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► **To cite this version:**

B. Diop, Yannick Mélinge, Laurent Molez, Raoul Jauberthie, A. Bouguerra. Durability Of Mortars With Natural Fillers In Aggressive Environnement. Structural Faults and Repair 2008, Jun 2008, Edinburgh, United Kingdom. hal-01421244

HAL Id: hal-01421244

<https://hal.science/hal-01421244>

Submitted on 21 Dec 2016

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DURABILITY OF MORTARS WITH NATURAL FILLERS IN AGGRESSIVE ENVIRONNEMENT

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KEYWORDS

durability, SEM, X-ray diffraction, sulphatic solutions, volcanic tuffs.

ABSTRACT

Huge quantities of volcanic tuffs are found in Mako area (western Senegal). The high content of SiO₂ is responsible of the pozzolanic properties of the tuffs when mixed to Portland cement. However, tuffs alterations could limit the pozzolanic effect: for example, the formation of clay decreases this effect but decrease also the porosity.

The aim of this study is to highlight the benefits of this admixture when mortars where exposed in different sulphatic solutions. In sulphuric acid, specimen surfaces are covered with a superficial formation of gypsum and with amonium sulphate solution only few needles of gypsum appears. As it is shown by SEM observations and X-ray diffraction, deeper alterations decreases when cement is partially substituted with tuff.

INTRODUCTION

Volcanic tuffs, like amorphous silica or silica with poorly crystallised phases (silica fume, fly ash, natural materials: diatomite), exhibit pozzolanic properties in mortars when cement is partially substituted (Papadakis & Tsimas, 2002). In a similar study, volcanic tuffs from Galatean Province of Central Turkey where used by Turkmenoglu et al, 2002. Their investigations with petrographic observations highlight the influence of different parameters (proportion of glass, clay minerals) in the pozzolanic effects. Workability was modified when cement is partially substituted with tuff. Investigations of Uzal & Turanli, 2003 showed the influence of grinding times, particle size distribution and the super-plasticizer influence to maintain good workability. In this case, the compressive strength is lowered when tuff is added and alkali silica expansion is reduced. Compression and flexural strengths of concrete containing tuff and cured in hot weather condition decrease in comparison with normal concrete (Ujhelyi et al, 1991).

The use of tuffs from Mako Province in western Sénégal was tested in our laboratory by Coatanlem et al, 2004. However, tuff alterations could limit the pozzolanic effect: for example, the formation of clay decreases this effect. This paper highlights the deteriorations of mortars with tuffs subjected to sulphate environments. The action of sulphuric acid and ammonium sulphate solutions with 3 concentrations is considered. Similarly, normal mortars where tested by Jauberthie & Rendell, 2003 and by Rendell et al, 2002.

SAMPLES PREPARATION

Mixture compounds

The samples used in the test program were 4cm x 4cm x 16cm prisms based on a standard cement mortar mix. The mortar used in the test is a normal mortar conforming to EN196. The aggregate type being standard sand conforming to ISO 679 and the method of manufacture was carried out in accordance to

EN 196. The mixture details were the following: aggregate-to-cement ratio was 3.0 and the water-to-cement ratio was 0.5. The cement type used is a normal portland cement – CEM 1 52.5 CP2 manufactured in SAINT-PIERRE-LA-COUR (France). An analysis of the cement type is summarised in Table 1. In the second series of samples, the volcanic tuff, ground below 50µm, replaced partially the cement. An analysis of the volcanic tuff is summarised in Table 2. SEM examinations of this tuff are shown in figure 1. The X-Ray diffraction analysis (figure 2) reveals the presence of illite and kaolin: 2 clay minerals, as well as quartz and goethite.

Table 1. Oxide analysis of the cement used in the tests

Ins	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO
0.26	20.15	5.18	2.76	65.13	0.69
K ₂ O	Na ₂ O	SO ₃	P.F.	CaOfree	
0.99	0.17	2.85	1.51	1.31	

Table 2. Oxide analysis of the tuff used in the tests

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	Na ₂ O
82.54	7.18	9.08	0.27	0.58	0.34

