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Alkaline activation as an alternative method for the consolidation of earthen architecture



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ABSTRACT

The majority of historic buildings and archaeological remains made of earth exhibit important conservation problems, which require consolidation. Most conventional consolidation treatments used in the past have not succeeded in providing a long-term solution because they did not tackle the main cause of degradation, the expansion and contraction of constituent clay minerals in response to humidity changes. Clay swelling could be reduced significantly by transforming clay minerals into non-expandable binding materials with cementing capacity using alkaline activation. It is demonstrated that a significant degree of clay mineral dissolution and transformation is achieved upon activation with 5 M NaOH and 5 M KOH solutions. Furthermore, adobe test blocks impregnated with either 5 M NaOH or 5 M KOH solution experience an important improvement in water resistance and mechanical strength. The influence of structural and compositional differences of clay minerals commonly present in earthen structures on their reactivity under high pH conditions and the consolidation efficacy of the various alkaline solutions are discussed. Also addressed are possible side effects of the alkaline treatment such as colour change and the formation of potentially damaging salts.

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1. Introduction

Earth has been used as a building material since ancient times and examples of earthen architecture can be found in many parts of the world. Famous examples include Chan-Chan in Peru, Shibān in Yemen, the Rebkung monastery in Tibet and the Alhambra in Spain. In dry climates, earthen structures are in general well preserved with little maintenance. In more humid regions, however, deterioration is a common problem and consolidation treatments are required to ensure the survival of important monuments or archaeological sites. Especially, the action of water of different sources (rain, condensation, capillary rise) can cause severe damage to earthen architecture including cracks and granular disintegration or may even result in the total loss of the structure [1,2].

The susceptibility of earthen structures towards water is a result of the presence of clays in soils used for construction. Clays act as a binder for other constituents such as sand and silt. However, changes in the structure's moisture content cause swelling and contraction of the clay minerals, which in turn results in the above mentioned damage phenomena. Especially deleterious are

expandable clays such as smectites or mixed-layer clays, which are commonly found in earth used for construction [3].

Conventional consolidation treatments using synthetic polymers or alkoxysilanes had, often, only limited success and, occasionally, led to a total failure due to esthetical, chemical or physico-mechanical incompatibility. Sometimes the applied product simply does not penetrate sufficiently into the earthen material and only consolidates a superficial outer layer, which typically detaches and falls off. Apart from limited penetration, conventional consolidation treatments do generally not tackle the cause of the problem, the expansion and contraction of the clay minerals, but rather only diminish the alteration effects, thus being ineffective in the long-term [4].

The above shows the urgent need for the development of an alternative, more effective method that allows the *in situ* consolidation of earthen architecture by reducing the swelling capacity of clays. The swelling capacity could be reduced by transforming clays into non-expandable binding materials such as calcium silicate hydrates or aluminosilicates with cementing capacity, using alkaline activation. Ca(OH)₂, NaOH or KOH might be suitable as alkaline activators.

Lime, Ca(OH)₂, has been used since ancient times as a stabilizer for earthen architecture. Ca(OH)₂ provokes pozzolanic reactions at high pH (> 12) at which clay minerals or other aluminosilicates are partially dissolved and transformed into calcium silicate hydrate

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(CSH), calcium aluminate hydrate (CAH) and/or zeolite-like phases. These phases have been recognized in antique roman mortars [5]. They can act as cementing agents and improve the durability and mechanical strength of earthen architecture. This is demonstrated by the soundness and resistance of ancient lime-amended rammed earth structures such as the Alhambra fortress [6].

NaOH and KOH might also be suitable for the alkaline activation of aluminosilicates. Both have been widely applied in the synthesis of zeolites since the mid-1930s [7]. Al and Si sources for zeolite synthesis include clay minerals and feldspathoids [8]. However, zeolite synthesis takes generally place at relatively high T ($\geq 60^\circ\text{C}$) and P (autoclave), reaction conditions which are not applicable in architectural conservation interventions.

More recently the geochemistry that yields the synthesis of zeolites has been applied for the production of alkaline activated cements, which are termed geopolymers [9], low-temperature aluminosilicate glass [10] or hydroceramics [11]. Often comparisons are drawn between zeolite chemistry and geopolymer chemistry due to the similarities between the two systems, and the geopolymeric gel phase is frequently described as a zeolitic precursor [12]. Commonly, alkaline activated cements are cured at T between 60 – 90°C [10,11].

