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Compressibility and microstructure of kaolin in varying thermo-hydro-mechanical states imposed by energy geostructures

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\textbf{ABSTRACT}

The thermal volumetric behaviour of soils plays a critical role in designing energy geostructures, withstanding temperature fluctuations. This study examined, for the first time, the thermal deformation of the partially saturated kaolin clay (matric suction of 0–300 kPa) within the operating temperature range of energy geostructures (8°C to 45°C), subjected to varying most recent stress histories (normal stress of 0–400 kPa). The thermal cycle has been applied to samples with identical hydro-mechanical stress histories initiated by heating or cooling from room temperature. Scanning electron microscopy (SEM) tests have been performed to investigate the impact of temperature on soil microstructure as a potential determining mechanism of thermal deformation. The volumetric deformation associated with thermal cycles is analysed, considering the concurrent role of the overconsolidation ratio and the most recent stress. Secondary thermal consolidation (i.e., particle rearrangement) mainly depends on the most recent stress history, occurring only in samples heated beyond the yield limit, with cooling preventing secondary thermal consolidation. A clear relationship between thermal volume change and matric suction was observed, with higher matric suction resulting in less pronounced particle rearrangement. Further SEM analysis revealed that heating beyond the yield limit alters the soil microstructure permanently, with cooling showing no impact.

\textbf{1. Introduction}

Climate hazards and the associated risks, such as sea level rise, heatwaves, droughts, wildfires and permafrost thaw, cascade across sectors and regions, disseminating impacts along coasts, urban centres, and mountainous areas (IPCC, 2022). As a result, key infrastructure and services, such as energy supply and transmission, may be disrupted. To face this massive challenge, adaptive and mitigating strategies should be developed. However, the chance for adaptation to many climate risks will likely diminish as the climate risks escalate, emphasising the importance of mitigating strategies (IPCC, 2022). Energy geostructures are thus expected to play a vital role in energy production in the coming years as a sustainable, eco-friendly solution, mitigating the adverse impact of global warming (Laloui and Sutman, 2020; Hashemi et al., 2023).

The operation of energy geostructures is not without risks, with temperature and water content fluctuations at the interface being of utmost significance in determining the volumetric behaviour of the surrounding soil. Therefore, analysing the volumetric behaviour of soils subjected to varying thermo-hydro-mechanical (THM) loads is crucial in the design and analysis of energy geostructures. The hydro-mechanical behaviour of unsaturated soils at ambient temperature (Blatz et al., 2005; Khosravi et al., 2020), as well as the thermo-mechanical behaviour of saturated soils (Vega and McCartney, 2014; Di Donna and Laloui, 2015; Houhou et al., 2020), are extensively investigated. Nonetheless, few studies have examined the coupled effect of water content and temperature variations on the volumetric behaviour of soils (Uchai-pichat and Khalili, 2009; Coccia and McCartney, 2016; Ng et al., 2016). These studies have revealed that the soil nature, the stress history, and the thermal loading path govern the non-isothermal volumetric behaviour of partially saturated soils (Loria and Coulthabiy, 2021; Hashemi and Sutman, 2022).

The literature has traditionally focused on thermally induced deformation of fine-grained soils, owing to their more sensitive behaviour to temperature change (Saix et al., 2000; Tang et al., 2008; Di Donna and Laloui, 2015). In these studies, over consolidation ratio (OCR) has been identified as a primary factor addressing the effect of

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stress history on thermally induced deformations (Laloui et al., 2014; Hashemi and Sutman, 2022). These studies have shown that normally consolidated (NC) soils may experience plastic contraction upon heating, whereas overconsolidated (OC) soils dilate elastically as the temperature increases (Abuel-Naga et al., 2007a; Di Donna and Laloui, 2015; Loria and Coulibaly, 2021).

The role of cooling is also crucial in determining the volumetric behaviour of soils, in addition to heating, especially considering that the operating temperature of energy geostuctures can range from 5 °C to 50 °C (Hashemi et al., 2022). A number of studies have examined the effect of subsequent cooling after the initial heating, leading to thermal contraction continuing to increase as the temperature decreases (Campbell and Mitchell, 1968; Di Donna and Laloui, 2015). On the other hand, recent studies have shown that subsequent cooling might remove a small part of the thermal contraction caused by initial heating (Shetty et al., 2019). However, as soils in thermally-active geotechnical systems are commonly subjected to elevated temperatures, the focus of the literature has been dedicated to studying the thermal deformation of soils resulting from heating above room temperature (Romero et al., 2003; Shetty et al., 2019). Therefore, the thermal deformation of fully/partially saturated soils subjected to cooling-heating cycles (i.e., initial cooling and subsequent heating), with different most recent stress histories, which is of paramount importance in designing and analysing energy geostuctures, is not yet studied in detail.

Additionally, a more detailed observation has revealed a thermally induced plastic strain in saturated soils achieved overconsolidation via reloading (i.e., reloading overconsolidation, ROC) (Burghignoli et al., 2000; Towhata et al., 1993). Indeed, the thermo-elastic dilative behaviour is mainly observed for heavily overconsolidated soils in fully/partially saturated state, achieved via unloading (i.e., unloading overconsolidation, UOC) (Tang et al., 2008; Uchaipichat and Khalili, 2009). Thus, the most recent history has been recently proposed as the primary determinant of the thermal volumetric behaviour of soils, with the sign of the secondary compression index serving as the representative variable (Coccia and McCartney, 2016). Though it is shown that this approach is equally applicable to soils characterised by any degree of saturation ($S_s$), limited data exists confirming its application to partially saturated soils (Loria and Coulibaly, 2021). Furthermore, to the best knowledge of the authors, no experimental result is available examining the effect of matric suction on the thermal deformation of ROC soils, necessitating further research into this behaviour.

