

Vol. 36, No. 06, pp. 1006-1019/Diciembre 2023

## Effect of curing conditions and formulation on the adhesive strength of stonesiporex mortar composite

# Efecto de las condiciones de curado y formulación sobre la resistencia adhesiva del compuesto de mortero stone-siporex

Harith E. Ali<sup>1,\*</sup>, Xavier Brunetaud<sup>2</sup>, Kevin Beck<sup>2</sup>, Naima Belayachi<sup>2</sup>, Malek Balawi<sup>2</sup>

Northern Technical University(NTU), Engineering Technical College, Mosul, Iraq. email: \*hbz\_alhaded@yahoo.com , Orcid: 0000-0002-0574-6851.
Laboratoire de Mécanique Gabriel Lame, Polytech Orleans-Université d'Orleans, France. email: xavier.brunetaud@univ-orleans.fr, Orcid:0000-0001-8470-2402. email: kevin.beck@univ-orleans.fr, Orcid:0000-0001-7573-5974. email: naima.belayachi-belaiche@univ-orleans.fr, Orcid: 0000-0003-4112-3368. email: maleklabs@hotmail.com, Orcid: 0009-0002-3448-582x

(recibido/received: 30-julio-2023; aceptado/accepted: 25-octubre-2023)

#### **ABSTRACT**

Background: Adhesive strength is one of the most important properties of the hardened mortar, and influenced by mortar workability (i.e. mortar water content), and substrate roughness surface degree. **Objective:** This study aims to measure the adhesion strength between three different hydrated aerial lime mortars, and the two most common building units in old and modern building in Loire valley in France: Tuffeau and Siporex, under different relative humidity (12,66,98%) and curing periods (7,28,90 days). Materials and Methods: These mortars include: B/F (1:5.6: Moisture=46 %, aggregate stone powder pass sieve NO.2mm), B/W (1:5.6: Moisture=44 %, aggregate stone powder pass sieveNO.2mm and retained on sieve NO. 0.16mm), and B/F/M (1:5.6: Moisture=35%: superplasticizer water reducer Melment F10 (2.5% of lime amount), aggregate stone powder pass sieve NO.2mm). Numerous complementary techniques have been used to achieve this study, such as: x-ray diffraction, coupled plasma optical emission spectrometry, mercury intrusion porosimetry, mortars axial expansion by consolidation apparatus, and Proceq-DY-215 apparatus for adhesive strength measurement. **Results:** The results showed that expansion of B/F, B/W, and B/F/M has little effect on their adhesion strength with Tuffeau /Siporex substrates. Also, the adopted relative humidity variations had a limited effect on the adhesion strength of B/W along the curing periods (7, 28, and 90 days), with noticeable effect for the other mortars. Conclusions: The conclusions referred to the important impact of the degree of substrate surface roughness on the adhesive strength of all used mortars, where the use of Siporex as substrate recorded a clear advantage compared to Tuffeau under all used relative humidity and curing periods.

Keywords: Hydrated aerial lime, Melment, Adhesive strength, Tuffeau, Siporex, Relative Humidity.

## **RESUMEN**

**Antecedentes:** La resistencia adhesiva es una de las propiedades más importantes del mortero endurecido y está influenciada por la trabajabilidad del mortero (es decir, el contenido de agua del mortero) y el grado

de rugosidad de la superficie del sustrato. Objetivo: Este estudio tiene como objetivo medir la resistencia a la adhesión entre tres morteros de cal aérea hidratada diferentes y las dos unidades de construcción más comunes en edificios antiguos y modernos en el valle del Loira en Francia: Tuffeau y Siporex, bajo diferentes niveles de humedad relativa (12, 66, 98%) y períodos de curado (7, 28, 90 días). Materiales y **métodos:** Estos morteros incluyen: B/F (1:5.6: Humedad = 46%, agregado de polvo de piedra que pasa por tamiz NO.2mm), B/W (1:5.6: Humedad = 44%, agregado de polvo de piedra que pasa por tamiz NO.2mm y retenido en tamiz NO. 0.16mm), y B/F/M (1:5.6: Humedad = 35%: plastificante reductor de agua Melment F10 (2.5% de la cantidad de cal), agregado de polvo de piedra que pasa por tamiz NO.2mm). Se han utilizado numerosas técnicas complementarias para llevar a cabo este estudio, como la difracción de rayos X, la espectrometría de emisión óptica de plasma acoplado, la porosimetría por intrusión de mercurio, la expansión axial de morteros mediante un aparato de consolidación y el aparato Proceq-DY-215 para medir la resistencia adhesiva. Resultados: Los resultados mostraron que la expansión de B/F, B/W y B/F/M tiene poco efecto en su resistencia adhesiva con sustratos de Tuffeau / Siporex. Además, las variaciones de humedad relativa adoptadas tuvieron un efecto limitado en la resistencia adhesiva de B/W a lo largo de los períodos de curado (7, 28 y 90 días), con un efecto notable en los otros morteros. Conclusiones: Las conclusiones se refieren al impacto importante del grado de rugosidad de la superficie del sustrato en la resistencia adhesiva de todos los morteros utilizados, donde el uso de Siporex como sustrato registró una clara ventaja en comparación con Tuffeau bajo todas las condiciones de humedad relativa y períodos de curado utilizados.

