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# Potential of clay-rich waste and industrial by-products as effective inorganic soil amendments to enhance soil physical properties

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## Abstract

Clay minerals play a crucial role in regulating the structure and functions of soils because of their chemical nature and their interactions with other soil constituents. In this context, a key objective of the research described in this article was to assess the extent to which the addition of various types of recycled clay-rich materials to soils prone to crusting and erosion increase their clay content and improve their physical properties. Excavated soil (ExS), considered waste under European Union law, and quarry sludge (QS), an industrial by-product of the washing process in mining operations, were used as soil amendments. ExS is dominated by swelling clay mineralogy such as interstratified illite-smectite, whereas QS contains high level of non-swelling clay minerals such as kaolinite and illite, and a low level of interstratified illite-smectite. Lysimeters were filled with topsoil (0–30 cm) from an agricultural field. Treatments where the soil was amended with these materials were compared with a control without amendment. Lettuce was cultivated for three growing seasons. After 250-days, the aggregate stability and water retention of the topsoil were measured. The equivalent pore-size distribution (PSD), derived from the soil water retention curve, was analysed to predict soil water and aeration status. The application of QS to the topsoil enhanced soil structure without compromising lettuce productivity compared with the control and other treatments. However, amending topsoil with ExS decreased the stability because of the abundance of swelling clay minerals. Soil water retention at saturation increased from 27% in the control to 42% and 36% with ExS and QS amendments, respectively. At a pressure head >15,000 cm (residual pores <0.2 μm), water retention rose from 6% in the control to 19% with ExS and 14% with QS. The application of the clay-rich material increased small pores compared with larger ones. This led to a minor increase in porosity responsible for water movement, aeration and root growth. This observation highlights the limitations of clay application benefits, which depend on the quantity and mineralogy of the clay and the presence of other binding agents. Further research is needed to investigate the combined effects of organic matter

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and clay-rich materials on improving the soil structure and optimizing the pore size distribution for multiple functions.

#### KEYWORDS

clay minerals, remediation, soil degradation, soil erosion, soil organic matter, structural stability

## 1 | INTRODUCTION

Over the past decade, concern has been mounting about the degradation of soils worldwide, and their continued ability to produce the food we need and to deliver the ecosystem services on which human society depend (Kopittke et al., 2019; Oldeman et al., 1991). In metropolitan France, for example, 18% of soils are at risk of high to very high erosion. The regions of Picardy, Nord-Pas-de-Calais, Upper-Normandy and Brittany present risks of soil losses greater than 5 t/ha/year over more than 10% of their territory (Cerdan et al., 2010; SDES, 2021). Simultaneously, climate change is leading to longer periods of drought interrupted by extremely intense rainfall events, which could accelerate soil losses by erosion. As a result, the soils of these regions have exhibited a decline in their inherent properties, including a reduction in their capacity to filter water and an increased tendency to form surface crusts (Antoni et al., 2011; Cerdan et al., 2010; Lemerrier et al., 2011).

Foremost among the approaches to alleviate the progressive degradation of soils, researchers have for many years attempted to enhance their structural stability, using a number of different avenues. Experimental studies of the structure of soils, frequently referred to as their architecture in recent years (Baveye et al., 2022; Vogel et al., 2022), have shown that its stability is influenced by both inorganic and organic factors, such as the presence of Fe–Al oxides, clay content and the type and amount of soil organic matter (SOM) (Bossuyt et al., 2001; Lynch & Bragg, 1985; Six, Elliott & Paustian, 2000; Six, Paustian, et al., 2000). Enhancing soil structural stability by applying organic and inorganic amendments has long been suggested as a method to reduce soil erodibility in agroecosystems and ensure food security (Yan et al., 2022).

The increase in aggregate formation and stability resulting from organic waste application is often related to increases in soil organic matter (SOM), which is mainly controlled by the application duration and rate (Annabi et al., 2011; Chandra & De, 1982). SOM has a high content of resistant, hydrophobic humic substances (Chenu et al., 2000; Piccolo & Mbagwu, 1999) and hence may make the soil hydrophobic, which would lead to a reduction of the wetting rate and slaking. SOM acts as a bonding agent through the formation of clay-humic associations bridged

by polyvalent cations (Tisdall & Oades, 1982). Soil organic matter is an essential but transient component of the soil that controls many physical, chemical and biological properties of the soil (Carter, 1996). Other organic compounds, in particular synthetic polymers, such as Polyacrylamide, have also been added to soils to improve their resilience (Özdemir et al., 2015).

The use of inorganic amendments to increase aggregate stability of soils susceptible to erosion has also been examined to some extent. Natural zeolites were found effective to control runoff and erosion because of their characteristics including large surface area and adsorption capacity (Behzadfar et al., 2017; Colombani et al., 2016; Özdemir et al., 2015). Clay amendments were also observed to be effective in stabilizing erodible soil (Ayoubi et al., 2022; Diouf et al., 1990; Pi et al., 2020). Soils with little clay tend to have a simple structure and can be difficult to manage in practice. Experience suggests that soils containing 15%–25% clay are often the most productive and contain enough clay to provide an adequate surface for interactions with water and nutrients and to have good structure (Newman, 1984; Yu et al., 2022). The addition of clay to degraded and coarse-textured soils, such as sandy and loamy sand soils, is becoming more common among landholders in southern and Western Australia. This practice aims to improve soil properties, including increasing aggregate stability and enhancing carbon sequestration in soils (Churchman et al., 2014; Djajadi et al., 2012; Schapel et al., 2019). The clay used for this purpose is excavated from subsoil, making the incorporation of clay-rich subsoil into topsoil a relatively onerous process. The excavation of the clay, necessitating the use of heavy machinery, and its transportation to the site where it is eventually used significantly increase costs.

