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Microstructural study of Styrene Polyacrylic (SPA) latex modified mortars

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ABSTRACT

In this paper, the influence of the styrene polyacrylic (SPA) latex polymer on the microstructural properties of limestone mortars has been studied. For this purpose, five mortars were developed with different dosages of the SPA latex (0%, 2.5%, 5%, 7.5% and 10%) by weight of cement. This research was carried out using XRD, FTIR, and SEM analyses. The results of XRD and FTIR studies showed that the addition of SPA latex can increase the portlandite content of polymer-modified mortars (PMMs), compared to the control mortar. In addition, the moist environment promotes the $Ca(OH)_2$ consumption in PMMs at early age and accelerates the hydration. Moreover, the SEM analysis revealed that the cement hydrate structure of the reference mortar is loose. In contrast, the hydrates of the PMMs were covered by a polymer film or membrane, and the pore structure is significantly affected by the filling effect the micropores by the latex particles.

1 Introduction

For decades, the use of polymers in the form of aqueous colloidal suspensions (latexes) in cement-based materials has been very popular to improve the different properties of conventional mortars and concretes such as workability, adhesion, flexural and tensile strengths, impermeability and durability [1-4]. These advantages are the results of the combination of the organic matrix (polymer) and the inorganic matrix (Portland cement) in the fresh state [5], and the interaction between the polymer film formation properties and the cement hydrates in the hardened state [5-8]. It is clear that the complex microstructure of polymer-modified cement systems is generally of considerable importance because it has a profound influence on the hydration, the physico-mechanical properties and the durability of these systems. This topic has attracted the careful attention of many researchers to elucidate the polymer modification process [3, 9-12]. In this context, Afridi et al. [9] compared the development of polymer films of the aqueous and powdered polymer modified mortars. They concluded

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that aqueous polymers form a higher quality polymer film as compared to that observed for the powdered polymers. In addition, these formed polymer films can acquire different structures. Tian et al. [13] reported that the polyacrylate (PA) latex is not uniformly dispersed in the modified mortar, which can be named as the localization of polymer modification, due to the chemical reaction between PA particles and calcium ions. As a result, the polymer film exists in different morphologies. Moreover, various previous studies have shown that the incorporation of latex polymers into cement systems prevents the hydration process and also modifies the composition, intensity, and morphology of hydration products [14-16]. Moreover, the polymer latex filled the cracks of cement hydration systems leading to the decrease of the porosity of these systems [5,17]. In addition to this physical action, chemical reactions occur between the polymer latex and the cement [13,18]. Indeed, the microstructure of cement systems modified by different latexes, for example styrene butadiene rubber (SBR), is widely studied [19-21]. However, little work has studied on the influence of styrene polyacrylic latex (SPA) on the microstructure of mortars. Therefore, the objectives of this research are to evaluate the effect of the SPA latex addition on the microstructure of a somewhat special mortar, which is the limestone mortar, and then to link the macro-properties to the internal structure of this mortar. It should be noted that various techniques have been adopted for this microstructural study such as X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectrometry and scanning electron microscopy (SEM). Finally, the contribution of the curing effects to the microstructure of the studied mortars was also discussed where two cure process were chosen, mixed cure and wet cure.

Along with the hydration, polymer covered the surface of cement particles and hydration products, or filled the cracks of cement hydration system. These physical functions could improve the porosity of cement. Let us note that, except the physical action between polymer latex and cement, chemical reactions are also present.

2 Materials and experimental methods

2.1 Materials used

The materials used in this experimental study are cement, SPA latex, limestone sand, superplasticizer and mixing water. Ordinary Portland cement (CEMI, 42.5 CRS) from Biskra (Algeria) was used in all mixtures. Its apparent and specific densities are 1.03 and 3.1 respectively and its Blaine surface area is 3200 cm^2/g. Its chemical and mineralogical compositions have been reported in Table 1, while Fig. 1 shows that the main compound of this cement is silicates (C_3S and C_2S).

