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Review



# Use of simple non-destructive techniques to evaluate the hydration reactions of PVA-modified cement pastes



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#### HIGHLIGHTS

• Hydration reaction of PVA-modified cement paste is measured.

• Simple non-destructive techniques are used to measure the hydration reaction of cement pastes.

• Adiabatic temperature rise, ultrasound transit time and permittivity decrease are compared.

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#### ABSTRACT

The rapidity of the hydration reaction defines the final properties of cement-based materials. Global techniques show the overall hydration reaction and make it easier for users to evaluate it in a simple nondestructive way, to assess the product and to foresee its final properties. In this paper, three global, simple non-destructive techniques (adiabatic temperature rise, ultrasonic pulse transit time and permittivity increase) showed the same fifth stages in the hydration of two different cement pastes, being able to tell apart their singular hydration reactions. Polymers, such as PVA, are widely used in the construction industry. These affect significantly the hydration reaction of the cement based materials. This paper demonstrates the validity to measure it for PVA-modified cement pastes by means of these techniques giving the building construction industry the chance to use the most available one to evaluate its material hydration reaction as a quality control tool.

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#### 1. Introduction

The hydration reaction of the cement paste and the events associated to it, such as heat generation, strength development and mass contractions, are the result of chemical, physical and mechanical processes. Deeply understanding it is a very important tool to predict the behaviour of the material in the long term [1–2].

Polymer modified mortars are widely used in the ready-mix industry. Polymers combined with cement-based materials generate a synergetic action that improves the cement properties and broaden its applications [3–15]. Polyvinyl alcohol (PVA) is a water-soluble polymer that forms a film in the cement matrix. This film forms during the hydration of the cement based materials behaving as an adhesive to aggregates [4,6] and improving the material performance given its low elasticity modulus and high tensile strength (68.5 MPa and 22.5 MPa, respectively [14]). This is a polymer among many others used in the constructing industry.

It is known that the addition of small amounts of water-soluble polymers to the cement paste can significantly influence in the hydration reaction of the modified cement paste [8,11–13]. This can be a setback when using these materials, then controlling the hydration reaction rate is advisable.

In previous works, the hydrating reaction of cement paste and paste modified with polymers was evaluated by measuring techniques such as temperature rise in semiadiabatic conditions, thermal analysis, infrared spectroscopy and scanning electron microscopy [16–19]. These are only some of many existing techniques to evaluate cement hydration, every time more accurate and analysed with very sophisticate instruments.

In this work, three global, non-destructive and simple techniques are studied: the temperature rise in adiabatic conditions, the transit time of ultrasonic pulses that travels through the cement paste while it hardens and the decrease of the conductivity of the cement paste during the hydration reaction. These techniques are characterized by their robustness and simplicity to perform and to analyse. They have what the building construction industry needs to evaluate the hydration.

Regarding the adiabatic temperature rise, the kinetics and mechanisms of hydration of cement pastes can be investigated satisfactorily by measuring it in adiabatic conditions and with other thermal techniques widely studied in the literature such as isothermal calorimetry, thermogravimetry and differential scanning calorimetry [7,17,20–22]. The adiabatic temperature has the particularity of reflecting the amount of temperature that the cement pastes can reach in a condition where there is no heat loss. This method is very useful to prevent, for example thermal cracking and to design mass concrete [23]. It can also reflect the chemical reactions that occur in the hydrating cement paste illustrating, as well, whether the addition of polymers to the cement paste accelerates or retards the hydration rate [7,16–17,20–22].

The hydration process of cement pastes can also be assessed by measuring the transit time of ultrasonic pulses passing through the matrix under study in a non-destructive way [24–29]. Initially the transit time of the ultrasonic pulse is very close to that of water, since water is the predominant phase. Then the pulse rate begins to diverge. This moment is known as the solid percolation threshold and corresponds to the initial setting time of the cement paste [25–27,30]. Hydration products form a network that prevents the free flow of the water solution in the cement matrix [31]. When this network is formed the hardening of the cement paste begins. The liquid phase ceases to be the predominant phase of the system, which is at this moment, the hydration products structure. The transit time will continue to increase until reaching a maximum value, and then it remains almost constant over time.

transit time value is related to the final elasticity modulus of the sample.

Finally, measuring the conductivity of cement pastes provides a non-destructive technique for monitoring the development of its hydration. This method has the advantage that it can be performed in situ [31–32]. The conductivity monitoring is based on the assumption that a system that goes under physical and chemical changes shows variations in its electrical parameters [31,33–34]. The amount of water of the cement paste and its interaction with the anhydrous cement has a significant influence on the dielectric response [32–33,35]. Similar to the strength, the dielectric properties of cement mixtures still vary over several months after mixing, although the most significant variations will occur within the firsts few days [32]. By monitoring the conductivity, it is possible, with the help of experimental and theoretical correlations, to assess the degree of hydration of cement-based materials [29,32,34–35].

Levita et al. [31] studied the relationship of the loss of connectivity of the macroscopic porosity, which they called "depercolation", with this technique and found a coincidence between it and the Vicat Test. They established that this method was able to lead to a more general comprehension of the evolution of the microstructure of the system that could be incorporated in process and quality control protocols which is an essential requirement nowadays.

The aim of this work is to evaluate the hydration of two cement pastes, pristine cement paste and PVA-modified cement paste, with very different hydration behaviours, by means of three simple non-destructive techniques. To analyse weather these techniques are sensible enough to evaluate and differentiate their hydration and to assess them as quality control tools.

#### 2. Materials and methods

#### 2.1. Materials

Pure portland cement class 50, (CPN 50 according to the Argentinean regulation: IRAM 50000), equivalent to CEM I 52.5, from Loma Negra S.A. was used. Its chemical composition was provided by the company and is detailed in Table 1. It has an ignition loss of 1.67% and a Blaine surface of 442 m<sup>2</sup>/kg.

Poly(vinyl alcohol) (PVA) was used to modified the cement paste. Its commercial name is *Celvol 823* from the company Celanese Chemicals. Celvol 823 is a poly(vinyl acetate) hydrolysed an 87–89%. In Fig. 1 its chemical structure can be appreciate.

PVA was received as pellets. It was dissolved in water at 80 °C by means of stirring. For homogenization, the solution was

Table 1   Chemical composition of CPN50.				
Main phases	%			
C <sub>3</sub> S	61.2			
$C_2S$	13.3			
C <sub>3</sub> A	2.7			
C <sub>4</sub> AF	13.4			
Oxides	%			
CaO	63.53			
SiO <sub>2</sub>	20.71			
Fe <sub>2</sub> O <sub>3</sub>	4.40			
$Al_2O_3$	3.81			
SO <sub>3</sub>	2.87			
MgO	0.79			
K <sub>2</sub> O	0.92			
Na <sub>2</sub> O	0.06			
Loss on ignition	1.67			
Insoluble residue	0.26			

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