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Physicochemical, Morphological, and Thermal Characterization of Selected Clay Samples from Southern Libya

Aishah Ramadan Mohamed[1,2]

[1.] *Environmental Science Department, Faculty of Environment and Natural Resource, Wadi Alshatti University,*
[2.] *Libyan Center for Studies and Research in Environmental Science and Technology*

Corresponding author E-mail: *aishahr84@yahoo.com*

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ABSTRACT

Eight clay samples collected from southern Libya were characterized using descriptive observation, light-microscope examination, Munsell color estimation, and selected physicochemical tests. The samples showed clear differences in color, texture, particle appearance, moisture content, pH, electrical conductivity, particle density, shrinkage behavior, exchangeable cations, and water-extractable phosphorus. Moisture contents were low in all samples, ranging from 0.09-1.06%, which indicates that the samples were close to air-dry condition before testing. The pH values ranged from 5.49-7.31, indicating slightly acidic to near-neutral conditions. Electrical conductivity ranged from 0.20-5.27 dS.m^{-1}, with Sample 2 showing the highest value, most likely because of a higher concentration of soluble salts. Particle density ranged from 1.88-2.31 g.cm^{-3}, while the shrinkage/expansion coefficient ranged from 1.81-6.59%. Water-extractable phosphorus varied widely from 0.97-87.6 mg.Kg^{-1}, indicating substantial differences in soluble phosphorus among the samples. Drying-curve data showed limited mass loss below 200 °C, followed by more pronounced losses at higher temperatures. By 800 °C, the total mass loss ranged from approximately 8.8-15.7%, with Sample 8 showing the highest total loss. These results provide an initial basis for evaluating southern Libyan clay as a local natural resource, but further mineralogical and application-specific testing is required before industrial or environmental use can be recommended

التوصيف الفيزيائي–الكيميائي، والمورفولوجي، والحراري لعينات مختارة من الطين من جنوب ليبيا

عائشة رمضان محمد[2,1]

[1] قسم علوم البيئة ، كلية البيئة والموارد الطبيعية –جامعة وادي الشاطي ، ليبيا

[2] المركز الليبي لدراسات وبحوث علوم وتكنولوجيا البيئة

المُخلص

تم توصيف ثماني عينات من الطين جُمعت من جنوب ليبيا باستخدام الملاحظة الوصفية، والفحص بالمجهر الضوئي، وتقدير اللون وفق دليل منسل، إضافة إلى بعض الاختبارات الفيزيائية والكيميائية المختارة. أظهرت العينات اختلافات واضحة في اللون، والقوام، ومظهر الجسيمات، ومحتوى الرطوبة، والرقم الهيدروجيني، والتوصيل الكهربائي، وكثافة الجسيمات، وسلوك الانكماش، والكاتيونات المتبادلة، والفوسفور القابل للاستخلاص بالماء. كان محتوى الرطوبة منخفضًا في جميع العينات، إذ تراوح بين 0.09% و1.06%، مما يشير إلى أن العينات كانت قريبة من حالة الجفاف الهوائي قبل إجراء الاختبارات. وتراوحت قيم الرقم الهيدروجيني بين 5.49 و7.31، مما يدل على ظروف تتراوح بين الحموضة الخفيفة والقرب من التعادل. أما التوصيل الكهربائي فقد تراوح بين 0.20 و5.27 ديسيسيمنز/م، وسجلت العينة رقم 2 أعلى قيمة، ويرجح أن ذلك يعود إلى ارتفاع تركيز الأملاح الذائبة فيها. تراوحت كثافة الجسيمات بين 1.88 و2.31 جم/سم^3، في حين تراوح معامل الانكماش/التمدد بين 1.81% و6.59%. كما أظهر الفوسفور القابل للاستخلاص بالماء تباينًا واسعًا، إذ تراوح بين 0.97 و87.6 ملجم/كجم، مما يشير إلى وجود اختلافات كبيرة في محتوى الفوسفور الذائب بين العينات. أظهرت بيانات منحنيات التجفيف فقدانًا محدودًا في الكتلة عند درجات حرارة أقل من 200°م، تلاه فقدان أوضح في الكتلة عند درجات الحرارة الأعلى. وعند درجة 800°م، تراوح الفقد الكلي في الكتلة تقريبًا بين 8.8% و15.7%، حيث سجلت العينة رقم 8 أعلى فقد كلي. توفر هذه النتائج أساسًا أوليًا لتقييم طين جنوب ليبيا كمورد طبيعي محلي، إلا أن إجراء اختبارات معدنية إضافية واختبارات مرتبطة بالتطبيقات المستهدفة يعد ضروريًا قبل التوصية باستخدامه في المجالات الصناعية أو البيئية.