A possible alternative to use clay that is specifically mined to improve soils would be to use readily available, clay-rich waste and industrial by-products. Their use would be low-cost and environmentally friendly. Some weaknesses of this alternative could include the variability in composition and the potential presence of contaminants. Excavated soil (ExS), e.g., soil extracted from construction sites, represents the largest source of waste generated annually in the European Union (Panagiotakis & Dermatas, 2022), however its management as a resource

and not as a waste destined to be landfilled (Coussy et al., 2019; Hale et al., 2021) still suffers from a deficit of legislation. Excavated material may consist principally of clay; however its exact nature closely depends on the local geological conditions and anthropogenic activities. In quarries in sand and gravel deposits, mining operations generate a by-product known as quarry sludge (QS), primarily composed of a slurry of silt and clay. At present, this material is principally either stored in a lagoon on site or stockpiled at an offsite processing plant (Wainwright et al., 2002). The application of ExS and QS as soil amendments can serve a dual purpose by revalorizing waste and by-products while potentially improving soil quality. To our knowledge, no previous studies have used this type of clay-rich waste as an amendment to improve the physical properties of soil.

In this general context, the key objective of the research described in this article was to carry out preliminary lysimeter experiments to determine whether the amendment of eroded soils with various ExS and QS, differing in clay mineralogy, or clay- and oxide content, can improve the physical properties of soils, and in particular their structural stability.

## 2 | MATERIALS AND METHODS

### 2.1 | Selection of soil, waste and by-products materials

The soil was collected from the surface horizon (0–30 cm) of an agricultural field in Saint-Pol-de-Léon, Brittany, France. The sampling was conducted using a hand auger, ensuring the removal of plant residues and other debris from the surface prior to collection. The collected soil was air-dried and stored at room temperature for the experiment. Soils in this region present a high risk of erosion (Antoni et al., 2011). The soil is classified as a Luvic Cambisol according to IUSS Working Group WRB (2022). For this study, two clay-rich materials were selected: ExS, classified as waste under EU law and QS, an industrial by-product. ExS and QS were collected from Lanvollon in the Brittany region and a Lafarge quarry located near Château-Gontier in the Pays de la Loire region, respectively. ExS is characterized by high proportions of 2:1 clay mineralogy, such as interstratified illite-smectite. The QS was selected based on its mixed (2:1 and 1:1) mineralogy, which includes high levels of kaolinite and illite and a low level of interstratified illite-smectite. The selection process followed the criteria outlined in the national research program SITERRE-ADAME, focusing on pedological engineering development (Vidal Beaudet et al., 2017). These criteria include ensuring sufficient production volume,

availability across various regions of France, low toxicity and the potential fertility of the materials as both mineral and organic constituents for soil (Rokia et al., 2014).

### 2.2 | Material characterization

The physico-chemical characteristics of the waste, by-products materials and the soil were assessed to determine how much waste or by-products should be incorporated into soil and ensure their safety. Trace metal elements were analysed using ICP-MS after treatment of the solid samples in aqua regia. Mineralogical characterization was carried out using X-ray diffraction on all samples, as well the fine fraction in each case ( $<2\mu\text{m}$ ). Diffractograms were obtained using a Bruker D8 Advance DA VINCI diffractometer equipped with a  $\text{CuK}\alpha$  source ( $\lambda = 1.54\text{\AA}$ ). Quantitative analyses were performed using the Rietveld refinement method (Rietveld, 1967, 1969) and the SiroQuant™ program, version 4. The pH of the soil and materials was measured in a 1:5 (volume fraction) suspension of soil in deionized water according to NF ISO 10390 (AFNOR, 2005). The total organic carbon content (TOC) was determined by dry combustion analysis according to NF ISO 14235 (AFNOR, 1998). Grain size distribution was determined according to the pipette method NF X 31-107 (AFNOR, 2003). Cation exchange capacity (CEC) and exchangeable cations were measured according to NF X31-130 (AFNOR, 1999).

### 2.3 | Clay-rich material mixing procedure

The initial soil contained 9.5% of granulometric clay ( $<2\mu\text{m}$ ) by mass weight. To determine the optimal ratio of waste and by-product materials to be added as an amendment, we aimed for a target range of 15%–25% of granulometric clays in the final soil, as suggested by Newman (1984) and Yu et al. (2022) for favourable soil structure. Two weight ratios were tested for each material: 34% and 11%, aiming for ca. 21% and 13%–14% granulometric clay content in the final soil, respectively. After identifying the suitable mix ratio, we explored applicable mixing methods. Singer et al. (1992) and Attou et al. (1998) have shown that wetting clay-rich material before mixing it with silt material enhances soil aggregation. One common method for modifying soil clay content in an agricultural context involves adding clay to the topsoil through spreading, using tools such as scrapers or specialized machinery (Davenport et al., 2011). Following these methods, we mixed the clay-rich material with water (collected rainwater) at a 50/50 weight ratio to facilitate dispersion.

To ensure homogeneity, the mixture was thoroughly combined with the initial soil before application in the lysimeter. Once applied, the topsoil was again ploughed to a depth of 15–25 cm using a manual chisel ploughing.