Palabras claves: Cal aérea hidratada, Melment, Resistencia adhesiva, Tuffeau, Siporex, Humedad relativa.

#### 1. INTRODUCTION

Adhesive strength is one of the most important properties of the hardened mortar, and influenced by mortar workability (i.e. mortar water content), and substrate roughness surface degree (Costigan & Pavía, 2010; Hendry & Khalaf, 2017). The use of lime mortars with the historic and modern calcareous units are more compatible than any other mortars (Cizer et al., 2010; Costigan & Pavía, 2010; Mathey & Rossiter, 1988). Lime mortars are usually applied to the substrate surface, and are penetrate within its pores by capillarity and forming a mechanical attachment (Hansen et al., 2003). However, rough substrate surface is receptive to the wet lime mortar and increase adhesion more than smooth surfaces (BIA, 2003; Costigan & Pavía, 2012; Pathanatecha, 2019). Moisture transfer between lime mortar and substrate are needed to develop adhesive strength, where it is largely governed by the mortar water content. From other side, the excessive water content of lime mortar can reduce adhesion at the mortar-substrate interface because of the low mortar workability (i.e, too fluid mortar), also, in this context, lime mortar expansion may occurs due to absorbed moisture and CaCO<sub>3</sub> formation by an amount of 1.35gm per 1 gm of Ca(OH)<sub>2</sub> within carbonation activity when it placed continuously under wet conditions, the produced swelling stresses can reduce the adhesive strength ((PCA), 1994; Chang et al., 1997; Costigan & Pavía, 2012; Pavía & Hanley, 2010; Pavía & Toomey, 2008). Another factor reduce adhesive strength produced from the incompletely filling of substrate surface pores by poorly graded lime mortar, turned these pores to be a weak spots, where water can permeate into and conversely bring about delamination or corrosion (Pathanatecha, 2019; Pavía & Toomey, 2008; Stefanidou & Papayianni, 2005). Adhesive strength between hydrated lime mortar and substrate is controlled by two mechanisms:(I) Chemical adhesion: bond between pozzolanic/carbonation reactions productions (i.e, calcium silicate/aluminate hydrate and CaCO<sub>3</sub> crystals) and substrate pores(Lanas & Alvarez-Galindo, 2003; Ngoma, 2009). This mechanism is inversely related with particle aggregate size (El-Turki et al., 2009). (II) Mechanical adhesion: interlocking effect between hydrated lime mortar and substrate, and directly related with substrate surface roughness (R. M. Lawrence et al., 2007; Pathanatecha, 2019). Pozzolanic and carbonation reactions are responsible for lime mortar hardening. Pozzolanic reaction precede carbonation, and occur between the dissolved calcium hydroxide Ca(OH)<sub>2</sub> (i.e. included in the hydrated lime mortar mineralogical composition), and silica/ alumina in the lattices of the clay minerals in highly alkaline environment, and produce calcium silicate/aluminate hydrates (R. M. H. Lawrence, 2006). Carbonation reaction proceeds by several steps: CO<sub>2</sub> diffusion within the mortars, followed by the reaction between the dissolved Ca(OH)<sub>2</sub> and CO<sub>2</sub> in the pore water of mortars, then CaCO<sub>3</sub> formation, which acts as a binding agent due to its interconnected microstructure (Beruto et al., 2005; Cizer et al., 2012). The degree and order of Pozzolanic/carbonation reactions will depend primarily on environmental temperature and relative humidity (Cizer et al., 2010; El-Turki et al., 2007, 2010; Kirk et al., 2015; Morgan & Ball, 2013; Van Balen, 2005). Carbonation occurs most favourably at a relative humidity ranged (40%-80%) where 100% of the lime mortar pore surface will be available (Stoian et al., 2015), but below 20% relative humidity carbonation cannot occurs because of insufficient pore water present for either Ca(OH)2 or CO2 to be dissolve, while with relative humidity above 90%,less than 50% of the pore surface will be available for carbonation process (25). However, the optimum carbonation speed is found at 20°C, with a carbonation depth directly proportional to the square root of time. This study concerns the adhesive strength between different compositions of hydrated lime mortars and the surfaces of Tuffeaue/ Siporex building units, which represent the main building units of old and modern buildings respectively in France. The adhesion strength has been determined after subjecting the mortars-buildings composite units to relative humidity (12,66,98 %) and curing periods (7,28,90 days).

#### 2. EXPERIMENTAL PROGRAM

#### 2.1. Materials

#### 2.1.1. *Mortars*

Three different mortars have been adopted in this study, their compositions consist of: hydrated aerial lime, aggregate stone powder from Saint-Cyr-en-Bourg Tuffeau stone which is available in the Loire Valley of France, and Melment F10 superplasticizer water reducer based on melamine formaldehyde resin (Sheet, 2002), see Table (1). The effective concentration of the hydrated lime had been recommended by numerous researchers to be (1:4-1:5.6, i.e. in term of aggregate/binder ratio) regarding mortars for restoration purposes and in order to gain more rigid mortar with fewer cracks (Beck & Al-Mukhtar, 2008; Pinto et al., 2017), thus, (1:5.6, i.e., 15%) had been used in this study. The hydrated lime powder is classified as calcium lime and designated as CL90 according to European standard (EN 459-1, 2001), and supplied by Saint-Astier, France based company. Two sizes of aggregate stone powder were selected in order to identify presence and absence of fine materials on adhesive strength properties, these sizes expressed by stone powder size pass sieve NO.2mm, and another size ranged (2mm-0.16mm). However, the dose of Melment F10 was (2.5% by weight of lime amount). The moisture content of the mortars was controlled by keeping the flow table extension (15±1cm) according to (ASTM C230/C230M-08) (Astm, 2008).

