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Roman cement mortar prepared by a multi-stage mixing process

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ABSTRACT: Roman cement is the predecessor to modern Portland cement. Nowadays, it is a very promising product with lower CO_2 emissions, frequently used to restore historical objects. However, there are still many practical problems as a setting that can be affected in several ways. One possibility is the multistage mixing of fresh mortar, a practical historical method that has not yet been scientifically investigated. This article presents an experimental study investigating the effect of multistage mixing on the properties of fresh and hardened mortar. The properties and structure of the mortar were compared with a reference mortar (retardened by citric acid). Multistage mixing affects fresh mortars with optimal consistency and a workability time of 120 minutes. The influence of mixing on the hydration process and structural formation is characterized by isothermal calorimetry and SEM. Comparison of reference and modified mixing mortars exhibits differences in hydration process, structure, and initial strength, but no significant effect at 90 days strength.

KEY WORDS: Roman cement; Mortar; Mixing process; Setting time.

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RESUMEN: *Mortero de cemento romano preparado mediante un proceso de mezcla multietapa.* El cemento romano es el predecesor del cemento Portland moderno. Actualmente es un producto muy prometedor con menores emisiones de CO_2, utilizado con frecuencia para restaurar objetos históricos. Sin embargo, sigue presentando muchos problemas prácticos, como su fraguado que puede verse afectado de varias maneras. Una posibilidad es la mezcla multietapa de mortero fresco, un método histórico práctico que aún no se ha investigado científicamente. Este artículo presenta un estudio experimental en el que se investiga el efecto de la mezcla multietapa sobre las propiedades del mortero fresco y endurecido. Las propiedades y la estructura del mortero se compararon con las de un mortero de referencia (retardado con ácido cítrico). La mezcla multietapa afecta a morteros frescos con una consistencia óptima y un tiempo de trabajabilidad de 120 minutos. La influencia de la mezcla sobre el proceso de hidratación y la formación estructural se caracteriza por calorimetría isotérmica y SEM. La comparación de los morteros de mezcla de referencia y los morteros modificados muestra diferencias en el proceso de hidratación, la estructura y la resistencia inicial, pero ningún efecto significativo a los 90 días de resistencia.

PALABRAS CLAVE: Cemento romano; Mortero; Proceso de mezcla; Tiempo de fraguado.

1. INTRODUCTION

OPC production is related to large-scale extraction of non-renewable raw materials, has high CO_2 emission values and the energy consumption in the production process is high (1, 2). The operation and production technologies of cement plants worldwide are gradually being modified to eliminate negative environmental impacts (3, 4, 5). Along with the trend of innovation in OPC production, current research in this area is focused on the development of new hydraulic binders with low impact categories (6,7,8,9). Historical building structures are also inspiring in this regard (10, 11, 12, 13). As it turns out, historical hydraulic mortars resist various degradation factors and achieve high durability in the long term (14, 15). Currently, worldwide, there is interest in studying these historic materials and technologies for at least three reasons:

1) Development of specific materials for the restoration of cultural heritage (16)
2) Development of environmentally acceptable modern building materials
3) Carbon sequestration

Natural cement, also known as Roman Cement in Europe, was a popular building material in the 19th century and early 20th century in Europe and America. Raw material with a higher proportion of clay was used to produce NC and the calcination temperature was significantly lower than that of modern OPCs (17, 18, 19, 20).

The calcination temperature of the raw material in current commercially produced NCs (21) depends on its composition and is approximately in the range of 800-1200°C. The specific composition of the raw material, calcination temperature, calcination time, mode of cooling and other factors influence the final phase composition of the NC (22, 23, 24). For this reason, the structure and properties of these binders are highly variable. This fact is a certain disadvantage in the practical application of NC in mortars and concretes and in relation to current standardization.

The main hydraulic components of NC are crystalline belites (dicalcium silicates - C_2S) and amorphous aluminates. The stable belites in cements calcined at lower temperatures are largely of the $\alpha'C_2S$ type. At higher calcination temperatures, βC_2S is stabilized. The proportion of belitic phases can be up to 60 % (23, 25, 26).

In terms of practical applications of NC in mortars and concretes, the strategic challenge is to retard the start of the mortar setting while maintaining their acceptable consistency and workability time. At the same time, it is necessary to achieve the designed flexural tensile strength and compressive strength parameters of the hardened mortar or concrete.