## 2.4 | Experimental device

The experiment was conducted at the Economical and Technical Action Committee (CATE) facility in the Brittany region (48.65° N, 3.98° W). CATE is a research station that specializes in vegetables and ornamental crops. The study site has a temperate humid oceanic climate, with cumulative autumn and winter precipitation ranging from 693 to 1088 mm and a mean annual air temperature of 11°C (Akkal-Corfini et al., 2021). Five lysimeters, each with a surface area of 1 m<sup>2</sup> and a depth of 0.83 m, were equipped with a drainage network connected to glass tanks to collect drainage effluent (Figure 1). The bottom 45 cm of each lysimeter was filled with the original soil. For the top 30 cm, four lysimeters were filled with soil mixed with either QS or ExS at two different mass ratios (66:34, 89:11). The fifth lysimeter served as a control, the topsoil was filled only with the original soil. These treatments are referred to as QS 34%, QS 11%, ExS 34%, ExS 11% and control, respectively.

The experiment took place between February 24th and November 24th, 2022 under real climatic conditions.

Three consecutive crops of Little Gem lettuce (cv. Alborada) were cultivated during the following growing periods: from March 8th to May 12th, 2022; from May 23rd

to July 11th, 2022 and from August 17th to October 14th, 2022. Lettuce seeds were initially sown in seed trays and later transplanted to lysimeters once the seedlings had developed four true leaves. The distance between rows and plants was 0.25 m, resulting in a density of 20 plants/m<sup>2</sup>. Soil preparation was completed prior to transplanting the lettuce. All treatments had similar soil management practices. Similar amounts of basal fertilizers and irrigation schedules were applied for all treatments. For the first cycle, harrowing to a depth of 10 cm was performed. For the second and third cycles, chiselling was carried out to a depth of 25 cm, followed by levelling of the surface. After 274 days (ca. 9 months), soil samples were collected from the top layer (0–30 cm) of each lysimeter for chemical and physical analysis using the same methods used for initial materials characterization. Three replicates were conducted for each measurement. Soil aggregate stability and water retention measurements were performed on these samples and are described in subsequent paragraphs. The aerial biomass of the aboveground portion of plants was measured across the entire surface of each lysimeter for each crop cycle. All measured data were subjected to ANOVA, and the Bonferroni test at  $p < .05$  was used to determine the significant differences between the treatments.

## 2.5 | Soil structure stability assessment

The assessment of the structural stability of soils is currently the object of an intense debate in the soil science community (Baveye et al., 2022; Vogel et al., 2022) and no



**FIGURE 1** (a) First growing season observed in treatment amended with 11% of excavated soil (ExS 11%), (b) glass tank collecting drainage effluent, (c) experimental setup, (d) third growing season of lettuce a few days before harvest, (e) total yield, which is the sum of all fresh weights of lettuce harvested from each treatment at the end of the experiment and (f) fresh weight of individually harvested lettuce heads.

doubt methods to address this parameter will evolve in years to come. For the time being, however, it is expedient to use the method proposed by Le Bissonnais (2016) and included in the NF ISO 10930 (AFNOR, 1993). This method consists of three tests designed to assess the resistance of soil aggregates to various breakdown mechanisms by water: (i) slaking caused by the compression of entrapped air because of fast wetting, (ii) microcracking by differential swelling during slow wetting and (iii) mechanical breakdown by raindrop impact. Slaking occurs when dry aggregates are immersed in water or rapidly wetted, leading to an entrapped air and pressure buildup within the aggregates. In contrast, when the soil is capillary rewetted, the air has time to escape, resulting in no buildup of air pressure and more intact aggregates. The mechanical breakdown test characterizes the resistance of pre-wetted aggregates.

One kilogram of soil from each treatment was air-dried and sieved to select aggregates whose size ranged from 3 to 5 mm. The fast and slow wetting tests were conducted on dried aggregates, and the mechanical breakdown was performed on aggregates wetted with ethanol before immersion in water. After a test, the samples were transferred to a set of six sieves (2, 1, 0.5, 0.2, 0.1 and 0.05 mm). For each test, the mean weight diameter (MWD), i.e., the sum of the mass fraction recovered from each sieve multiplied by the average aperture between two adjacent sieves, was calculated and expressed in millimetres. Three replicates were performed for each test, and the subsequent results were presented as the averages of replicates, along with their root-mean-square deviation. The aggregate stability test protocol allowed separating the aggregates among four size fractions based on the aggregate hierarchy model of Tisdall and Oades (1982). The relative abundance of each aggregate size fraction was then expressed as its proportion by mass: large macroaggregates (500–3000  $\mu\text{m}$ ), macroaggregates (200–500  $\mu\text{m}$ ), microaggregates (50–200  $\mu\text{m}$ ) and silt- and free clay <50  $\mu\text{m}$ .

## 2.6 | Soil water retention data and equivalent pore-size distribution

Sample preparation for soil water retention measurements followed the standard NF ISO 11464 (AFNOR, 2006). Soil was air dried in the laboratory at 38°C, and large clods were broken into smaller aggregates. The soil was then dry sieved through a 2 mm sieve. Subsequently, soil samples were placed in metal rings measuring 3.5 cm in diameter and 1.5 cm in height. Samples were saturated and, using a pressure membrane apparatus, gravimetric water content was

measured at four soil pressure heads ' $h$ ' of 100, 316, 3162 and 15,849 cm. Soil bulk density was calculated at each soil suction to convert gravimetric water content to volumetric units. Three replicates of each soil treatment were prepared.

To evaluate changes in soil pore system after incorporating clay-rich materials, the equivalent pore-size distribution (PSD) was characterized, as it is a reliable indicator of soil structural condition and useful for assessing soil water storage and aeration (Topp et al., 1997). PSD is commonly derived from the soil water retention curve function (Dexter, 2004; Durner, 1994), modelled using the van Genuchten function (van Genuchten et al., 1991). The RETC code was used to fit the van Genuchten function to the measured pairs of ( $\theta$ ,  $h$ ).

