Temporal evolution of dry-mixed soft sensitive clays

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1. Introduction

Supplementary Cementitious Materials (SCMs) have gained prominence in recent years, as sustainable partial replacements to conventional lime and cement [1]. Among these SCMs, calcined clays have attracted considerable attention due to their abundant availability and potential reactivity [2]. Despite their growing use in structural concrete, the potential of these calcined clays in ground improvement techniques such as dry soil mixing remains largely unexplored.

During application of dry soil mixing in engineering practice, lime and cement are incorporated into soft clays as dry powders [3] and are commonly added in a 50:50 ratio [4]. Although introduced simultaneously, the reaction mechanisms of lime and cement differ significantly. Cement hydration proceeds rapidly through primary clinker reactions, forming early strength providing C–S–H [5], whereas lime stabilisation depends on slower pozzolanic reactions with clay minerals, resulting in gradual strength development [6]. The addition of calcined clays further modifies the system through a multi-stage process: initially acting as fillers, then providing nucleation sites that accelerate hydration, and subsequently participating in pozzolanic reactions with portlandite to form additional cementitious products [1]. The rate of these reactions is further influenced by factors such as binder composition, curing conditions, homogeneity of mixing and dosage [7]. As a result, the overall hydration and microstructural development of clay–binder systems are governed by a combination of physical and chemical processes that evolve progressively during curing.

Conventional laboratory testing methods are often insufficient to simultaneously capture the evolution of micro- and macro-scale changes during curing alongside strength development with minimum sample preparation. To address these limitations, this study employs a combination of Electrical Impedance Spectroscopy (EIS), which is sensitive to microstructural changes during curing, and miniature Cone Penetration Tests (mCPT), which assess the resulting mechanical response at the laboratory scale (deci-meter) at the end of the curing period, for calcined clay–based binder systems stabilising natural clay.

2. Materials and sample preparation

The natural clay used in this study was obtained from Kärra region of Gothenburg with water content of approximately 80%. Cement and lime used in this study were CEM 1 and slaked lime powder ($Ca(OH)_2$) respectively. Calcined clay used in this study was calcined kaolinite (Speswhite kaolinite activated at 800°C). A constant total binder content of 80 kg/m³ was used in all mixes.

Samples for EIS and mCPT were prepared by mixing natural clay with varying proportions of lime, cement, and calcined clay as seen as in Figure 1 (represented by blue dots in the ternary diagram). The mixes were then cast into cylindrical plastic rings with a diameter of 50 mm and a height of 20 mm. After casting, the samples were sealed with end caps to prevent moisture loss and left to cure until the required curing age. EIS was tested on 14, 28, 36 and 56 days while the strength response was measured using the mCPT at the end of 56 days on the same sample.

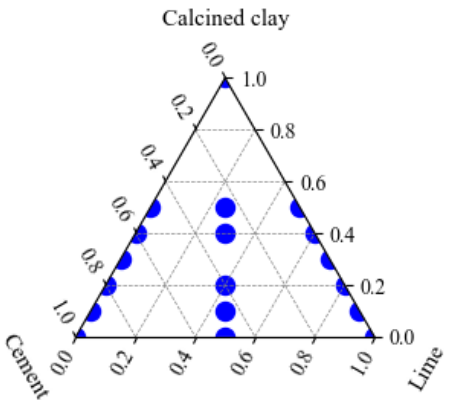

Figure 1. Binder compositions studied using EIS and mCPT.

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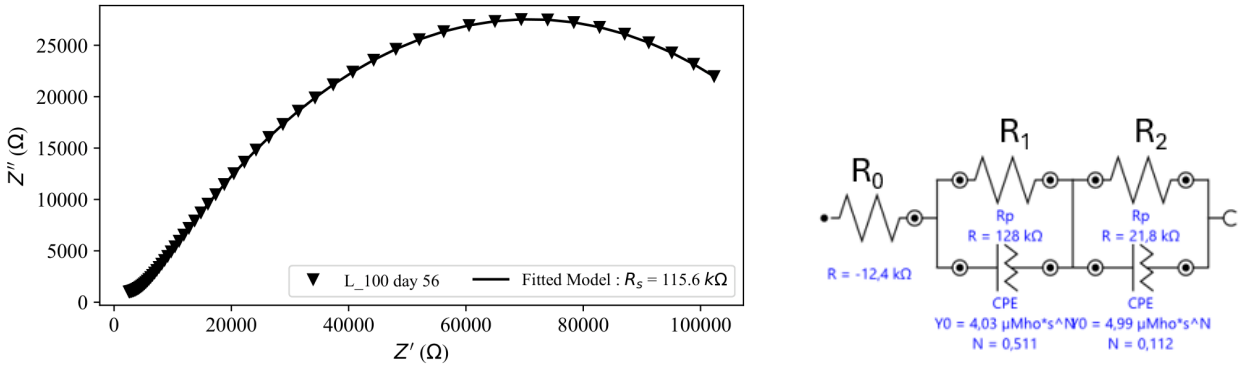
3. Methods