Natural cement is characterized by rapid setting, usually within 7-10 minutes of adding water. This is very inconvenient from the point of view of applying this material to construction.

Various methods are currently used to retard the start of setting of fresh mix (also referred to as retardation). The most widely used method is the application of retarding additives such as: citric acid, sodium citrate, potassium citrate, sodium gluconate, gypsum, lime, etc.

As expected, citric acid inhibits the formation of C-A-H (calcium aluminate hydrates), AF_m phases (hexagonal calcium ferro-aluminate hydrates); ettringite formation has been shown to be affected. The observation is consistent with the assumption that citrate ions act as a chelating agent due to their preferential coordination at Ca^{2+}, Al^{3+} or Fe^{3+} centres. This reaction produces very stable chelate complexes of iron (II or III) hydroxocitrates with aluminium, which affect the nucleation and subsequent crystal growth of AF_m or AF_t (trigonal calcium ferro aluminate hydrates) phases (27-30).

However, some retarders can adversely affect the strength of hardened mortar (21, 31, 32). Conversely, some retarders and retardation techniques can increase the long-term strength of hardened mortar, although in a specific way (24, 33). The suitability of using a particular chemical retarder and/or retardation technique for all types of NC cannot be conclusively confirmed.

Among the historical retardation technologies for NC-based mortars, the pre-hydration method is described. The pre-hydration method has been experimentally investigated by Starinieri et al (33, 34). The authors of this study were able to show that for NC made from raw materials originating from Gartenau, Austria, the start of the mortar setting can be extended to several hours by the pre-hydration process. However, there are a number of factors that influence both the retardation time and the final properties of the hardened mortar. In this study, the authors state that the pre-hydration method is not suitable for all NCs. For example, the natural cement PROMPT (Vicat, France) did not respond convincingly to this method (34).

The workability time of the mortar can be extended by increasing the value of the w/c ratio. However, for NC-based mortars, the increase in workability time by this method is not very significant. The value of the w/c ratio is also related to the effect on the structure and final functional properties of the hardened mortar (35, 36).

Another historical mortar retardation technology is remixing (hereafter remixing = repeated and/or prolonged mechanical mixing of fresh concrete and mortar). This practical experience has been mostly orally transmitted since the 19th century. Previously, when the fresh mortar mixture began to set on site, the plasterers would remix it several times, thus 'reviving' the mortar. This experience in sample preparation is also described by Starinieri et al (34) but they do not investigate or discuss this phenomenon further.

The effect of remixing is described in older literature (37-39). Bechyně (38) states that the processing of fresh concrete (or mortar with hydraulic binder) is not necessarily completed before the start of the mortar setting. The concrete mix can be processed if it is "live" (if the mix remains ductile). Bechyně further states that mechanical intervention in the setting mix, although brutal and requiring special machinery and adequate formwork design, is very effective. After curing, the concrete remixed in this way shows high densities and significantly higher strength values (38).

In recent years, there has been a growing interest among researchers in verifying and explaining the effects of remixing and/or modifying the mixing process in fresh concrete, cement mortars and pastes (40-44). This comes especially in the context of the development of new, more environmentally attractive construction materials and technologies.

This paper publishes the results of a case study where a specific historical mortar remixing technology based on PROMPT natural rapid-setting cement (Vicat, France) was experimentally verified. The remixing effect was investigated and compared with a setting retardation technology using citric acid (TEMPO product). Citric acid is recommended by the manufacturer of PROMPT natural cement for setting retardation (45).

2. MATERIALS AND METHODS

2.1. Mortar samples and materials for sample production

For the purpose of this case study, 3 sets of mortar samples were produced (Table 1). For each set, 12 samples were produced. The samples were 40 x 40 x 160 mm in size. The mortar samples labelled RR were reference samples, the R samples were affected by citric acid (supplied by VICAT under the label TEMPO), the A samples were prepared by a special procedure prolonged mixing (Chapter 2.2).

The binder was the commercial product PROMPT Natural Cement (hereafter PROMPT). The filler in the mortars was quartz (standard) sand, the sand grain size was 0.08-2 mm. The w/c ratio of the mortars was high w/c = 1. The mortar ingredients were proportioned by weight. Drinking water from the water supply system was used as mixing water.

Table 1. Sample designation and mortar composition.