Soil pressure head  $h$  (cm) and the equivalent pore diameter ( $d$ ,  $\mu\text{m}$ ) were related by (Warrick & Or, 2002):

$$d = \frac{2980}{h} \quad (1)$$

Pores were classified as 'drainage or air capacity' pores ( $d > 30 \mu\text{m}$ ), 'storage or available water' pores ( $0.2 \mu\text{m} \leq d \leq 30 \mu\text{m}$ ) and residual pores ( $d < 0.2 \mu\text{m}$ ) (Cameron & Buchan, 2006; Dexter, 1988; Greenland, 1981). Total porosity was determined from the saturated water content  $\theta_s$ , assuming that the soil water is entirely retained in the pores.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Physico-chemical characteristics of amendments and amended soils

The initial soil contained 9.5% clay, 39.1% silt, 49.1% sand and 2.8% organic matter (Table 1). The two clay-rich materials that were used as soil amendments to improve the physico-chemical properties of the soil had contrasting properties (Tables 1 and 2). The ExS showed a high CEC of 20.2 meq/100 g and its clay fraction was primarily composed of 2:1 clay minerals, with 32.4% of interstratified illite-smectite. In contrast, the QS had a CEC of 12.2 meq/100 g, which was half as small as that of ExS, largely because of differences in clay mineralogy. QS exhibited a mixed (2:1 and 1:1) clay mineralogy, primarily consisting of kaolinite (45.4%), followed by interstratified illite-smectite (13.7%) and illite (10.4%). Additionally, QS contained approximately twice the concentration of goethite compared with ExS. The analysis of ExS and QS showed that the trace elements are within the regulatory limits set by French regulatory guidelines for heavy metals in reused of ExS (Coussy et al., 2020). This indicates that the levels of

	Control	ExS 34%	ExS 11%	QS 34%	QS 11%
Sand 50–2000 $\mu\text{m}$ (%)	49.1 <sup>ab</sup> $\pm$ 0.2	42.2 <sup>cb</sup> $\pm$ 2.2	53.5 <sup>a</sup> $\pm$ 1.8	40.4 <sup>c</sup> $\pm$ 1.0	48.5 <sup>ab</sup> $\pm$ 3.0
Silt 2–50 $\mu\text{m}$ (%)	39.1 <sup>a</sup> $\pm$ 4.5	39.3 <sup>a</sup> $\pm$ 0.7	33.6 <sup>a</sup> $\pm$ 1.5	38.8 <sup>a</sup> $\pm$ 2.9	36.9 <sup>a</sup> $\pm$ 2.3
Clay <2 $\mu\text{m}$ (%)	9.5 <sup>c</sup> $\pm$ 0.2	16.1 <sup>ab</sup> $\pm$ 1.6	10.9 <sup>c</sup> $\pm$ 0.7	18.8 <sup>a</sup> $\pm$ 3.7	12.4 <sup>bc</sup> $\pm$ 0.8
CEC (meq/100 g)	7.8 <sup>c</sup> $\pm$ 0.4	10.9 <sup>a</sup> $\pm$ 0.2	8.6 <sup>c</sup> $\pm$ 0.5	9.7 <sup>b</sup> $\pm$ 0.1	8.2 <sup>c</sup> $\pm$ 0.1
pH	8.0 <sup>b</sup> $\pm$ 0.0	8.2 <sup>a</sup> $\pm$ 0.0	8.2 <sup>a</sup> $\pm$ 0.0	8.1 <sup>ab</sup> $\pm$ 0.1	8.2 <sup>ab</sup> $\pm$ 0.0
Organic matter (%)	2.8 <sup>a</sup> $\pm$ 0.1	2.6 <sup>ab</sup> $\pm$ 0.1	2.4 <sup>b</sup> $\pm$ 0.0	2.3 <sup>b</sup> $\pm$ 0.1	2.5 <sup>ab</sup> $\pm$ 0.1

Note: Means followed by the same letter are not significantly different ( $p < .05$ ).

TABLE 2 Physico-chemical properties and mineralogy of waste and industrial by-product.

Physico-chemical properties								
	Sand 50–2000 $\mu\text{m}$ (%)	Silt 2–50 $\mu\text{m}$ (%)	Clay <2 $\mu\text{m}$ (%)	OM (%)	CaCO <sub>3</sub> (g/kg <sup>-1</sup> )	pH	CEC (meq/100 g)	
ExS	24.0 $\pm$ 3.0 <sup>a</sup>	39.5 $\pm$ 2.9 <sup>a</sup>	35.2 $\pm$ 0.8 <sup>b</sup>	1.3 $\pm$ 0.2 <sup>a</sup>	1 $\pm$ 0 <sup>a</sup>	5.6 $\pm$ 0.1 <sup>a</sup>	20.2 $\pm$ 1.4 <sup>a</sup>	
QS	23.5 $\pm$ 5.7 <sup>a</sup>	29.1 $\pm$ 1.7 <sup>b</sup>	46.8 $\pm$ 4.2 <sup>a</sup>	0.55 $\pm$ 0.1 <sup>b</sup>	1 $\pm$ 0 <sup>a</sup>	5.6 $\pm$ 0.8 <sup>a</sup>	12.2 $\pm$ 0.7 <sup>b</sup>	
Mineralogy (%)								
	Quartz	Kaolinite	Smectite/ interstratified illite	Illite	Feldspath	Plagioclase	Amphibole	Goethite
ExS	24.5	6.3	32.4	4.3	3.3	23	1.5	4.9
QS	18.1	45.4	13.7	10.4	5	<0.5	-	7.2

Note: Mean values followed by the same letter are not significantly different ( $p < .05$ ).

Abbreviation: OM, organic matter content.

trace elements are within the range of values commonly observed in French soils, and therefore do not indicate any contamination issue (see Appendix S1).