Once the non-isothermal volumetric behaviour of soils along both heating and cooling paths is characterised, it is necessary to identify the factors governing thermal deformation to determine the impact of thermal loads on the behaviour of soils and soil-energy geostucture interfaces. The thermal deformation of soils is generally attributed to three primary mechanisms. While the expansion of soil components and the increase in repulsive forces between clay platelets are identified to be responsible for the reversible dilative behaviour of UOC soils, particle rearrangement at the microscopic scale is introduced as the potential mechanism leading to the thermally induced irreversible contraction of NC soils (Pedrotti and Tarantino, 2018; Hashemi and Sutman, 2022). The disturbance in the equilibrium between the Van der Waals attractive and the electrostatic repulsive forces is suggested as the potential mechanism leading to a change in soil microstructure and, thus, particle rearrangement (Casarella et al., 2021a; Tarantino et al., 2021). This assumption necessitates further experimental investigation of soil microstructure alteration due to thermal loading, which is not examined to date.

Therefore, a thorough understanding of thermally induced deformations of fully/partially saturated soils is required to predict and address any unfavourable response of soils and soil-structure interfaces to temperature fluctuations. It is also crucial to investigate potential microstructural modifications that could contribute to thermal deformations. Therefore, this study initially examines the thermal deformation of kaolin clay, following both heating-cooling and cooling-heating cycles, employing temperature-controlled oedometer devices. The tests have been carried out on NC, UOC, and ROC soils in partially and fully saturated states, addressing the potential impact of matric suction on thermal deformation. In these tests, the secondary compression index is monitored as the potential determining variable of thermal deformation. Then, the change in soil microstructure in response to thermal and hydraulic loads is investigated using a scanning electron microscope (SEM). A discussion of results is finally presented to identify the potential underlying mechanisms determining the thermal behaviour of fully/partially saturated soils, with the role of microstructure alteration studied in detail.

2. Experimental program

2.1. Tested material

Polwhite-E kaolin, a temperature-sensitive clay, was used in this experiment, with the particle size distribution shown in Fig. 1. The kaolin clay had a specific gravity ($G_s$) of 2.61, a liquid limit ($w_l$) of 51%, a plastic limit ($w_p$) of 32%, and a plasticity index ($PI$) of 19% at room temperature (i.e., $24 \pm 2$ °C). As shown in Table 1, two sets of samples with different initial states, chosen after a series of oedometer tests, were prepared in this experimental study to ensure that collapsing/swelling due to wetting is kept to a minimum (i.e., $\Delta e \leq 0.01$) (Hashemi et al.,...
2022). Thus, no significant changes in soil structure occur during the wetting stage, a key parameter in soil mechanical behaviour (Garakanli et al., 2018). G1 and G2 samples were prepared with relative compaction levels of 78% and 72%, respectively, considering a maximum dry unit weight of 14.94 kN/m$^3$ and optimum water content of 26.8%.

To prepare the samples, the oven-dried kaolin clay mixed with distilled water was first stored in an airtight container for at least three days to ensure moisture equilibrium at desired initial water content (i.e., $\theta_0$) (Ravera et al., 2020; Hashemi et al., 2022). The soil mixture was then compacted into the oedometer ring by imposing a gradual variable static force via a piston in the strain-controlled press to achieve the desired initial void ratio (i.e., $e_0$). At this stage, recording pre-consolidation stress ($\sigma_c$) of <80 kPa for both groups of samples (i.e., G1 and G2) implied that imposing normal stress (i.e., $\sigma_n$) >100 kPa guarantees the NC state of the samples. For SEM tests, as soon as the THM consolidation was complete, the oedometer samples were cut into smaller pieces (e.g., 5 × 5 × 20 mm) and subjected to freeze/drying (Pedrotti and Tarantino, 2018). Further details regarding soil microstructure preservation prior to SEM testing are provided in Section 2.3.

### 2.2. Experimental setup

The experiments were carried out using a conventional oedometer (CO), a suction-controlled oedometer (USO: Unsaturated Oedometer), and an SEM setup. To perform non-isothermal oedometer tests, each oedometer, shown schematically in Fig. 2, was placed in an environmental chamber capable of controlling the temperature between 5 °C and 60 °C. Calibration tests with one thermocouple in the centre of the specimen and one in the designated place, the annulus space for the CO and next to the cell for the USO setups, revealed no significant discrepancies in the measured temperatures. Thus, the temperature was monitored and logged into the hard drive without disturbing the sample throughout the tests.

The axis translation technique was employed to introduce matric suction into the soil samples, consolidated in the USO setup. Combined with a hanging column, axis translation can be considered a precision approach to control low suction regimes with a low resolution as low as 0.01 kPa (Ahmadinezhad et al., 2019). However, two independent systems were utilized in this study to control pore air pressure and pore water pressure. The pore water pressure was regulated by a high air-entry value (HAEV) ceramic disc placed beneath the soil sample, using a GDS digital pressure/volume controller, which provided precision to 1 mm$^3$ of volume. On the other hand, a compressed air system equipped with air regulators was used to apply pore air pressure to the upper porous stone via a conduit in the loading ram. More details are given in (Haghighi, 2011).

As shown in Fig. 3(a), the temperature of the chamber was set to 47 °C and 49 °C for the CO and USO setups, respectively, to achieve 45 °C in the middle of the specimens. The non-uniform air conditioning explains this slight difference in the set and measured temperatures, where the temperature of the specimens is lower than that of the chambers. The thermal deformation of each setup at various temperatures was also examined. The change in the readings of the vertical LVDTs, shown in Fig. 3(b), was used to calibrate the vertical thermal strain of the sample. Alternately, the diametrical thermal strain of oedometer rings will be compensated by soil particle dilation, thus resulting in a negligible effect on the void ratio (Romero et al., 2003; Salager et al., 2008). The LVDTs were also tested for potential changes in readings due to mechanical and hydraulic loads, with negligible variations observed after several calibration tests. The calibration tests are described more in detail in Haghighi (2011).

This study also used an SEM setup to examine soil microstructure alteration upon THM loading. Extensive efforts were made to preserve the soil microstructure prior to SEM testing, including reducing the time from extraction to scanning and treating the samples with liquid nitrogen. To quickly submerge the glass jar, including the trimmed consoliated sample, a liquid nitrogen container was placed next to the consolidation setup to freeze the sample. Freeze-drying of the frozen samples was performed to avoid any further changes in structure or porosity caused by vacuum drying in the SEM. Finally, the samples were kept in a sealed glass jar on top of Silica Gel to limit moisture adsorption before running the scans. More details are given in Section 2.3.