Table 1 – Chemical and mineralogical composition of cement (%)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI*
20.99	4.14	3.45	63.84	2.82	3.16	0.65	0.23	0.71
C_3S		**C_2S**		**C_3A**			**C_4AF**	
58.54		16.34		5.14			10.49	

*Loss On Ignition.

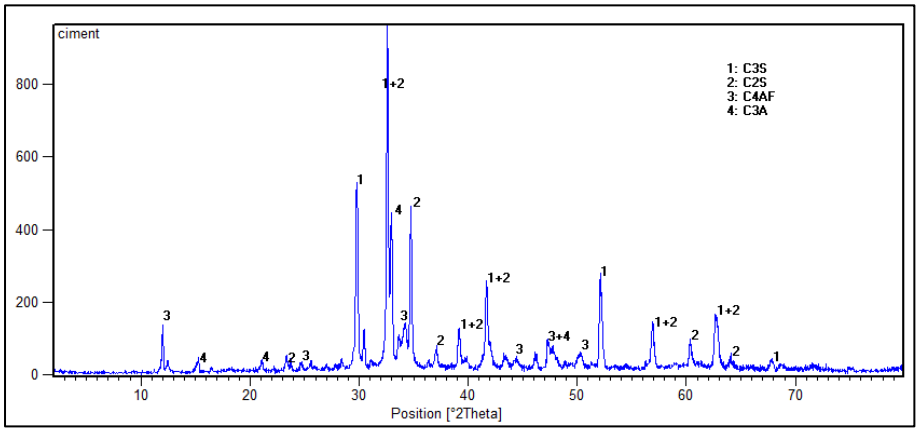


Fig. 1 – XRD analysis of cement used

The SPA used latex is a milky white aqueous dispersion supplied by TECKNACHEM company from ALGERIA (as shown in Fig. 2). It is known under the trade name TEKWELD. The solids content of the SPA latex is 26% (± 2%), its pH value (at 20°C) is 4 (± 1) and its density (at 20°C) is equal to 1.040 (± 0.02) [22].

Fig. 2 – General aspect of SPA latex

The limestone sand of granular class (0-5 mm) from the Amouri quarry in Laghouat (Algeria) was used. The chemical and physical characteristics of the used limestone sand are grouped in Tables 3 and 4 respectively. Fig.3 illustrates that the particle size distribution of this sand is continuous and it is located within the specific spindle of the material. X-ray diffraction analysis revealed its almost pure limestone nature (Fig. 4).