The application of clay-rich waste and by-products at a rate of 34% significantly increased the granulometric clay fraction of the amended soil. Specifically, it increased by up to 68% and 97% in ExS 34% and QS 34%, respectively, compared with the control. The clay fraction of soil amended with 11% of clay-rich material increased by about 14% and 30% respectively in ExS 11% and QS 11%, although these differences were not statistically significant compared with the control.

The addition of mineral amendments also contributed to an increase in the CEC of the different soil treatments compared with the control. The CEC increased by up to 39%, 9%, 24% and 4% respectively in ExS 34%, ExS 11%, QS 34% and QS 11%. The treatments receiving higher amounts of clay materials (ExS 34% and QS 34%) showed a significant increase in CEC. However, the addition of these materials had minimal effect on soil pH. Soil organic matter decreased slightly in soil amended with QS 34% and ExS 11% compared with the control. The study assessed the impact of clay amendment on lettuce production in comparison with the control treatment. The results indicate that the average head weight and total yield of fresh lettuce ranged from 141 to 340 g plant<sup>-1</sup> and from 26 to 41 t ha<sup>-1</sup>, respectively (Table 3, Figure 1e,f). Lettuce

yields did not vary significantly among the different treatments and did not show a clear trend in relation to the application of the clay amendment.

### 3.2 | Impact of clay amendment on soil structure stability

After 250 days of cultivation, soil aggregate stability was measured in the different soil treatments. The results showed that application of QS at a rate of 34% improved aggregate stability compared with the control soil. Initially, aggregates formed in the control were unstable, as indicated by the low MWD values, which ranged from 0.70 to 0.73 (Figure 2a). The MWD values in the QS 34% treatment ranged from 0.90 to 1.04, indicating medium-stability aggregates compared with the unstable aggregates observed in the control soil. However, the addition of ExS at the same rate resulted in a deterioration of soil aggregate stability, with MWD values ranging from 0.39 to 0.50 for ExS 34%. Soil treatments containing QS and ExS at a rate of 11% did not show a significant difference in aggregate stability compared with the control soil.

The soil management practices were similar across all soil treatments, therefore differences in soil aggregation between treatments cannot be attributed to differences in that respect. The clay content and type of applied material

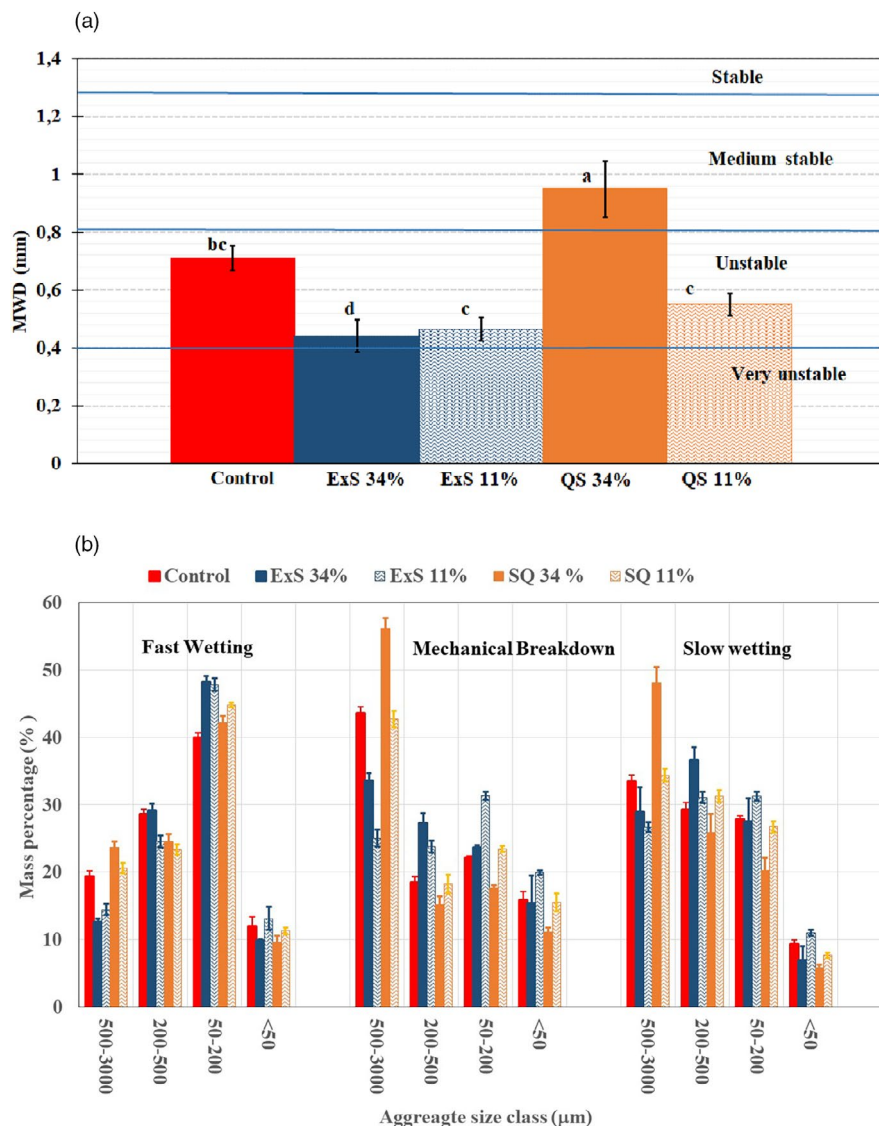
TABLE 1 Physico-chemical characteristics of the control soil and the soil amended with ExS and QS at two rates (34% and 11%).