Sample	PROMPT : Water : Sand	Citric acid (TEMPO)
RR/7,14,28,56	1:1:1	-
R/7,14,28,56	1:1:1	0.6% by weight PROMPT
A/7,14,28,56	1:1:1	-

Natural Cement PROMPT

The properties of this commercial product are specified in the technical documentation (45, 46), further information, e.g. chemical and mineralogical composition with loss of ignition at temperature 975 °C (Tables 2 and 3), is available in publication (47).

The chemical composition of PROMPT has been verified for the purpose of sample production by X-ray fluorescence spectroscopy (XRF), see Table 4. The differences in the composition of PROMPT (cf. Table 2 and Table 4) are slight.

Table 2. Chemical composition of PROMPT by manufacturer in mass % (39).

LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O
9.28 %	18.09 %	7.24 %	3.2 %	53.07 %	3.84 %	3.24 %	1.16 %	0.28 %

Table 3. Mineralogical composition of PROMPT by manufacturer in mass % (39).

C_3S	C_2S	C_3A	C_4AF	$C_{12}A_7$	C_4A_3S	Periclase	Free lime	Calcite	Sulfates	Others, incl. amorphous phases
5-15%	40-60%	6±2%	9±2%	3±1%	3±1%	4±1%	2±2%	10-15%	3±1%	10-15%

Table 4. Control analysis of the chemical composition of PROMPT before sample production in mass %.

LOI	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	P_2O_5	MnO	TiO_2
8.14%	18%	7.68%	3.2%	55.1%	3.69%	2.38%	0.97%	0.14 %	0.072 %	0.065%	0.28%

2.2. Sample production

The samples were produced in the laboratory. A specific procedure different from the standard was used to produce the mortar samples (48). A standardized programmable laboratory mixing HOBART device fulfilling the specific requirements of the standard EN 196-1 (49) was used to prepare the mortars.

Initially, for all samples, the dry ingredients were mixed by hand. After a period of 10 minutes, water was added, in the case of the R samples, water with dissolved TEMPO retarder. The time of water addition was recorded as t = 0, the mixing device was started.

The RR and R samples were mixed at low speed according to the standard EN 196-1 for 3 minutes and then the mortar samples were placed in the moulds (48). Compaction of the mortars in the moulds was done by tapping to avoid segregation of the ingredients. Despite this, segregation was observed in the R samples.

The A samples were mixed using a procedure that was suggested based on previous laboratory experiments and the authors' observations. The mortar was "revived" a total of 3 times during mixing – "remixed" using the following procedure:

- The time of water addition was recorded as t = 0, mixing at a slow speed for 2 minutes.
- At time t = 2 minutes, mixing was stopped, the walls of the vessel were wiped with a spatula.
- At time t = 4 minutes, mixing was continued at slow speed for 1 minute, then the mixing was stopped.
- At time t = 7 minutes, solidification of the mixture was observed.
- At time t = 8 minutes, mixing was started at a slow speed for 1 minute and then stopped.
- A change in the consistency of the mixture was observed.
- At time t = 12 minutes, mixing was continued at a slow speed for 1 minute and then mixing was stopped.
- The mortar was placed in the moulds.

Other information about sample production:

The RR samples set within 7 minutes after the addition of water. All samples were stored in an environment according to standard (48). The A samples were still formable at 7 days in the moulds. After 14 days, samples A and R were soft and unsuitable for demoulding. The samples were removed from the moulds after a period of 28 days. The samples in the moulds and samples after removal from the moulds were conditioned in a controlled environment of an air conditioning unit at a temperature of 20 ± 1°C and a relative humidity greatet than 90 %.

2.3. Properties of fresh mortars

The following properties were determined on the fresh mortar samples:

- determination of the normal consistency of fresh mortars (50),
- determination of the bulk density of fresh mortars (51),
- determination of the air content of fresh mortars (52),
- the workability of fresh mortars was determined according to EN 1015-9 (53) by both methods.

2.4. Analysis of the development of the heat of hydration

Isothermal conductivity calorimetry test was performed on pastes (without sand, see Table 1) in a TAM Air (TA Instruments), for 50 days. From approximately day 46 onwards, the calorimetric curves of the paste samples did not change (Figure 1). Curves are shown for normalized heat flux values (mW/g), which is the conversion of heat flux to sample mass.