The main objective of this study is the evaluation of alkaline activation as an alternative consolidation treatment for clayey material such as rammed earth or adobe. Consolidation treatments for earthen architecture are generally applied at room T . Thus, an important aspect of this study is to determine the extent of mineral transformation under high pH conditions at room T . The evaluation will also have to consider the efficacy of different activator solutions, e.g. $\text{Ca}(\text{OH})_2$, NaOH and KOH. In the preparation of alkali-activated cements and in the synthesis of zeolites, clay minerals are directly mixed with the alkaline solution. In contrast, alkaline activation in a conservation treatment has to be performed by impregnation with the alkaline solution, as it is commonly done when applying conventional consolidants. Thus, it has to be determined whether an adequate penetration depth can be achieved using alkaline solutions and whether the alkaline solution concentration is sufficiently high to promote mineral transformation and stabilization. The suitability and performance of this consolidation method as an *in situ* treatment is evaluated here by applying alkaline solutions to adobe test blocks. The ultimate goal of this research is to determine whether alkaline treatments can improve the water resistance and mechanical properties of earthen materials. Furthermore, the damage potential of salts, which might form during alkaline activation will be assessed.

2. Materials and methods

2.1. Materials and sample preparation

For our study we chose the soil from the hills close to the city of Granada (Alhambra Formation), which was historically used as a construction material for rammed earth structures [6,13]. In order to evaluate the reactivity of this soil, 5 g of powdered clay ($< 2\ \mu\text{m}$ fraction separated using centrifugation) was subjected to alkaline activation using 100 mL of the following solutions:

- 0.025 M $\text{Ca}(\text{OH})_2$, this concentration corresponds to a saturated solution at room T ;
- 5 M NaOH;
- 5 M KOH.

Two batches were prepared for each alkaline solution. The clay samples were kept in tightly capped polypropylene bottles and stored in the laboratory at 20°C . Bottles were stirred periodically

and samples for analysis were taken at predetermined time intervals (1 week, 1, 2.5, 4, 6 months, 1 year).

Additionally, adobe test blocks (size: $4 \times 4 \times 4\ \text{cm}$) were prepared from the $< 1\ \text{mm}$ fraction of the Alhambra Formation soil (soil/water mass ratio = 3/1) using wooden molds. The blocks were treated by impregnation with the various alkaline solutions (6 blocks per solution) during 20 minutes and cured in sealed plastic bags for 50 days as a proxy to an *in situ* treatment. Afterwards, the bags were opened to facilitate slow drying at $\sim 20^\circ\text{C}$ and $\sim 45\%$ RH until a stable weight was reached after ~ 40 days. Note that during the drying phase, adobe blocks were exposed to atmospheric CO_2 . Subsequently, they were submitted to further testing in order to evaluate the efficiency of the treatment.

The interactions of highly concentrated alkaline solutions with clay minerals are manifold and may include:

- the exchange of cations in the clay minerals for the dominant cation in the alkaline solution [14,15];
- flocculation induced by highly concentrated alkaline solution which results in decreased osmotic and intracrystalline swelling of clays, depending on the electrolyte concentration [16];
- clay mineral dissolution and transformation [17].

In order to determine the main causes leading to modifications in the blocks water resistance and mechanical strength upon alkaline activation, additional adobe samples were treated with saturated (4.65 M) KCl solution following the same protocol as in the case of the alkaline solution. Whereas cation exchange and flocculation are facilitated by a treatment with KCl, mineral dissolution and transformation will not occur due to the neutral pH of the KCl solution.

Furthermore, a salt crystallization study was performed in order to obtain a ranking of the damage potential of salts, which might form during alkaline activation. For this test, calcarenite blocks ($3 \times 3 \times 25\ \text{cm}$) from Santa Pudia, a quarry in the province of Granada, were used. This limestone has a known high susceptibility to salt damage. Adobe blocks could not be used in this test due to their poor water resistance. The porosity of the limestone ($27.7 \pm 0.1\%$) is practically identical to the one of the treated adobe blocks ($27.6 \pm 0.9\%$). However, the volume of pores with a diameter $< 1\ \mu\text{m}$, which is decisive in producing salt damage [18], is slightly higher in the treated adobe blocks ($\sim 79\%$) than in the limestone ($\sim 63\%$). Thus, the damage patterns observed in the limestone might not be identical in adobe which is assumed to have a higher susceptibility to salt damage as a result of the lower mechanical resistance and the higher volume of small pores. However, the ranking of the salts damage potential is not likely being influenced by differences between both porous materials.

The stone blocks were partially immersed in saturated salt solution. The surface of the salt solutions was covered with paraffin in order to promote the migration of the salt solution through the pore system of the stone and to avoid creeping and excessive solution evaporation. The salt solutions included here were Na_2CO_3 , K_2CO_3 and KHCO_3 . These carbonates might form upon alkaline activation. Their damaging potential was evaluated by visual observations and by the determination of material loss as well as of the amount of salt crystallized as efflorescence and/or subflorescence.

2.2. Analytical methods

The chemical composition of the clay fraction and the $< 1\ \text{mm}$ fraction of the untreated soil was determined using a commercial wavelength dispersive X-ray fluorescence spectrometer (BRUKER S4 Pioneer).

The mineralogical evolution was studied with X-ray diffraction (XRD) using a X'Pert PRO (PANalytical B.V.) equipped with

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