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Extended ICL study for lime stabilisation: Navigating pH changes over prolonged time and differentiating the effects of lime aging

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ABSTRACT: This study extends the conventional Initial Consumption of Lime (ICL) test methodology to investigate lime requirements for the stabilisation of Mercia mudstone and London clay soils. This would address the limitations of standard ICL test, such as oversimplified assumptions, reliance on short reaction times, and the inability to account for soil heterogeneity and lime aging. The extended approach incorporates curing durations of up to 240-hours at a standard temperature of 25°C, alternative target pH, varying lime content from 0.25% to 6%, and the effects of lime aging and mixing techniques. The incorporation of aged lime samples addressed the impact of reduced lime reactivity on stabilisation efficiency. Based on the experimental findings, London clay exhibited a clear decreasing trend in pH after the first hour with increasing lime content with a slower pH reduction over time at higher doses, whereas Mercia mudstone showed a less consistent pattern. Notably, a consistent trend of reduced pH values associated with aged lime across both soils further emphasizes the impact of lime ageing. The findings highlight the need for improved methods beyond pH based criteria for reliable lime content determination.

1 INTRODUCTION

Soil stabilisation improves engineering properties of soil by increasing pH and triggering two reaction stages. Immediate reactions, occurring within hours, involve cation exchange and flocculation, reducing plasticity and improving workability. Long-term pozzolanic reactions form cementitious compounds such as calcium-silicate-hydrates (CSH) and calcium-aluminate-hydrates (CAH), enhancing strength and durability over time (Herrin and Mitchell, 1961; Eades and Grim, 1966; Bell, 1996; Beetham, 2015). These reactions depend on the mineral composition of the soil, with montmorillonite being highly reactive to lime and kaolinitic clays exhibiting lower reactivity (Bell 1996).

The increase in soil pH due to lime addition enhanced the solubility of siliceous and aluminous compounds, facilitating the disintegration of clay particles. The resulting cementitious matrix transformed the soil into a rigid, impermeable layer with a high load bearing capacity. This transformation begins within few hours of lime addition and continues for years, progressively enhancing the mechanical properties of soil (Petry and Lee 1988; Amadi and Okeiyi 2017; Eades and Grim. 1966). Since both immediate and long-term reactions depend on adequate lime availability to sustain the underlying chemical processes, determining the optimum lime content (OLC) is a key factor for soil stabilisation. The Initial Consumption of Lime (ICL) test developed by Eades and Grim (1966) for determining optimum lime content (OLC) required for stabilisation was adopted by American Society for Testing and Materials (ASTM D6276, 2019). While the ICL is quick and easy to perform, it focuses on short term pH changes, potentially overlooking factors such as, pH fluctuations, longer reaction time, and durability of the final product (Rogers, Glendinning and Roff, 1997; Cherian and Arnepalli, 2015; Akula, Naik and Little, 2021).

This study aims to extend the conventional ICL test by investigating pH changes over an extended period, mixing method, and distinguishing between the effects of lime aging. By examining these factors, this study focuses on enhancing the understanding and accuracy

of lime content determination method (ICL) for soil stabilisation.

2 LITERATURE REVIEW

The pH of soil is one of the most important factors that controls the process of lime stabilisation. A highly alkaline environment is required to dissolve reactive silica and alumina from clay minerals, which then participate in pozzolanic reactions responsible for improving the strength and stiffness of soil (Eades and Grim, 1966). When lime is added, the dissociation of hydroxyl ions increases the pH of the soil solution. This promotes cation exchange, flocculation, and the rearrangement of soil particles, which in turn reduces plasticity and improves compaction characteristics (Khattab, Al-Mukhtar and Fleureau, 2007; Cherian and Arnepalli, 2015).

The reactivity of lime with soil minerals is largely dependent on pH. At higher pH values, silica and alumina dissolve more effectively and allow the formation of cementitious calcium-silicate-hydrate (C-S-H) gels, which bind particles together and improve the mechanical properties of the soil (Cherian and Arnepalli, 2015). However, in acidic soils the reaction is less efficient, as part of the lime is consumed in neutralising acidity before pozzolanic activity can occur. As a result, higher amounts of lime are required, yet the overall strength gain is often lower (Ghobadi, Abdilor and Babazadeh, 2014). On the other hand, if the pH becomes excessively high, problems of over-stabilisation may develop, such as shourinkage, reduced permeability, and a decline in long-term durability (de Brito Galvão, Elsharief and Simões, 2004; Ghobadi, Abdilor and Babazadeh, 2014).

