

Article

Influence of Pore Water Chemistry on Particle Association and Physical Properties of Lime-Treated Bentonite

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Abstract: In the present work, an investigation on the influence of the chemical environment on the sedimentation behaviour of bentonite suspensions is performed with particular reference to the effect of lime addition on the clay particle arrangement. The role of lime content, cation valence and source of calcium ions is considered in the experimental work. At the microscale, particle interaction is analysed by means of zeta potential measurements. Soil fabric formation during sedimentation and its physical properties are inferred from dynamic light scattering measurements, sedimentation tests and Atterberg limits. The addition of cations to pore water promotes the flocculation of montmorillonite particles favouring the formation of particle aggregates, whose dimension depends on ion valence and concentration. The final height of sediments reflects the combined effect of the mutual interactions among particles and the development of secondary phases due to pozzolanic reactions. The influence of clay mineralogy and its effects on the physical properties of lime-treated bentonite is highlighted by comparison with experimental evidence on lime-treated kaolin.

Keywords: sedimentation; pore water chemistry; lime treatment; clay particle interactions; bentonite; microstructure; Atterberg limits



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1. Introduction

Understanding the chemo-physical mechanisms controlling the behaviour of lime-treated soils is a relevant issue due to the strong impact that the use of lime has on the physical and hydro-mechanical properties of treated soils as a result of the chemical reactions taking place after the treatment [1–6]. The study of the colloidal behaviour of clay and the factors influencing the chemo-physical interactions between clay particles provide a basis for understanding the soil fabric arrangement and its influence on the engineering properties of soils. Unbalanced force fields at the interfaces between clay and water cause interactions between soil particles, dissolved ions and water, which govern the behaviour of soils. The sedimentation behaviour of clay suspension is closely related to the electro-chemical interactions acting at a particle level. As discussed by several authors, the changes in the pore water chemistry affect the extent of the attraction and repulsion forces among particles and therefore control the microstructure of the sediment formed from suspensions [7–14]. The sedimentation tests, thus, can be a useful tool for understanding the influence of pore water chemistry on the microstructural features of soil skeleton formed in the very short term.

An experimental study on the influence of pore water chemistry on the sedimentation behaviour of lime-treated kaolin was performed by Vitale et al. (2016) [15], which provided a first attempt to investigate the effects of chemo-physical evolution of lime-treated kaolin on the microstructural arrangement of the soil skeleton during sedimentation. As shown by the authors, two typical curves can be identified from sedimentation tests. The settling

of a flocculated system is shown in Figure 1a; the suspension height progressively reduces over time, leaving a clear supernatant fluid. Figure 1b shows a dispersed system, which maintains its configuration over time with the height of the suspension not relevantly modified and a not-well-recognisable partition between the suspension height and a clear supernatant fluid.

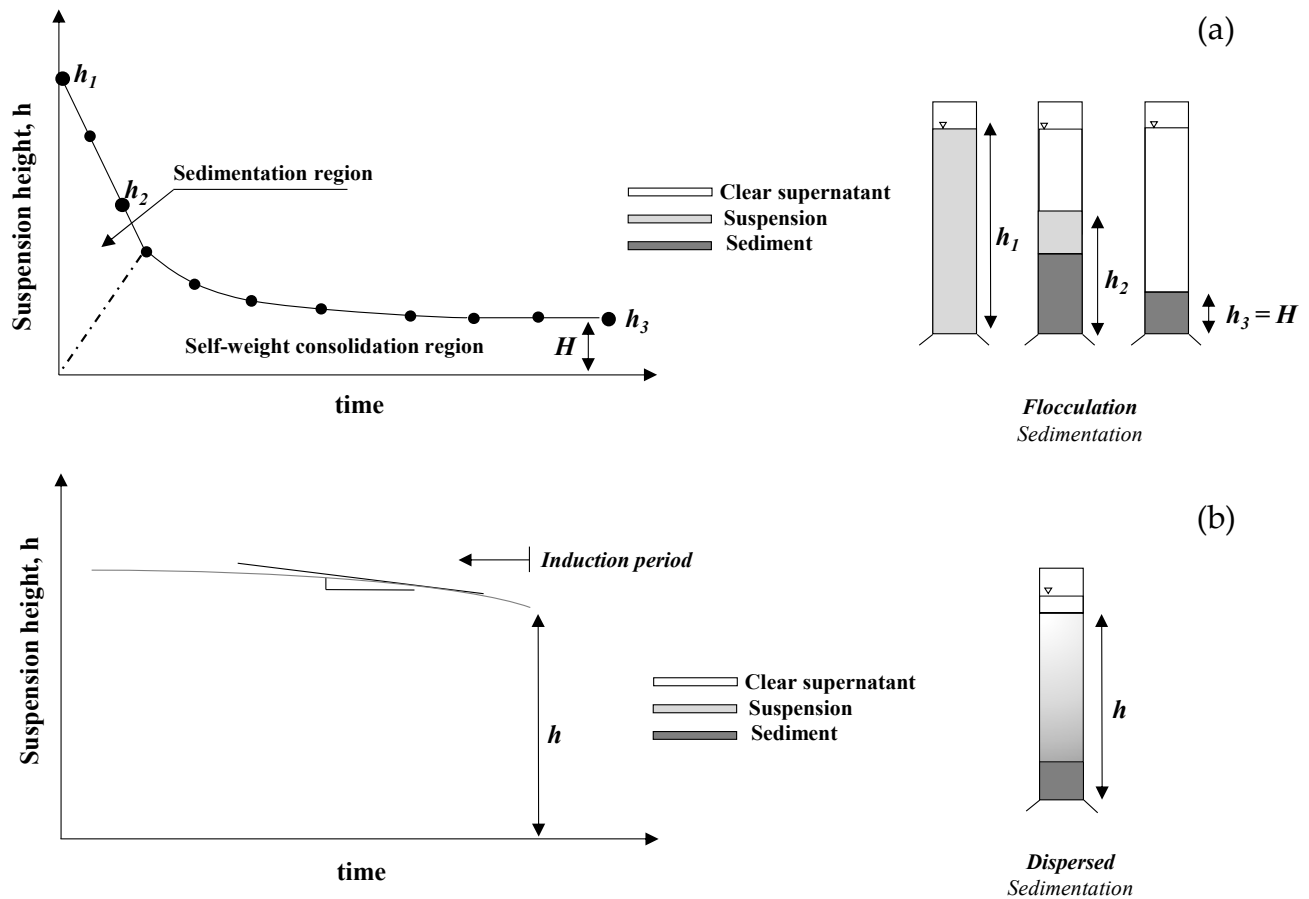


Figure 1. Effect of lime content on sedimentation behaviour of suspensions (a) settling of flocculated system; (b) settling of dispersed system (adapted from Vitale et al., 2016 [15]).