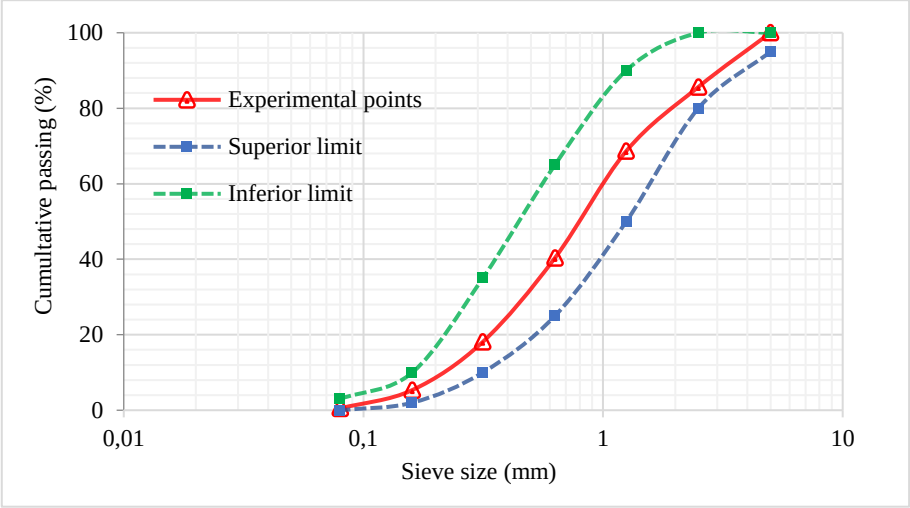


Fig. 3 – Grading curve of limestone sand used

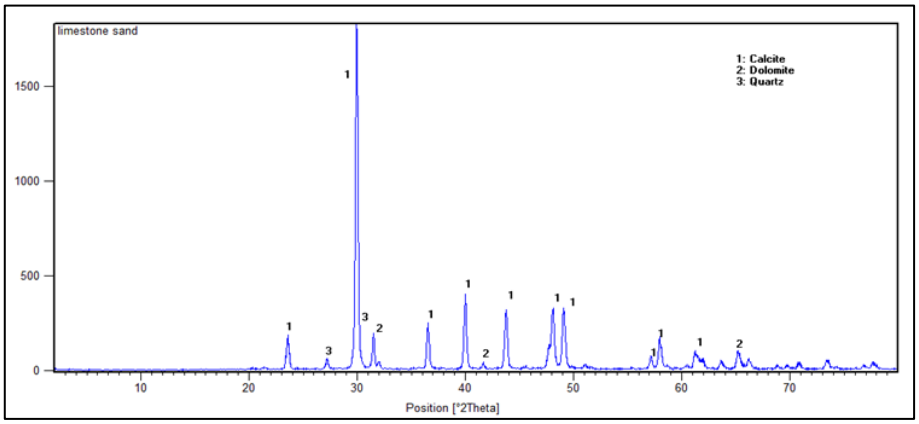


Fig. 4 – XRD analysis of limestone sand used

Table 2 – Chemical composition of limestone sand used

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI
0.51	0.33	0.31	54.82	0.89	0.053	0.001	0.16	42.92

Table 3 – Physical properties of limestone sand used

Characteristics	Standard	Value found
Absolute density (g/cm^3)	NF EN 1097-6 [23]	2.59
Apparent density (g/cm^3)		1.49
Sand equivalent: SE (%)	NF EN 933-8 [24]	66
% of elements <0,08 mm	NF EN 933-1 [25]	7.5
Finesse module: FM	NF EN 12620 [26]	3.21
Absorption coefficient: WA_{24} (%)	NF EN 1097-6 [23]	4.70

The use of a superplasticizer is important in the limestone mortars modified by SPA latex [27]. Therefore, a superplasticizer marketed under the name "SUPERIOR 9WG" from the company “TECKNACHEM” of ALGERIA was used. This adjuvant is composed of the new generation polycarboxylates. Its normal range of use varies, according to the technical sheet, between 0.6% and 2% (in dry extract) of the weight of cement. It is in liquid form of the brown color of 33% (± 2%) of dry extract. Its density is 1.10 (± 0.03), and its pH (at 20°C) is 5.5 (± 1) with a chlorine content of less than 0.1%.

2.2 Preparation of samples

Five samples of mortars were prepared according to JIS A 1171-2000 (Japanese Industrial Standard) [9,28,29], with a polymer/cement mass ratio (P/C) of 0%, 2.5%, 5%, 7.5% and 10% in dry extract and with the cement / sand (C/S) weight ratio of 1/3. For all the mixes, the W/C ratio has been adjusted for a constant plastic workability (the flow time at 8±2s). This formulation is more widespread and her name is "iso-rheology" [30-32]. It is important to note that the amount of water required must take into consideration the part of water introduced into SPA latex. After the mixing process, the mixtures were poured and compacted in prismatic molds of 4×4×16 cm^3 (Fig. 5). To avoid evaporation of water, the specimens were covered with a plastic film [33]. The demolding was done after 24 hours. In order to study the effect of different cure conditions on the microstructure of studied mortars, the samples were subjected to two different cure procedures. The first is a wet cure (20 ± 2°C, 90 ± 5% RH) [34-36]. The second is mixed cure consisting of 6 days of water immersion and subsequent drying in the laboratory environment (20 ± 3°C, 50 ± 10% RH) [34,37-40]. It should be noted that the reference samples (without the addition of SPA latex) are immersed in water. This is a recommended cure for pure hydraulic mortars (EN 1504) [38,41]. Table 4 summarizes the compositions of the studied mixtures.