To determine the required amount of lime for stabilisation, the Initial Consumption of Lime (ICL) test developed Eades and Grim (1966) is widely used. This test identifies the minimum lime content needed to achieve a pH of 12.4 in a soil-lime-water system, which is considered sufficient for pozzolanic reactions to proceed. The ICL method has become popular due to its simplicity, rapid procedure, and inclusion in standard specifications (ASTM D6276, 2019).

However, the method has also been the subject of criticism. One major limitation of the ICL test is the assumption that cation exchange is complete within 1-hour (Cambi *et al.*, 2016). In practice, soils often require more time for lime reactions to develop, particularly those with complex mineralogy or high organic matter (Cherian and Arnepalli, 2015). As a result, the test may underestimate lime demand. Researchers have noted that short reaction times are not sufficient to capture soil–lime interactions in full, which questions the accuracy of results obtained using the standard procedure (Rogers and Glendinning, 2000; Beetham, 2015).

Moreover, the test does not account for the wide variability found in natural soils, including differences in mineralogy, initial pH, and heterogeneity, all of which influence lime reactivity (Cambi *et al.*, 2016). Another drawback is that the ICL test focuses only on immediate chemical behaviour and does not consider long-term durability. In field conditions, stabilised soils are subjected to cyclic wetting and drying, freeze–thaw cycles, and variable curing environments, which have a major effect on strength and performance (Sherwood, 1993; Hebib and Farrell, 2017). Lime slaking over time can also alter its reactivity, further reducing the reliability of 1-hour predictions (Rogers, Glendinning and Roff, 1997).

Despite these limitations, the ICL test remains a key method for lime stabilisation because of its practicality and ease of use. However, Rogers, Glendinning and Roff (1997) recommended that the test be continued until the pH stabilises and gets constant, recognising that some soils require longer reaction times to reach equilibrium. Subsequent studies, such as Cambi et al. (2016) have highlighted the need to adapt the procedure for different soil types, while other research by Beetham (2015) has shown that factors like reaction time and soil mineralogy can significantly influence lime–soil interactions. Together, these findings suggest that, while the ICL test has greatly contributed to lime stabilisation practice, its shortcomings highlight the need for further refinement to ensure reliable predictions of both short-term and long-term performance.

3 MATERIALS AND METHODS

3.1 Materials

Two clay soils, Mercia mudstone and London clay were selected for experimentation. Particle size distribution testing (Figure 1) was performed in accordance with (BS 1377:2, 1990) using dry sieving and wet washing. The Mercia Mudstone sample is a well-graded soil with <5% of particles finer than 0.01 mm, whereas the London Clay sample is dominated with 98% of particles finer than 0.01 mm, resulting in very low permeability and high plasticity.

Mercia mudstone comprises mixed-layer clay minerals (illite, chlorite, illite-smectite), quartz, feldspars, carbonates and evaporites (Hobbs *et al.*, 2002), whereas London clay contains smectite, illite, kaolinite, chlorite, quartz, feldspars, carbonates, pyrite and minor marine-derived evaporites (Kemp and Wagner, 2006).

Quicklime supplied by Tarmac UK was used in two forms: fresh quicklime (FQL), acquired recently, and aged quicklime (AQL), which had been stored in the laboratory for more than three years in airtight containers. A simple lime reactivity test, as outlined by Dathathreya and Rama Rao (1997), confirmed that FQL showed rapid hydration and heat release, while AQL exhibited a slower response due to reduced reactivity over time (Figure 2). The soil properties were tested according to (BS 1377:2, 1990) (Table 1).

