the calcination of silica rich limestone directly quarried or synthetically mixed. Hydraulic lime mortars harden by evaporation, carbonation of lime and the reaction between lime and pozzolans or the hydraulic phases in the presence of water. This reaction produces calcium silicate hydrates and calcium aluminate hydrates, allowing setting under water and impart higher earlier strength to the hydraulic lime mortars [5].

International centers, such as ICOMOS or ICCROM, have recommended the use of materials similar in composition and properties to the original ones for the restoration works of historical buildings [6, 7]. The use of lime mortars in restoration of historical buildings was described in several previous works [8]. Determination of historic lime mortar characteristics became an important subject in the second half of the 20th century. The studies on historic lime mortars and plasters are compiled by Hansen et al. [9] in an extensive bibliography and provide a source for conservators and conservation scientists. Among the studies of historic lime mortars, the achievement of hydraulic properties of historic mortars is usually described as the process of mixing pozzolans with high calcium lime [10–15].

There has recently been increased scientific interest in lime-based mortars and their uses, as they show greater compatibility with ancient building materials and fulfil the recommendations of ICCROM [18] about the use of materials similar to the original ones in repair work. [19–23]. Lime mixed with Pozzalanic additions were used extensively in the past as mortars for the construction of historical and traditional buildings. Mortar/plaster or concrete produced by the mixtures of lime and calcinated clay supplied from ground or broken clay tiles. Therefore, analogous materials should be used in today's interventions on the historic buildings in order to assure compatibility of the restoration mortars to authentic ones.

One of the major problems of selecting the appropriate pozzolan used as a pozzolanic addition in restoration mortars is its reactivity. The use of highly reactive pozzolans as an additive to lime mortars produces hydraulic, durable mortars with sufficient mechanical strength, similar to historic ones. [22-25]. The present paper aims at investigating the effect of modifying lime mortar by several natural local minerals on the physical and mechanical of the mortars. On the other hand, providing wide selection of binding mortar's strengths introduces different solutions for conservation applications and ancient materials.

EXPERIMENTAL WORK

Several mixtures of air lime mortars were prepared -following BS EN 459-2:2010- with different percentage of either Kaolinite, Metakaolin, Hib (Heeba in Arabic), burned Heeba or fine crushed clay bricks (Homra in Arabic) as well as using controlled sand. In



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addition, the control mortar made of air lime were also prepared for comparison. Table 1 shows the composition of the 15 tested mortar mixtures.

Table 1: Mixing proportions (by weight) for mortar mixtures.

Mix ID	Hydrated Lime	Pale Kaoline	Dark Kaoline	Pale Meta Kaoline	Dark Meta Kaolinite	Homra	Heeba	Burned Heeba	Controlled Sand	Water
CNTRL	1	-	-	-	-	-	-	-	3	0.93
LPK33	1	0.5	-	-	-	-	-	-	3	0.83
LPK50	1	1	-	-	-	-	-	-	3	0.83
LDK33	1	-	0.5	-	-	-	-	-	3	0.855
LDK50	1	-	1	-	-	-	-	-	3	0.84
LPMK33	1	-	-	0.5	-	-	-	-	3	0.88
LPMK50	1	-	-	1	-	-	-	-	3	0.86
LDMK33	1	-	-	-	0.5	-	-	-	3	0.86
LDMK50	1	-	-	-	1	-	-	-	3	0.81
LHO33	1	-	-	-	-	0.5	-	-	3	1
LHE33	1	-	-	-	-	-	0.5	-	3	0.95
LHE50	1	-	-	-	-	-	1	-	3	0.89
LHE67	0.5	-	-	-	-	-	1	-	3	0.83
LBHE33	1	-	-	-	-	-	-	0.5	3	0.81
LBHE50	1	-	-	-	-	-	-	1	3	0.83

1. Materials, specimen manufacture and curing

1.1 Materials

1.1.1 Lime

The used Hydrated (slacked) air lime ($\text{Ca}(\text{OH})_2$) powder was a commercial product supplied by Hamaco [26] is of class EN 459-1 CL70-S satisfying BS EN 459-1[27].

1.1.2 Aggregate

The used sand was mainly of quartz and it was sieved to ensure that it satisfied the third grading zone of ES 1109/71, as shown in Table (2). Sand was also washed on sieve $0.75\mu\text{m}$ (200 mesh) to control very fine particles in aggregate. Finally sand was used in saturated surface dry (SSD) condition in all mortar mixes.

1.1.3 Mineral Additives

Two different types of Kaolinite and Metakaolin were used for the proposed modification in addition to Hib (Heeba in Arabic), Burned Hib and fine crushed clay bricks (Homra in Arabic). Metakaolin, and Burned Heeba were prepared by burning Kaolinite and Heeba on oven was obtained by heat treating of $850-900^\circ\text{C}$.