2.5. Strength of hardened mortars

The bending and compressive strength was determined on mortar samples after 28, 56 and 90 days after mixing. The samples had standardized dimensions according to EN 196-1 (49) with width 40 mm, height 40 mm and length 160 mm. Testing was performed on equipment FORM+TEST 10kN for determining the strength properties of hardened mortars by loading speed 50N/s. The samples were loaded to failure with loading scheme according to EN 196-1 (49).

2.6. SEM analysis

Scanning electron microscopy (SEM) analysis were performed for the morphology evaluation on a JEOL JSM-7610Fplus. Samples were coated by a very thin layer of Pt (30 nm) to make sample surface conductive and prevent charge accumulation.

3. RESULTS AND DISCUSSION

3.1. The properties of fresh mortars

The test results are given in the tables (Tables 5, 6, 7, 8). As expected, the reference mortar (RR samples) set within 7 minutes of the addition of water. Therefore, the normative methods used were not appropriate and did not produce the expected results. In comparison to the reference mortar, both retardation methods (samples R and A) achieved the target and both mortars had a significantly longer fresh mortar workability time. Samples R with TEMPO (citric acid) additive showed a liquid consistency with a clear segregation of the ingredients and the workability time was higher than 180 minutes (test was terminated after 180 minutes). The remixed fresh mortar (samples A) had an ideal plastic consistency and a workability time of >120 minutes. The air content was twice as high in mortar A compared to mortar R. However, the bulk density was almost identical. These results may have been influenced by the segregation of the components in mortar R.

Table 5. Consistency of fresh mortars.

Sample	Flow value
RR	Note 1
R	Note 2
A	180 mm

Note 1: Not measurable by the method, setting occurred within 7 minutes from t=0; does not allow the test to be performed
Note 2: Liquid mixture with clear segregation of components does not allow the test to be performed

Table 6. Bulk density of fresh mortars.

Sample	Bulk density ρ_m (kg/m^3)
RR	Note 3
R	1 690
A	1 680

Note 3: Not measurable by method, setting occurred within 7 minutes of t=0 (addition of water); does not allow the test to be performed.

Table 7. Air content of fresh mortars.

Sample	Air content of fresh mortar (%)
RR	Note 3
R	2.2
A	4.5

Note 3: Not measurable by method, setting occurred within 7 minutes of t=0 (addition of water); does not allow the test to be performed.

Table 8. Workability of fresh mortars.

Sample	Method A (min) (45)	Method B (min) (45)
RR	Note 3	Note 3
R	> 180	> 180
A	> 120	> 120

Note 3: Not measurable by method, setting occurred within 7 minutes of t=0 (addition of water); does not allow the test to be performed.

3.2. Analysis of the development of the heat of hydration

For the samples without additive (RR, A), an almost identical curve is evident, with the development of hydration heat starting after approximately three days, with a significant peak in values at day 5 of hydration. The normalized heat flux curve of the sample R (TEMPO retarder) has a significantly different shape. The hydration heat evolution is significantly lower compared to samples RR and A, starting after 10 days with a peak at approximately day 18. The calorimetry results (Figure 1) express a different hydration mechanism in mortar R, where the influence of citric acid on the formation of ettringite and early hydration products was evident, as described above in Chapter 1.

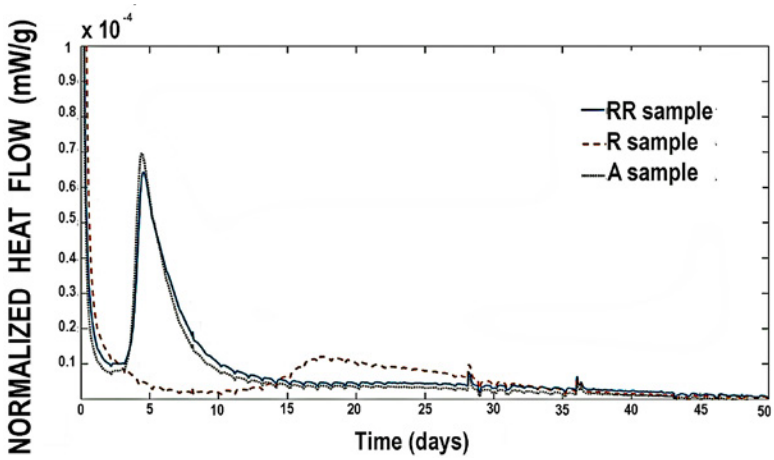


Figure 1. Calorimetric curves.