### 2.3. Experimental programme

This experimental programme has studied the thermal deformation of fully/partially saturated kaolin clay subjected to varying normal stresses and the most recent history. Furthermore, as presented in Table 2, SEM tests were conducted to investigate further the potential contribution of soil microstructure alteration to thermal deformation. A number of tests were repeated for each group of tests, ensuring the repeatability of results.

Fig. 4 presents the thermomechanical (TM) paths followed, employing the CO setup to examine the compressibility (ASTM.D2435/2435M-11, 2011) and microstructure alteration of fully saturated soil. All the samples were initially inundated with distilled water at room temperature (i.e., point $s_0$) and kept submerged in water throughout the testing period to ensure saturation (Ravera et al., 2020; Hashemi et al., 2022). All tests showed a degree of saturation over 92%, indicating quasi-saturation afterwards. The samples were then mechanically loaded (L), unloaded (UL), or reloaded (RL) to achieve the desired OCR. Fig. 4(a) shows that the mechanical load was increased to points $b$, $c$, or $d$ for the NC samples. UOC samples were subjected to an initial mechanical load increase to point $d$ (i.e., $s_0=400$ kPa), followed by a mechanical unloading to either $a$ or $c$, depending on the desired OCR, as shown in Fig. 4(b). Furthermore, ROC samples achieved the desired OCR by unloading from $d$ to $R_0$ (i.e., $s_0=25$ kPa), and then reloading to either $a$ or $c$. After two days of equilibrium, a thermal load was applied. For samples following the HC path (i.e., HC samples), the temperature was increased to 45 °C, decreased to 24 °C, then to 8 °C, and finally increased to 24 °C. Following the CH path (i.e., followed by CH samples), the

![Fig. 1. Kaolin clay grain size distribution (Hashemi et al., 2022).](image-url)
temperature was initially reduced to 8 °C, then raised to 24 °C and 45 °C, and then lowered back to room temperature. The heating/cooling rate was limited to 3 °C/h, ensuring a drained condition throughout the tests (Cekerevac and Laloui, 2004; Hashemi et al., 2022).

SEM tests were carried out on saturated NC samples, consolidated to point c (i.e., \( \sigma_n = 200 \) kPa), and loaded thermally following paths \( c \rightarrow c' \), \( c \rightarrow c' \rightarrow c \), and \( c \rightarrow c' \rightarrow c \rightarrow c' \rightarrow c \) or remaining at point c (i.e., room temperature). For all tests, the thermal loading phase lasted for 24 h, with the sample without thermal loads kept at room temperature for the same amount of time. This allowed distinguishing the effect of creep deformation from that of thermal loading on the soil microstructure. Following the thermal consolidation, a trimmed specimen was immediately submerged in a liquid nitrogen bath at \(-196 \) °C for about half an hour to rapidly freeze the pore fluid, minimising the change in soil microstructure due to unloading and the subsequent temperature variation (Jaradat et al., 2017; Houhou et al., 2020; Cotecchia et al., 2020).

Even though it has been revealed that the impact of unloading and subsequent rebound on pore distribution is negligible (Cetin, 2004), the extraction process was kept as brief as possible to reduce the potential inevitable effect on soil microstructure. The comparisons in this study

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**Fig. 2.** Schematic diagram of (a) the conventional oedometer setup and (b) the unsaturated oedometer setup.
are even more valid as the unloading stage was repeated identically for the samples regardless of the thermal loading path followed (Houhou et al., 2020).

The specimens were then dried using the freeze-dryer at a temperature of $55^\circ C$ and a vacuum of 0.03 mbar to prevent any structural alteration during dehydration and any capillary effect (Delage and Lefebvre, 1984). Soil microstructure was then captured for uncoated, low-vacuum samples with fractured surfaces using SEM equipped with a range of detectors and stages. The images are segregated into pores and no pores (i.e., solids) zones by performing the interactive thresholding module in ImageJ to quantitatively capture the change in the area occupied by the pores (Jaradat et al., 2017). The selected lower and upper thresholds for the grayscale, representing the pores, were set in accordance with the brightness and contrast levels to achieve the best fit for each image. For the comparative analysis carried out in this study, the potential unavoidable thresholding inaccuracy that could result in a minor systematic error, equal magnitudes, and similar signs is insignificant (Darbari et al., 2017).

The thermal deformation of the partially saturated soil was also examined using the USO setup, following the THM loading path shown in Fig. 5. First, the samples were wetted by decreasing the initial matric

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### Table 2
A summary of the testing criteria for the consolidation and SEM tests.

<table>
<thead>
<tr>
<th>Testing setup</th>
<th>OCR</th>
<th>Net normal stress, $\sigma_n$ (kPa)</th>
<th>Matric suction, $s$ (kPa)</th>
<th>Thermal loading path</th>
<th>Number of samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>SO</td>
<td>1</td>
<td>L: 0 to 400</td>
<td>None</td>
<td>None</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>8 &amp;</td>
<td>UL: 50 &amp; 200</td>
<td>0</td>
<td>HC/CH</td>
<td>4</td>
</tr>
<tr>
<td>SO + SEM</td>
<td>1</td>
<td>L: 200</td>
<td>0</td>
<td>HC/CH/None</td>
<td>5</td>
</tr>
<tr>
<td>USO</td>
<td>1</td>
<td>L: 0 to 400</td>
<td>None</td>
<td>None</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>UL: 200</td>
<td>0, 100, 300</td>
<td>HC/CH</td>
<td>1</td>
</tr>
<tr>
<td>USO + SEM</td>
<td>1</td>
<td>L: 200</td>
<td>300</td>
<td>None</td>
<td>1</td>
</tr>
</tbody>
</table>

* The sample was initially heated to 45 °C and then cooled back to room temperature (partial HC path).

** The sample was initially cooled to 8 °C and then heated to room temperature (partial CH path).

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The thermal deformation of the partially saturated soil was also examined using the USO setup, following the THM loading path shown in Fig. 5. First, the samples were wetted by decreasing the initial matric

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Fig. 3. (a) Calibration of the thermal loading system and (b) the calibration of the measured vertical displacement due to the thermal deformation of the cell structure.

Fig. 4. The TM loading path followed for (a) normally consolidated and (b) overconsolidated specimens using the conventional oedometer setup.