Table 1. Codes and Compositions of Mortar used

Mortar	Mortar Compositions
Code	
B/F	1:5.6:Moisture=46 %, aggregate stone powder pass sieve NO.2mm
B/W	1:5.6:Moisture=44 %, aggregate stone powder pass sieveNO.2mm and retained om sieve NO.0.16mm
B/F/M	1:5.6: Moisture=35 %:Melment(2.5% of lime amount), aggregate stone powder pass sieve NO.2mm

#### 2.1.2. Substrates

Two types of calcareous substrates materials have been used in this study: Tuffeau stone and Siporex unit building. Both Tuffeau and Siporex materials represent the commonly used construction material in old castles/houses and modern buildings respectively in Loire valley in France (Andolsun, 2006; Beck et al., 2003). The used Tuffeau is a yellowish white porous sedimentary calcareous limestone extracted from a quarry of Saint-Cyr-en-Bourg, this stone could be described as a low-density building material with a high total porosity and is easily workable (Beck & Al-Mukhtar, 2010b). While Siporex is a lightweight insulated autoclaved aerated concrete, which is mix of calcareous materials such as cement-siliceous quartz sand fine materials-calcined gypsum-lime-aluminium powder and water (Guid, 2015; Mathey & Rossiter, 1988).

### 2.2. Characterization of the Tuffeau, Siporex and hydrated aerial lime

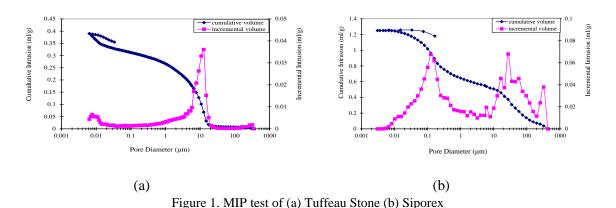
Table 2 and 3 present the physical, mineralogical composition by X-ray diffraction test, and chemical characteristics of Tuffeau, Siporex, and hydrated aerial lime by coupled plasma-optical emission spectrometry(ICP-OES) test, while Figure (1) show the pore size distribution of Tuffeau stone and Siporex by Mercury Intrusion Porosimetry test (MIP). The principal minerals present in Tufeau stone are calcite (CaCO<sub>3</sub>) and Silica (SiO<sub>2</sub>) in the form of Opal and quartz, with mica and clays minerals. Tuffeau stone has wide pore size distribution (from 6 nm to 20 µm) with an average pore diameter of 5µm (Beck & Al-Mukhtar, 2010a) (Figure 1a). In this context, the main minerals of Siporex are Tobermorite group of calcium silicate hydrates (CSH), in addition to Ettringite mineral (Andolsun, 2006). The pores sizes present in Siporex structure are ranged (5nm to 400µm) with medium pore diameter of 35µm, these pores can be divided according to their size in to (18.5% micro pores<0.1µm) and (81.5% meso-macro pores 0.1-400 um) (Figure 1b). The hydrated aerial lime was made from a pure limestone (i.e.95% of CaCO<sub>3</sub>), with portlandite (Ca(OH)<sub>2</sub>) content equal to 92.2%. From another side, the pozzolanic reaction in (B/F, B/F/M) mortars was found to be possible between hydrated lime and clay minerals/silica fine grains present in Tuffeau stone powder by an amount of 20% of lime weight (Beck & Al-Mukhtar, 2008), while this reaction is probably less efficiently in (B/W) mortar due to absence most of the fine materials from stone powder composition.

Table 2. Physical Properties of Hydrated lime, Tuffeau, and Siporex

Parameter	Tuffeau stone	Siporex	Hydrated aerial lime
Mineralogical compositions	Calcite, Opal CT	C <sub>3</sub> SH <sub>2</sub> ,Tobermorite,	Portlandit content
	Quartz,Clay and Mica	Ettringite, CSH	[ Ca(OH) <sub>2</sub> =92.2%
Skeletal density(g/cm <sup>3</sup> )	2.62	2.42	
Bulk dry density (g/cm <sup>3</sup> )	1.204	0.678	
Porosity(%) by hydrostatic method [8]	54	72	

Table 3. Chemical Compositions of Hydrated lime, Tuffeau, and Siporex

	$SiO_2$	CaO	$Al_2O_3$	$Fe_2O_3$	MgO	Na <sub>2</sub> O	$K_2O$	$P_2O_5$	$TiO_2$	LOI*
	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Hydrated aerial lime	0.24	73.7	0.11	0.21	0.35	0.00	0.01	0.00	0.00	24.81
Tuffeau stone	41.41	29.75	2.14	0.87	0.5	0.05	0.64	0.00	0.16	24.00
Siporex	32-58	18-36	2.4	2	< 2	Alkali	s=< 1%	Other	s=1-4 %	Weight loss=8-12%



## 2.3. Mortars expansion detection

B/F, B/W, and B/F/M mortars have been moulded inside consolidation ring (i.e. diameter=7cm, thickness=2cm) without compaction, and stored inside closed sacks for 7 days in controlled room conditions (i.e.  $20~^{\circ}\text{C}\pm~2$ , Relative Humidity (RH)= $50\%\pm5$ ) to firm up (Beck & Al-Mukhtar, 2008). Later, they transferred to the consolidation apparatus and simultaneous synchronous measuring of both axial free expansion and absorbed water have been done until all readings get stable.