3.3. Strength of hardened mortars

The number of mortar samples in each set for determining the bending strength of was 3. Each set for determining the compressive strength of mortars contain 6 samples.

The mean values for the sets are presented in the form of graphs (Figure 2 and 3). The statistical parameters of the sets are presented in Table 9 and 10. According to the assumptions, the highest strength values were achieved for the reference mortar RR. The strength of the mortars R and A increases with the increasing age of the mortar. The statistical evaluation (Table 9 and 10) provide interesting information in view of retarded mortars strength which are more stable than the data for the reference mortar. This fact can be associated to the formation of a hydrated structure of materials, rapid solidification of the mortar RR, etc. and deserves more attention in subsequent research.

Table 9. Bending strength. Statistical data evaluation.

Sample	Standard deviation			Range		
	28 days	56 days	90 days	28 days	56 days	90 days
RR	0.13	0.23	0.27	0.017	0.055	0.073
R	0.20	0.06	0.09	0.041	0.004	0.008
A	0.16	0.32	0.16	0.027	0.101	0.024

Table 10. Compressive strength. Statistical data evaluation.

Sample	Standard deviation			Range		
	28 days	56 days	90 days	28 days	56 days	90 days
RR	0.30	0.65	0.52	0.091	0.425	0.266
R	0.08	0.16	0.20	0.007	0.026	0.039
A	0.01	0.07	0.15	0.00017	0.005	0.021

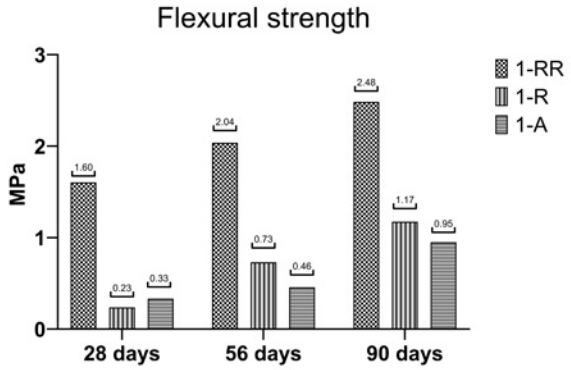


Figure 2. Mean value of bending strength.

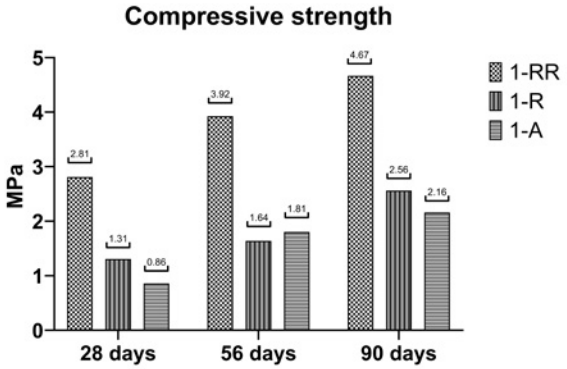


Figure 3. Mean value of compressive strength.

3.4. SEM analysis

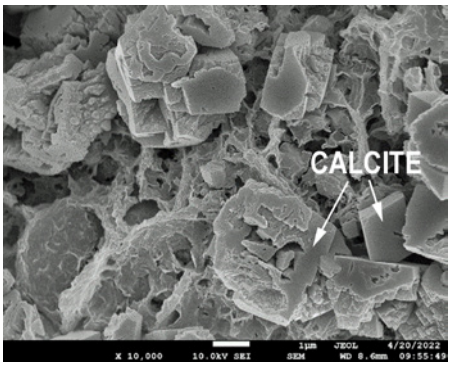
The differences in the development of the hydrated structure are evident from the SEM (scanning electron microscopy) pictures.

The minerals of calcite group formation are evident for 7-days old reference mortar samples RR. The calcite crystals progressed considerably by 14-days from the mixing with apparent ettringite formations are also evident (Figure 4a, 4b).

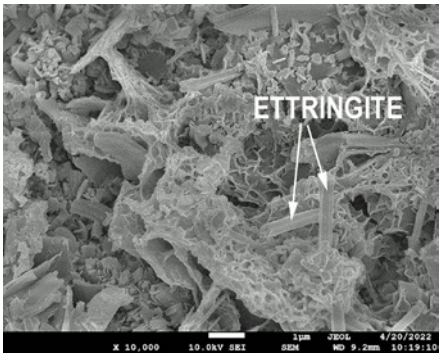
After the dormant period, 56-day-old mortar samples showed leaf-like formations within the structure (Figure 4c), which have also been described in the literature as a 'house-of-cards' arrangement. The formation of hydration products is apparently slowed down for the citric acid retarded mortar (samples R), and significantly underdeveloped or deformed ettringite crystals are already visible at 14 days (Figure 5a). The formation of a crystalline structure (belite crystal shape, or larnite-like) is evident for R samples aged 56 days (Figure 5b).