Three stages of the sedimentation process can be identified for a flocculated system, namely induction period, sedimentation and self-weight consolidation stage (Figure 2). During the induction period, particles and groups of particles interact with each other to form aggregates. The duration of this initial phase depends on the nature and intensity of the mutual interaction forces between clay particles. The sedimentation and self-weight consolidation stages begin when the flocculated soil particles settle under gravity, forming a deposit of increasing height that undergoes self-weight consolidation. As shown in Figure 2, at generic time t^* , these two phenomena occur simultaneously. The sedimentation and the self-weight consolidation regions are delimited by the soil formation line, characterised by a constant value of the void ratio e_m , namely the soil formation void ratio [16]. At time t_s , corresponding to the intersection between the soil formation line and the line representing the upper interface of the suspension, an abrupt reduction in settling velocity occurs, indicating the completion of the sedimentation stage [11,17]. During the subsequent self-weight consolidation stage, the sediment reduces its height at a decreasing rate until the final height is reached.

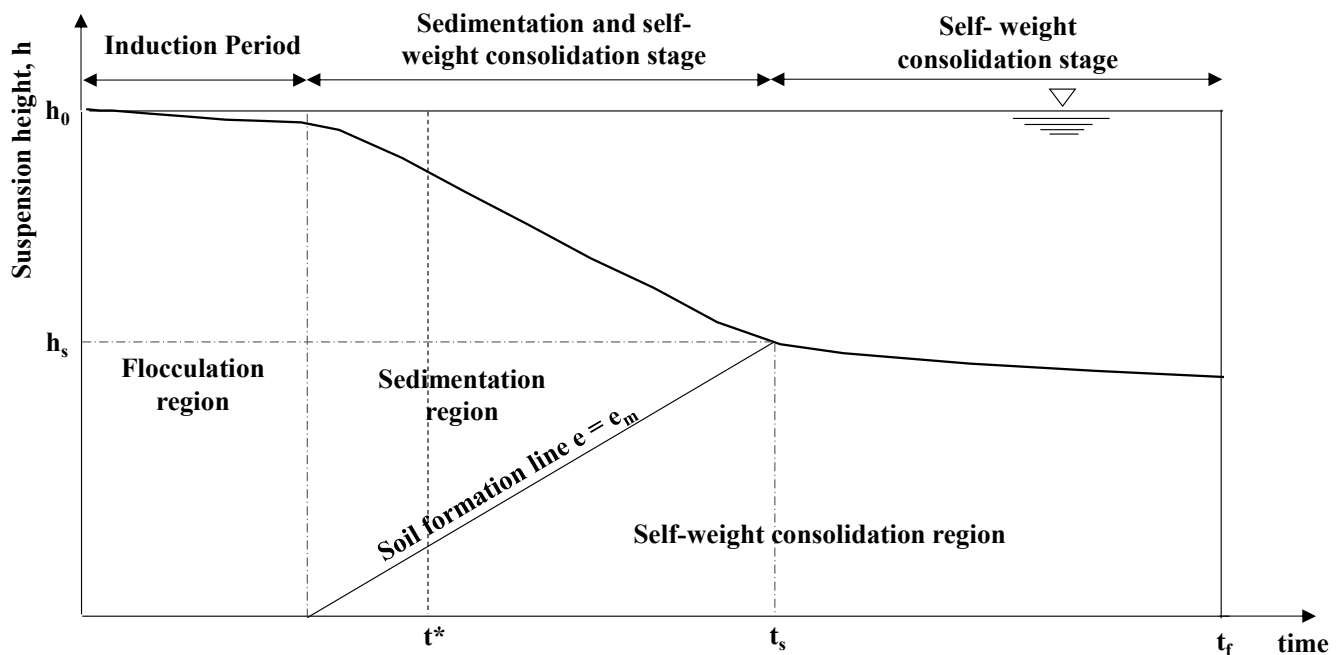


Figure 2. Different sedimentation stages for suspensions (adapted from Vitale et al., 2016 [15]).

The effects of salinity on the sedimentation behaviour of bentonite suspensions have been investigated in the literature [18–21], whereas no detailed studies have been found on the influence of particle aggregation on the settling characteristics by varying pore water chemistry, in particular after the addition of lime. In the present work, an investigation on the sedimentation behaviour of bentonite suspensions under different chemical environments is proposed, focusing on the chemo-physical interactions controlling the clay particle arrangement and hence the soil fabric formation. The effects of lime content, cation valence and source of calcium ions on the electro-kinetic properties of clay particles are investigated by means of zeta potential measurements. Soil fabric evolution and its effects on the physical properties are inferred from dynamic light scattering measurements, sedimentation tests and Atterberg limits. The link between the microscale investigations and the macroscopic evolution of soil properties allows an interpretation of the mechanisms governing the behaviour of treated bentonite. The addition of lime plays a key role in the mechanical improvement of soil behaviour due to the combined effects of the chemical reactions taking place after the treatment (i.e., cation exchange and pozzolanic reactions), whose time scale depends on clay mineralogy. An insight into the influence of clay mineralogy on particle association and its effects on the physical properties of treated soils is given by comparison with similar experimental tests on lime-treated kaolin.