- **Kaolinite:** Kaolinite [$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$] is a naturally forming clay that is widely used in ceramic industries. Upon sintering, kaolinite undergoes a phase transformation process over a relatively broad temperature range and this thermal behavior has been extensively studied [28-31]. Kaolinite, with the 1:1-type layered structure has high crystallinity and unique structure: one side of the interlayer space is covered with



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hydroxyl groups of the $Al_2(OH)_4$ octahedral sheets and the other side is covered by oxygens of the SiO_4 tetrahedron.[32].

- **Metakaolin:** The $<2\mu m$ kaolinite fraction was calcined in a programmable muffle furnace at $900^{\circ}C$ for 2 h, and the obtained calcined kaolinite (metakaolinite, Cal K) [33].
- **Heeba:** from the clay containing calcitic soil deposits.
Hib has historically been used in plasters in the Theban area, and while precise source locations for the raw materials of ancient plasters is difficult to determine after the passage of 3000 or more years [34].
- **Homra:** homra is the common Arabic name for the powder produced by crashing old clay fired bricks. Nowadays, homra is produced from modern red bricks prepared from shale "Tafla". Tafla is a silty sediment containing mainly the same clays minerals to mention montmorillonite, kaolinite, illite.

The burning of the clayey minerals forms vitrified products containing more reactive silica and alumina than the original clay like the volcanic earths that were molten with the lava (35). It was found that burning the slightly reactive kaolinite in clay while manufacturing clay bricks gives place to meta kaolinite which is a very reactive mineral (36). Table (3) shows the chemical composition of the used lime and mineral additives. The chemical analysis was carried out according to ASTM C114-2013.

1.1.4 Preparation of strength specimens

Samples were prepared using a pre-mixing calcium hydroxide powder (CH) with mineral additive (replaced) by the ratios given in Table (1) (mass.%) .

The amounts of water given in Table (1) were designed so that each mortar had comparable consistencies (i.e. flow range of 160–170mm as shown in Table (4)). In the case of the mortars made with lime putties, workability sufficient for filling molds even with low consistency values. All the mortars were mechanically mixed in a laboratory mixer using a standard sequence of operations.

Mortars mixtures compacted in the molds with twenty stricken for each of the two layers used to fill the molds. Nine cubes [50*50*50 mm], three prisms [40*40*160 mm] and three cylinders [40×80 mm] were prepared for each mixture. Surfaces of the filled molds were then slightly pressed to remove any air bubbles, voids and to finish the surface of specimen. Specimens were released from the molds after 24 hours and curing was carried out in controlled environmental conditions (RH $65 \pm 5\%$ and $2 \pm 23^{\circ} C$) until the test day. For the first 28 days the mixtures were kept in the controlled environmental chamber. During the last 6 days before testing, all samples were kept in a relatively constant laboratory environment (T = $(22 \pm 3)^{\circ}C$, RH = $(30 \pm 2)\%$) (Fig.1).



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Fig.1: The controlled environmental chamber

(Table 2) Grading of the used sand

Sieve size mm	9.51	4.75	2.83	1.41	0.707	0.354	0.177	0.075
% Passing	100	100	100	94.33	71.48	15.7	3.75	0
% Passing ES 1109/71	100	90-100	85-100	75-100	60-79	12-40	0-10	0

(Table 3) The chemical composition of the used lime and mineral additives

Component	%							
%	*Hydrated Lime	Pale Kaolinite	Dark Kaolinite	Pale Meta Kaolinite	Dark Meta Kaolinite	Hib	Burned Hib	Homra
(SiO ₂)	0.7003	63.6690	64.3729	70.7901	72.561	37.3666	43.0601	48.8901
(Al ₂ O ₃)	0.1213	21.7919	17.8449	24.521	21.012	42.3109	48.7578	26.0009
(Fe ₂ O ₃)	0.0062	0.8959	3.0557	0.9951	3.4221	3.8494	4.436	5.8882
(CaO)	71.8745	0.9254	1.1008	1.061	1.232	0.8723	1.0053	2.5508
(MgO)	0.5170	0.4113	0.5964	0.271	0.660	0.2435	0.2806	0.7256
Cl ⁻	0.0103	0.0761	0.0282	0.091	0.032	0.0802	0.0924	0.1006
(P ₂ O ₅)	0.0084	0.0108	0.0113	Nil	Nil	Nil	Nil	Nil
(k ₂ O)	0.1672	0.1679	0.1275	0.282	0.141	0.1786	0.2059	0.5554
(Na ₂ O)	0.3599	0.6372	0.5890	1.2201	0.6701	1.0786	1.234	1.1083
(TiO ₂)	Nil	0.0032	Nil	Nil	Nil	Nil	Nil	Nil
(SO ₃)	0.0399	0.0622	0.0070	0.2203	0.0081	0.2181	0.2514	0.2055
(L.O.I)	26.1254	11.2124	12.1263	0.2101	0.4403	13.2221	0.5766	13.9563
Total	99.9311	99.8633	99.9211	99.944	99.738	99.4125	99.9001	99.9817
Ca(OH)₂	96.8745	Nil	Nil	Nil	Nil	Nil	Nil	Nil
Active – SiO ₂ %	Nil	44.9553	40.005	49.989	44.853	13.5914	15.6223	21.511

*Comply with EN 459-1 CL70-S Limits ((CaO+MgO) ≥ 65% , MgO ≤ 7% , SO₃ ≤ 2.5%)



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2 TEST RESULTS AND DISCUSSION

2.1 Determination of Flow

The flow of mortar mixtures were determined to ensure a uniform consistency. Flow was determined according to EN 196-1 (BS EN 459-2:2010 - 6.8) and the flow table test results are show in (Table4)

Table4: Flow (mm) Table Test Result.

