

The pozzolanic properties of inorganic and organomodified nano-montmorillonite dispersions

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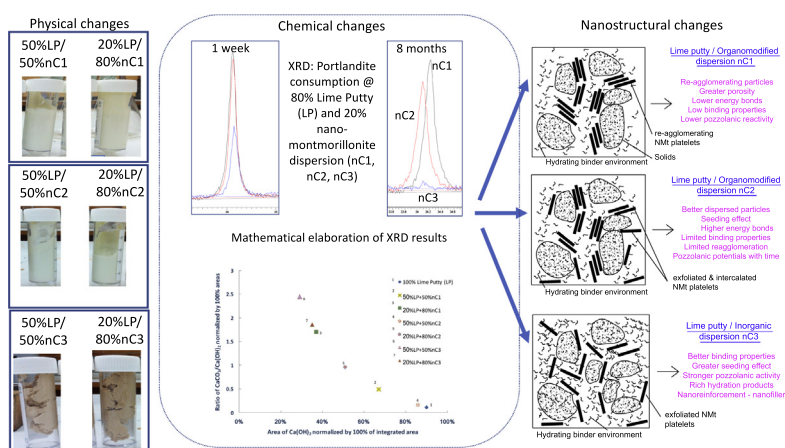
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HIGHLIGHTS

- Chappelle tests were applied to nano-montmorillonite (NMt) dispersions.
- The pozzolanicity of inorganic or organomodified lime putty/NMt pastes was compared.
- A new criterion to assess the pozzolanic potential of NMt dispersions was devised.
- Inorganic NMt dispersions offer solutions for the production of sustainable binders.
- Level of exfoliation of NMt affects pozzolanicity.

GRAPHICAL ABSTRACT



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ABSTRACT

The pozzolanic potentials of three non-thermally treated, nano-montmorillonite (NMt) dispersions were investigated by a new method involving the analysis of NMt/lime putty pastes via TGA/dTG and XRD crystallographic and semi-quantitative analysis. The criterion conceived was validated at eight days and eight months and was additionally verified via the Chappelle method. The inorganic NMt dispersion showed the most pronounced pozzolanic behaviour promoting Ca(OH)₂ consumption towards calcium silicate/aluminate hydrates formation and binding behaviour. The two organomodified NMt dispersions exhibited pozzolanicity increasing with time. The results can pave the way for advances in cement science and restoration mortars development for historical structures rehabilitation, where low CO₂-footprint, natural inorganic materials are a prerequisite.

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1. Introduction

Montmorillonite (Mt) is one of the main minerals found in bentonites, a naturally occurring clay. Mt is rich in stacks of

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layers/platelets held together by interlayer cations, by van der Waals forces, by electrostatic force or by hydrogen bonding inhibiting the solubility or miscibility [1]. Moreover, Mt is a 2:1 layered silicate, implying that each layer is composed of two silicon tetrahedral sheets bonding with one octahedral sheet of alumina between them. Isomorphous substitution takes place in the octahedral sheet where most hydroxyl groups are located apart from the broken sides of each particle [2]. Surface modifiers, such as quaternary ammonium salts (quats) inserted in the interlayer space can fully separate these platelets (exfoliation or if at a lower extent, intercalation), creating individual nano-thick, plate-like particles. These particles, hereafter referred to as nanomontmorillonite (NMt), can engage in chemical reactions analogous to their specific surface area.

Apart from these organic modifiers, which usually cause platelet separation by cation exchange, and produce a pulverized product, Mt can be exfoliated in water in its pristine condition. In fact, even in earlier research focused on polymer-clay nanocomposites it has been stated that apart from organomodification, small additions of water were adequate for clay exfoliation [3]. Therefore, if Mt is dispersed in water, it can maintain its inorganic nature and platelets can remain dispersed with the help of inorganic surfactants. Inorganic surfactants promote the homogenous dispersion of the NMt platelets in the aqueous environment. NMt platelets dispersed in water are easier to handle compared to those in powder form, because agglomeration of particles into clusters (usually of micron scale or bigger) is avoided. In addition, NMt dispersed in water, provides greater miscibility with other cementing constituents.

The nanoengineering process of Mt layer separation and the NMt products have attracted the attention of cement scientists who seek to use the exfoliated NMt particles as a means of additional nucleation sites or nano-scale reinforcement in the hydrating cement matrix [4]. The nature, nanostructure, production methods, effect of modifiers and dispersants and the configurations of the NMt platelet separation for use as polymer nanocomposites or adsorption materials can be found in the literature [3,5–7]. However, limited research is presented on the use of NMt and this research is applied in cement binder formulations [4,8–10].