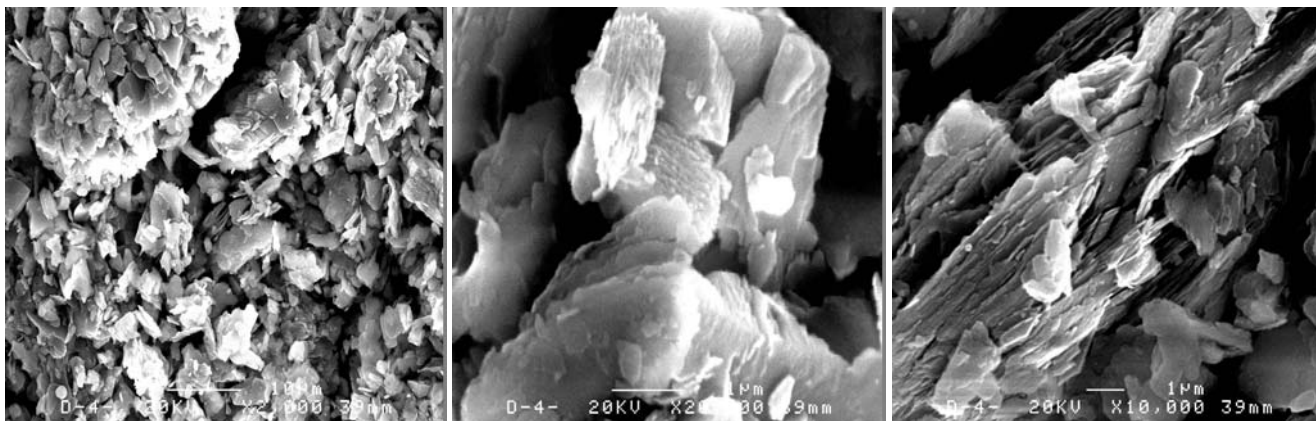


Fig.1. SEM micrographs of the volcanic tuff

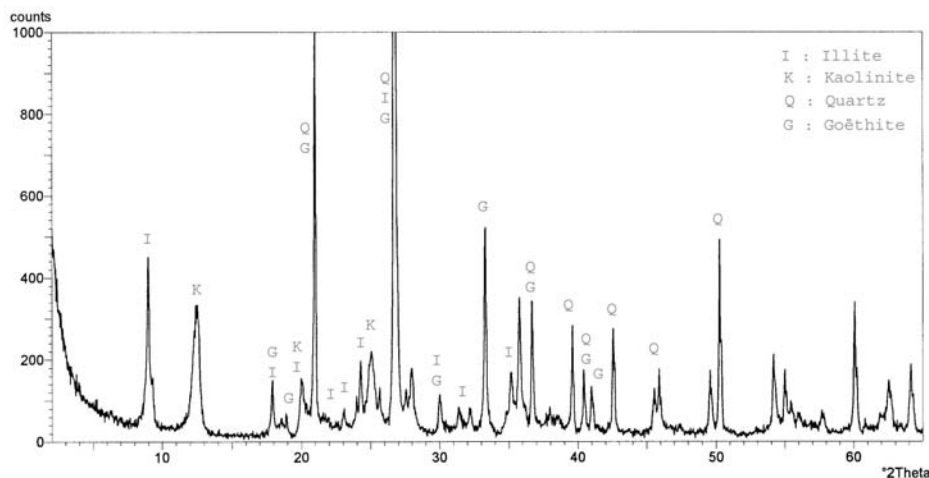


Fig.2. XRD pattern of tuff (λ k_{α} Cu , filtered)

In the investigation, the two series of samples were produced and subjected to identical regime of exposure. Mortar composition, flexural and compression strengths of samples MO (normal mortar) and MT (mortar with tuff) measured after 2 years (stored in an air conditioned room 20°C, 50%RH) are summarised in Table 3. A pozzolanic effect of tuff is expected. But, our tuff is altered and which explain a content of clay (illite and kaolinite). The tuff content increase induces lower strength due to the competition between pozzolanic effect and fillers effect.

Exposure tests in sulphate environment

After 2 years storage in an air conditioned room (20°C, 50%RH), the following stage in the program consisted in the comparison of the characteristics of the mortars with and without tuff throughout 2 months exposure in sulphate solutions. Six curing environments were used in the test:

- Stored in sulphuric acid, 0.1M, 0.25M, 0.5M,
- Stored in ammonium sulphate, 0.1M, 0.25M, and 0.5M.

Table 3. Mortar properties of reference samples after 2 years

	Composition	by weight (g)	Flexural strength (MPa)	Compressive strength (MPa)
MO	Sand	2700	11.2	66.5
	Cement	900		
	Tuff	0		
	Water	450		
MT	Sand	2700	7.2	42.5
	Cement	630		
	Tuff	270		
	Water	450		

TESTS RESULTS

Sulphuric acid

It was noted that during the test period, the samples in the sulphuric acid solution were covered in a soft white deposit. The degree of samples surface deterioration was very evident (figure 3) as it can be seen in table 4 were the size changes of the samples are reported. The section of normal mortar decrease largely when the sample is in the acidic solution. When the concentration increases, the size decreases.