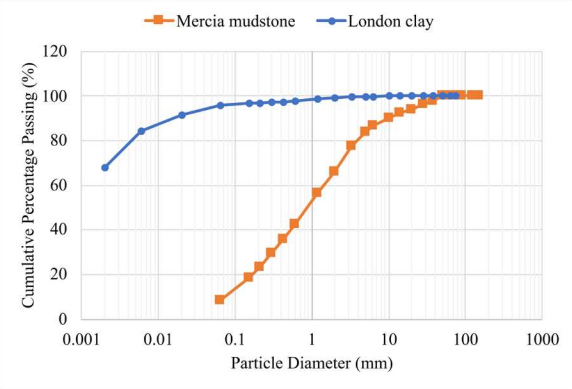

Figure 1 Particle size distribution curve of tested soils

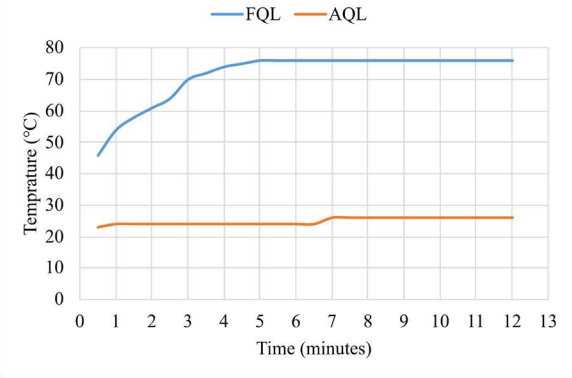

Figure 2. Rate of temperature increase over time of FQL and AQL

Table 1 Properties of the used soils.

Property	**Mercia Mudstone**	**London Clay**
Liquid Limit %	32.00	71.00
Plastic Limit %	16.50	27.00
Plasticity Index %	15.50	44.00
OMC %	12.30	26.00
MDD Mg/m^3	1.95	1.52
Organic Matter %	2.05	0.75
Particle Density Mg/m^3	2.66	2.72
Initial pH without lime	6.95	7.95

3.2 Extended ICL Framework

The extended Initial Consumption of Lime (ICL) test was designed to address criticisms of the standard method, including the short 1-hour duration and assumptions of complete cation exchange. In this study, soil–lime mixtures were monitored over 240-hours to observe pH evolution and better capture the progression of pozzolanic reactions. A target pH of 10 was chosen, as pozzolanic activity can occur below the conventional 12.4 threshold (Diamond and Kinter, 1965). (Figure 3) presents the framework of the experimental plan.

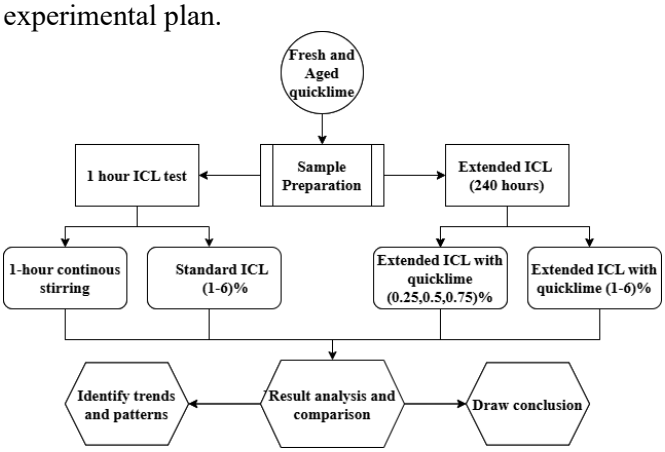

Figure 3. Extended ICL experimental plan

3.3 Experimental Procedure

Samples were prepared with quicklime concentrations ranging from 0.25% to 6%, using both FQL and AQL and (Figure 4). For each soil type, 28 samples were tested to determine the extended Initial Consumption of Lime (ICL). Additionally, 22 samples were prepared for both soils to compare the effect of continuous stirring for 1-hour versus shaking on pH. Each test used 20 g of soil below 25 micrometre size. Quicklime was thoroughly mixed with the soil in the dry state to ensure homogeneity, followed by the addition of 100 ml of purified water to initiate the reaction and form a slurry.

The pH meter was calibrated daily using standard buffer solutions, and at least three readings were taken for each measurement; the mean value was used as the final result, with negligible standard deviation. To prevent calcium deposition on the probe, it was periodically rinsed with diluted hydrochloric acid in accordance with standard guidelines. Replicate samples were tested under identical conditions to confirm the reliability and consistency of the results.