Mix ID	CNTRL	LPK 33	LPK 50	LDK 33	LDK 50	LPMK 33	LPMK 50	LDMK 33	LDMK 50	LHO 33	LHE 33	LHE 50	LHE 67	LBHE 33	LBHE 50
Flow	161	160	165	165	163	168	170	161	168	170	170	169	163	169	160

2.2 Setting time test

The setting time test were used to measure the initial and final setting time of the paste specimens, according to BS EN 459-2:2010 - 6.5.4. The setting time test results are show in (Table5). The results show that all mixture's additives remarkably increased the initial setting time except specimen LPMK50 where the initial setting time was slightly decreased. On contrary, using all the proposed additives decreased the final setting time. Further reduction of the final setting time was monitored with increasing the content of the additives for pale and dark kaoline and pale and dark metakaolinite, while increasing the content of Heeba and Bunt Heeba increased the final setting time.

2.3 Lime Combination Test

The purpose of the lime combination test is to determine the relative Pozzalanic activity of minerals. The test method appears in A. D. Cowper's Lime and Lime Mortars in 1927. They referred to the test as a "Practical Test for Pozzalanic Properties" [32]. The test measure the calcium aluminum – silicate hydrated which from when a mineral in mixed with lime and water as the increase of volume of solid matter that can be visually observed in a test tube after one day (Fig.2). Table (6) show that the highest activity is by the Metakaoline mixture. Here, it has to be streamered that the volume increassated after one day varies significantly. This seems to be inversely to the final setting time. A modification of the test would be needed to measure the volume at final setting time then to determine the final increase in the volume as an indication of the Pozzalanic activity. The results show that all mixtures additives resulted in increasing the final volume with maximum increase with 115.79% with LLMK50 and minimum increase 7.7 % with L33HE76.



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Table 5: Physical properties of fresh mortar mixtures

Formulation	Standard water	Setting time		
		Average Initial Set (hours)	Average Final Set (hours)	Δt
CNTRL	0.93	2:45	40:10	37:25
LPK33	0.83	5:15	27:30	22:15
LPK50	0.83	3:35	24:30	20:55
LDK33	0.855	3:55	25:10	21:15
LDK50	0.84	3:20	24:05	20:45
LPMK33	0.88	3:20	25:15	21:55
LPMK50	0.86	2:35	20:05	17:30
LDMK33	0.86	3:50	25:45	21:55
LDMK50	0.81	2:55	21:33	18:38
LHO33	1	4:30	26:20	21:50
LHE33	0.95	3:15	31:45	28:30
LHE50	0.89	5:10	34:30	29:20
LHE67	0.83	5:30	39:55	34:25
LBHE33	0.81	3:20	23:55	20:35
LBHE50	0.83	5:20	34:50	29:30



Fig.2: Lime Combination Test

Table 6: Lime Combination Test Results

Formulation	Average Vol. Day 1 (mL)	Average Final Vol. (mL)	Increase (%)
LCONTLM	1.9	1.9	Nil
LLK33	1.7	1.9	11.76
LLK50	2	2.4	20
LDK33	1.8	2	11.12
LDK50	2	2.5	25
LLMK33	1.6	3.1	93.75
LLMK50	1.9	4.1	115.79
LDMK33	1.7	3.2	88.24
LDMK50	2	4.4	120
LHO33	1.6	2.5	56.25
LHE33	1	1.5	50
LHE50	1.2	1.6	33.34
L33HE76	1.3	1.4	7.7
LBHE33	1.4	1.6	14.29
LBHE50	1.7	2	17.65

3. Mechanical Properties of Mortar Mixtures

3.1 Compression test

Compression test was complied with EN 196-1:2005 and was implemented using a Tecnotest® compression-testing machine see (Fig.3). The maximum load was automatically recorded and converted into stress. Initially, compressive strength of all mortar samples were explored according to EN 196-1:2005& ASTM C109 using 9 cubes [50×50×50 mm] cubic samples (3 samples for each age). The compressive strength test was carried out at different ages up to 296 days age as shown in Table (7).