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suction (i.e., $s_i$) to the desired suctions presented in Table 2. By achieving the hydraulic equilibrium, the NC samples were mechanically loaded to 400 kPa, whereas the OC samples were loaded to 800 kPa and then unloaded or reloaded (after being unloaded to 50 kPa) to 200 kPa to achieve an OCR of 4. The normal load was maintained at this stage to obtain the equilibrium criterion of $\varepsilon_n < 0.025\%$/day (Romero et al., 2003). Finally, the soil samples were thermally loaded along either the HC or CH path, maintaining each temperature until no significant thermal strain was observed (i.e., the equilibrium criterion was met). A similar SEM analysis was carried out on specimens consolidated by the USO setup, as summarised in Table 2, to investigate the impact of matric suction on soil microstructure. For this purpose, two NC samples with no thermal history but varying matric suctions (i.e., 0 and 300 kPa) were scanned after being loaded mechanically to 200 kPa.

It is worth noting that both CO and USO tests have been carried out along the wetting path by decreasing matric suction for unsaturated samples (using the USO setup) and by inundation for saturated samples (using the CO setup). Accordingly, shrinkage caused by drying of samples is unlikely to occur in this study. Furthermore, for radial deformation, it is observed that as the temperature change is limited to 20 °C with a maximum of 45 °C, the radial deformation does not significantly impact the $K_0$ condition (Romero Morales, 1999). The diametrical thermal strain of oedometer rings will be also compensated by soil particle dilation, thus resulting in a negligible effect on the void ratio (Romero et al., 2003; Salager et al., 2008).

3. Test results and interpretation

3.1. Conventional oedometer tests

In the context of energy geostuctures, thermal consolidation can be analysed in accordance with the analogy of the consolidation process caused by mechanical and thermal loads (Campanella and Mitchell, 1968; Shetty et al., 2019; Morteza Zeinali and Abdelaziz, 2021). Fig. 6 presents the thermal consolidation of saturated NC kaolin clay subjected to thermal loading following HC or CH paths, as loaded mechanically to 400 kPa. In Fig. 6(a), the HC sample initially dilated as the temperature increased to 45 °C, owing to the thermal expansion of the soil constituents. The end of the heating phase was marked by the beginning of primary thermal consolidation, accompanied by the complete dissipation of the excess pore water pressure generated during the heating phase (Ravera et al., 2020). The relatively low permeability of the clay and the impedance effects of the ceramic disc led to delayed drainage (Campanella and Mitchell, 1968; Romero et al., 2003; Ravera et al., 2020). As excess pore water dissipates completely, the contractive strain continues to develop, though at a slower rate, referred to as secondary thermal consolidation (Campanella and Mitchell, 1968; Shetty et al., 2019).
Subsequent cooling stages, from 45 °C to 24 °C and then to 8 °C, are characterised primarily by elastic contraction of the soil components, with no pronounced secondary thermal consolidation (i.e., no potential associated particle rearrangement) (Hueckel and Pellegrini, 1996; Cekerevac and Laloui, 2004). Thus, the initial contraction in response to the temperature increase to 45 °C was not recovered in the subsequent cooling stages, resulting in a permanent plastic contraction. Once back to room temperature, soil constituents initially expanded elastically, followed by excess pore water dissipation and no secondary thermal consolidation. The initial plastic contraction owing to prolonged exposure to the fixed high temperature (i.e., 45 °C), leading to the thermal-induced overconsolidation, explains this behaviour (Sultan et al., 2002; Ghahremannejad, 2003; Laloui and Sutman, 2020).

A comparable thermal volumetric behaviour was observed for the CH sample, as shown in Fig. 6 (b). The initial cooling stage led to an elastic thermal contraction, with no evidence of secondary thermal compression, while the low temperature was maintained for a prolonged time. The initial elastic contraction of constituents provided a stable configuration of the solid particles, leading to no potential particle rearrangement (Ravera et al., 2020). The subsequent temperature increase to 24 °C resulted in elastic expansion compensated with the primary thermal consolidation and no significant secondary contractive deformation, leading to a less pronounced expansion (Fig. 6 (b)). Once the temperature increased to 45 °C, secondary thermal consolidation appeared to play a more prominent role in determining thermal strains. This observation implies the significant role of the thermal yield, with thermal collapse occurring as the sample is heated beyond the thermal yield temperature (Laloui and Sutman, 2020; Maghsoodi et al., 2020).

In comparison with the HC sample (εT=0.6%), the CH sample showed less plastic deformation at the end of the thermal cycle (εT=0.5%), which can be attributed to the stiffer structure (i.e., a more stable configuration of the solid particles) resulting from the initial cooling phase (Ravera et al., 2020). This behaviour is also observed in samples subjected to HC and CH paths, but at varying net normal stresses (σn=100 or 200 kPa), which will be further discussed in the following discussion. Alternatively, the secondary thermal consolidation that occurred during the initial heating phase appears to play a more pronounced role in plastic deformations of the HC sample at the end of the thermal cycle. Fig. 6 also shows that cooling samples from 24 °C to 8 °C resulted in thermal strains of 0.29% and 0.27%, following both HC and CH paths, respectively. It is evident from this observation that the thermal deformation by cooling is independent of the thermal path followed.

A framework focusing on the role of the most recent stress history, in which variations in the void ratio would occur even under isothermal conditions, can be used to interpret the macroscopic thermally induced deformation of fine-grained soils (Loria and Coulibaly, 2021). Fig. 7 presents the thermal deformation of saturated OC kaolin samples, with varying OCRs achieved via unloading or reloading, following the HC path. All OC samples experienced thermal expansion upon initial heating, regardless of the mechanical loading path followed. However, long-term exposure to high temperatures resulted in different secondary thermal consolidation mechanisms. The UOC samples with an OCR of 8 clearly showed no secondary thermal consolidation, as shown in Fig. 7 (d), while the ROC samples (regardless of OCR) and the slightly UOC sample experienced limited thermal consolidation, like the NC samples. No secondary thermal consolidation was observed during the subsequent cooling and heating stages for all samples.