Supplementary cementitious materials (SCM) used in lime or cement mortars can be classified as (i) pozzolanic (ii) latent-hydraulic or (iii) fillers. Pozzolanic SCM are the most common. A pozzolanic SCM consists of a material rich in amorphous alumina or silica that is non-reactive with other compounds and water as is to form additional hydration products. However, in the alkaline medium created by the dissolution of calcium hydroxide in water the silicate or aluminosilicate networks break down to form calcium silicate and/or calcium aluminate hydrates. The relative pozzolanicity of a material depends on a high content of amorphous phases and a high specific surface area.

With respect to pozzolanic studies on clays, at present only the pozzolanic activity of thermally and mechanically treated kaolin [2,11,12] or halloysite nanoclay particles [13] has been confirmed. There is disagreement on the pozzolanic potential of Mt with some researchers stating that calcined Mt mineral exhibits limited pozzolanic activity depending on the calcination temperature [2], while others claim that natural and calcined Mt contribute to pozzolanic reactions [14]. That is to say, Mt so far has only been investigated in its calcined form. However, the high temperatures involved for calcinations increase the CO₂ footprint of the material. With the evolution of nanotechnology, which allows us to manipulate matter at levels slightly above the atomic, another option rather than calcination arises; the nanomodification of Mt, i.e. the breaking of the forces holding its platelets together so that the nanoplatelets will then be individually available for reactions,

as explained above. With respect to NMt, the incorporation of Cloisite®30B to waste glass powder cement mortars exhibited enhanced pozzolanic reactivity leading to improved mechanical properties [15]. In another study the incorporation of Cloisite®30B to ordinary Portland cement showed pozzolanic potentials [16]. Still, one element of the process is to nanomodify the Mt and another part is to disperse it in water, so as to render it more compatible with binders, more easily usable with higher potentials for mass production. So far, only the authors' team has presented research on the effect of various NMt dispersions in the hydrating cement paste [4,8–10] and of this work only part of it has proven the pozzolanic potentials of the inorganic NMt dispersion in ternary Portland cement-limestone binders [8]. Therefore, although the pozzolanic contribution of NMt in cement binders has been confirmed, the pozzolanic behaviour of NMt as a raw material is yet to be scrutinized. However, given the complex nature of NMt dispersions, containing not only Mt but also modifiers and dispersing agents, a criterion taking into account the decomposition of various components within the same temperature intervals is yet to be presented. This elaborate research will provide sound calculations of pozzolanicity in more intricate matrices such as those of NMt enhanced cement binders.

The currently widely used pozzolanic additions such as fly ash, are being depleted [17] and others such as silica fume are difficult to handle and increase the total cost of the binder. The abundance of bentonite in nature, from which Mt can be received, and the ease of Mt exfoliation in water in the case of inorganic dispersions [9,10], make NMt a potentially interesting alternative SCM. Furthermore, the reaction of the exfoliated platelets around hydrated Portland cement particles can enable a tortuous microcrack propagation pattern, hence providing nanoreinforcement at the nanolevel to the microlevel, as confirmed by a series of experiments [10]. Other clays, such as metakaolin, get activated by heating above 700 °C, increasing the embodied CO₂ of the binder. Therefore, avoiding calcination and nanomodifying Mt, comprises a way of producing sustainable low carbon additions for the future. Given that the filler effect of Mt is confirmed [2], what remains to be assessed is the pozzolanic effect of NMt dispersions, which are easier to handle than their powder counterparts, however exhibit an inherent difficulty because of the presence of the modifier and surfactant.

The depletion of natural and man-made pozzolanas and the need to lower the carbon dioxide footprint by avoiding pozzolanas produced by calcination, is calling for new materials and methods to be developed. From the above, it can be established that neither the pozzolanic activity of non-calcined NMt nor the pozzolanicity of non-calcined NMt dispersions has been examined previously. Such a discovery could be proven valuable for cement science, because it will allow the use of NMt dispersions, whose main constituent, montmorillonite, is abundant in nature and environmentally friendly, as SCM, avoiding the otherwise added environmental cost of calcination. Therefore, this research programme was designed to investigate the pozzolanic properties of two different aqueous organomodified NMt dispersions and one aqueous inorganic NMt dispersion. Characterization techniques for this standalone methodology of assessing the pozzolanic behaviour only necessitated: thermal gravimetry analysis and differential thermogravimetry (TGA/dTG), X-ray diffraction (XRD) crystallography and Semi-quantitative analysis based on XRD at 8 days and 8 months.

The application of this knowledge is suited for the characterization of NMt as a potential pozzolanic SCM can open a new horizon for the understanding of the effect and further development of nanoclays and NMt in cementitious composites and lime binders. Lastly, this study can further be elaborated for the development of new binders for cultural heritage conservation, referred to in detail in the discussion.

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