Fig.3: Compression test setup



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Table 7: Compression Strength Test Results (Mpa)

Age (days)	CNTRL	LPK33	LPK50	LDK33	LDK50	LPMK33	LPMK50	LDMK33	LDMK50	LHO33	LHE33	LHE50	LHE67	LBHE33	LBHE50
25							2.185								
27								3.194							
34								0.725							
36															0.505
37			0.620												
39					0.675										
41														0.765	
44						3.605									
51	0.325											1.390			
86													1.290		
91											0.455				
92															
93										0.964					
105				0.582											
106		0.804													
166							3.393								
168								4.477							
175								0.803							
177															0.586
178			0.810												
180					0.946										
182														0.957	
185						5.196									
192	0.540														
215							3.718								
217								4.949							
224								0.812							
226															0.623
227			0.814									1.797			
229					0.972										
231														0.985	
232															
233											0.520		1.406		
234										1.153					
241	0.577					5.412									
246				0.693											
247		0.860													
276												1.837			
281													1.479		
282										0.568					
283									1.165						
295				0.720											
296		0.880													

Figures (4) through (9) present the compressive strength at different ages for various mixtures show in table (7). It can be seen that the highest compressive strength (5.2 MPa) was obtained by replacing 33 % of the lime by the pale Metakaoline. In order to achieve close strength, 50 % replacement by dark Metakaoline (4.95MPa). The Results show compressive strength was decreased by 10% by replacing 33 % of the lime with Hib. On the other hand, replacing 50 % of the lime with burned Hib increased the maximum compressive strength increase by 8%. By replacing 33 % of the lime by the pale Metakaoline the compressive strength was increased more than eightfold, while replacing 50 % of the lime by the dark Metakaoline compressive strength was increased more than sevenfold. The chemical composition of the additives had remarkable influence on the compressive strength differences in addition to their expected higher activity by burning. All the presented compressive strengths were compared after 230±10 days.

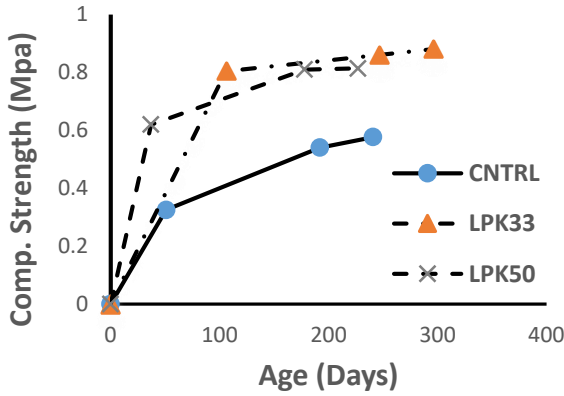


Fig.4: Compressive strength of control line with different percent of pule kaoline

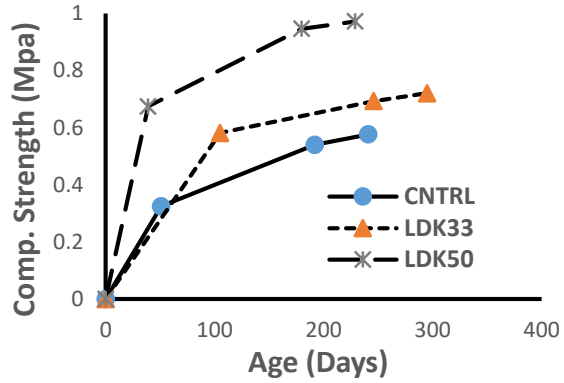


Fig.5: Compressive strength of control line with different percent of dark kaoline

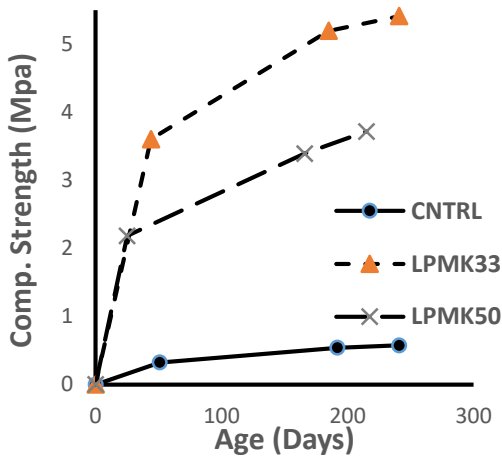


Fig.6: Compressive strength of control line with different percent of pule meta kaoline

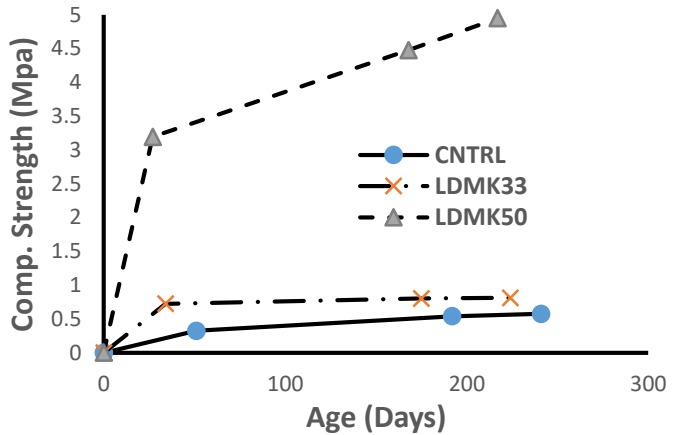


Fig.7: Compressive strength of control line with different percent of dark meta kaoline