**TABLE 3** Head weight and total yield for the three growing seasons and different treatments: control and soil amended with ExS and QS at two rates (34% and 11%).

	Head weight (g plant <sup>-1</sup> )			Total yield (t ha <sup>-1</sup> )		
	First season	Second season	Third season	First season	Second season	Third season
Control	340 ± 56 <sup>a</sup>	205 ± 23 <sup>a</sup>	171 ± 62 <sup>ab</sup>	31	41	26
ExS 34%	185 ± 50 <sup>c</sup>	165 ± 25 <sup>b</sup>	193 ± 34 <sup>a</sup>	29	33	33
ExS 11%	237 ± 58 <sup>bc</sup>	205 ± 25 <sup>a</sup>	180 ± 28 <sup>ab</sup>	28	41	31
QS 34%	258 ± 54 <sup>ab</sup>	141 ± 22 <sup>b</sup>	173 ± 30 <sup>ab</sup>	34	28	37
QS 11%	270 ± 86 <sup>ab</sup>	166 ± 32 <sup>b</sup>	157 ± 30 <sup>b</sup>	35	33	35

Note: Head weight is the average fresh weight of individual harvested lettuce heads. Total yield is the sum of all fresh weight of lettuce harvested from each treatment at the end of the experiment as the weight per growing area of lysimeter. Mean values followed by the same letter are not significantly different ( $p < .05$ ).

**FIGURE 2** (a) Mean weight diameter (MWD) measured with fast wetting, mechanical breakdown and slow wetting tests on aggregates of 3–5 mm collected from control and soil amended with excavated soil (ExS 34%, ExS 11%) and quarry sludge (QS 34% and QS 11%). Values followed by different letters are significantly different at  $p < .05$  by the Bonferroni test. (b) Fragments size distribution recorded after the three tests of aggregate stability.



appear to be more likely causes of the evolution of soil aggregate stability. An increase in soil clay mineral content can have both positive and negative effects on aggregate stability (Lado et al., 2004). On one hand, it can act as a cementing material and enhance stability. For

the mechanical breakdown and slow wetting tests, QS 34% was the most stable. The greatest response in stable macroaggregate formation occurred in the soil treatment receiving QS characterized by its high clay and oxide content. Eighty four percent of this material consists of



clay minerals. Various authors (Ayoubi et al., 2022; Diouf et al., 1990; Pi et al., 2020) have reported that amending soil with phlogopite, bentonite and kaolinite increases soil shear strength and aggregation because of their binding effects as soil clay content increased.

On the other hand, soil mineral content can promote differential swelling and increase the volume of entrapped air, which can lead to aggregate slaking and microcracking. The disaggregation of ExS 34% samples, which has a high inter-stratified illite-smectite content, occurred when aggregates were immersed in water or wetted because of the effect of entrapped air and differential swelling (Figure 2b). The stability of the aggregates in QS 34%, with mixed (2:1 and 1:1) clay mineralogy, is highest because differential swelling is limited because of the low content of swelling clay. Ayoubi et al. (2022) showed that different clay types have different mechanical behaviours, such as soil dispersion. They observed that swelling clay, such as montmorillonite, forms aggregates more easily but can also easily break down because of swelling upon wetting and lead to increased splash erosion. Several studies have reported that soils with mixed clay mineralogy have higher aggregate stability compared with other soils dominated by swelling clay (2:1) (Six, Elliott & Paustian, 2000). Mixed mineralogy can result in different binding mechanisms, such as electrostatic binding between 2:1 clays and organic matter functioning as a binding agent between 2:1 and 1:1 clays (Denef et al., 2002). The presence of a high amount of oxides in QS material can also lead to additional binding mechanisms, such as between 1:1 clays and oxides (i.e., mineral-mineral bonds) (Oades & Waters, 1991; Tisdall & Oades, 1982). Oxides play a crucial role as cementing and binding agents of clay and soil organic matter because of their large specific surface area and variable charge (Fukumasu et al., 2022). Our results are also consistent with previous studies by Mermut et al. (1997) and Burroughs et al. (1992), who found that soils dominated by swelling clays have low aggregate stability, while highly stable aggregation is attributed to oxides and kaolinite clays.

The comparison of aggregate size distributions observed with slow wetting and breakdown tests showed that soil treatment ExS 34% had the highest proportion of microaggregates (50–200  $\mu\text{m}$ ) and macroaggregates (200–500  $\mu\text{m}$ ). On the other hand, QS 34% had the highest proportion of large macroaggregates >500  $\mu\text{m}$ . More than 50% of the total aggregates were >500  $\mu\text{m}$ . The results indicate that different clay minerals have contributed differently to the formation and stabilization of different aggregate size classes, which is consistent with the findings of Fernández-Ugalde et al. (2013). These authors observed that clay minerals were selectively distributed within different aggregate size classes in the soil. For example, the accumulation of swelling clay minerals in microaggregates was greater than in macroaggregates. The