The evolution of thermal strain with temperature is thoroughly investigated in Fig. 8, focusing on the role of normal stress, OCR, thermal path, and the most recent stress history. As shown in Fig. 8 (a), NC kaolin samples, subjected to normal loads of 100 and 200 kPa, exhibited thermal strains of 0.13% and 0.12% as heated from 24 °C to 45 °C, as the first phase of the HC path, respectively. This observation confirms the
findings of other studies regarding the stress level independency of thermal deformation (Sultan et al., 2002; Abuel-Naga et al., 2007b). For the CH samples, normal stress does not affect thermal strain either after cooling down from 24 °C to 8 °C, confirming that cooling is independent of stress levels, as well. It is worth noting that thermal deformations are shown to be repeatable in Fig. 8(a), where two samples with identical loading paths (e.g., HC-σn=200 kPa) showed similar thermal deformations. The results also show the parallel cooling paths for all the samples, implying that the thermal expansion coefficient of the solid skeleton is independent of the thermal path and the net stress (Uchai-pichat and Khalili, 2009).

The test results in terms of thermal strain against temperature for both NC and OC samples following varying thermal paths are presented in Fig. 8. As shown in Fig. 8(b), the contractive strain upon heating to 45 °C becomes dilative for HC samples as the OCR goes beyond unity, which is less pronounced for the ROC sample. On the other hand, the first cooling phase of CH samples seems to be independent of the most recent stress history, as a thermal strain of roughly 0.22% is determined for NC, ROC and UOC samples. A thermal path dependency of final thermal deformation can also be observed for the NC samples, with HC samples exhibiting a more significant permanent deformation at the end of the thermal cycle. This observation is in line with the thermal consolidation of the NC sample, subjected to a normal load of 400 kPa, studied in Fig. 6.

The role of the most recent stress history and OCR on the thermal deformation of OC soils is examined in more detail in Fig. 8(c) and (d). The results show that the higher the OCR, the smaller the contraction due to the thermal cycle. Therefore, contractive strains shift to dilative strains as the OCR increases (e.g., a shift from blue to black lines). Although both ROC and UOC samples showed similar thermal volume changes, ROC samples exhibited more contractive strains at the end of the thermal cycle. The role of secondary thermal consolidation during the heating period beyond the yield temperature might account for this observation.

In this regard, Fig. 9 investigates the evolution of thermal strain associated with secondary thermal consolidation with temperature (Shetty et al., 2019). The highly overconsolidated ROC sample (i.e., with an OCR of 8) exhibited contractive strains at 45 °C, as shown in Fig. 9(a). On the other hand, the UOC sample showed limited thermal expansion as heated to 45 °C, emphasising the role of the most recent stress history (Burghignoli et al., 2000; Coccia and McCartney, 2016). A more pronounced change in thermal strains is evident for slightly overconsolidated HC samples, shown by blue lines, where plastic contractive strains increased from 0.025% for the UOC soil to 0.065% for the ROC soil. Thermal strains have a relatively small absolute value, but a >100% increase in contractive strains emphasises the importance of the most recent stress history. Subsequent cooling and heating stages appear not to cause pronounced secondary thermal consolidation, regardless of the loading history.

On the other hand, CH samples demonstrated less tendency to undergo secondary thermal consolidation than HC samples, as shown in Fig. 9(b). The first cooling and subsequent heating stages, from room temperature to 8 °C and then back to room temperature, were identified with no significant thermal strains for all the samples with varying normal stress and most recent stress history. However, as the temperature increased beyond the yield limit (i.e., increased to 45 °C), limited thermal contractive strains were observed, which were more pronounced for the ROC samples with lower OCR. These observations confirm that thermally induced deformations result mainly from an increase in the rate of secondary compression due to higher temperatures (Coccia and McCartney, 2016).

The Estimation of Thermally Induced Rearrangement of Soils (EThIReS) methodology introduces a novel method to analyse the deformation-time curve in thermal consolidation (Shetty et al., 2019). In this approach, the contributions of both mechanical (ΔVs) and thermal (ΔVT) loadings responsible for the volumetric deformation of the
specimen is considered by tracking the total volume change, $\Delta V$, over time. It has been observed that $\Delta V_T$ shares similarities with $\Delta V_M$, where the dissipation of $\Delta u_M$ and $\Delta u_T$, caused by mechanical and thermal loadings, respectively, are responsible for mechanical and thermal consolidation. This extended approach allows for a thorough analysis of the volumetric behaviour in response to thermal loading, distinguishing between primary thermal consolidation ($\Delta V_{pT}$) and secondary thermal compression ($\Delta V_{sT}$) (Shetty et al., 2019). Therefore, the secondary thermal compression index ($C_T$) is calculated for each heating/cooling phase by monitoring the evolution of secondary compressive strains over time through the same method used to determine the secondary mechanical compression index ($C_M$).

In this study, the difference between $C_T$ and $C_M$ (referred as relative creep rate), measured prior to the thermal loading, against temperature is presented in Fig. 10. The change in the secondary mechanical compression index of the sample kept at room temperature for an extra 24 h (i.e., the period of thermal loading for other samples) is also measured. Therefore, the thermal volumetric behaviour of soils can be better understood by excluding the effect of soil mechanical creep. In Fig. 10, positive values denote that the behaviour has become more contractive, less dilative, or has shifted from dilative to contractive. A 0.05%/day drop in mechanical creep rate was measured for the sample, which did not undergo thermal loading, meaning that it contracted more slowly. The secondary thermal compression index appears to be more affected by heating than cooling in terms of temperature. In NC samples subjected to a normal load of 200 kPa, the creep rate increased by 0.12%/day as temperature increased to 45 $^\circ$C, while cooling had a limited impact of 0.02%/day, confirmed by repeating tests two times. Although the change in creep rate is more significant for UOC samples than ROC ones as heated to elevated temperatures, studying the thermal deformation of the samples unravels varying impacts of temperature on creep rates. As ROC samples experience contractive mechanical strains before heating, the higher creep rate means an enhanced contractive behaviour. Conversely, the UOC samples dilating prior to heating tend to dilate more slowly or slightly contract as the temperature increases.