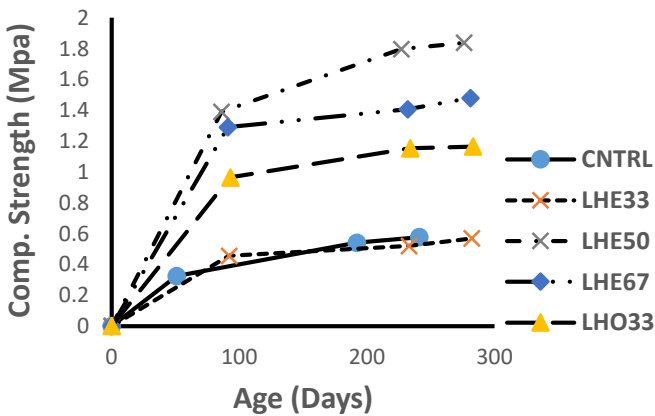


Fig.8: Compressive strength of control line with different percent of Hib & Homra

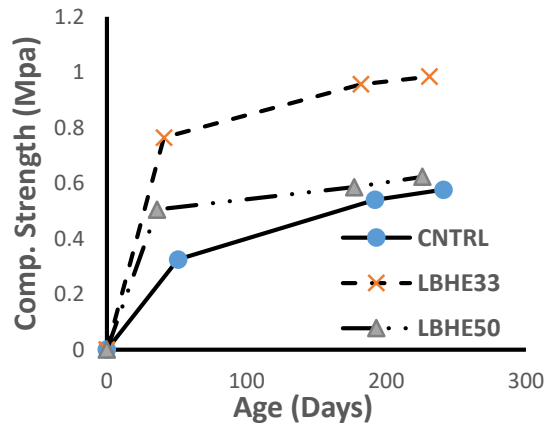


Fig.9: Compressive strength of control line with different percent of burned Hib

The Flexural strength test was carried out as shown in Figure (10). Figure (11) shows the flexural strength of different mixtures at 324 days. It can be seen that highest flexural strength was obtained with lime replacement of 50% pule Metakaoline and 50 % dark Metakaoline which are compatible with the compressive strength's results. The minimum increase of flexural strength was achieved by replacing lime with 33% of Dark koline and Homra having 88.5%, and 120.7% increase respectively.



Fig.10: Flexural strength test setup

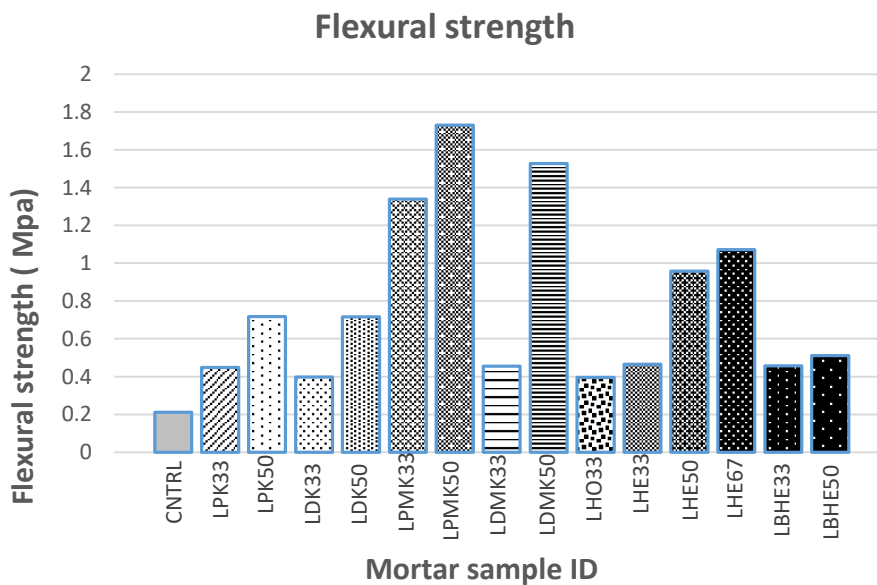


Fig.11: Flexural strength test result for 15 mix



3.3 Splitting Tensile strength

To study the effect of different additive materials on splitting tensile strength on hydrated lime, seven 40×80 mm cylinders were prepared –for each mortar mixture- by the same proportions mentioned in Table1. The cylinders were tested according to Brazilian splitting test at the age of 220 ± 10 days by using A Tecnotest® compression-testing machine as shown in figure (12). The results are presented in Table (9) while Figures (13) through (18) provide the splitting tensile strength progress up to 240 days. It can be seen that highest splitting tensile strength was achieved with 33% dark kaolin and 33 % pule kaolin lime replacement having 45.2%,24% increment respectively which is not compatible with the results of the compressive strength. The result show that decrease of splitting tensile strength from control mix with -12.89 % by replacing 33 % of the lime by Homra, when we replacing 33% of the pule kaolin splitting tensile strength decrease by -9.07 %. It can be seen that Compressive strength/splitting tensile strength result ratio between 1.29 % with 33 % pule Metakaoline to 15.47 % with 33% Hib. See table (8).