Ammonium Sulphate

Lee (1970) suggests that ammonium sulphate is the most destructive of the sulphate salts: a 5% solution is able to causing 3.8% linear expansion in a cement mortar after 12 weeks. He proposes that the expansion may be due to the formation of an expansive double salt $\text{CaSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot \text{H}_2\text{O}$. Considering the general reaction between an ammonium salt and lime it can be seen that one would expect the formation of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ gypsum and a release of ammoniac; this gas liberation being common to most ammonium salts. It has been observed that during the period of immersion of a PC CEM I mortar in ammonium sulphate solution there is the formation of needle like crystals over the surface of the concrete, these have been identified as gypsum (Rendell & Jauberthie, 1999; Rendell et al, 2000)

Samples stored in ammonium sulphate present some needle like crystal at the surface (figure 4) and the samples sizes were not significantly modified (Table 5).

Analysis of the product resulting from the attack

After the acidic attack, the solid residue is separated by filtration. After dehydration at 25°C, the X-Ray diffraction analysis shows that the attack by the sulphuric acid transforms the hardened cement paste into gypsum similarly with normal mortar (figure 5) and tuff + cement mortar (figure 6). Naturally, the quartz is found in the 2 samples, and clay minerals traces (illite and kaolin) were detected. Those residues have to be dried at a temperature lower than 40°C to avoid the transformation of gypsum into hemi hydrate.

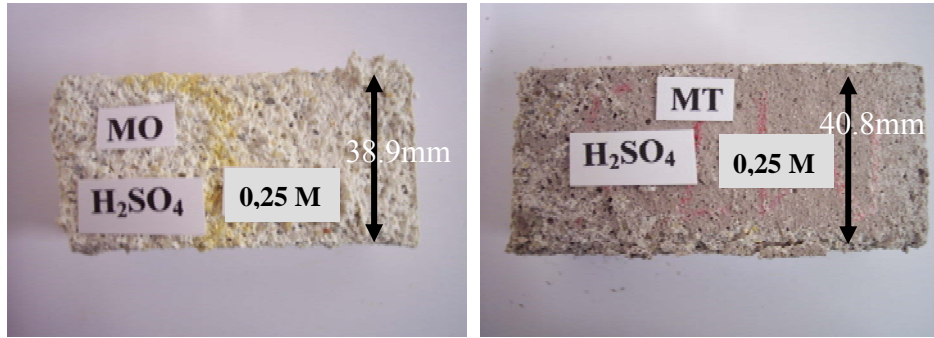


Fig.3. Samples stored in H₂SO₄ (0.25M)



Fig.4. Samples stored in (NH₄)₂SO₄ (0.25M)

Table 4. Evolution of the section of the samples v/s solutions (H₂SO₄)

	Stored solution	Size (mm)
MO	H ₂ SO ₄ 0.1 M	40.5
	H ₂ SO ₄ 0.25M	38.9
	H ₂ SO ₄ 0.5 M	36.5
MT	H ₂ SO ₄ 0.1 M	40.5
	H ₂ SO ₄ 0.25M	40.8
	H ₂ SO ₄ 0.5 M	41.8

Table 5. Evolution of the section of the samples v/s solutions ((NH₄)₂SO₄)

	Stored solution	Size (mm)
MO	(NH ₄) ₂ SO ₄ 0.1 M	40.1
	(NH ₄) ₂ SO ₄ 0.25 M	40.2
	(NH ₄) ₂ SO ₄ 0.5 M	40.4
MT	(NH ₄) ₂ SO ₄ 0.1 M	40.1
	(NH ₄) ₂ SO ₄ 0.25 M	40.1
	(NH ₄) ₂ SO ₄ 0.5 M	40.1

SEM observations

SEM observations (figure 7) of the sample of normal mortar stored in sulfuric acid (H_2SO_4 0.25M) confirm the X-Ray diffraction results. A formation of scale (fig. 7 detail (0)) on the sample surfaces is noted. Such scale induces a penetration of acidic solution deeper in the section. The falling of scales explains the section decreases observed in figure 3. Large prismatic crystals of gypsum cover the surface and fill the pores. Behind this layer, an ettringite front appears. Fine needle like crystals are present to 1.5 mm depth. Portlandite layer are not observed in this area. At 2 mm depth, portlandite is slightly altered (fig. 7, detail (5)). Deeper in, traditional hydrated mineral phases of classic Portland Cement are observed.