## 2.4. Adhesive Strength

## 2.4.1. Samples Preparations

The mortars adopted in this study have been placed on the surface of Tuffeaue/Siporex cylindrical samples (diameter=5cm, height=3.5 cm) without pressure, with (3mm) thickness in order to minimize  $CO_2$  diffusion path and accelerate carbonation reaction front from mortars exposed surface, toward interlocking spots with Tuffeaue/Siporex substrates (Çizer, 2004; Cizer et al., 2012), see Figure (2a). These mortars/substrates composite samples stored inside sealed sacks for 7 days in controlled room conditions (i.e.,  $20 \, ^{\circ}C\pm 2$ ,  $RH=50\%\pm 5$ ) in order to firm up the mortars pastes shape (Beck & Al-Mukhtar, 2008). Then the samples unpacked and kept in incubators at RH: 12, 66, and 98% at temperature ( $20 \, ^{\circ}C\pm 2$ ), for curing periods of: 7, 28, and 90 days. In this context, a mortars disks sets with thickness=3mm, and diameter=5cm, have been made and stored with the same manner as mortars/substrates samples, then transferred to incubators (see Figure 2b), in order to identify inner mortars moisture content inside the incubators at the end of the curing periods (Ali et al., 2011; Harith Al-Hadedy Suhail Khattab, 2017).

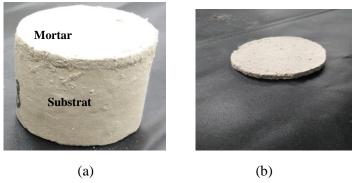


Figure 2. Adhesive Test Samples: (a) Mortar/Substrate composite, (b) Mortar Disk

#### 2.4.2. Adhesive test

After cured the mortars/substrates composite samples inside the incubators, these samples have been tested to identify their adhesive strength using Proceq-DY-215 apparatus, with tensile loading rate 0.005Mpa/sec (EN1015-12, 2001). Epoxy glue is used to fix the two opposite surfaces (i.e., mortar with the test disc/substrates with the base), see Figure (3). The results in this study are the average of four multiple tests with value variations of  $\pm 0.01$ 

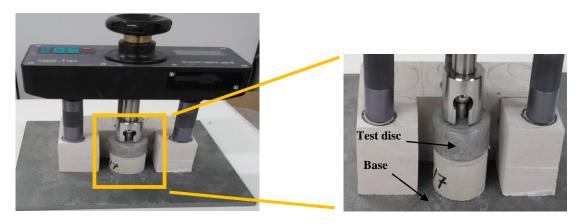


Figure 3. Adhesive Test Apparatus with Glued Surfaces

#### 3. RESULTS AND DISCUSSION

The final moisture contents of B/F, B/W, and B/F/M mortars after curing condition were :44.32, 42.9, and 34.2 %, with total densities: 1.73,1.65, and 1.82 gm/cm³ respectively. B/F mortar could be considered as reference mortar for comparison with the other (i.e., B/W and B/F/M), because of its well graded and presence of pozzolanic/carbonation reactions. In this context, B/W mortar can be describe as poorly graded mortar because of constrain its stone powder grains by size (2-0.16mm), in turn, this gain the mortar more open structure, and possibility of decrement its pozzolanic and carbonation activity (El-Turki et al., 2009; Stefanidou & Papayianni, 2005). On the other hand, B/F/M mortar, showed higher density than B/F mortar, this could be attributed to Melment additive role in reducing the pores amount generated within mortar structure (KOŤÁTKOVÁ et al., 2018).

## 3.1. Mortars Expansion

Figures (4 a and b) show the axial expansion/absorbed water properties of B/F, B/W, and B/F/M mortars. The axial expansion spent (60) days to get stable, and showed highest value of B/F by about (0.3%), followed by B/W with a value (0.225%), and then (0.15%) for B/F/M. The reduction in B/W expandability, probably be attributed to B/W open structure, which provide adequate space for CaCO<sub>3</sub> formation, in addition to the positive role of coarse grain in B/W structure volume stability(Çizer, 2004; Costigan & Pavía, 2010; R. M. Lawrence et al., 2007). From other side, the presence of Melment additive within B/F/M compounds, played an important role in mortar expansion reduction compared to B/F by (50%) because of Melment decrement of mortar porosity and capillary absorption (Fredlund & Rahardjo, 1993; KOŤÁTKOVÁ et al., 2018). Generally, the limited expansion of the used mortars could be considered to have little effect on their adhesive strength.