2. Materials and Methods

2.1. Bentonite

Natural bentonite clay, mainly composed of montmorillonite, is supplied by SSB Srl, Sardinia, Italy. The specific gravity is 2.53 g/cm^3 , and the surface area determined by nitrogen adsorption (BET) is $99.38 \text{ m}^2/\text{g}$. The pH value of the soil is about 7.55. The liquid and plastic limits are 143% and 69%, respectively, given a plastic index IP of 74%. The mineralogical composition was determined by X-ray analysis performed on a randomly oriented powder using a Brucker AXS D8 Advance Diffractometer (Elk Grove Village, IL, USA) with $\text{CuK}\alpha$ ($\lambda = 0.154 \text{ nm}$) radiation and a step size of 0.021 degrees. The X-ray diffraction pattern of the bulk sample is shown in Figure 3a. X-ray diffraction patterns of the air-dried and ethylene glycol-saturated samples are shown in Figure 3b for fractions lower than $2 \mu\text{m}$. The soil is formed by Ca-montmorillonite as a major phase, as confirmed by the shift of its characteristic peak after treatment with ethylene glycol. The minor phases

identified in the bentonite sample include quartz and calcite. The chemical composition of bentonite is reported in Table 1.

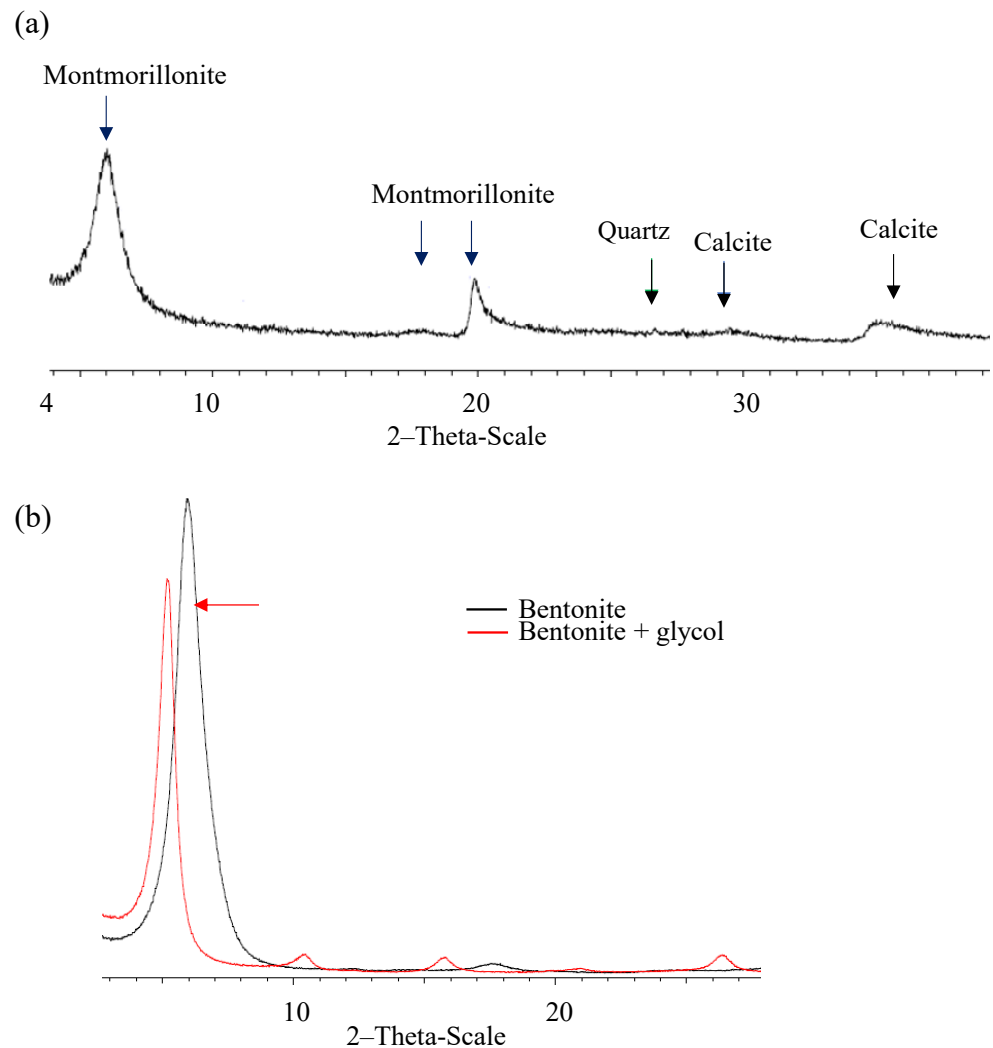


Figure 3. X-ray diffraction patterns: (a) bentonite; (b) bentonite—clay fraction (<2 μm).

Table 1. Chemical composition of bentonite.

Chemical Components	wt%
SiO ₂	64.85
Al ₂ O ₃	24.33
Na ₂ O	0.48
CaO	2.14
K ₂ O	0.21
TiO	0.38
MnO	0.05
MgO	3.18
FeO	4.21
ZrO ₂	0.11

2.2. Sedimentation Tests

Suspensions for sedimentation tests are prepared in graduated cylinders with a mixture of 50 g of dry bentonite at 100% water content. Different amounts of CaO, CaCl₂ and KOH are added to reach the required pH and electrolyte concentrations. Additional water is added to reach an initial suspension volume of 1000 mL. The solution used to

fill the cylinder has the same chemical composition as the soil pore water in order to avoid leaching effects. Bentonite suspension treated with CaCl_2 solution is prepared at the concentration of a saturated lime solution ($[\text{Ca}^{2+}] = 22 \text{ mmol/L}$). The pH value is increased to 12.4 by adding KOH, which is the pH of calcium-saturated solution. During the test, the settlement of the interface between the suspension and the clear supernatant above is monitored over time by readings taken with a transparent ruler fastened to the sedimentation jars. Visual observations allow the suspension heights to be measured with an accuracy of 0.5 mm [16]. A sequence of images of different settlement stages during the sedimentation test is reported in Figure 4. Table 2 lists the suspensions analysed.



Figure 4. Time evolution of suspension height during the sedimentation test.

Table 2. Composition of bentonite suspensions.