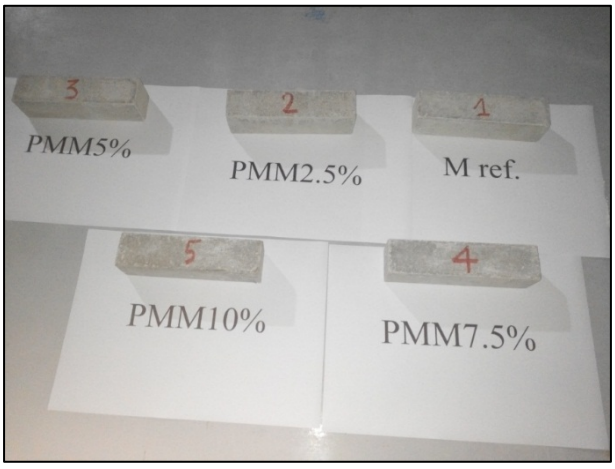


Fig.5 – General appearance of specimens

Table 4 – Compositions of mixtures studied

Designation	C/S	P*/C (%)	W/C	Sp*/C (%)	Flow time (s)
M ref.	1/3	0	0.5	0.8	10.03
PMM2.5%		2.5	0.492		9.42
PMM5%		5	0.481		9.37
PMM7.5%		7.5	0.463		8.89
PMM10%		10	0.45		8.44

*in dry extract.

2.3 Test methods

This experimental study was carried out using various techniques such as: XRD, FTIR and SEM. At the age of 90 days of curing, these analyzes were performed. The XRD analysis was made with Kα1 copper radiation having a wavelength λ (kα1Cu) equal to 1.5418Å. The diffracted intensity of the KαCu radiation was recorded between 20° and 60° (2θ angle range). A scan was performed with a step length of 0.1° and a time of 3s for each step (the scanning speed is: 2°/min).

In addition, the FTIR spectra were recorded by a "Perkin Elmer Spectrum One" spectrometer. The powder samples, previously dried, were mixed with KBr. This analysis was performed in the range of 4000 to 400 cm^{-1} with a resolution of 4.0 cm^{-1}.

The microscopy of the studied mortars was made using a VEGA3 TESCAN type scanning electron microscope (acceleration voltage is 15kV). Before the test, the studied samples were coated with a thin gold film using a vacuum metallizer to render their surfaces conductive.

3 Results and discussion

3.1 XRD analysis

The portlandite ($Ca(OH)_2$) is one of the main phases of cement hydration, and its content can reach, by volume, 25% of the total mass of hydrates [21,42]. In addition, the portlandite can be influenced by the presence of additives such as latex polymer [42]. In this study, we are much more interested in the effect of SPA latex on the behavior of $Ca(OH)_2$. Moreover, the portlandite content is an indicator of the degree of hydration [38,43].

The XRD curves of SPA latex modified limestone mortars, with wet and mixed cure procedures, and control mortar in a range of 2θ=28.5°-31.5° are presented in Fig. 5. Based on the identification of crystalline phases made software "X'Pert HighScore Plus version 2.1", it has been reported that the maximum intensity of the portlandite of the reference code (ICDD PDF no 00-050-0008) was located in the previous range. In this way, the integrated results of XRD peak of the portlandite including this peak position, the interplanar spacing (d), the full-width half-maximum (FWHM), the peak height (I. max.), the integral intensity (I. integ.) and the ratio of the $Ca(OH)_2$ content in the modified mortar to that of the control mortar (R), calculated from the integral intensity [16 ,21, 44], are detailed in Table 5. The ratio R is defined as follows:

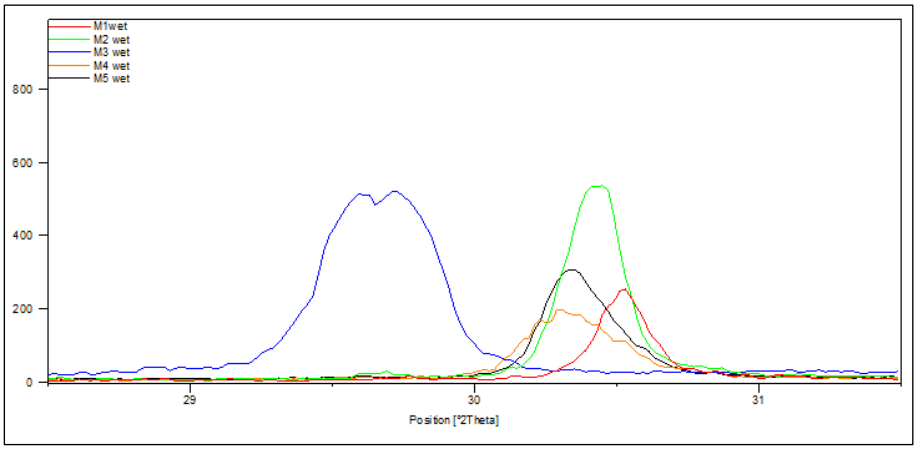
$$R = \frac{\text{I.integ.}_{(P/C=x\%)}}{\text{I.integ.}_{(P/C=0\%)}} \quad (1)$$

The XRD analysis, in the range of 2θ = (20°-60°), showed the absence of chemical interaction between the cement and the SPA latex, since there is no formation of new product. From Table 5, it can be seen that the portlandite content is lowest in the control sample except for a PMM2.5% sample subjected to a mixed cure. In addition, the R-value of SPA latex modified mortars, regardless of the cure mode, varies between 1.23 and 7.06. According to the literature, it is important to note that the portlandite content has been gradually increased with the time of hydration for all the studied pastes. However, unmodified paste also exhibited the maximum R-value compared to those of pastes modified by SBR latex at an early age. This is due to the fact that latex apparently delays hydration process at 3 days [21, 44]. Nevertheless, this quantitative study allows us to show that the retarding action of the SPA latex on the portlandite content, and consequently on the cement hydration degree,

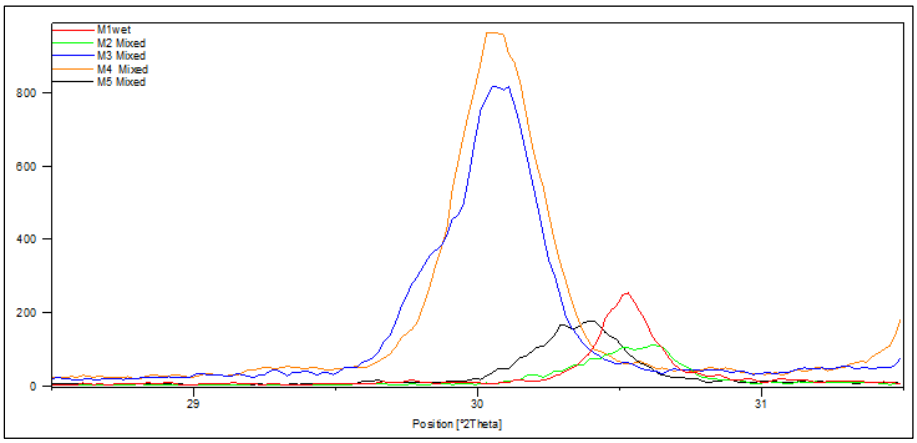
was not evident in the long term (90 days later). This is probably due to the better dispersion of the cement particles in the water of SPA latex modified mixtures. This finding is in accordance with that of mortars modified with water-soluble polymers such as poly (vinyl alcohol-acetate) (PVAA), methylcellulose (MC) and hydroxyethylcellulose (HEC) [45].