Table 8: Compressive strength/splitting tensile strength result (%)

MIX	CNTRL	LPK 33	LPK 50	LDK 33	LDK 50	LPMK 33	LPMK 50	LDMK 33	LDMK 50	LHO 33	LHE 33	LHE 50	LHE 67	LBHE 33	LBHE 50
C./S. %	14.79	12.25	9.88	16.20	7.39	1.29	2.55	9.31	1.53	12.90	15.47	5.12	6.88	8.55	12.53



Fig.12: Flexural strength test setup

Table 9: Splitting Tensile strength Test Results (Mpa)

Age (days)	CNTRL	LPK33	LPK50	LDK33	LDK50	LPMK33	LPMK50	LDMK33	LDMK50	LHO33	LHE33	LHE50	LHE67	LBHE33	LBHE50
82							0.047		0.023						
84			0.040									0.063			0.047
89					0.038					0.062					
94													0.054		
140				0.045		0.040252		0.034			0.051			0.056	
143	0.049	0.056													
180							0.082		0.074						
182			0.078									0.091			0.078
189					0.069					0.066					
190															
192													0.096	0.081	
194			0.080		0.072					0.067				0.084	
198		0.102		0.112							0.076	0.094	0.097		0.082
199						0.066	0.095	0.058	0.076						
206		0.077													
238				0.117		0.070		0.071			0.080				
241	0.085	0.105													

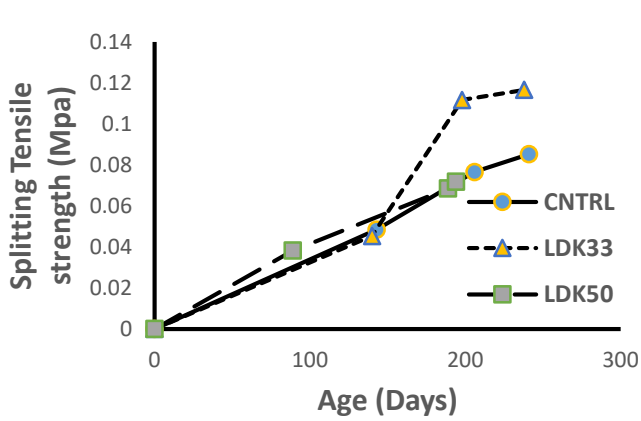


Fig.13: Splitting strength of control lime with different percent of pule kaoline

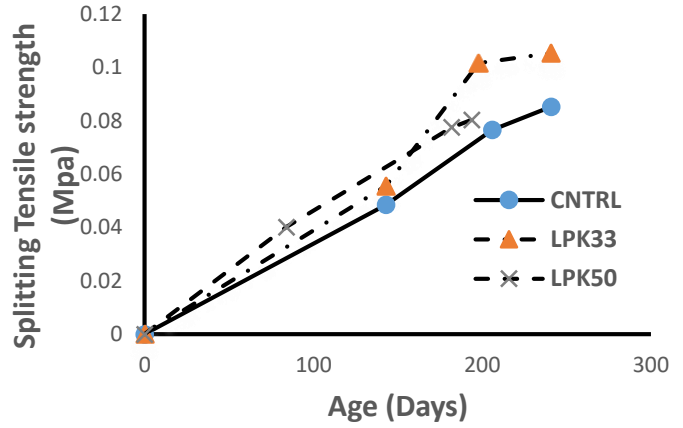


Fig.14: Splitting strength of control lime with different percent of dark kaoline

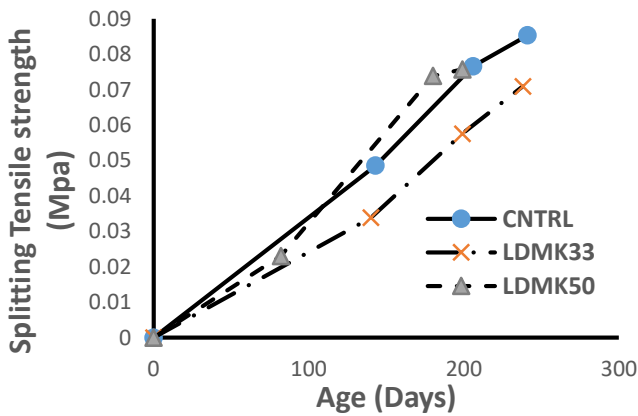


Fig.15: Splitting strength of control lime with different percent of pule meta kaoline