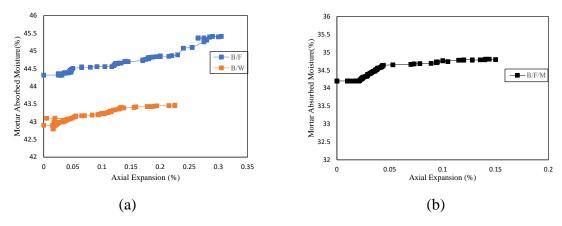


Figure 4. Axial Expansion Behaviour of: (a) B/F, B/W, (b) B/F/M

## 3.2. Mortars Adhesive Strength

When B/F, B/W, and B/F/M mortars placed on Tuffeau /Siporex substrates surface, their components will be spread on and within substrates pores. Later, after excess mortars moisture evaporate, reactions will be create between dissolved Ca(OH)<sub>2</sub> presence in mortars and :(I) silica/alumina of both mortar fine aggregate, and substrates compositions (i.e. pozzolanic reaction), (II) diffuses CO<sub>2</sub> through mortars (i.e. carbonation process). Therefore, factors such as: (i)ambient RH (ii) the quality of substrate material and its ability to react with mortar (iii) degree of Substrate Surface Roughness (SSR) (Figure (5)), will control adhesive strength between used mortars and substrates.

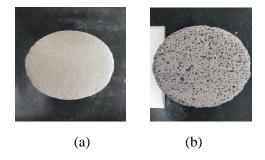


Figure 5. Surface Roughness of Substrates SSR: (a) Tuffeau (b) Siporex

Figures 6 and 7 show the adhesive strength between B/F, B/W, B/F/M mortars and Tuffeau/Siporex substrates respectively, at curing periods 7,28, and 90 days, under RH=12,66, and 98%. In general, the adhesive strength over all curing periods showed high values for B/F, followed by B/F/M, and then B/W. Also the adhesive strength characterized by its relatively small values at RH=12%, and its noticeable increase at 66%, and then its decrease at 98%, where this variation was evident in B/F, B/F/M mortars, but had little effect in B/W mortar.

## 3.2.1. Adhesive Strength of Mortars at RH=12%

Table 4, shows the inner moisture content of B/F, B/W, B/F/M mortars at the adopted curing periods and relative humidity conditions, while Figures (6 and 7) show the adhesive strength of these mortars with Tuffeau/ Siporex substrates. In case of B/F mortar, its inner moisture content at 7 days curing period with all relative humidity values may probably be enough for pozzolanic reaction accompanied with slow CO<sub>2</sub> diffusion within B/F structure for carbonation activity (Cizer et al., 2012; R. M. H. Lawrence, 2006), this produced low adhesive strength with Tuffeau and Siporex substrates (see Figures 6a,7a). With the reduction

of B/F inner mortar moisture along (28 and 90 days), CO<sub>2</sub> diffusion will be more active, and consequently increase mortar adhesive with both substrates. Here, it is worth mentioned that B/F adhesive with Siporex at 90 days is 18% more than its adhesive with Tuffeau, because of Siporex high SSR (see Figure 8a).

Mortar Moisture Content (%) Mortar Code RH=12% RH=66% RH=98% 28 days 90 days 7 days 28 days 90 days 7 days 28 days 90 days 7 days B/F 13.69 6.08 23.56 29.11 22.87 13.12 30.9 13.74 36.76 B/W 18.15 13.44 12.81 4.3 28.75 22.55 12.2 34.84 27.82 B/F/M 8.76 27.05 9.24 3.3 26.45 14.46 10.78 22.91 17.28

Table 4. Inner mortars moisture content in term of RH and curing periods

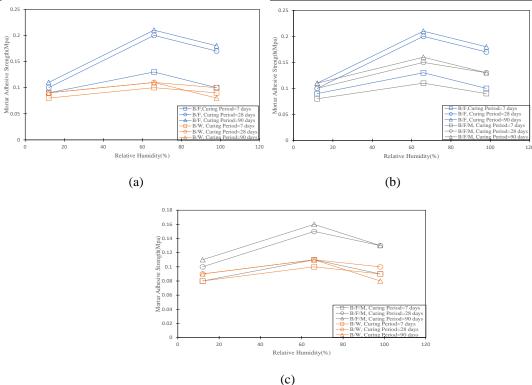


Figure 6. Adhesive Strength of B/F, B/W, and B/F/M with Tuffeau Stone Substrate

In comparison between B/W and B/F mortars, adhesive strength of B/W with Tuffeau substrate decreased by (18%) at 90 days curing period, this come due to the fine materials excluding from B/W composition, which decreased effectiveness of pozzolanic reaction and limited its adhesive strength value with the carbonation reaction (Pathanatecha, 2019; Stefanidou & Papayianni, 2005), in addition to the weak penetration of B/W mortar through Tuffeau substrate pore size ( $\leq$ 0.16mm), leaving these pores almost empty. From other side, the large pore size of Siporex substrate surface allows easy B/W mortar to penetration, which developed its adhesive strength by (22%) for 90 days curing period compared to Tuffeau substrate (Figures 7a, 8b).