3.1. Electrochemical impedance spectroscopy (EIS)

Electrochemical Impedance Spectroscopy (EIS) was performed using a two-electrode configuration with an electrode area ratio of 1:5 (counter electrode : working electrode), fabricated from printed circuit boards (PCB) with exposed gold-plated surfaces. A sinusoidal voltage of 1 V was applied across the specimen using a Metrohm PGSTAT204 potentiostat equipped with a FRA32M EIS board, and the corresponding impedance response was measured over a frequency range of 0.1 to 1 × 10^5 Hz. The resulting impedance data could be represented in a Nyquist plot, in which the imaginary component of the impedance (Z″) is plotted against the real component (Z′) to visualise the electrochemical response of the material. Here the real part Z′ denotes the resistive component associated with the ionic mobility while the imaginary part denotes the capacitive component associated with solid–liquid interfaces and microstructural development during hydration.

The Nyquist plots were fitted using an equivalent circuit model as suggested by Christensen *et.al.* [8] for hydrating cement pastes (Figure 2b). The quality of the fit was evaluated using the chi-square (χ^2) criterion, where the acceptable fits satisfied the condition $\chi^2 < 0.2$. Figure 2 presents the Nyquist plot and the corresponding fitted circuit for sample L100, in which natural clay was mixed with 80 kg/m³ of lime and cured for 56 days. According to Christensen *et.al.* [8], the bulk electrical resistance R_s of the mixes, determined as $R_0 + R_2$, is the most representative parameter for monitoring microstructural evolution in cement pastes. An increase in bulk resistance was observed with curing time. This increase is attributed to the combined effects of pore water consumption and the progressive blocking of capillary pores due to the formation and deposition of calcium silicate hydrate (C–S–H) during binder hydration.



(a) (b)

Figure 2. (a) Example of Nyquist plots with fitted equivalent circuit models at 56 days of curing for mix L100 and (b) equivalent circuit fitting model [8].

3.2. Miniature cone penetration test (mCPT)

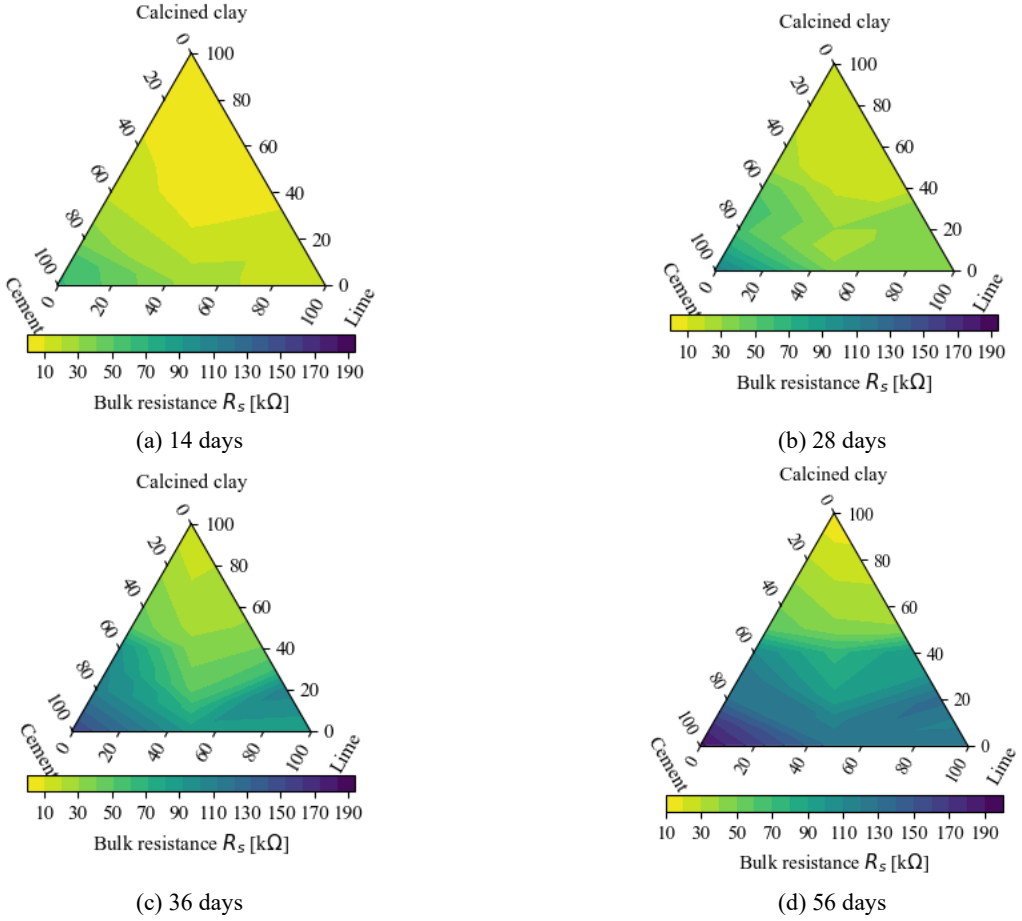
Miniature Cone Penetration Tests (mCPT) were employed as a simple and quick means of assessing strength variations associated with different binder compositions at the end of curing at 56 days. The actuator of the mCPT setup consisted of modified components from a Nemesys mid-pressure pump. A stainless-steel needle probe, attached to the actuator, with a 60° cone tip angle and a base diameter of 2 mm was used for the tests. The load response was measured using a load cell of 100 ± 1 N capacity and the displacement was measured using external LVDTs (resolution 0.001 mm). Penetration was performed at a constant rate of 1 mm/s. The peak force recorded at a displacement of 4 mm (F) was used to calculate the peak cone resistance ($q_{c,peak}$), which was calculated as F/A where A represents the cross-sectional area of the needle probe. This penetration depth of 4 mm was selected to enable repeatable measurements from both sides of the sample, thereby accounting for the inherent heterogeneity of dry-mixed clays, while minimising potential boundary effects.