Table 5 – Integrated results of XRD peak of portlandite of mortars studied

P/C (%)	Cure mode	Position 2θ (°)	d (Å)	FWHM	I. max. (cts)	I. Integ. (cts.°)	R
0	Wet	30.498	2.929	0.182	229.63	41.754	1
2.5		30.466	2.934	0.217	519.49	110.935	2.66
5		29.810	2.997	0.197	430.10	83.496	2
7.5		30.311	2.946	0.331	186.14	55.706	1.33
10		30.316	2.948	0.236	290.28	67.624	1.62
2.5	Mixed	30.571	2.924	0.394	104.97	40.757	0.98
5		30.021	2.977	0.217	752.20	160.631	3.85
7.5		30.069	2.972	0.315	948.68	294.672	7.06
10		30.387	2.942	0.315	165.46	51.394	1.23



(a) wet cure



(b) mixed cure

Fig. 5 – XRD curves of mortars studied for portlandite characteristic peak

On the other hand, by varying the P/C ratio between 5% and 7.5%, it has been observed that the $Ca(OH)_2$ content in the case of the PMMs subjected to a mixed cure is higher than that formed in the PMMs at moisture. This suggests that wet treatment accelerates the hydration process of PMMs at an early age and promotes the consumption of the portlandite. The PMM7.5% subjected to a mixed cure presented the maximum content of $Ca(OH)_2$ where the R ratio reaches 7.06. This result is consistent with that obtained in the compressive strength study at 90 days (not mentioned here).

3.2 FTIR analysis

Indeed, FTIR spectrometry is a versatile tool often used for molecular characterization, but it has been exploited to elucidate the hydration reaction of the studied mortars and also to identify the influence of latex polymers on hydrates [46]. To specify the chemical bonds between the SPA latex polymer and the cement hydrates, the spectral analysis on the cement mortar and the mortar modified with 5% of the SPA latex was performed. Fig. 6 shows the FTIR spectra recorded for the control mortar and PMM5% subjected to a mixed cure. As shown in Fig. 6, all spectra characterized on the SPA latex modified mortar can be observed in the reference mortar. This allows concluding that no newly formed chemical bonds in the cement-latex SPA co-matrix. Nevertheless, the comparison between the recorded spectra indicates that the addition of 5% of the SPA latex resulted in modifications on the cement hydration, in particular, a slight increase in the intensity of the signal at 3645 cm^{-1} which corresponds to the O-H free bond from calcium hydroxide. This indicates that the $Ca(OH)_2$ content is slightly increased after 90 days of hydration due to the delayed hydration. This result is compatible with XRD analysis. After this peak, wide and flat shape of the bands between 3500 cm^{-1} and 3000 cm^{-1} corresponding to the crystalline water can be observed for the two samples studied. This is due to low crystallinity after 90 days of hydration. However, it appears that these absorption peaks show an increase with the incorporation of the SPA latex in comparison with the control mortar. This increase indicates that PMM5% has a better water retention property [47, 48]. On the other hand, the infrared spectrum has peaked at 1456 cm^{-1} and 877 cm^{-1} attributed to the vibration of the C-O bond due to the presence of CO_3^{-2} ion which is mainly associated with calcium carbonate ($CaCO_3$) [49]. These peaks are increased with the addition of SPA latex. In addition, the peak at 1122 cm^{-1}, corresponding to ettringite, was detected in both composites and its intensities are similar [40]. A significant peak was observed for both composites at 968 cm^{-1}. This explains the polymerization of SiO_4^{-4} present in C_3S and C_2S and indicates that the SiO_4 tetrahedron structure modifies and the calcium silicate hydrate (C-S-H) is influenced [38].

The limestone sand, which represents 68 to 75.5% by total weight of the mortar, was used in the composites studied. The characteristic spectra of calcite are (3050 - 2750, 2680 - 2380, 2260 - 2080, 1950 and 1794 cm^{-1}) [49]. In the samples studied, the peaks corresponding to calcite appear at 2362 and 2335 cm^{-1}.