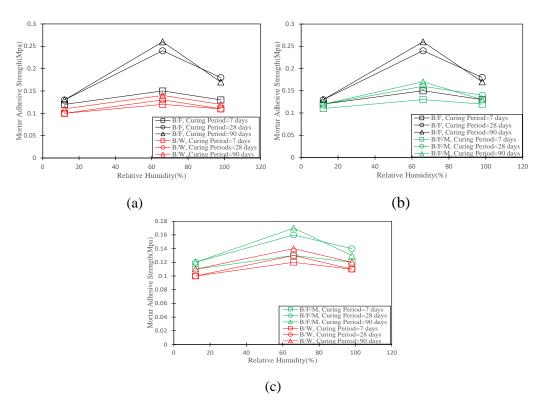


Figure 7. Adhesive Strength of B/F, B/W, and B/F/M with Siporex Substrate

In case of B/F/M, it can be said that the adhesive values of B/F/M with tuffeau/siporex substrates are close to those of B/F, and greater than the others in B/W mortar (Figures 6b-6c,7b-7c), this could be attributed to the decrement of both absorbed moisture and pore amount inside B/F/M structure due to Melment addition, and its reflection on carbonation/pozzolanic reactions .It is worth noting that the Siporex SSR, played an important role in increment B/F/M adhesion strength by an about (9% for 90 days curing period) compared with Tuffeau (Figure 8c) .

#### 3.2.2. Adhesive Strength of Mortars at RH=66%

Here, the moisture content of B/F, B/W and B/F/M, will be in an ideal state for pozzolanic activity, and provide (100%) of mortars pore surface for CO<sub>2</sub> diffusion to catalyse carbonation process (Stoian et al., 2015), later, these reactions produced different values of adhesive strength with Tuffeau substrate depending on mortar compositions, and given high values in B/F mortar, then decreased by 23.8% in B/F/M mortar, followed by reduction amount of 47.6% in case of B/W mortar at 90 days curing period(Figures 6a, 6b,6c). In this context, a similar behaviour was observed for B/F, B/W, and B/F/M mortars with Siporex substrate (Figures 7a,7b,7c), but with higher adhesive amount by about 23.8%, 27.3, and 6.25% respectively at 90 days curing period compared with Tuffeau substrate (Figures 8a, 8b,8c). This latter behaviour could be attributed to the SSR of Siporex.

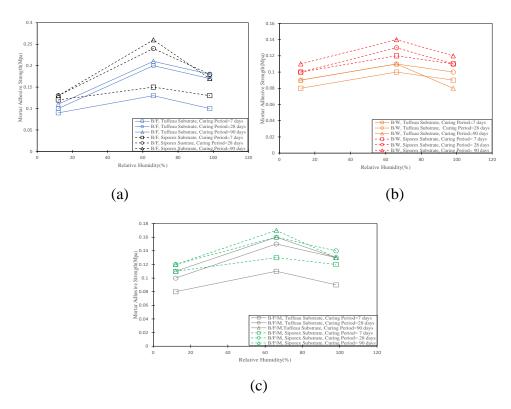


Figure 8. Adhesive Strength of Mortars with Tuffeau/Siporex Substrates

#### 3.2.3. Adhesive Strength of Mortars at RH=98%

In this high RH, 50% of mortars pore surface might be available for  $CO_2$  diffusion to catalyse carbonation process in addition to pozzolanic reaction, also there is probability of concentration the high moisture within empty or semi-empty spots between B/F, B/W,B/F/M and Tuffeau/Siporex substrates (Stefanidou & Papayianni, 2005), all these factors reduced the adhesive strength between these mortars compared to their adhesive ability at RH=66%, by about 14.3%, 27.3%, and 18.75 respectively with Tuffeau , and by 34.6%, 14.28%, and 23.5% respectively with Siporex at 90 days curing periods (Figures 8a,8b,and 8c). It is necessary to note that the reduction in adhesive strength was evident for B/W mortar with Tuffeau , more than with Siporex, this could be related to the incomplete penetration of B/W mortar within Tuffeau substrate pores ( $\leq 0.16$ mm).

#### 4. CONCLUSIONS

After viewing materials, technique used, and discussed the results obtained, the following points may be concluded:

1- The expansion of B/F, B/W, and B/F/M used has little effect on their adhesion strength with tuffeau and siporex substrates, due to their limited axial expansion obtained. The addition of Melment F10 to B/F/M mortar, had a significant role in reducing its expandability by 50% compared to B/F basic mortar, while the exclusion of the fine materials from aggregate stone powder in B/W mortar, also reduced its axial expansion but with lesser amount compared to B/F/M mortar.

- 2- The variation in the relative humidity used (i.e. 12, 66, and 98%) had a limited effect on the adhesion strength of B/W mortar along the curing periods (7, 28, and 90days), while had a noticeable effect for the rest of the used mortars (i.e. B/F, B/F/M). The relative humidity (RH=66%) can be considered the perfect humidity that will provide optimum moisture content and CO<sub>2</sub> diffusion condition through mortars structure for carbonation and pozzolanic reactions. While the relative humidity values (RH=12, 98%) had a negative impact on mortars adhesion strength due to the lack of moisture content required to complete these reactions, or the excessive moisture content that hinders CO2 diffusion, and concentrate inside empty pores in substrates- mortars interlock zone respectively.
- 3- The degree of substrate surface roughness showed an important impact on adhesive strength of all mortars. The use of Siporex as substrate recorded a clear advantage compared to Tuffeau for all used mortars under relative humidity and curing periods.