Suspensions	Composition
B_pH = 7.55	Raw bentonite
B_KOH_pH = 12.4	Bentonite mixed with KOH solution, pH value 12.4
B_1%CaO	Bentonite treated with 1%CaO
B_3%CaO	Bentonite treated with 3%CaO
B_5%CaO	Bentonite treated with 5%CaO
B_7%CaO	Bentonite treated with 7%CaO
B_CaO_pH = 12.4	Bentonite mixed with CaO at pH value 12.4
B_CaCl ₂ _pH = 12.4	Bentonite mixed with CaCl_2 suspension, pH = 12.4 regulated by KOH addition

2.3. Zeta Potential and Dynamic Light Scattering (DLS) Measurements

The zeta potential measurements are performed at 25 °C in a capillary cell (DTS1061) with a Malvern Nano Zetasizer apparatus and evaluated from the electrophoretic mobility using the Smoluchowski formula [22]. Diluted suspensions with 100 mg/L solid content are prepared at different pH values ranging between 2 and 12.4 by adding either HCl, KOH or $\text{Ca}(\text{OH})_2$ solutions. Bentonite suspensions are prepared at increasing calcium ion concentrations with both $\text{Ca}(\text{OH})_2$ and CaCl_2 solutions. With reference to CaCl_2 bentonite suspensions, the pH is regulated by adding KOH.

The hydrodynamic size of soil particles is determined using the dynamic light scattering (DLS) technique. This is the most used tool for the size characterisation of spherical particles; however, the technique can also be used for the size characterisation of non-spherical particles in suspension [23,24]. With reference to montmorillonite particles, as well as their aggregates, reliable measurements of the relative sizes under given experimental conditions are provided by Furukawa et al. (2018) [25]. Hydrodynamic size measurements of soil particles using the dynamic light scattering (DLS) technique are performed at 25 °C

and laser scattering at 173° in a capillary cell with a Malvern Nano Zetasizer apparatus. The bentonite suspension (100 mg/L) is prepared in deionised water and at pH 12.4 by considering different concentrations of $\text{Ca}(\text{OH})_2$, KOH and CaCl_2 solutions.

2.4. Nitrogen Adsorption Measurements

The specific surface area of the samples is determined from the volumetric adsorption isotherm at 77K of N_2 gas using ASAP 2010 physisorption Analyse (Micromeritics, Norcross, GA, USA). The samples are first outgassed at 110 °C by applying heat and vacuum to remove adsorbed contaminants in the porosity and surface acquired from atmospheric exposure.

3. Results

Figure 5 shows the interface heights of bentonite suspensions monitored over time at increasing lime contents. The sedimentation curve of bentonite suspension in deionised water is characterised by a dispersed behaviour. Clay particles remain suspended for a long time interval, exhibiting negligible settling. A change in the sedimentation characteristics is observed after the addition of lime. Bentonite suspensions exhibit flocculated behaviour. A closer examination of the sedimentation curves, shown in Figure 6, evidences a progressive shortening of the induction period and an increase in the settling velocity at increasing lime contents. The values of t_s , representing the time at which an abrupt reduction in settling velocity occurs, and the related sediment height h_s , are derived from sedimentation curves of lime-treated suspensions and reported in Table 3. Lower values of the sediment height h_s , corresponding to the completion of sedimentation stage t_s , are detected by increasing the divalent cation concentration (i.e., amount of lime in the clay–water suspensions).

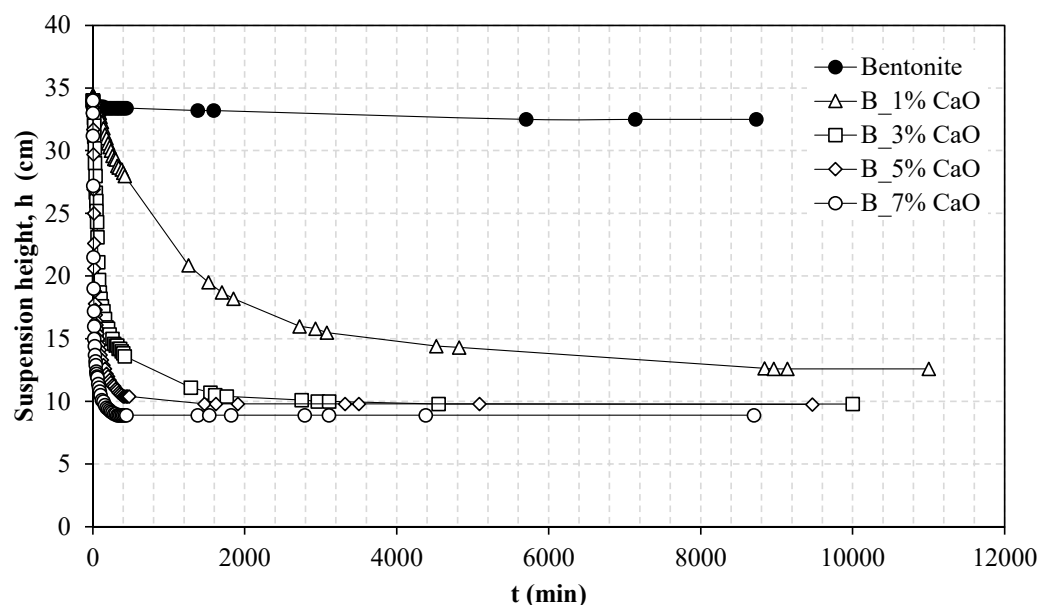


Figure 5. Effect of lime content on sedimentation behaviour of bentonite suspensions.

Table 3. t_s and h_s values of lime-treated bentonite suspensions.