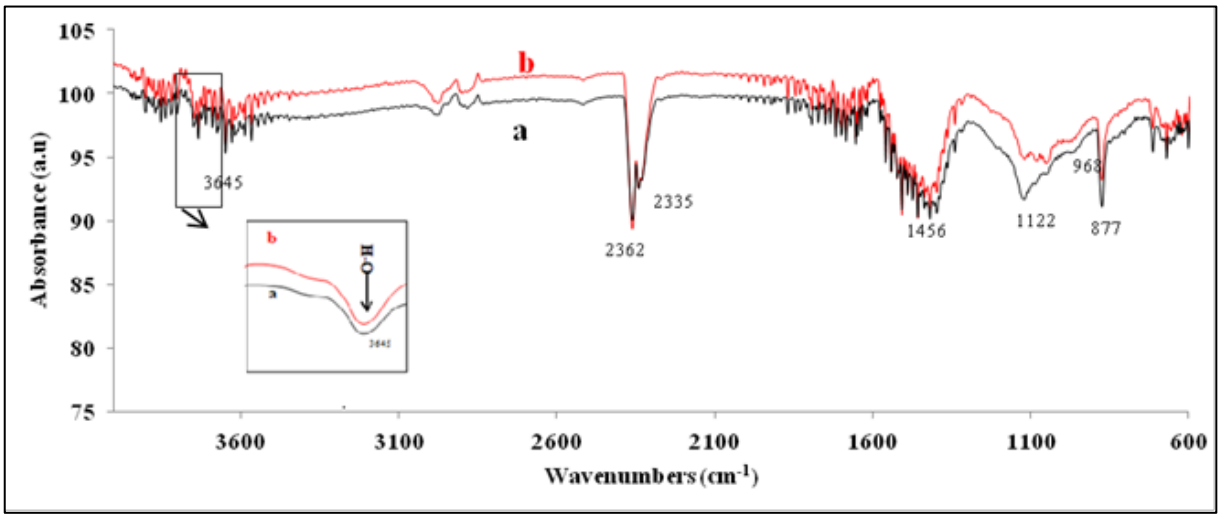
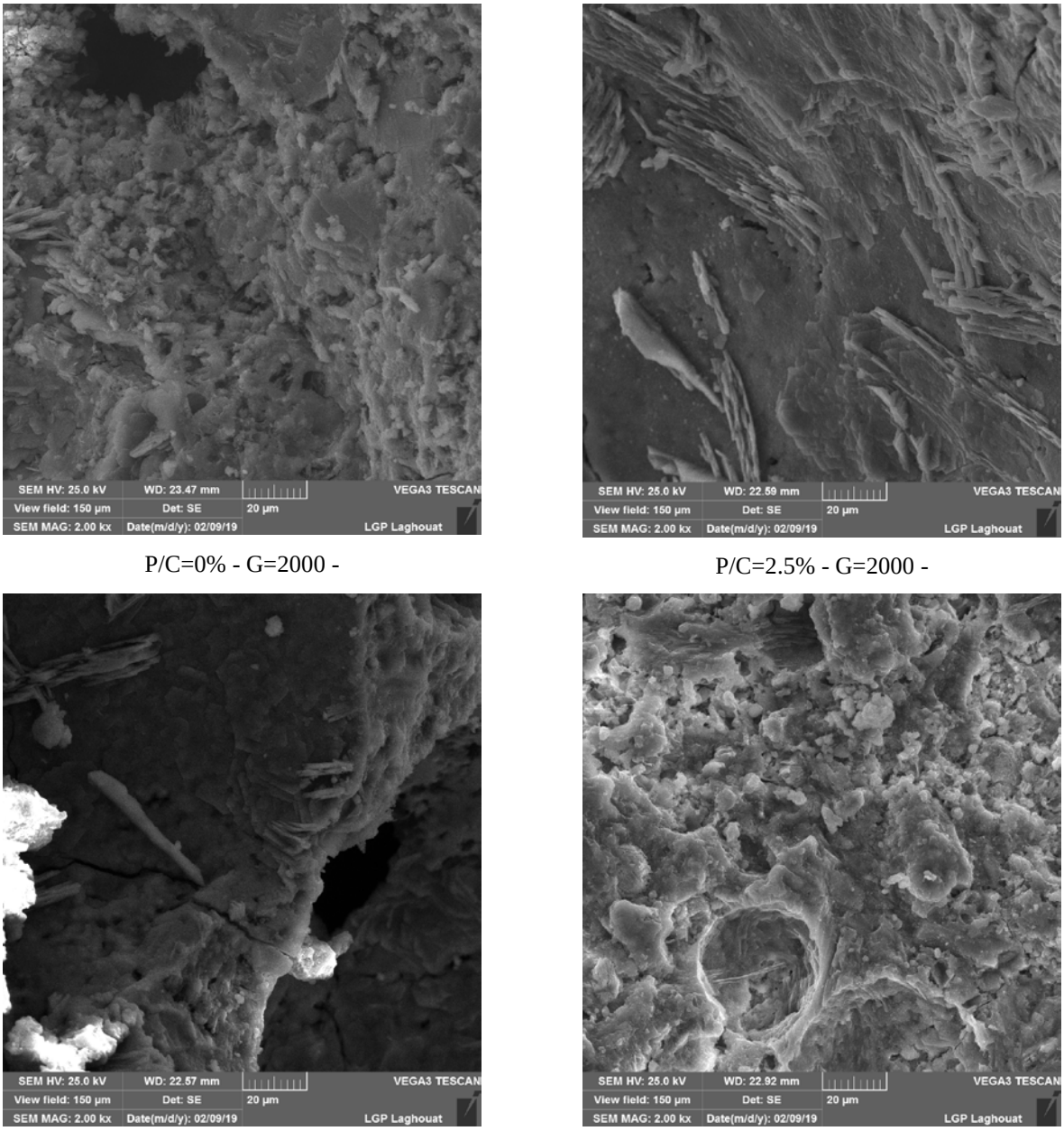