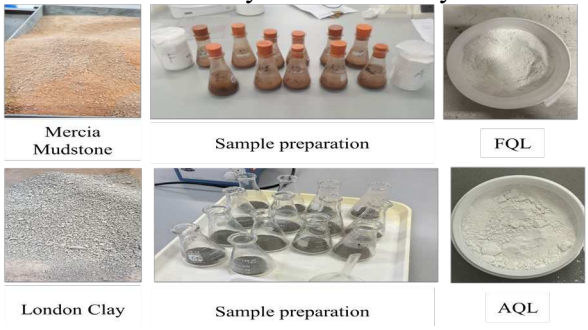

Figure 4. Sample preparation using quicklime for Mercia mudstone and London clay

4 RESULTS & DISCUSSION

4.1 pH response of FQL and AQL during standard and extended ICL testing

(Figure 5 and Figure 6) illustrate the variation in pH with increasing lime content (0–6%) for FQL and AQL. In both cases, pH increased with lime addition; however, FQL consistently produced higher values across all lime contents. For both Mercia Mudstone and London Clay, the standard Initial Consumption of Lime (ICL) test revealed differences in lime requirement. Mercia Mudstone, with an initial pH of 6.95, required approximately 6% lime to reach the target pH of 12.4 within 1-hour using FQL. In contrast, London Clay, with a higher initial pH of 7.95, achieved the target pH of 12.4 with only 4.5% FQL. For AQL, Mercia Mudstone did not reach the ICL thoureshold even at 6% lime, while London Clay required about 5.5% lime to achieve a pH of 12.4.

Despite the addition of high lime contents up to 6%, a constant-pH plateau was not observed in either soil within the arbitrary 1-hour testing period. This directly contradicts the modified definition of the ICL proposed by Rogers, Glendinning and Roff (1997), who characterised the ICL as the minimum lime content required to until pH curve flattens. This defines two opposing errors that Mercia mudstone exhibits a delayed peak, meaning the 1-hour value underestimates its alkaline potential, while London clay exhibits a rapid peak followed by decline, meaning the 1-hour value overestimates its sustained alkalinity.

Mixing conditions also influenced pH achieved in 1-hour in both soils (Figure 5 and Figure 6). Continuous stirring created a more dynamic mixing environment, improving lime dispersion and accelerating early-stage reactions. However, stirred samples exhibited slightly lower pH values within the first hour compared to shaken samples, suggesting faster lime dissolution but incomplete short-term equilibrium. For Mercia Mudstone (Figure 5), the average and peak pH values were higher than the corresponding 1-hour ICL results, indicating that lime hydration and soil reactions continued beyond the standard test period. FQL achieved peak pH values around 12.5–12.6 at 4–5% lime, whereas AQL reached lower peaks (12.3–12.4) even at 6% lime, reflecting reduced reactivity due to aging. In contrast, for London Clay (Figure 6), the peak pH was attained within 1-hour; thus, the extended ICL results matched the standard test. Nevertheless, pH gradually decreased after 1-hour for both FQL and AQL, with smaller reductions observed at higher lime contents.

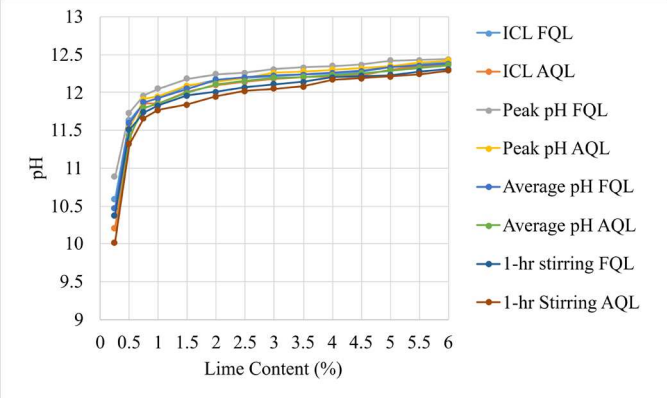

Figure 5. Comparison of Peak, Average, and 1-hour pH response for FQL and AQL in Mercia Mudstone

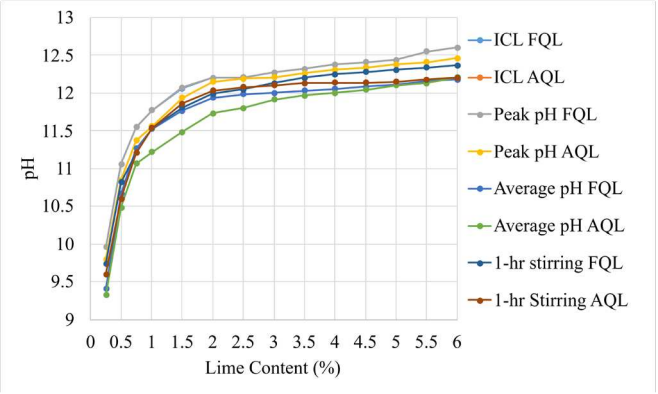

Figure 6. Comparison of Peak, Average, and 1-hour pH response for FQL and AQL in London Clay