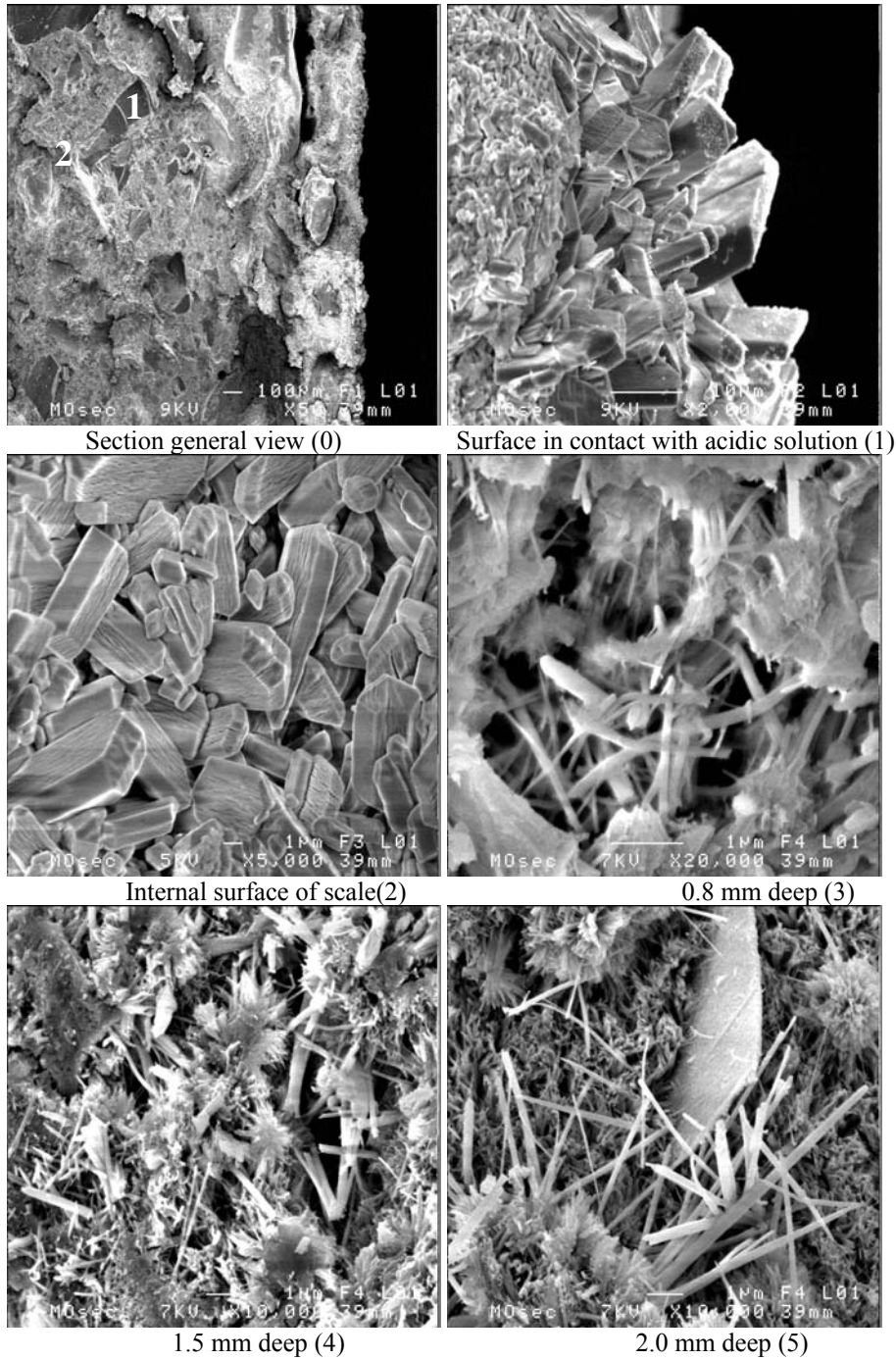


Fig.7. MO Samples stored in H_2SO_4 (0.25 M)

SEM observations of the sample of mortar containing tuff and stored in sulfuric acid (H_2SO_4 0.25M) are presented on figure 8. Same gypsum mineralization is observed at the sample surface. However, no scale appears (figure 8 detail (3)). At 0.5 mm depth, the portlandite layers are detected and at 1.3 mm the portlandite is less altered. The formation of new mineral phases without spalling explain the expansion of samples (table 4).

The tuff addition induces two type of effect. A pozzolanic effect is due to silica. This effect is limited because of natural tuff alteration. Induced clays bring a filler effect, not interesting regarding strength but reducing the global pore size. Combination of those two effects lead to delay of the sulphatic attack.