Fig. 6 – FTIR of (a) reference sample (b) PMM5% sample (with mixed cure)

3.3 SEM Observation



P/C=0% - G=2000 - P/C=2.5% - G=2000 -

P/C=5% - G=2000 - P/C=10% - G=2000 -

Fig. 7 – SEM images of the studied samples

Fig. 7 shows SEM images at a magnification G = 2000 of the reference mortar, the PMM2.5%, the PMM5% and the PMM10%. This analysis allows us to observe that the reference mortar has greater porosity and lesser homogeneity than the PMMs. Furthermore, the hydrates in the control mortar are loosely bound and these hydrates have been clearly shown as CSH in the form of needles. In contrast, the microstructure of the PMMs is dense and the hydrates have been encapsulated by a polymer film or membrane, which makes the observation of the hydration products difficult [32,38,50]. This film improves the bond between cement hydrates; even the pore structure is significantly affected by the filling effect of SPA latex particles in the micropores [51]. This improves the impermeability of the PMMs. The PMM5% presents a poor microstructure with cracks compared to other PMMs. This revealed that the addition of 5% SPA latex induces discontinuities in the microstructure of the mortar and thus reduces the physico-mechanical properties. Moreover, for PMM10%, large cavities were observed indicating the existence of the excess SPA latex.

4 Conclusion

In this research, five mortar mixtures containing 0%, 2.5%, 5%, 7.5% and 10% of the SPA latex were tested to study the effect of SPA latex on the evolution of the microstructure of limestone mortars. The main conclusions of this study are summarized below.

The $Ca(OH)_2$ content in the studied mortars was determined by XRD. The portlandite content of PMMs, regardless of the mode of cure, is significantly higher than that of the control mortar. This result is compatible with FTIR- results. This increase can be explained by the fact that the SPA latex retards hydration at early age, which allows the hydration to strongly continue even after 90 days, compared to the control mortar. The wet treatment accelerates the hydration of the PMMs at early age and promotes the consumption of portlandite.

The FTIR analysis shows that no newly formed chemical bond in the cement-latex SPA co-matrix. However, the addition of latex alters the intensity of cement hydrates such as CSH.

The SEM analysis shows that the hydrate structure of the unmodified mortar is loose. In contrast, the PMMs components are densely attached. In addition, the PMMs hydrates have been covered by a polymer film or membrane, and the pore structure is significantly affected by the filling effect of the latex particles in the micropores.

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