4.2 Effect of lime content on pH evolution over time

Mercia Mudstone (Figure 7 and Figure 8) exhibited a gradual rise in pH, peaking at 24-hours regardless of lime age, with FQL reaching a pH of 12.4 at 4.5% lime content and AQL requiring 5.5% lime. Beyond 3% lime, pH trajectories converged such that increases from 3% to 6% lime yielded only marginal gains (<0.2 pH units) compared to the >5-unit rise from 0% to 3%. All effective lime doses stabilised by 120-hours and maintained constant pH to 240-hours, although higher lime contents (>3%) reached the plateau earlier than lower doses.

Conversely, London Clay (Figure 9 and Figure 10) reached peak pH at 1-hour for all lime concentrations, followed by a consistent decrease over time. Following the initial peak, pH declined steadily until 120-hours, after which it stabilised and remained constant to 240-hours, with higher lime contents (>3%) achieving a plateau before 120-hours and maintaining nearly constant pH thereafter.The 1-hour ICL test fails to capture soil-specific pH kinetics as chemical equilibrium is not achieved within 1-hour, even at 6% lime addition. Both soils pH stabilised by 120-hours regardless of percentage lime content and lime aging.

4.3 Impact of lime aging

The aging of lime affected both soils similarly, though the magnitude of its impact varied. FQL demonstrated higher initial reactivity, achieving higher pH values more rapidly than AQL. In both soils, AQL exhibited a delayed reaction, resulting in lower pH values across all lime contents (Figure 7 to Figure 10).

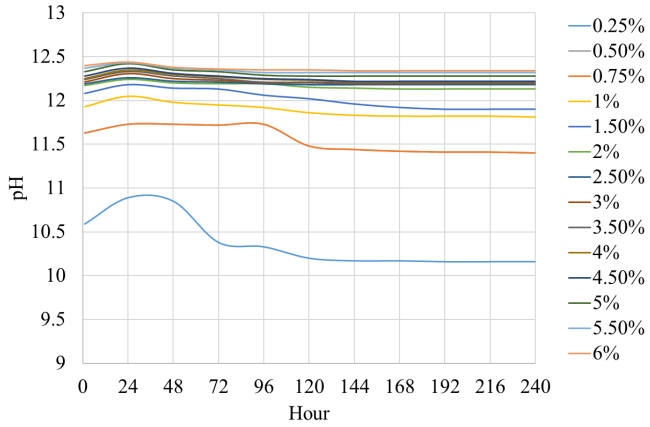

Figure 7. Effect of Lime Content on pH evolution over time for Mercia Mudstone using FQL

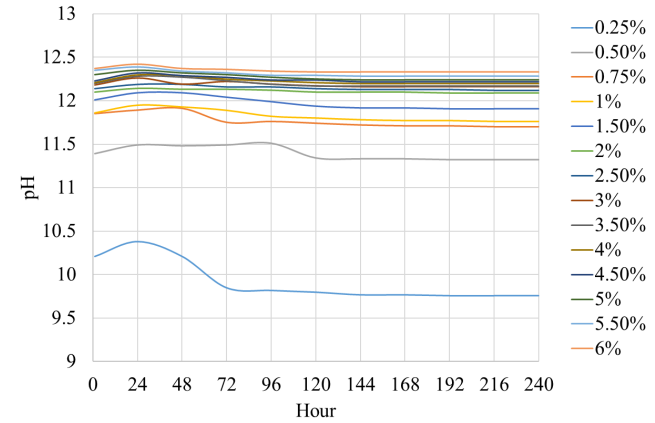

Figure 8. Effect of Lime Content on pH evolution over time for Mercia Mudstone using AQL

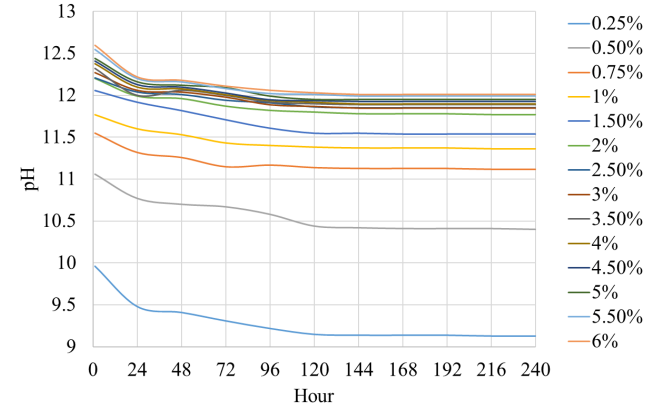

Figure 9. Effect of Lime Content on pH evolution over time for London clay using FQL