The mortar prepared by prolonged mixing process (samples A) shows slower and less distinct portlandite formation at 7 and 14-days old samples (Figure 6a, 6b), but ettringite formations are already present at 7 days (Figure 6a). The presence of "leaf-like formations" is visible at 56 days of age, but portlandite formation still seems to be inhibited by some factor (Figure 6c).

(a)



(b)



(c)

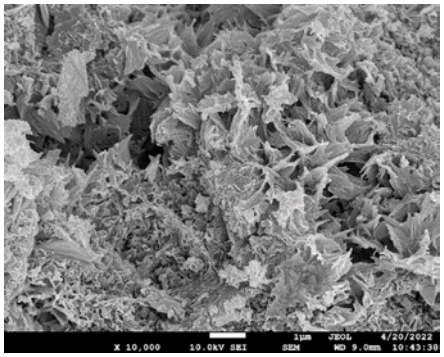


Figure 4. Microstructure of RR mortar samples (reference samples): (a) 7 days, (b) 14 days, (c) 56 days.

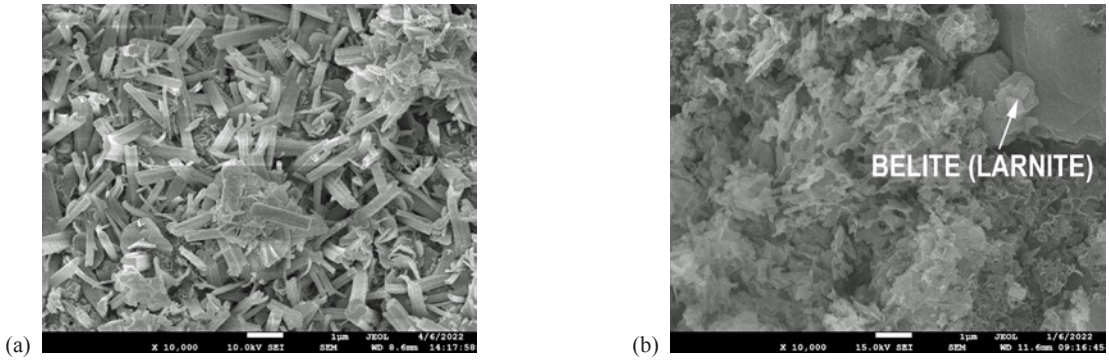


Figure 5. Microstructure of R mortar samples (affected by citric acid): (a) 14 days, (b) 56 days.