#### **ACKNOWLEDGMENT**

The author Harith E. Ali, expresses his gratitude to the Iraqi ministry of higher education and scientific research/Northern Technical University, and to the laboratory of Mechanics Gabriel Lamé/Orleans university/ France, for their support provided to complete this study.

#### REFERENCES

- (PCA). (1994). Portland Cement Association.
- 459-1, E. (2001). Chaux de construction-Partie 1.
- Ali, H. E., Khattab, S. A., Beck, K., & Al-Mukhtar, M. (2011). Salt weathering in the Al-Namrud monuments in Iraq: characterization of historical stone and fresh stone treated with accelerated decay tests. 2nd International Conference on Salt Weathering on Building and Stone Sculpture (SWBSS), 19–22.
- Andolsun, S. (2006). A study on material properties of autoclaved aerated concrete (AAC) and its complementary wall elements: their compatibility in comtemporary and historical wall sections. Middle East Technical University.
- Astm, C. (2008). 230, Standard specification for flow table for use in tests of hydraulic cement. West Conshohocken, PA: ASTM International.
- Beck, K., & Al-Mukhtar, M. (2008). Formulation and characterization of an appropriate lime-based mortar for use with a porous limestone. *Environmental Geology*, *56*(3–4), 715–727.
- Beck, K., & Al-Mukhtar, M. (2010a). Evaluation of the compatibility of building limestones from salt crystallization experiments. *Geological Society, London, Special Publications*, 333(1), 111–118.
- Beck, K., & Al-Mukhtar, M. (2010b). Weathering effects in an urban environment: a case study of tuffeau, a French porous limestone. *Geological Society, London, Special Publications*, 331(1), 103–111.
- Beck, K., Al-Mukhtar, M., Rozenbaum, O., & Rautureau, M. (2003). Characterization, water transfer properties and deterioration in tuffeau: building material in the Loire valley—France. *Building and Environment*, 38(9–10), 1151–1162.
- Beruto, D. T., Barberis, F., & Botter, R. (2005). Calcium carbonate binding mechanisms in the setting of calcium and calcium–magnesium putty-limes. *Journal of Cultural Heritage*, 6(3), 253–260.

- BIA. (2003). The Brick Industry Association (BIA).
- Chang, T., Sproat, E. A., Lai, Y.-H., Shephard, N. E., & Dillard, D. A. (1997). A test method for accelerated humidity conditioning and estimation of adhesive bond durability. *The Journal of Adhesion*, 60(1–4), 153–162.
- Çizer, Ö. (2004). *Investigation of Lime Mortar Characteristics for the conservation of the Ottoman baths in Seferihisar-Urla Region*. Izmir Institute of Technology (Turkey).
- Cizer, Ö., Rodriguez-Navarro, C., Ruiz-Agudo, E., Elsen, J., Van Gemert, D., & Van Balen, K. (2012). Phase and morphology evolution of calcium carbonate precipitated by carbonation of hydrated lime. *Journal of Materials Science*, 47(16), 6151–6165.
- Cizer, Ö., Van Balen, K., & Van Gemert, D. (2010). Competition between hydration and carbonation in hydraulic lime and lime-pozzolana mortars. *Advanced Materials Research*, *133*, 241–246.
- Costigan, A., & Pavía, S. (2010). *Influence of mortar water content and workability on the mechanical behaviour of lime mortar masonry.*
- Costigan, A., & Pavía, S. (2012). Influence of the mechanical properties of lime mortar on the strength of brick masonry. In *Historic Mortars: Characterisation, Assessment and Repair* (pp. 359–372). Springer.
- El-Turki, A., Ball, R. J., & Allen, G. C. (2007). The influence of relative humidity on structural and chemical changes during carbonation of hydraulic lime. *Cement and Concrete Research*, *37*(8), 1233–1240.
- El-Turki, A., Ball, R. J., Holmes, S., Allen, W. J., & Allen, G. C. (2010). Environmental cycling and laboratory testing to evaluate the significance of moisture control for lime mortars. *Construction and Building Materials*, 24(8), 1392–1397.
- El-Turki, A., Carter, M. A., Wilson, M. A., Ball, R. J., & Allen, G. C. (2009). A microbalance study of the effects of hydraulicity and sand grain size on carbonation of lime and cement. *Construction and Building Materials*, 23(3), 1423–1428.
- EN1015-12. (2001). European standard.
- Fredlund, D. G., & Rahardjo, H. (1993). Soil mechanics for unsaturated soils. John Wiley & Sons.
- Guid, T. (2015). No Title.
- Hansen, E., Doehne, E., Fidler, J., Larson, J., Martin, B., Matteini, M., Rodriguez-Navarro, C., Pardo, E. S., Price, C., & de Tagle, A. (2003). A review of selected inorganic consolidants and protective treatments for porous calcareous materials. *Studies in Conservation*, 48(sup1), 13–25.
- Harith Al-Hadedy Suhail Khattab. (2017). *deterioration Mechanism of Stones in Iraqi Historical Buildings*. Noor Publishing. https://www.noor-publishing.com/catalog/details/store/ae/book/978-3-330-97678-8/deterioration-mechanism-of-stones-in-iraqi-historical-buildings
- Hendry, A. W., & Khalaf, F. M. (2017). Masonry wall construction. CRC Press.
- Kirk, V., Gabriel, F., Isabella, P., Mathieu, B., Magdalena, B., & Gaurav, S. (2015). *Direct Carbonation of Ca (OH) 2 Using Liquid and Supercritical CO2: Implications for Carbon-Neutral Cementation*.
- KOŤÁTKOVÁ, J., HLAVÁČ, Z., ROSNECKÝ, V. Í. T., Mohyla, R., & JANSA, J. (2018). The effect of superplasticizers on the properties of gamma irradiated cement pastes. *Ceramics–Silikáty*, 62(3), 306–310.
- Lanas, J., & Alvarez-Galindo, J. I. (2003). Masonry repair lime-based mortars: factors affecting the