Suspension ID	t_s (min)	h_s (cm)
B_1%CaO	1260	20.85
B_3%CaO	80	19.7
B_5%CaO	25	18.80
B_7%CaO	20	15.00

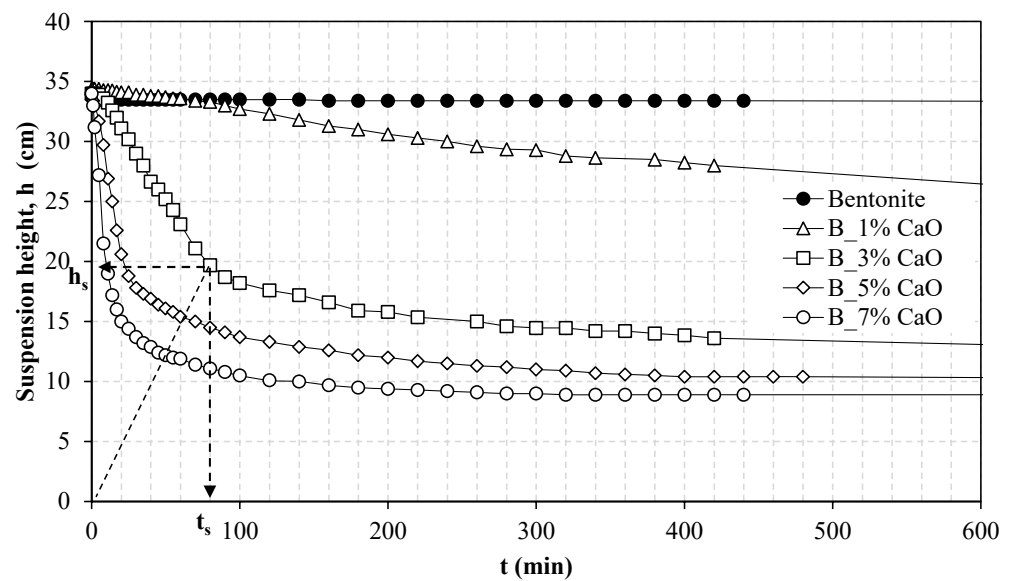


Figure 6. Sedimentation curves at increasing lime content—early stages of settling.

The effect of cation valence on the sedimentation behaviour of bentonite suspensions is shown in Figure 7. The suspension treated with KOH solution shows flocculated behaviour. Compared to the lime-treated suspension, the sample prepared with KOH solution at a pH equal to 12.4 shows a longer flocculation period characterised by a lower velocity of the interface (Figure 8). As shown in Table 4, in the presence of monovalent cations, the time t_s corresponding to the completion of the sedimentation stage ($t_s = 1440$ min) increases significantly compared to the case of divalent cations ($t_s = 25$ min with CaO; $t = 30$ min with CaCl_2).

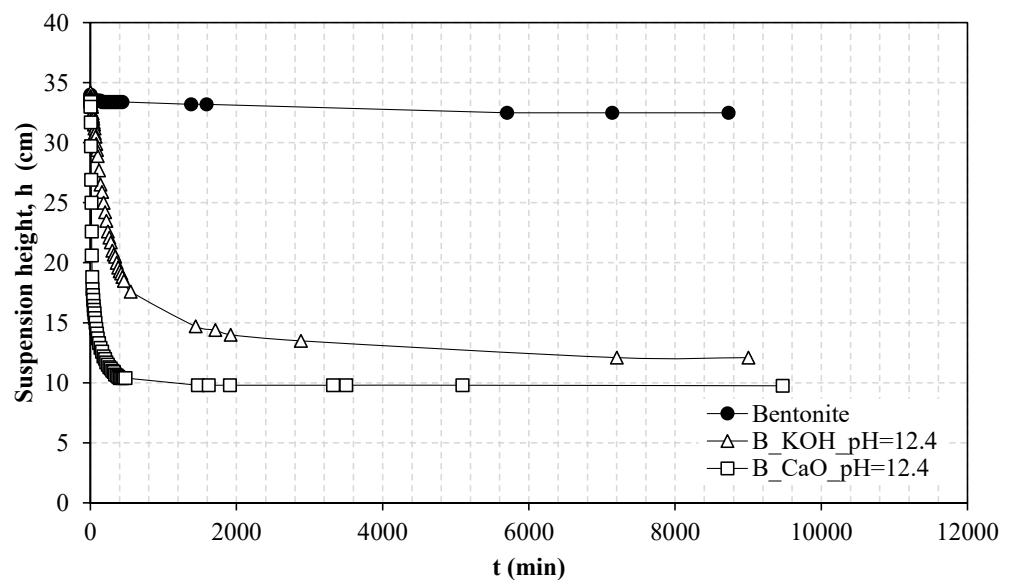


Figure 7. Effect of cation valence on the sedimentation behaviour.

Table 4. t_s and h_s values of bentonite suspensions.

ID Suspension	t_s (min)	h_s (cm)
B_KOH_pH = 12.4	1440	14.70
B_CaO_pH = 12.4	25	18.80
B_CaCl ₂ _pH = 12.4	30	24.80

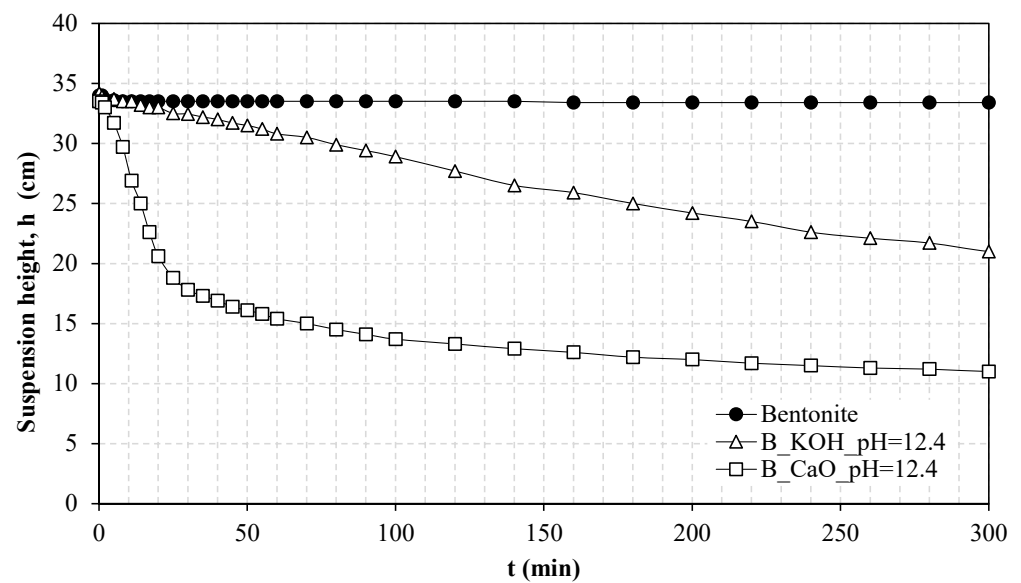


Figure 8. Sedimentation curves as function of cation valence—early stages of settling.