### 3.2. Unsaturated oedometer tests

The impact of matric suction on the mechanical behaviour of partially saturated kaolin is addressed in Fig. 11, with the normal compression line shifting toward higher net stresses as matric suction increases (Uchaipichat and Khalili, 2009; Bagheri et al., 2020; Pham et al., 2023). The suction hardening caused by the stiffening of the soil structure also led to lower swelling ($c_s$), and compression ($c_c$) indexes and higher preconsolidation stresses in higher matric suctions, as shown in Table 3 (Tang et al., 2008; Uchaipichat and Khalili, 2009; Garakani et al., 2018; Bagheri et al., 2020). The repeatability of the mechanical consolidation results of the partially saturated kaolin clay was further confirmed by repeating two tests at each suction.

To gain a comprehensive understanding of the non-isothermal volumetric behaviour of partially saturated soils, the evolution of thermal strain with temperature is examined in Fig. 12. The final thermal strain at the end of the thermal cycle appears to decrease with increasing matric suction, even though no consistent effect is observed during a single heating stage to 45 $^\circ$C (Fig. 12(a)) or a single cooling stage to 8 $^\circ$C (Fig. 12(b)) (Salager et al., 2008; Uchaipichat and Khalili, 2009; Coccia and McCartney, 2016). Suction-induced nonlinear increase in OCR might be the primary factor responsible for this observation (Alsherif...
Higher matric suction results in stiffer soil that is more resistant to mechanical loads, resulting in larger pores and less intense consolidation (Romero and Simms, 2008; Garakani et al., 2018). Although the stiffer structure of the soil may resist the thermal loads until a certain degree, thermal loads might affect the large pores in the sample with higher matric suction to a greater extent, resulting in a more significant thermal collapse (Zeinali and Abdelaziz, 2022; Houhou et al., 2020). Therefore, the thermal volumetric behaviour of clays is governed by the interplay between the suction-induced overconsolidation and the more pronounced impact of temperature on the larger pores within the soil medium. This phenomenon plays a crucial role in understanding the complex thermal behaviour of clays, particularly in higher matric suctions.

### Table 3
Preconsolidation stress and compressibility indices obtained for the different THM tests.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>$c_c$</th>
<th>$e_0$</th>
<th>$e_c$</th>
</tr>
</thead>
<tbody>
<tr>
<td>HC-s00</td>
<td>0.535</td>
<td>0.035</td>
<td>37</td>
</tr>
<tr>
<td>CH-s00</td>
<td>0.515</td>
<td>0.031</td>
<td>48</td>
</tr>
<tr>
<td>HC-s100</td>
<td>0.444</td>
<td>0.013</td>
<td>89</td>
</tr>
<tr>
<td>CH-s100</td>
<td>0.420</td>
<td>0.018</td>
<td>82</td>
</tr>
<tr>
<td>HC-s300</td>
<td>0.403</td>
<td>0.008</td>
<td>151</td>
</tr>
<tr>
<td>CH-s300</td>
<td>0.390</td>
<td>0.009</td>
<td>153</td>
</tr>
<tr>
<td>HC-s300-UOC</td>
<td>0.395</td>
<td>0.011</td>
<td>132</td>
</tr>
<tr>
<td>HC-s300-ROC</td>
<td>0.426</td>
<td>0.019</td>
<td>125</td>
</tr>
</tbody>
</table>

### Fig. 11.
Effect of thermal cycles on the oedometric curves of unsaturated kaolinite analysed in net stress framework: (a) the NC samples, and (b) the t OC samples.

### Fig. 12.
The total thermal strain evolution with temperature for samples (a) following the HC path, and (b) the CH path; as well as thermal strains associated with secondary thermal consolidation (c) following the HC path, and (d) the CH path.
The study of secondary thermal consolidation reveals the primary significance of matric suction in determining the thermal strains. Fig. 12 (c) and (d) demonstrate that secondary thermal consolidation occurs only once the unsaturated sample is heated beyond a yield temperature, with less pronounced thermal consolidation at higher matric suctions, similar to saturated samples. For instance, as shown in Fig. 12(c), the thermal strain associated with the secondary thermal consolidation upon heating to 45 °C was reduced from 0.29% to 0.12% as matric suction increased from 0 to 300 kPa. Therefore, the impact of suction-induced overconsolidation is notably significant in governing the thermal deformations associated with the secondary thermal consolidation.

As discussed in Section 3.1, the most recent stress history, rather than just OCR, is identified as the primary determinant of the thermal behaviour of soils. This phenomenon is investigated for HC samples in higher matric suctions (e.g., \(s = 300 \text{ kPa}\)), shown in Fig. 12(a), with the UOC sample showing limited dilation while the ROC sample contracted at the end of the thermal cycle. In fact, the increased matric suction resulted in a stiffer structure and, therefore, less deformation upon heating. However, the shift from dilative to contractive strains for the ROC sample is still evident. Additionally, Fig. 12(c) shows that the ROC sample has been subjected to limited secondary thermal consolidation in response to the temperature increase, emphasising the importance of the most recent stress history.

### 3.3. SEM tests

The result of SEM tests, capturing the effect of cooling on the microstructure of saturated NC kaolin clay, is presented in Fig. 13. As presented in Fig. 13, pores seem to occupy a smaller area of the thermally loaded samples, with the sample at 8 °C (Fig. 13(b)) showing fewer or smaller pores than the sample at 24 °C (Fig. 13(a)). This observation is better illustrated in Fig. 13(d) and (e), with the black parts representing the pores. A more detailed analysis of the impact of cooling on the soil microstructure revealed that the sample that followed the CH path partially (i.e., cooled to 8 °C and then heated to 24 °C) exhibited less pronounced changes in pore size (Fig. 13(c) and (f)). As presented in Table 4, the average area occupied by pores (AACP) decreased from 22.9% to 19.7% as the sample was cooled from 24 °C to 8 °C, increasing to 21.6% once the sample was heated back to room temperature. Even though cooling has led to a smaller pore area, no thermal collapse or particle rearrangement seems to occur. These findings are in agreement with the results presented in previous sections, where cooling led to elastic contraction accompanied by no significant secondary thermal consolidation or associated particle rearrangement.

![Fig. 13. A SEM image of the specimens (a) at 24 °C, (b) at 8 °C, and (c) after partial CH path, along with the analysed images of the specimens (a) at 24 °C, (b) at 8 °C, and (c) after partial CH path.](image-url)
The area fraction occupied by pores for the CO and USO thermally loaded samples using image analysis methods.