- mechanical behavior. Cement and Concrete Research, 33(11), 1867–1876.
- Lawrence, R. M. H. (2006). A study of carbonation in non-hydraulic lime mortars.
- Lawrence, R. M., Mays, T. J., Rigby, S. P., Walker, P., & D'Ayala, D. (2007). Effects of carbonation on the pore structure of non-hydraulic lime mortars. *Cement and Concrete Research*, *37*(7), 1059–1069.
- Mathey, R. G., & Rossiter, W. J. (1988). A review of autoclaved aerated concrete products.
- Morgan, R. C., & Ball, R. J. (2013). Environmental (wet and dry) cycling of hydraulic lime mortars. *Journal of the Building Limes Forum*, 20, 21–31.
- Ngoma, A. M. K. (2009). Characterisation and consolidation of historical lime mortars in cultural heritage buildings and associated structures in East Africa. KTH.
- Pathanatecha, W. (2019). A study of various parameters affecting adhesion of coatings to metal substrates.
- Pavía, S., & Hanley, R. (2010). Flexural bond strength of natural hydraulic lime mortar and clay brick. *Materials and Structures*, 43, 913–922.
- Pavía, S., & Toomey, B. (2008). Influence of the aggregate quality on the physical properties of natural feebly-hydraulic lime mortars. *Materials and Structures*, 41, 559–569.
- Pinto, A. P. F., Gomes, A., Silva, B., Candeias, A., & do Vale, F. (2017). Influence of water reducers on the early age properties of aerial lime mortars. *3rd International Conference on Protection of Historical Constructions*, 337–338.
- Sheet, T. (2002). Superplasticizers in Powder Form for Cementitious and Calcium Sulphate Based Dry Mix Mortars.
- Stefanidou, M., & Papayianni, I. (2005). The role of aggregates on the structure and properties of lime mortars. *Cement and Concrete Composites*, 27(9–10), 914–919.
- Stoian, J., Oey, T., Bullard, J. W., Huang, J., Kumar, A., Balonis, M., Terrill, J., Neithalath, N., & Sant, G. (2015). New insights into the prehydration of cement and its mitigation. *Cement and Concrete Research*, 70, 94–103.
- Van Balen, K. (2005). Carbonation reaction of lime, kinetics at ambient temperature. *Cement and Concrete Research*, *35*(4), 647–657.

## **AUTHOR BIBLIOGRAPHY**



**Harith E. Ali:** PhD. Civil engineering/Soil and Rock Foundation (Geotechnique). Northern Technical University(NTU), Engineering Technical College, Mosul, Iraq, email: <a href="mailto:hbz alhaded@yahoo.com">hbz alhaded@yahoo.com</a> . Researcher in soil /rock mechanics foundation and stone deterioration /conservation of historical buildings.



**Xavier Brunetaud**: PhD. professer Civil/Structural/ Materials Engineering, Université d'Orléans, LaMé Laboratoire de Mécanique Gabriel Lame, Polytech Orleans-Université d'Orleans, France, email: <a href="mailto:xavier.brunetaud@univ-orleans.fr">xavier.brunetaud@univ-orleans.fr</a>. Researcher in concrete durability-civil engineering materials- heritage conservation and 3D modelling.



**Kevin Beck:** PhD. Assistant Professor Civil/Materials Engineering, Université d'Orléans, Université de Tours, INSA-CVL Laboratoire de mécanique Gabriel Lamé LaMé – EA7494, France, email: <a href="mailto:kevin.beck@univ-orleans.fr">kevin.beck@univ-orleans.fr</a>. Researcher in conservation of historical heritage building-physico/chemical and hydromechanical and characterization of porouse materials



**Naima Belayachi:** PhD. Assistant Professor Civil/Mechanics/Materials Engineering Laboratoire de Mécanique Gabriel Lame, Polytech Orleans-Université d'Orleans, France, email: <a href="maima.belayachi-belaiche@univ-orleans.fr">naima.belayachi-belaiche@univ-orleans.fr</a>. Researcher in civil-mechanics-materials-building thermal-and aeraulic.



**Malek Balawi**: MSc. Civil Engineering, PhD. Student Civil/Materials Engineering. Laboratoire de Mécanique Gabriel Lame, Polytech Orleans-Université d'Orleans, France, email: <a href="maleklabs@hotmail.com">maleklabs@hotmail.com</a>. Research interest in civil-materials and conservation of historical buildings.