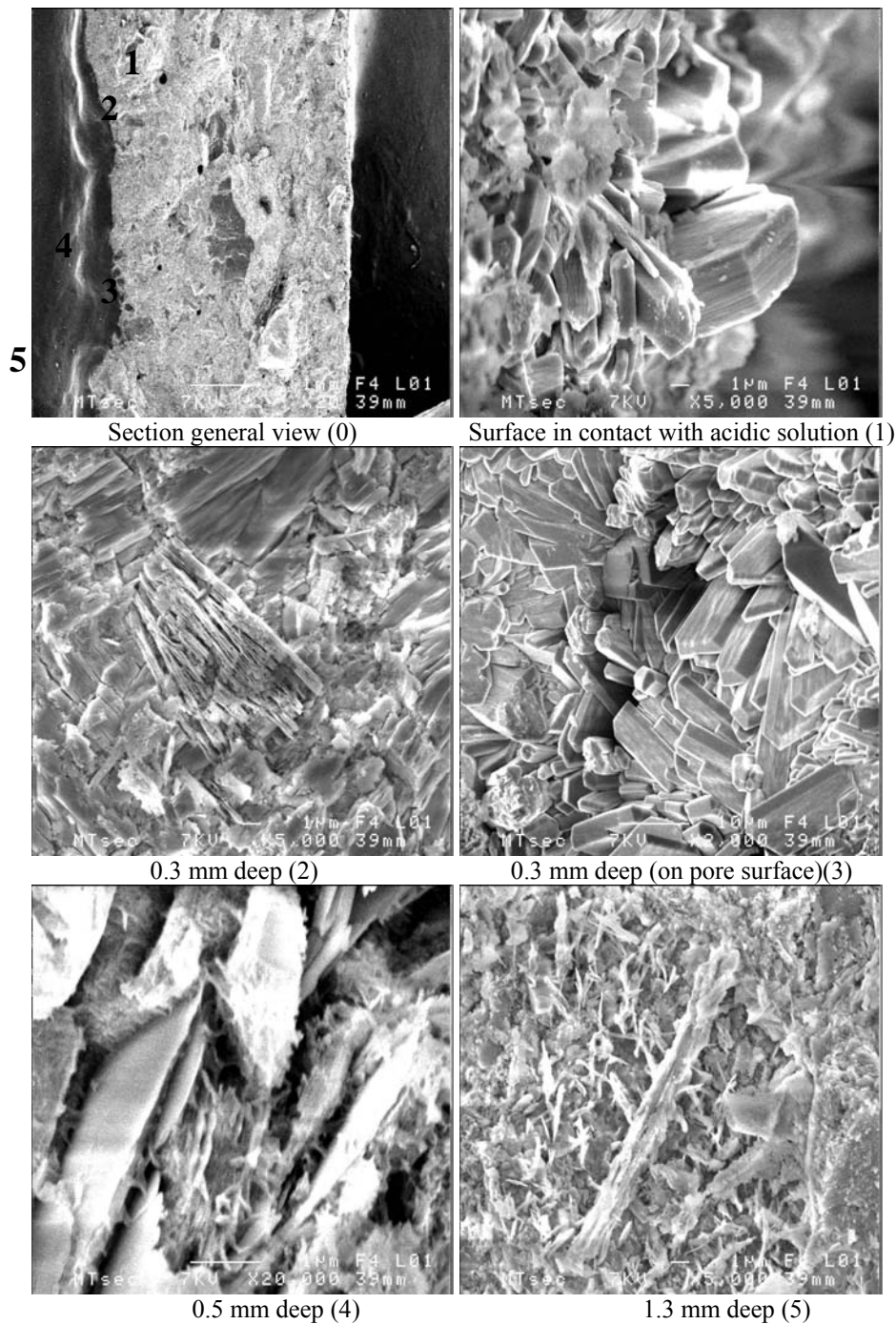


Fig.8. MT Samples stored in H_2SO_4 (0.25 M)

CONCLUSION

Huge quantities of volcanic tuffs are found in Mako area (western Senegal). The high content of SiO₂ is responsible of the pozzolanic properties of the tuffs when mixed with Portland Cement. However, tuffs alterations could limit the pozzolanic effect: for example, the formation of clay decreases this effect. Mechanical tests are displayed on Portland Cement + tuffs and Portland Cement samples: 30% of tuff/cement substitution leads to compressive strength reduced by 35%.

When mortars were exposed to different sulphatic solutions, tuff increase chemical resistance. In sulphuric acid, specimen surface are covered with a superficial formation of gypsum. The spalling of this layer is reduced when tuff is present in the mortar. With ammonium sulphate, only few needles of gypsum grow at the surface of sample without tuff. In this case, small cracks appears at the surface when samples were washed.

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