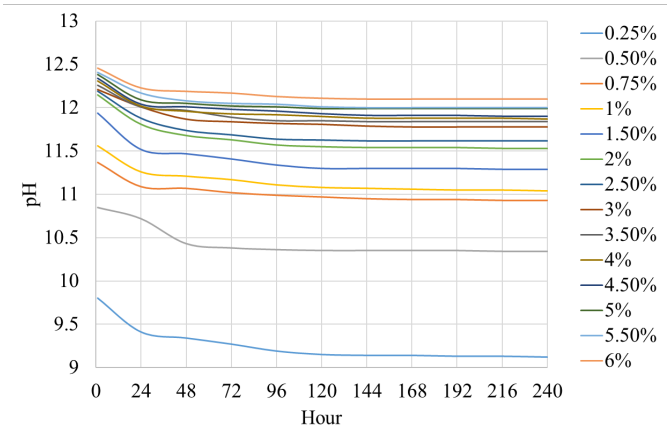

Figure 10. Effect of Lime Content on pH evolution over time for London clay using AQL

4.4 Target pH

Lime type, test duration, and the initial soil pH influence the ability to reach the standard target pH of 12.4. For extended ICL tests, a target pH of 10 was set for both soils as pozzolanic reactions can occur at pH values above 10 due to the dissolution of silica and alumina from clay minerals (Diamond and Kinter, 1965).

In Mercia Mudstone, the sample with FQL maintained pH >10 at all lime contents, while aged lime required ≥0.5%. In London Clay, both lime types maintained pH >10 except at 0.25%. Although low lime contents achieved pH >10, limited calcium may restrict amount of bonding and formation of cementitious products, despite the favorable chemical environment.

In the extended ICL, pH continued to change up to 240-hours, showing reactions persist beyond 1-hour.Additionally, the initial soil pH strongly affects the ability to reach 12.4; Mercia Mudstone starts at pH 6.95, while London Clay begins at pH 7.95. Therefore, the standard ICL test primarily reflects how quickly the lime–soil solution can reach the target pH of 12.4.

Interestingly, even though London Clay starts at a higher pH (7.95), its 0.5% FQL barely maintains pH above 10, whereas Mercia Mudstone, starting at 6.95, achieves a pH above 10 with just 0.25% FQL due to differences in soil mineralogy that the standard test does not take into account.