The influence of the calcium cation source on the sedimentation behaviour of bentonite suspension is shown in Figure 9. Compared to the sample prepared with CaCl_2 solution, lime-treated bentonite suspension exhibits a higher degree of particle association as highlighted in the early stage of the sedimentation curve (Figure 10) by (i) a shorter induction period, (ii) a higher settling velocity, and (iii) a faster beginning of the consolidation stage evidenced by the reduced value of t_s due to the more rapid completion of the sedimentation stage. At the same pH value, the presence of chloride anions in the CaCl_2 suspension influences particles' interactions by reducing their aggregations.

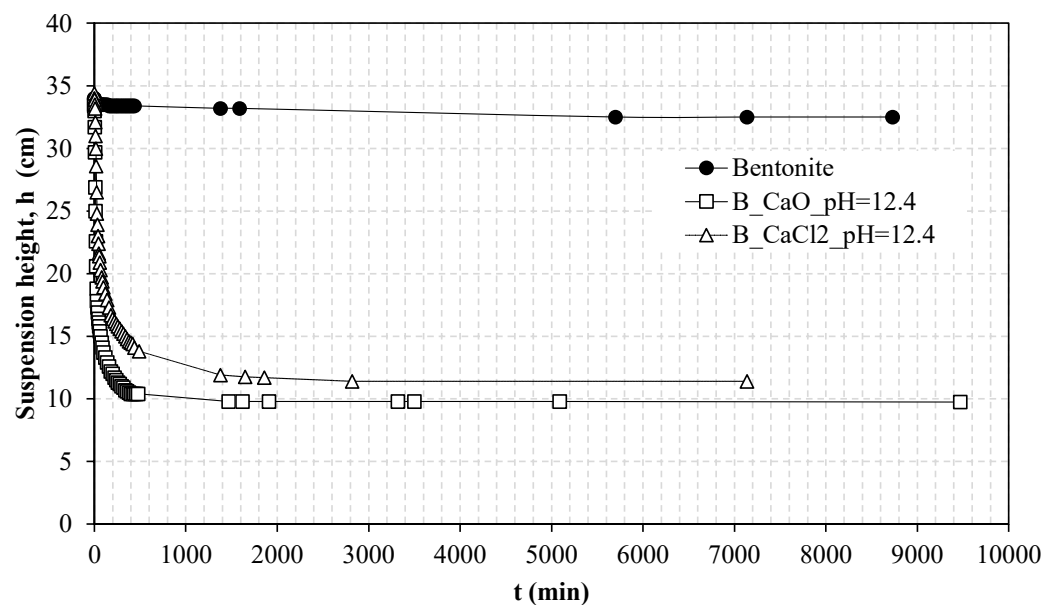


Figure 9. Effect of calcium ion source on the sedimentation behaviour of bentonite suspensions.

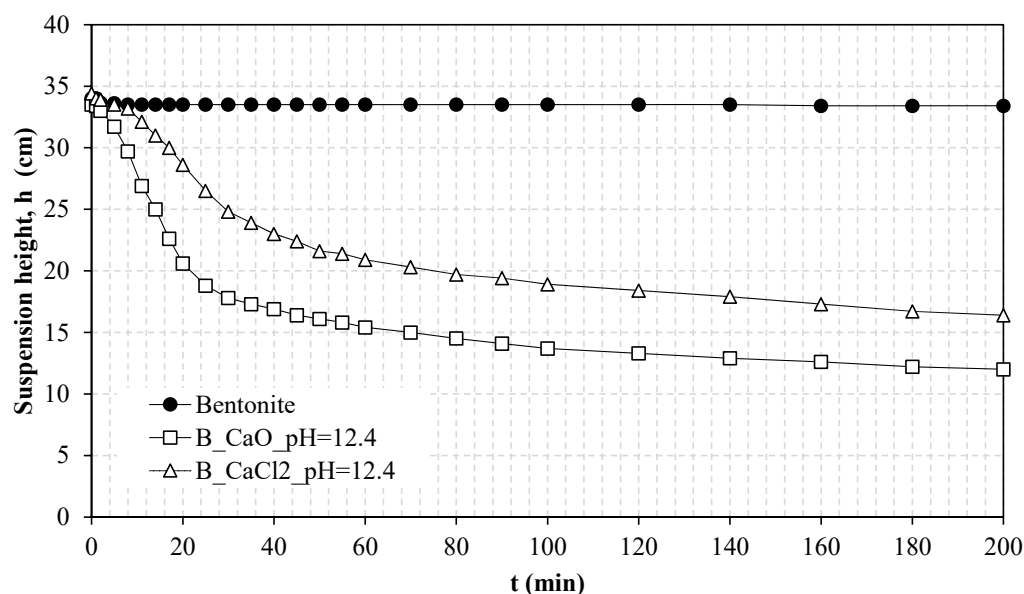


Figure 10. Effect of calcium ion source on the sedimentation behaviour—early stage of settling.

4. Discussion

The results of the sedimentation tests showed a dispersed behaviour of bentonite suspension in deionised water ($\text{pH} = 7.55$). Clay particles remain suspended for a long time interval, without any evolution of their aggregation, as confirmed by DLS measurements (Figure 11). The surface charge density of montmorillonite particles is mainly due to the permanent negative structural charge induced by isomorphous substitutions. As a result, electrostatic repulsions between negatively charged particles prevail, avoiding aggregation. A slight pH-dependent surface charge behaviour is detected from zeta potential measurements for bentonite suspension (Figure 12), as expected for 2:1 minerals [26]. The addition of KOH increases the pH up to 12.4 and induces flocculation, as evident from the sedimentation behaviour of bentonite suspension (Figure 7). Although the surface charge distribution of bentonite particles is slightly affected by the higher-pH environment, the presence of monovalent ions (K^+) promotes a reduction in double-layer repulsions and in turn the particle association, as confirmed by the slight increase in the average size of the suspended particles (Figure 11). The addition of lime induces an increase in the pore water pH divalent cation concentration (Ca^{++}), affecting the sedimentation behaviour of bentonite suspension. The large decrease in the absolute value of the zeta potential shown in Figure 12 is a consequence of the strong tendency of montmorillonite particles to adsorb divalent cations to neutralise the high negative surface charge [27–30]. As a result, the increase in the average size of the aggregated particles, detected by DLS measurements and shown in Figure 11, confirms a higher degree of particle flocculation [31].