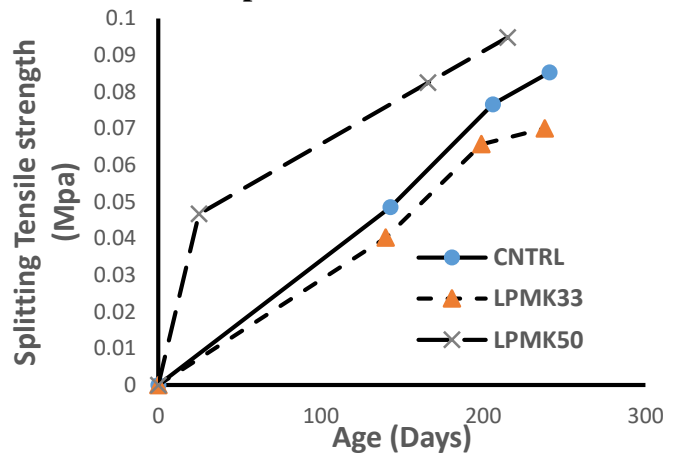


Fig.16: Splitting strength of control lime with different percent of dark meta kaoline

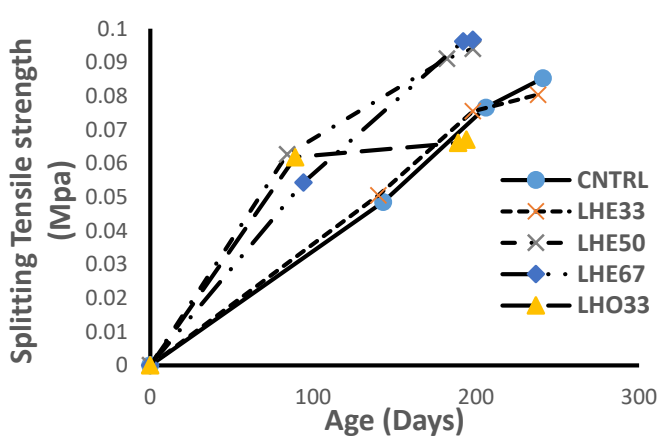


Fig.17: Splitting strength of control lime with different percent of Hib & Homra

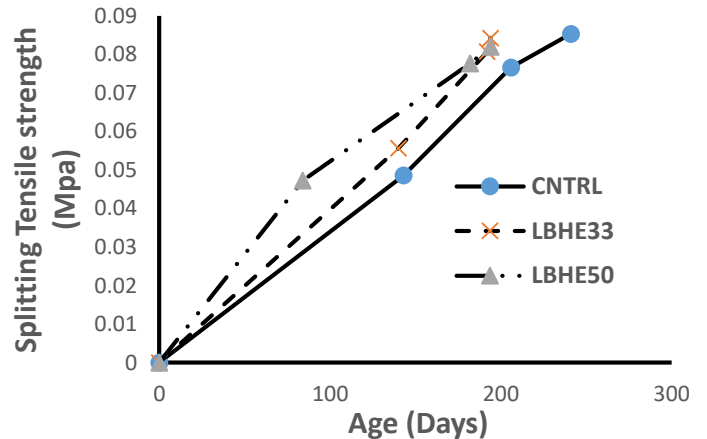


Fig.18: Splitting strength of control lime with different percent of burned Hib

4. Physical Properties

4.1 Unit weight – Absorption – Porosity

The dry unit weight was calculated for three compressive strength specimens as the cubes were weight in dry condition and their dimensions were measured before testing. Absorption was calculated for three cubes cast from each mix. Cubes were weighed in three conditions: saturated after being submerged in water for 24 Hr. (water made a shallow cover over specimens), dried in air, dried in oven. Thus, apparent and total absorption were calculated .while Porosity was calculated from the unit weight and the total absorption. [33-34]

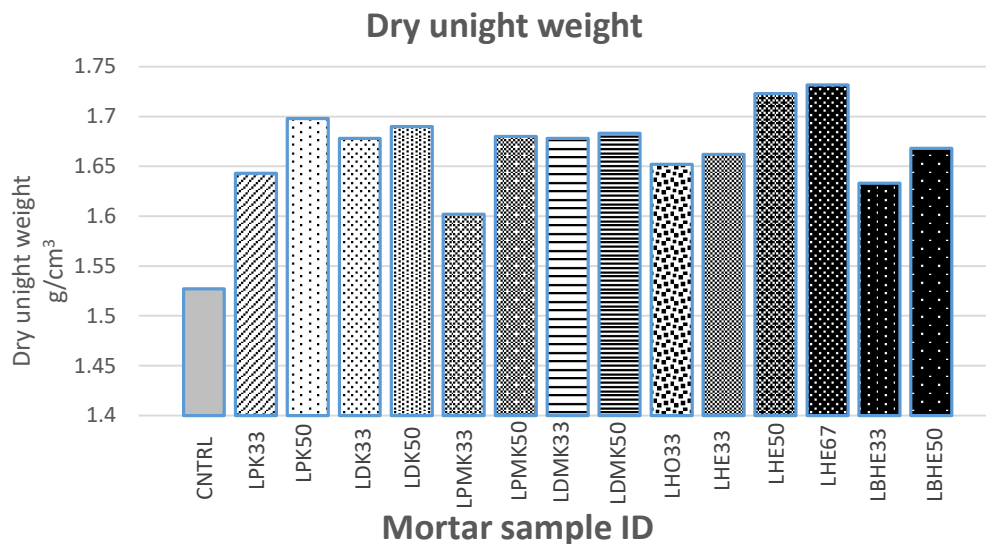


Fig.19: Dry Unit (volume) weight Test Result (G/Cm³)