<table>
<thead>
<tr>
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</tr>
</thead>
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<tr>
<td>CONSOLIDATION SETUP</td>
</tr>
<tr>
<td>SLICE NUMBER</td>
</tr>
<tr>
<td>--------------</td>
</tr>
<tr>
<td>SO-T24-1</td>
</tr>
<tr>
<td>SO-T24-2</td>
</tr>
<tr>
<td>SO-T5-1</td>
</tr>
<tr>
<td>SO-T5-2</td>
</tr>
<tr>
<td>USO-s300-T4-1</td>
</tr>
<tr>
<td>USO-s300-T24-2</td>
</tr>
</tbody>
</table>

The change in soil microstructure upon heating is investigated in Fig. 14, where a more significant drop in the area occupied by the pores is observed. As presented in Table 4, the AACP (i.e., black parts in Fig. 14 (d) to (f)) significantly decreased, from 22.9% to 14.4%, as temperature is observed. As presented in Table 4, the AACP (i.e., black parts in Fig. 14, where a more significant drop in the area occupied by the pores is observed as the temperature decreased to 24 °C, reflecting the impact of the two former mechanisms, and the secondary compression index, representing the latter, are the sensible parameters. Approaches that only consider one of these parameters have some significant limitations that prevent them from addressing all experimental results. For instance, the former approach fails to reflect the contraction of ROC soils, while the latter cannot account for expansion upon cooling (Burghignoli et al., 2000; Shetty et al., 2019; Loria and Coulibaly, 2021). Consequently, the experimental results should be analysed within a framework incorporating both OCR and secondary compression index.

4.1. Thermal deformation of NC kaolin clay

Experimental evidence indicates that thermally induced soil deformation depends on stress history, not stress level, with NC kaolin clay exhibiting comparable contractive deformation in varying normal stresses (Sultan et al., 2002; Abuel-Naga et al., 2007b). Since most thermal contraction occurs during the first thermal cycle, studying kaolin clay through one thermal cycle can provide a great deal of insight into soil thermal behaviour (Vega and McCartney, 2014; Zhou et al., 2017; Laloui and Loria, 2019).

The NC kaolin clay subjected to cooling, following either CH or HC paths, experienced elastic contraction without any secondary thermal compression, leaving the secondary thermal consolidation index unchanged. SEM scans of the cooled and room temperature samples support this finding, with visible small and large pores in both scans and limited shrinkage upon cooling. Nonetheless, the elastic contraction of pores resulted in a smaller occupied area. The elastic nature of deformations is unravelled by observing the further expansion of pores in the SEM scan of the sample subjected to heating after initial cooling, with the occupied area returning to its initial level. Despite the lack of SEM scans of unsaturated soil, negligible secondary thermal strains upon cooling for soils with varying matric suction imply no particle rearrangement.

The thermal deformation of NC kaolin clay heated beyond the yield temperature, regardless of the thermal history, is characterised by contractive strains (François and Laloui, 2008; Coccia and McCartney, 2016; Liu et al., 2021). The potential transient thermal pore pressures, induced by the difference in rate between pore pressure generation and water flow, are first dissipated, resulting in primary thermal consolidation (Vega and McCartney, 2014; Morteza Zeinali and Abdelaziz, 2021). Once the soil temperature stabilises, with no additional thermal pore pressure generation, secondary thermal contractive strains continue to develop (Morteza Zeinali and Abdelaziz, 2021). In this context, the thermal consolidation of soils is primarily determined by the secondary thermal compression index analysis. In this study, NC kaolin clay experienced a 0.15%/day increase in secondary thermal consolidation index as heated from 24 °C to 45 °C, which could be due to the decrease in the viscosity of water (Lu and Mitchell, 2019; Morteza Zeinali and Abdelaziz, 2021). However, the limited increase in the secondary thermal consolidation index of OC samples, exhibiting different thermal behaviours following identical thermal paths, suggests that other factors are at play.

Particle rearrangement at the microscopic scale is partly responsible for the irreversible thermally induced deformation of particles (Lu and Mitchell, 2019; Loria and Coulibaly, 2021; Tarantino et al., 2021; Casarella et al., 2021a; Jaradat et al., 2022). The balance between physicochemical forces (i.e., the Van der Waals attractive and the electrostatic repulsive forces) might be disrupted by the different rigidities and thermal expansion of the minerals (Loria and Coulibaly, 2021). Consequently, the shearing strength of inter-particle contacts, which is strictly linked to clay particles and water interactions, is reduced (Towhata et al., 1990). The slippage at the edge-to-face contact leads to particle rearrangement, generating the mechanism of plastic deformation (Santamarina, 2005; Pedrottì and Tarantino, 2018; Tarantino et al., 2021). The soil structure is reorganised until there are enough particle contacts to withstand stress at a higher temperature (Loria and Coulibaly, 2021; Hashemi et al., 2022). The pH of the soil results should be analysed within a framework incorporating both OCR and secondary compression index.
Electrolyte (e.g., water) is found to govern this phenomenon through its impact on the charge of clay platelets, with alkaline water preventing the process (Pedrotti and Tarantino, 2018; Tarantino et al., 2021; Wei and Abdelaziz, 2022). Thus, the thermal collapse was not prevented in this study, using water with a pH of 5.

The thermal collapse of NC kaolin clay in the present study is well demonstrated by SEM scans of saturated samples, in which the area occupied by pores drops dramatically at elevated temperatures. This drop is primarily associated with the reduction in the number and size of large pores (Jaradat et al., 2017; Pedrotti and Tarantino, 2018). Pore size distributions (PSD) of fine-grained soils subjected to elevated temperatures have shown that large pores are primarily affected by temperature, whereas small pores appear not to be affected much (Houhou et al., 2020; Zeinali and Abdelaziz, 2022). The additional reduction in the area occupied by pores after cooling the sample back to 24 °C suggests that the last thermal collapse may be responsible for plastic contraction, even though a clear shift from edge-to-edge to face-to-face contact cannot be detected. It is also worth noting that stiffer structures due to suction hardening compensated for the larger pores observed in SEM scans of the unsaturated sample in this study, resulting in higher thermal resistance. The secondary thermal consolidation was, therefore, less pronounced in unsaturated samples.