Bentonite suspension treated with CaCl_2 solution at $\text{pH} = 12.4$ (regulated by the addition of KOH) shows a lower reduction in the absolute value of the zeta potential (Figure 12) and a lesser degree of particle flocculation (Figure 11) compared to the lime-treated sample. These results seem to be consistent with the role of additional K^+ ions, reducing the swelling potential of montmorillonite, and with the presence of chloride anions, whose adsorption results in a more negative net surface charge [32–34]. Nevertheless, the differences between the sedimentation behaviour of CaCl_2 and CaO bentonite suspensions cannot be explained only by the different aggregation process of clay particles induced by pore water chemistry. As shown by Vitale et al., 2017 [35], new mineralogical phases are formed in lime-treated bentonite starting from 24 h of curing, as a consequence of the development of pozzolanic reactions. Sedimentation curves of lime-treated bentonite show the combined effect of two different reactions, namely cation exchange and pozzolanic reactions. The final height of the sediment at the end of the test is the result of the combined

effect of pore water chemistry, which influences the mutual interactions among particles and the development of secondary phases in the form of gel. In the very short term, the differences between the sedimentation behaviour of lime-treated bentonite suspensions at the stage of the test corresponding to 24 h of curing can be explained by different microstructural reorganisation of particles. A progressive reduction in the induction periods and an increase in the settling interface velocities at increasing lime contents are observed as a consequence of a higher degree of flocculation induced by increasing the amount of lime. A confirmation of the higher degree of flocculation is provided by nitrogen adsorption measurements (BET), which show a decrease in the specific surface area of the lime-treated samples as a function of lime contents at 24 h of curing (Figure 13). According to Sridharan and Prakash (2001) [36], the volume change undergone by montmorillonitic soils during settling and sediment formation is ruled by diffuse double-layer repulsions. The reduction in double-layer thickness surrounding the individual clay particles, due to the addition of increasing amounts of lime, enhances the van der Waals attractive forces [37] and the formation of larger aggregates with closer fabric (Figure 5). The closer fabric at increasing lime content is consistent with the height of the sediments after 24 h of curing (1440 min in the sedimentation tests). Compared to bentonite suspensions, Vitale et al. (2016) [15] highlighted different aggregation mechanisms of suspended lime-treated kaolinite particles. At increasing calcium ion concentrations, lime-treated kaolin shows a higher degree of flocculation of larger stacked particles, with a higher final height of the sediment.

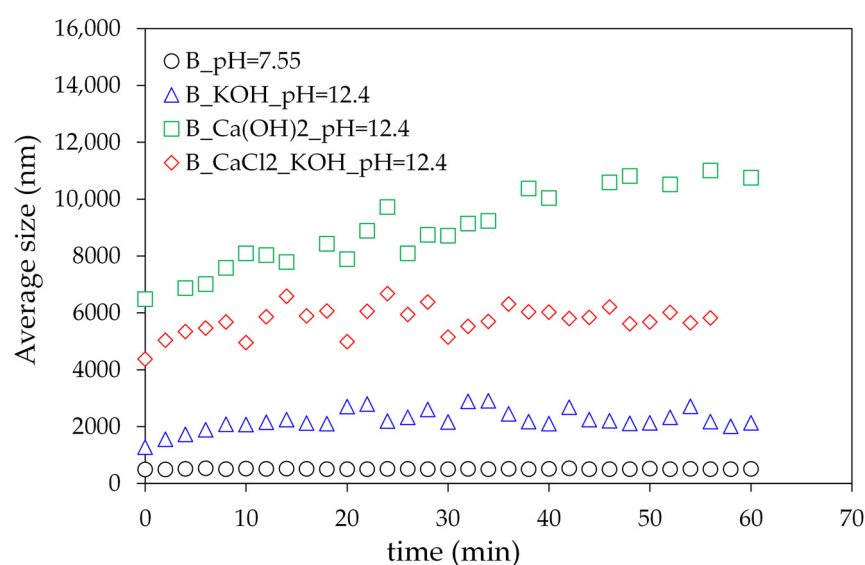


Figure 11. Evolution of the average floc size of bentonite suspensions under different chemical environments.

Chemo-physical interactions induced by lime addition on different clay mineralogy affect the physical characteristics of treated soils. As evidenced in Figure 14, lime-treated bentonite shows a decrease in liquid limit in the short term (i.e., 24 h of curing) with increasing lime content, which is consistent with the decrease in the interparticle repulsion depending on the double-layer thickness reduction induced by lime [38,39]. Conversely, lime-treated kaolin shows an increase in the liquid limit as a function of the lime content due to the aggregation of kaolinite particles induced by the increase in interparticle attraction forces [27]. With reference to plastic limit, both bentonite and kaolin-treated samples show increasing values of PL for higher lime contents (Figure 14b). The formation of aggregated structures for both the treated clays favours a decrease in the air-entry value of the treated clays controlling the PL, and in turn an increase in the plastic limit [39]. As a consequence, a trend of decreasing plasticity index is observed for lime-treated bentonite and an opposite tendency, with an increase in plasticity index, for lime-treated kaolin (Figure 14c).