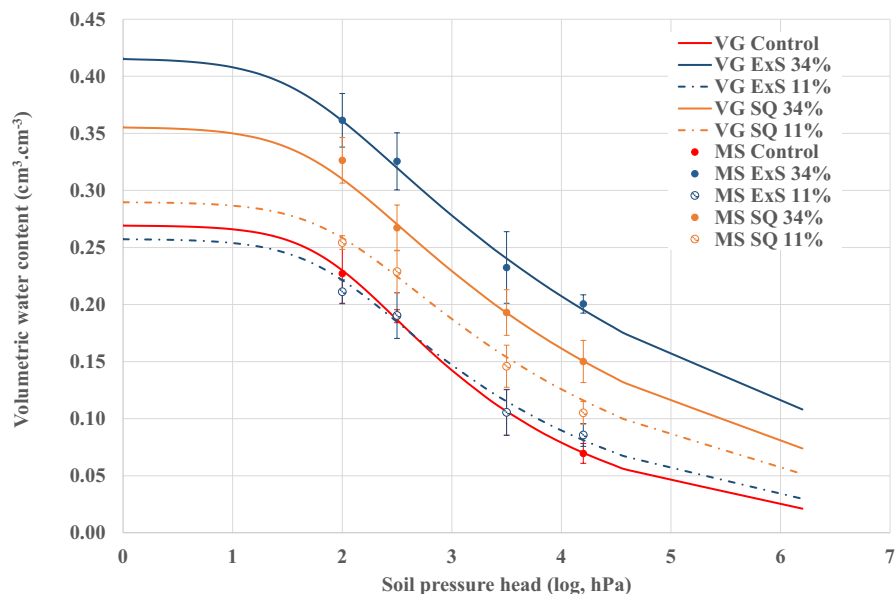
enrichment of microaggregates with swelling clay phases is explained by the reactivity of these mineral phases, such as their large specific surface area and mechanical properties, as well as the fact that they were observed to be smaller than illite and kaolinite particles in soils.

### 3.3 | Effect of clayey amendments on soil water retention and PSD

The application of 34% rich-clay materials to the soil significantly increased the water content at various soil pressure heads compared with the control soil (Figure 3). However, there were no significant differences for soil treatments receiving only 11% of these materials. The adjusted parameters of the van Genuchten model, particularly ' $\alpha$ ' (related to air-entry pressure head) and ' $\theta_s$ ' (water content at saturation), were higher in soils amended with 34% of materials compared with the control soil and in the case of soil treatments with 11% of materials, indicating an increase in porosity likely associated with the clay input (Appendix S2). Dempster et al. (2012) and Ogunniyi et al. (2021) showed that amendment with bentonite and kaolinite increased the water-holding capacity of the soil. Clay in the soil creates micropores and menisci, which generate capillary forces and increase the specific surface area of the soil matrix leading to greater water adsorption (Hillel, 1998). Capillarity and adsorption are the two phenomena that determine soil water retention. Therefore, soil treatments QS 34% and ExS 34%, which are characterized by high clay content and CEC, promote the retention of water. The addition of 11% clay materials has an insignificant effect on soil water retention. This can be explained by the fact that the clay content and CEC of the soil amended with 11% of waste was not significantly different from the control.

The PSD of the soil was altered by the addition of the clayey amendments compared with the control soil (Table 4). The total porosity increased proportionally with the amount of clay added, with a significant increase observed at a 34% application rate. ExS 34% and QS 34% showed a 51% and 34% increase in total porosity, respectively, compared with the control. The percentage of residual pores less than 0.2  $\mu\text{m}$  increased from 25% in the control to 46% for ExS 34% and 41% for QS 34%. The study revealed that only QS 34% showed a corresponding increase in storage pores (30–0.2  $\mu\text{m}$ ) compared with the control, although the effect was not significant. The addition of clay materials did not significantly affect drainage or air-filled pores (>30  $\mu\text{m}$ ). This suggests that this practice only resulted in a pronounced increase in small pores compared with larger ones, which can be attributed to two mechanisms. The first mechanism is that the added material introduces clay particles that consist mainly of small pores, such as

**FIGURE 3** Comparison of soil water retention curves (SWC) between treatment without mineral amendment (Control) and treatments amended with two mineral amendments (ExS, excavated soil; QS, quarry sludge) at two application rates (34% and 11%). SWC expressed as volumetric water content,  $\theta_s$  ( $\text{cm}^3 \cdot \text{cm}^{-3}$ ), versus Log matric potential head. VG and MS indicate respectively van Genuchten model and measured data.



**TABLE 4** Pore size distribution ( $\text{cm}^3 \cdot \text{cm}^{-3}$ ) in different treatments compared with the control soil.

	Control	ExS 34%	ExS 11%	QS 34%	QS 11%
Drainage or air filled pores ( $>30 \mu\text{m}$ )	$0.04 \pm 0.03^a$	$0.04 \pm 0.02^a$	$0.04 \pm 0.02^a$	$0.03 \pm 0.01^a$	$0.02 \pm 0.02^a$
Storage pores ( $30\text{--}0.2 \mu\text{m}$ )	$0.16 \pm 0.01^a$	$0.16 \pm 0.01^a$	$0.14 \pm 0.02^a$	$0.18 \pm 0.03^a$	$0.16 \pm 0.01^a$
Residual pores ( $<0.2 \mu\text{m}$ )	$0.06 \pm 0.01^d$	$0.19 \pm 0.01^a$	$0.08 \pm 0.00^{cd}$	$0.14 \pm 0.03^b$	$0.09 \pm 0.02^c$
Total porosity	$0.27 \pm 0.05^b$	$0.42 \pm 0.02^a$	$0.26 \pm 0.05^b$	$0.36 \pm 0.06^a$	$0.29 \pm 0.02^b$

Note: Values followed by different letters are significantly different at  $p < .05$ .

microporosity with radii  $<0.5 \mu\text{m}$  (Hurst & Nadeau, 1995; Wang et al., 2020). According to Giannetta et al. (2021), kaolinite, illite and illite-smectite have microporosity values of 42%, 67% and 65% of the total porosity, respectively. It seems that kaolinite has fewer micropores than the other clay samples. The difference in residual pore content between QS 34% and ExS 34% may be because of the higher kaolinite content in QS 34%, while ExS 34% has a higher content of illite and illite-smectite. Clay particles also have a tendency to fill pores larger than themselves, which can lead to the sealing or reduction of the size of existing larger soil pores.