4.2. Thermal deformation of OC kaolin clay

Soils heated above room temperature under drained conditions either exhibit a reversible expansion or an irreversible contraction, which is not recovered upon cooling (François et al., 2007; Alsherif and McCartney, 2016). In this study, the UOC samples that followed either the HC or CH thermal paths showed only elastic deformations upon heating and cooling with no secondary thermal consolidation. The elastic deformations are explained by the temperature-sensitive behaviour of repulsive forces between clay platelets, which tends to increase with temperature, and the elastic deformation of soil components (Sutman, 2016; Casarella et al., 2021b; Hashemi et al., 2022; Jaradat et al., 2022). The more stable soil structure and the lower mobilised force led to no reorganisation of the soil structure and, thus, no significant change in the secondary compression index at different temperatures (Loria and Coulibaly, 2021). Limited secondary thermal consolidation observed for slightly OC samples (i.e., OCR = 2) at 45 °C can be attributed to the change from expansive to contractive behaviour at temperatures above the transition temperature (Coccia and McCartney, 2016).

In contrast, the ROC samples showed an accumulation of plastic strain at the end of the thermal cycle, similar to NC soils, which was
more pronounced in the sample with a lower OCR. The overconsolidated nature of the ROC samples resulted in an initial elastic dilation when heated beyond room temperature. However, the end of the initial dilation was marked with secondary thermal consolidation, determined by the most recent stress history. Even though the latter consolidation did not fully compensate for the initial dilation, it was not recovered by subsequent thermal loading, leading to permanent plastic deformation. A difference in the clay content, sample preparation method, and plasticity index may explain the lower contribution of secondary thermal consolidation of ROC samples compared to other studies (Burghignoli et al., 2000; Loria and Coulibaly, 2021). There was also a limited increase in the secondary thermal consolidation index at elevated temperatures, implying a more contractive behaviour. Furthermore, ROC samples with higher matric suction exhibited a stiffer structure with no significant secondary thermal strains.

5. Concluding remarks

In this study, SEM and oedometer tests were carried out to examine the effects of temperature on the compressibility and microstructure of partially to fully saturated kaolin clay. Two oedometers, one for saturated and one for unsaturated testing, capable of conducting tests at varying temperatures were employed. To better understand the role of soil microstructure on the thermal volumetric behaviour of soils, a series of SEM tests were performed on samples subjected to varying thermo-hydro-mechanical (THM) loads. Samples were subjected to different THM loading paths to assess the effect of the most recent stress history, matric suction, and the thermal path. The key findings that could be considered in the design and analysis of energy geostructures are summarised below:

- Further analysis revealed that thermal strains associated with cooling or heating below yield temperature are independent of OCR and the most recent stress history. Alternately, the secondary thermal consolidation of clays at temperatures beyond the yield point is governed by both OCR and the most recent stress history, as evidenced by the enhance thermal secondary compression index of NC and ROC clays, and the elastic expansion of UOC clays. Therefore, a framework incorporating both the OCR and secondary consolidation index should be utilized for analysing thermal deformation in clays. Additionally, since the shear response of the interface depends on the thermal volumetric response of the interface, unlike UOC interfaces that exhibit temperature-independent shear behaviour, further investigation is needed to understand the effect of temperature on the shear response of the ROC soil-energy geostructures interface.

- The secondary thermal consolidation of clays is also affected by the thermal path followed, with the NC sample that followed the CH path exhibiting a less pronounced thermal contraction compared to that of the sample that followed the HC path. This phenomenon can be attributed to the stiffer and more stable structure of the sample resulting from previous cooling stages. It is important to highlight that the thermal path does not seem to govern the thermal strains resulting from cooling or heating below the yield temperature. Therefore, the thermal path appears to affect only the plastic deformation of clays, thereby altering boundary conditions to some extent in the framework of energy geostructures, with an operational temperature range of 8 °C to 45 °C.

- Matric suction plays a significant role in determining the non-isothermal volumetric behaviour of clays, essential for the analysis and design of energy geostructures, operation of which is accompanied by heat and water transfer. For the range of matric suction applied in this study, matric suction had a relatively small effect on the volumetric response of NC kaolinite upon a single heating stage. Nonetheless, analysis of secondary thermal deformation revealed the paramount role of matric suction, with samples with higher suction exhibiting less plastic contraction. The stiffer structure and suction-induced nonlinear increase in OCR may account for this phenomenon. The role of matric suction was also evident for the OC samples, though less significant, where samples with higher matric suction
showed less severe particle rearrangement compared to saturated samples.

- The thermally induced irreversible contraction of clays is attributed to particle rearrangement at the microscopic scale. This phenomenon is evident from the SEM scans of the heated samples exhibiting a significant drop in the occupied area accompanied by the loss or shrinkage of large pores, which was not recovered by subsequent cooling. On the other hand, the scans of cooled saturated NC kaolin clays revealed a drop in the pore area, which was recovered by subsequent heating, indicating the elastic nature of deformations. Furthermore, the porous area was also larger in the sample with higher matric suction than in the saturated one with an identical stress path. This observation supports the assumption that matric suction plays a significant role in stiffening soil structures while leading to a higher potential for thermal deformation due to the presence of larger pores.

- This paper aims to investigate the potential impacts of the thermal path and matric suction on the non-isothermal volumetric behaviour of soils, with a focus on studying the alteration of soil microstructure as a potential underlying mechanism. However, there were a number of limitations in fully comprehending the volumetric behaviour of soils in response to THM loads. While studying the THM behaviour of pure kaolinite is necessary to establish a fundamental understanding, it is important to note that the soils surrounding energy piles often consist of a mixture of fine-grained and coarse-grained soils. Furthermore, the soil surrounding energy geostuctures might have a different anisotropy and fabric than the reconstituted kaolinite used in this experiment. A more comprehensive investigation of the cyclic thermal deformation of partially saturated soil is also required, to provide a detailed understanding of the role of matric suction in characterising cyclic thermal deformation. Therefore, further experiments are needed to develop a comprehensive framework, considering all variables, THM paths, and soil combinations.

CRediT authorship contribution statement

Amirhossein Hashemi: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Validation, Visualization, Writing - original draft, Writing - review & editing. Melis Sutman: Supervision, Writing – review & editing, Project administration. Gabriela M. Medero: Supervision, Writing – review & editing. Jim Buckman: Investigation, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

References