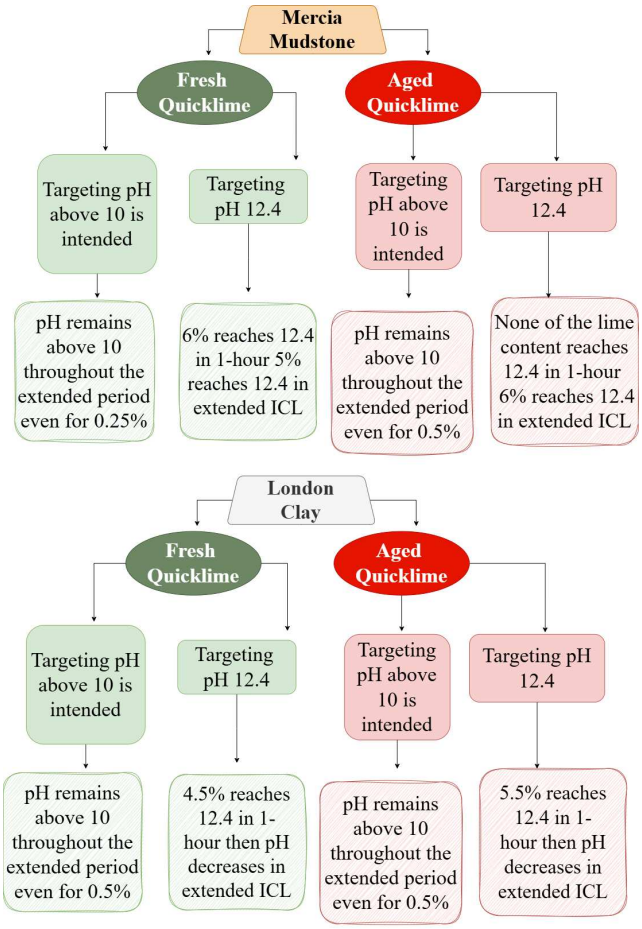

Figure 11. Alternative target pH for Mercia mudstone and London clay

4.5 Discussion

The results from the extended ICL reveal fundamental methodological and kinetic deficiencies in the standard 1-hour test, calling into question its continued use in geotechnical design and specification. Comparing continuous stirring with the standard intermittent shaking showed that the 1-hour pH reading is highly procedure sensitive, undermining its reliability. The standard‘s reliance on a single 20g soil sample also introduces a considerable risk of underrepresenting natural heterogeneity and spatial variability, an issue already noted in previous studies (Cherian and Arnepalli, 2015; Cambi *et al.*, 2016).

Moreover, The kinetic deficiency produces distinct types of error. The standard ICL either underestimates the true lime requirement in soils such as Mercia Mudstone, where maximum pH is not achieved until approximately 24-hours, and overestimates in soils like London Clay, where pH peaks within 1-hour but subsequently declines. Additional influences, including lime aging and mixing regime, which consistently reduced pH and required higher dosages, and the arbitrary adoption of a fixed pH target of 12.4, further compound the inaccuracy of the standard test.

Furthermore, the plateau observed at 120-hours indicates that the soil–lime system reaches a its equilibrium level. The constant pH observed after approximately 120-hours for all lime dosages indicates that the soil–lime system reaches chemical equilibrium only after extended curing. Notably, pH did not become constant within the standard 1-hour period, even at 6% lime addition, contradicting the modified ICL definition proposed by Rogers et al. (1997), which associates ICL with the point where the pH curve flattens. This demonstrates that the 1-hour ICL value reflects only a transient reaction stage rather than the true equilibrium behaviour of the system, as it does not account for the extended time required for cation exchange and soil–lime reactions to reach chemical equilibrium.

Collectively, these limitations demonstrate that reliance on a single instantaneous measurement gives a misleading representation of soil–lime interactions. This misrepresentation leads to inaccurate estimation of lime requirement in soils with contrasting mineralogical compositions and reaction rates, resulting in either excessive or insufficient binder application. Carbonation in AQL lowers pH values, yet this effect is not accounted for in the standard ICL test. Although the pH increase is smaller compared to fresh quicklime FQL, reactions between the lime and soil still proceed, as shown in (Figure 11). Even at low lime contents, the pH remains above 10, indicating that aged lime can slowly dissolve, buffer the system, and contribute to stabilisation though at a reduced rate, provided the system remains sufficiently alkaline.

A time-dependent protocol that accounts for sustained alkalinity and carbonation would enhance accuracy, reduce lime overuse and CO_2 emissions, and support the reuse of aged lime in line with sustainable geotechnical practice.

5 CONCLUSION

This study demonstrated that lime age, soil type, reaction time, and mixing conditions significantly influence pH development and the apparent lime requirement in Mercia Mudstone and London Clay. FQL showed higher reactivity, achieving the target pH of 12.4 at lower lime contents than AQL. Extended testing confirmed that pH continued to evolve beyond 1-hour, stabilising at approximately 240-hours, indicating that lime soil reactions persist over time.

The mixing method also affected early pH behaviour, with stirring promoting faster dissolution resulting in lower pH compared to shaking in 1-hour.

For both soils, an alternative target pH of 10 is maintained even for low lime contents showing that reactions can still occur below the standard thoureshold of 12.4. Due to fluctuations in pH beyond 1-hour, the standard ICL test essentially reflects how quickly the system can reach pH 12.4, rather than capturing the full reaction process.

Initial soil pH and soil mineralogy also influence the ability to achieve 12.4 and the progressive changes in pH over time. London Clay exhibited a clearer pH reduction trend after the first hour compared to Mercia Mudstone, and aged lime consistently produced lower pH values across both soils.

Overall, these findings indicate that relying solely on conventional ICL tests based on short-term pH measurement may be insufficient. Approaches that consider extended reaction times, soil-specific behavior, and direct indicators of pozzolanic activity are needed to accurately determine optimum lime content and achieve reliable, durable, and sustainable soil stabilisation.

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