The measured drainage or air-filled porosity ( $>30 \mu\text{m}$ ) ranged from 0.01 to  $0.06 \text{ cm}^3 \cdot \text{cm}^{-3}$ . An air-filled porosity value of  $\geq 0.10 \text{ cm}^3 \cdot \text{cm}^{-3}$  is recommended to achieve good soil aeration and minimal crop damage (Reynolds et al., 2009; Topp et al., 1997). The results indicate that all soil treatments, including the control, have poor drainage, which can adversely affect the ability of the soil to quickly drain excess water, reduce soil aeration, inhibit soil biochemical processes and impede root growth. The ability to store and provide water available to plant roots is indicated by ‘storage or available water’, which ranged from 0.14 to  $0.18 \text{ cm}^3 \cdot \text{cm}^{-3}$  across the different treatments. Values equal to or greater than  $0.15 \text{ cm}^3 \cdot \text{cm}^{-3}$  are considered ‘good’ for root growth and soil function. The addition

of clay-rich waste did not increase available water or drainage porosity compared with the control soil. This lack of increase can be attributed to the dynamics in soil porosity associated with clay-rich material application. It primarily increases residual soil pores, which retain water at a greater soil water potential than that what plants can generate to extract water (Lal & Shukla, 2005). Jiao et al. (2021) showed that small pore volume increases with soil clay content at the expense of the larger pores. The QS 34% and ExS 34% treatments exhibited a narrow or poorly graded PSD. A wide or well-graded PSD is better than a narrow or poorly-graded one because it can quickly drain off excess water, minimizing poor aeration, while also retaining water essential to the crop, thereby alleviating the negative effects of droughts (Topp et al., 1997).

### 3.4 | Interest and limits of our study

Our study focused on examining the isolated effect of adding clay to enhance soil properties. The results show that clay has the potential to improve soil physical properties related to soil erosion. However, this potential is limited by two main factors. First, the effect of adding clay depends on the amount and mineralogy of the clay introduced, as well

as the presence of other binding agents such as oxides. For example, ExS 34% had low aggregate stability because of the abundance of swelling clay minerals, whereas QS 34%, which had a mixed clay mineralogy and a significant oxide content, showed higher aggregate stability. Second, our findings suggest that the impact of clay addition is primarily observed at the scale of the smallest pores, such as residual pores. This observation may clarify the limitations of the benefits obtained from clay application. Soil aggregates have a hierarchical structure that is maintained by multiple types of binding agents operating at different levels of hierarchy. Fukumasu et al. (2022) investigated the relationships between soil organic carbon (SOC), clay content and PSD across a wide range of topsoil SOC and clay contents. These authors observed that soils with high clay content require a higher SOC content to increase plant-available water and soil hydraulic conductivity. Additionally, a large porosity in the 0.2–720  $\mu\text{m}$  diameter class is positively correlated with SOC content. Johannes et al. (2017, 2023) and Sauzet et al. (2024) also reported that with increasing clay content, a larger SOC content was necessary to obtain a resilient soil structure and promote soil pores responsible for drainage and storage. Boix-Fayos et al. (2001) suggested that the water stability of microaggregates is dependent on soil clay content, while the water stability of the macroaggregates is dependent on soil organic matter content. The significance of organic matter in 2:1 clay-dominated soils was demonstrated in two ways: first, as the primary binding agent because of polyvalent metal–organic matter binding with clay, and second, to reduce the extent of clay swelling because of the hydrophobic characteristics of soil organic matter, thereby reducing the extent of aggregate disruption (Chenu et al., 2000; Six et al., 2002, 2004; Zaher et al., 2005). Further research should explore the combined effect of adding organic matter and clay material to enhance soil structure and promote a PSD that fulfils multiple functions. Another area that would benefit from further research concerns the method used to characterize the structure of the soil. Instead of proceeding to an assessment of aggregate stability, it would be preferable to adopt methods, e.g., based on X-ray computed tomography imaging of soils and the quantitative evaluation of pore connectivity, that are suitable to describe the structure in its undisturbed state, as recommended by Vogel et al. (2022) and Baveye et al. (2022, 2024).

## 4 | CONCLUSION

One of the key observations made in the research described in this article is that amending a soil prone to crusting and erosion with clay-rich waste and industrial by-products had a beneficial effect on soil properties. The modification of soil properties was dependent on the amount and

mineralogy of the introduced clay, as well as the presence of other types of binding agents. Experimental observations suggest that an industrial by-product, characterized by a mixed (2:1 and 1:1) clay mineralogy and high oxide content, has the potential to improve soil aggregate stability and reduce soil erosion without compromising biomass production. The application of the clay-rich material modified the distribution of soil pore sizes, increased total porosity and soil water retention, and, especially, resulted in an increase in water content held at permanent wilting point. This leads to a minor increase in porosity responsible for water movement, aeration and root growth. We also observed that different clay minerals have varying efficiencies in building aggregates and that the effect of adding clay is primarily observed at the smallest pore scale. The application of clay material increases residual soil pores, which hold water so tightly even at high soil pressure head that it cannot be extracted by roots or soil organisms. However, as important as the increasing the clay content of soils might be, more is undoubtedly necessary to improve soil quality and make soils more resilient to erosion and degradation. Additional research is required to explore the combined effect of adding organic matter and clay material to enhance soil structure and promote a PSD that fulfils multiple functions, such as soil storage and facilitating drainage. Nevertheless, the research indicates that at least some QS, an industrial by-product, could be utilized in crop production systems to improve soil quality instead of being disposed of in landfills.

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## CONFLICT OF INTEREST STATEMENT

The authors declare no competing interests.

## DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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## SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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