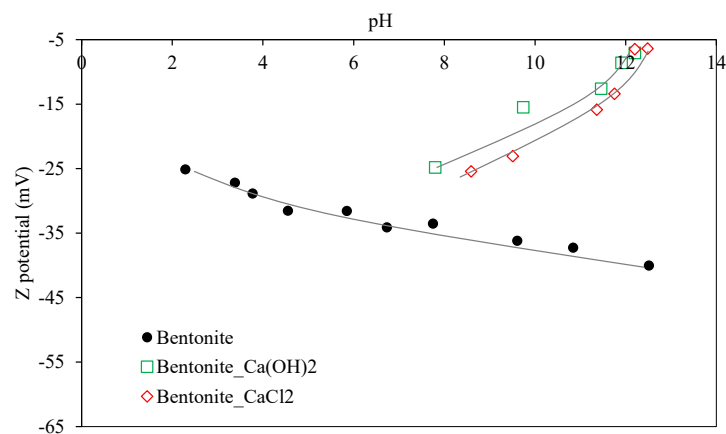


Figure 12. Zeta potential of bentonite suspensions as function of pH.

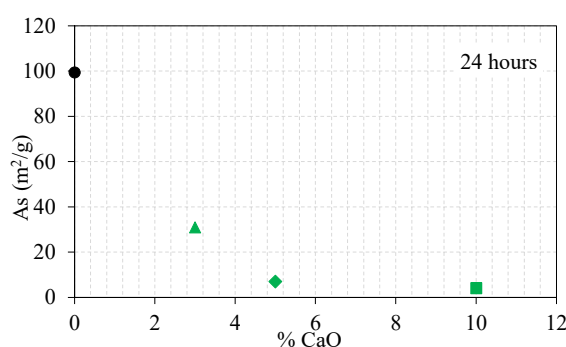


Figure 13. Specific surface area (As) of bentonite-treated samples as function of lime content after 24 h of curing.

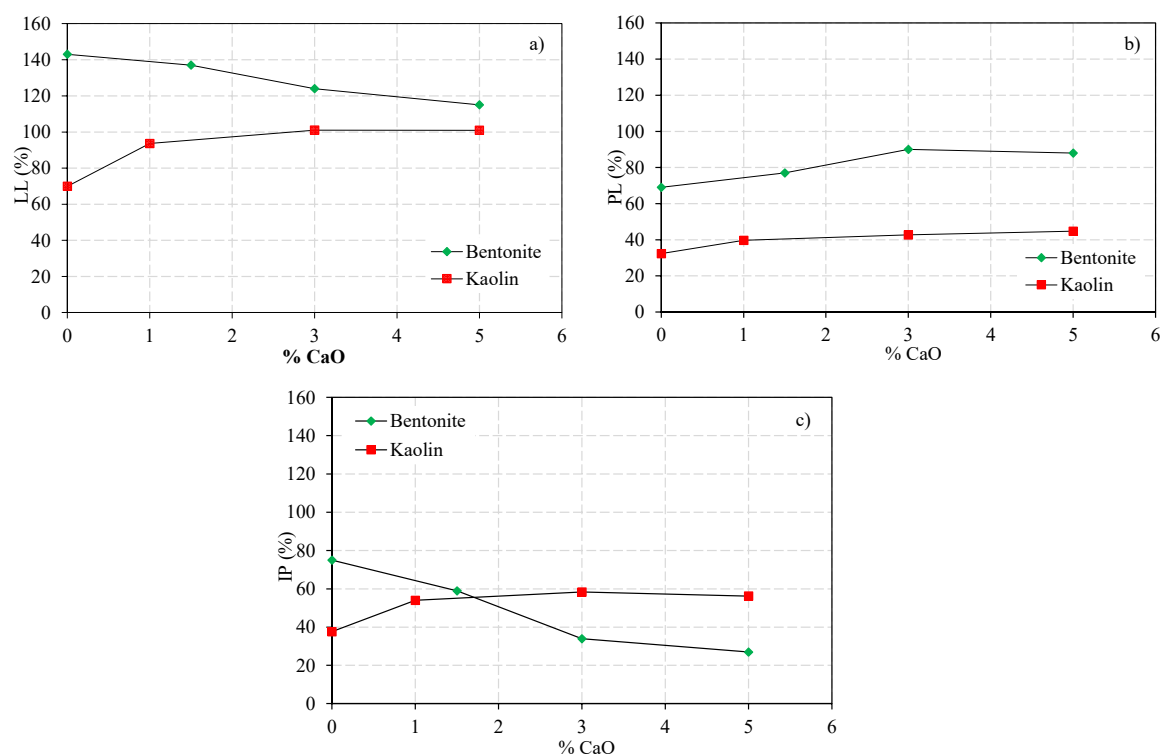


Figure 14. Evolution of plasticity properties of lime-treated bentonite and kaolin after 24 h of curing as function of CaO content: (a) liquid limit (LL); (b) plastic limit (PL); (c) plasticity index (IP).

5. Conclusions

In this paper, the sedimentation behaviour of bentonite suspensions is analysed, taking into account the role played by the chemical environment and, in particular, by lime addition on the reaction mechanisms affecting the clay particle aggregation and soil skeleton formation. An attempt to shed light on the influence of clay mineralogy on the chemo-physical interaction and on its effects on the physical properties of treated soils is also provided by comparing experimental tests on lime-treated bentonite and kaolin samples.

The main findings allow the following conclusions to be drawn:

- The arrangement of montmorillonite particles depends on cation concentration and it is slightly affected by pH due to the permanent negative structural charge induced by isomorphous substitutions;
- The presence of monovalent cations (K^+) in suspension promotes flocculation as a result of the reduction in electrostatic repulsion between negatively charged particles;
- The addition of lime induces an increase in divalent cation concentration (Ca^{++}), favouring a higher degree of particle flocculation evidenced by an increase in the average size of the aggregated particles;
- A lesser degree of particle flocculation is observed when calcium is added from a different source (i.e., calcium chloride) consistently with the presence of chloride anions, whose adsorption results in a more negative net surface charge, weakening particle aggregation as evidenced by the sedimentation test;
- The effects of the flocculated arrangement of montmorillonite particles induced by lime are reflected on the soil skeleton formation at the stage of the test corresponding to 24 h of curing;
- Over a longer curing time, the sedimentation behaviour of lime-treated bentonite depends on the combined effect of the mutual interactions among particles and the development of secondary phases due to pozzolanic reactions;
- Comparison with experimental evidence on lime-treated kaolin highlights the main role played by clay mineralogy in affecting the particle association process and plasticity properties of treated clays.

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