Each specimen was tested at four locations arranged symmetrically at 90° intervals within the 50 mm diameter sample on each side of the sample. The centre-to-centre spacing between adjacent test points, as well as the distance from the specimen edge to the nearest test point, was approximately 16.65 mm. This spacing was selected as an intermediate value based on scaling considerations for clays and sands to minimise boundary effects [9], as specific guidelines for dry-mixed clays are not currently available. The penetration resistance ($q_{c,peak}$) reported for each specimen represents the average of the eight measurements (4 on each side).

4. Results

Figure 3 presents the evolution of bulk electrical resistance R_s from EIS at 14, 28, and 56 days in the form of ternary plots. For mixes with varying proportions of calcined clay (points along the left axis), the resistance increases more rapidly with curing time. However, by 56 days, the lime control samples (points along the right axis) exhibit comparable resistance

values, suggesting similar degrees of microstructural densification, particularly in terms of C–S–H formation and pore refinement at later ages.



(a) 14 days (b) 28 days

(c) 36 days (d) 56 days

Figure 3. Evolution of bulk electrical resistance R_s at: (a) 14, (b) 28, (c) 36 (d) 56 days

Furthermore, at 56 days, the resistance contour corresponding to 30% replacement in the cement control mixes and 20% replacement in the lime control mixes aligns closely with that of the reference lime–cement (50:50) mix, indicating comparable microstructural development. This suggests that up to 30% of the cement in cement control mixes and 20% of the lime in lime control mixes can be replaced by calcined clay without compromising the degree of microstructural development achieved at 56 days of curing in a reference mix.

Figure 4 shows the ternary plot of the penetration resistance at 56 days from mCPT. The mechanical response in terms of the penetration resistance $q_{c,peak}$. The correspondence between electrical and mechanical responses under sealed curing conditions, indicates that progressive hydration resulting in reduced ionic mobility and increased matrix densification can be directly linked to strength development.

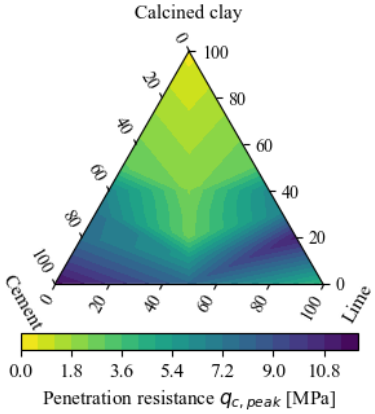


Figure 4. Ternary plot of penetration resistance q at 56 days.

5. Conclusion

Electrochemical Impedance Spectroscopy (EIS) and miniature Cone Penetration Tests (mCPT) have shown to be useful complementary methods for monitoring material scale (nm - μm) as well as laboratory scale (dm) evolution of properties in stabilised soft clays. EIS provides non-destructive insights into hydration and changes in pore structure and ionic conductivity through variations in electrical response, while mCPT captures the corresponding evolution of strength with different compositions of binders. Together, these techniques offer a robust framework for assessing the performance of conventional and alternative binders and for optimising mix designs in ground improvement.

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