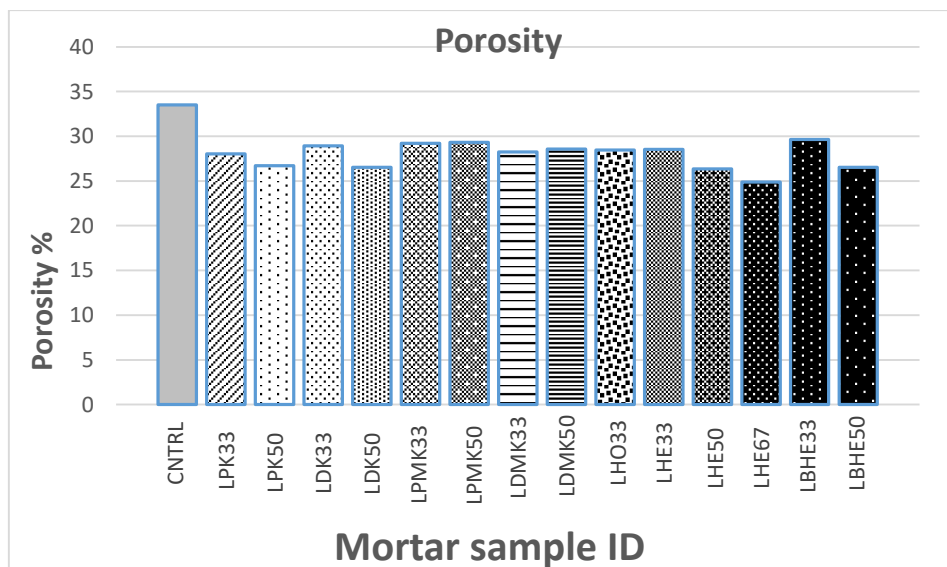


Fig.20: Porosity Test Result (%)



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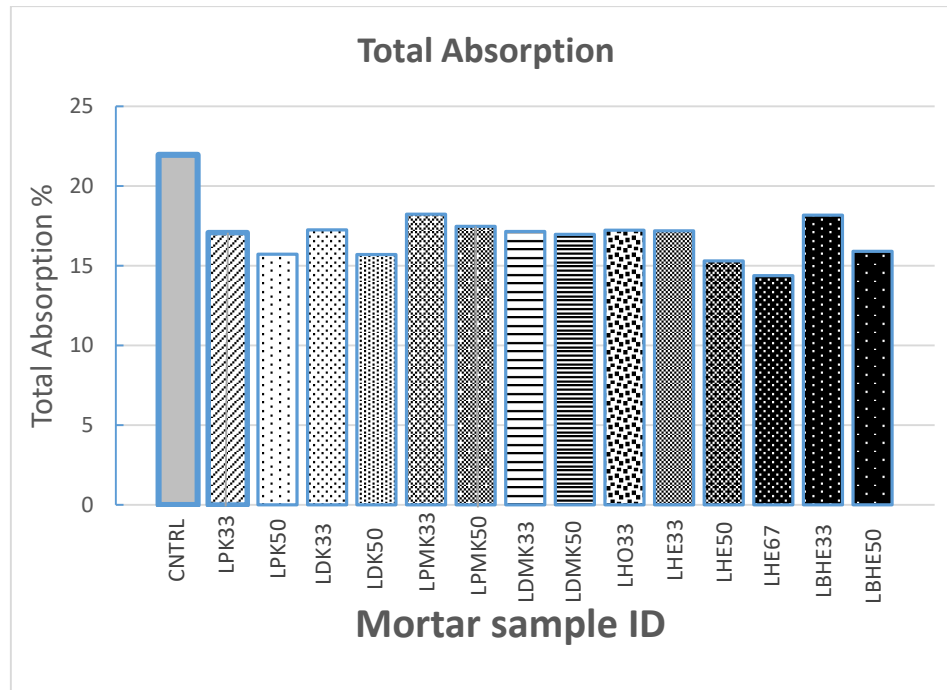


Fig.21: The Total Absorption Test Result (%)

The result of the tests are show in Figures (19) through (21). It can be seen that the porosity and absorption of the modified mortars are less than that the control air lime mortar leading to expectedly more durable mortars. On the other hand the dry unit weight of the control air lime mortar is less than the other modified mortars. This increment ranged between 5% and 13.4% for 33 % pule meta kaolin and 67% Hib replacement ratios respectively.

5. CONCLUSIONS

From the point of view of historical buildings conservation, it's important to achieve different ranges of physical, mechanical and hydraulic properties for lime-base mortars using natural additives and without using any ratio of cement. The strength increase can be turned to reproduce the chemical properties of the historic mortar in order to be compatible with the original materials. In addition the porosity and absorption of modified lime mortars were found to be either comparable or less than the respective properties of the classical lime mortars.

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