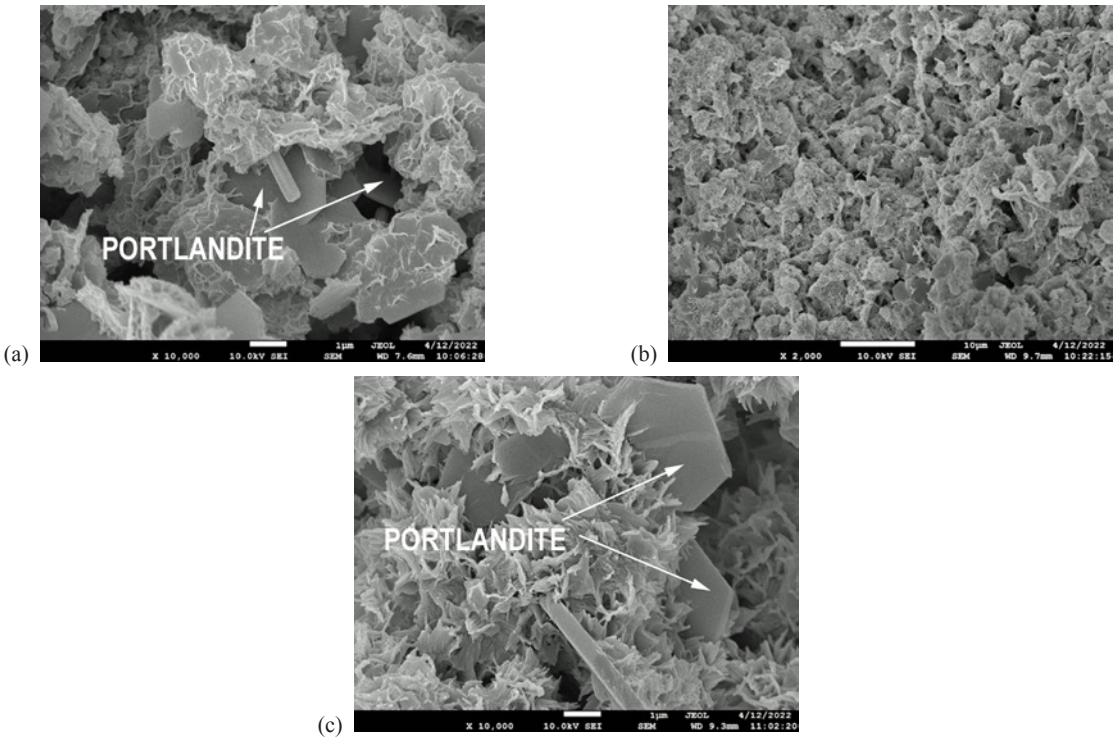


Figure 6. Microstructure of mortar samples A (prolonged mixing process): (a) 7 days, (b) 14 days, (c) 56 days.

4. CONCLUSIONS

The results of a case study analyse the properties of mortars based on natural cement PROMPT (Vicat, France), in which two selected techniques for retarding the setting of fresh mortar were used.

The rapid onset of setting in this type of mortar is a particularly practical problem. Both selected techniques for retarding setting were effective, and both techniques significantly extended the workability time of fresh mortar. However, each of the tested methods has its advantages and disadvantages.

At present, the retardation method with citric acid (TEMPO product, Vicat France) is preferred. As shown in this study, mortars retarded with citric acid have lower initial strengths. In this mortar, the structure and development of the structure of the hydrating mortar are affected. For this reason, it is necessary to respect the specific application requirements, especially for the initial strength of the mortar, and to take into account the dosage of citric acid already when designing the mortar composition. This study shows that at a higher w/c ratio (here w/c = 1) the fresh mortar mixture is very sensitive to the dosage of citric acid and the components separate (blend). In real application conditions, it is usually difficult to accurately dose a small amount of citric acid (only 0.6% of the cement weight) into the mixture. This is a disadvantage for ensuring the quality of the construction product.

The technology of setting retardation by the modification of the mixing process is based on historical experience. As the results of this study show, the initial strength of the mortar is lower. However, the properties of the fresh mortar offer higher application comfort. There is no separation of components (blending), the mortar has an optimal consistency even at a high w/c ratio.

By modifying the mixing process when preparing fresh mortar based on PROMPT natural cement, the workability time can be significantly extended, but the initial strength of the mortar is significantly lower compared to the reference mortar.

The results of SEM analysis and calorimetry in this study do not prove, but only suggest, that both technologies of setting retardation affect hydration and microstructure formation of mortars in a different way. The effect of citric acid on the formation of hydration products has been described (see Chapter 1). However, the mechanism of hydration in mortar retarded by modification of the mixing process is not yet explained and described in this study. On the other hand, the authors will continue further research with the aim of explaining the hydration process. However, it can be hypothesized that in mortars with prolonged mixing, hydration is influenced by the addition of CO_2 to the structure of the mortar.

Abbreviations

C_3S	tricalcium silicate, or alite
C_2S	dicalcium silicate, or belite
C_3A	tricalcium aluminate
C_4AF	tetracalcium aluminoferrite, or brownmillerite
$C_{12}A_7$	dodecacalcium hepta-aluminate, or mayenite
C_4A_3S	anhydrous calcium sulfoaluminate

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Authorship contribution statement

Jana Daňková: Conceptualization, writing, review, validation and research, data curation.
Pavel Mec: Data curation, investigation, formal analysis.
Roman Gabor: Data curation, investigation.
David Bujdoš: Data curation.
Tereza Majstríková: Writing-original draft.
Adéla Valentová: Writing-original draft.
Jiří Šafrata: Data curation.

Declaration of competing interests

The authors of this article declare that they have no financial, professional or personal conflicts of interest that could have inappropriately influenced this work.

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