الكلمات المفتاحية: توصيف الطين، جنوب ليبيا، الخصائص الفيزيائية والكيميائية، لون منسل ***(Munsell color)*** ، منحنى التجفيف، الانكماش

1. Introduction

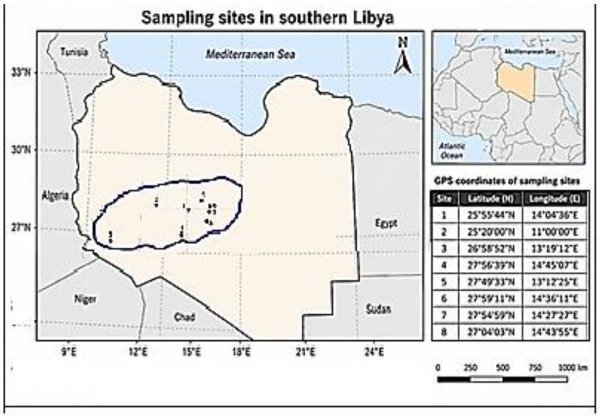
Clayey materials originate from the physical and chemical weathering of primary minerals and parent rocks. During weathering, water promotes the alteration, transport, and redistribution of mineral particles and dissolved ions, leading to the formation of clay minerals such as kaolinite, smectite, chlorite, and mica. These minerals strongly influence soil texture, water retention, ion exchange, plasticity, shrinkage, and swelling behavior (Kumari & Mohan, 2021). In soil science, the clay fraction usually refers to particles smaller than 2 µm, whereas industrial and geological definitions of clay also consider mineralogical composition and plastic behavior. Clay minerals are commonly described as hydrated aluminum silicates with a sheet-like structure. Their small particle size, large specific surface area, and surface charge make them important for nutrient retention, pollutant sorption, ceramics, cement, refractory products, and environmental remediation (Al-Ani & Sarapää, 2008; Uddin, 2017).

Desert environments can contain clayey horizons formed through long-term weathering, aeolian deposition, episodic rainfall, and downward movement of suspended particles and dissolved materials. In arid and semi-arid regions, clay-rich materials may retain water and nutrients more effectively than sandy soils, but they may also show poor drainage, volume change, salinity effects, and engineering limitations (McAuliffe et al., 2022; Peila et al., 2016). Libya contains a range of geological raw materials, including clay resources. The southern region is particularly important because of its large area, arid climate, and the samples may warrant further investigation for environmental or industrial applications, (Dhahri et al., 2024). Therefore, the present study aimed to provide a preliminary characterization of selected clay samples from southern Libya by describing their morphology, color, moisture content, pH, electrical conductivity, particle density, shrinkage/expansion coefficient, selected soluble and exchangeable ions, water-extractable phosphorus, and thermal mass-loss behavior.

2. Materials and Methods

Eight bulk clay samples, each weighing approximately 1 Kg (in three replicates), were collected from selected sites in southern Libya, including areas reported by the authors as *Marzuq, Ghat, Obari, Al-Shati,* & *Sebha*. The sample coordinates are presented in Figure 1. After collection, the samples were air-dried for three days, gently ground, sieved, and stored in dry cardboard containers until laboratory analysis (Chima *et al*., 2017).

Microscopic observations were conducted using a light microscope, and representative images were recorded for each sample (Rochow & Tucker, 1994). Color was determined in dry and wet conditions using Munsell soil color charts (Munsell Color Company, 1994). Moisture content was measured gravimetrically by weighing the sample, drying it to constant mass, and calculating water content as a percentage of the original mass (Shukla *et al*., 2014; Wuddivira *et al*., 2012). The shrinkage/expansion coefficient was determined from volume change during drying according to the approach described by Cáceres *et al*. (2021). Particle density was measured using a pycnometer. The extraction procedure was then carried out using the extract at a soil-to-solution ratio of 1:1, following standard soil analysis methods. Soil pH was measured using a calibrated pH meter, and electrical conductivity (EC) was measured using a conductivity meter and reported as Ds.m^{-1} (Corwin & Yemoto, 2019; Wang *et al*., 2025). Calcium and magnesium were estimated by EDTA titration, sodium by flame photometry, potassium using the stated soil-test method, and water-extractable phosphorus according to the phosphorus determination procedure cited in the original manuscript (Hardy *et al*., 2013; Lok *et al*., 2024; Mogashane *et al*., 2025). Thermal mass-loss behavior was assessed by heating samples at selected temperatures up to 800 °C and recording mass loss (Yu *et al*., 2024; Yariv, 2005).



GPS coordinates of sampling sites

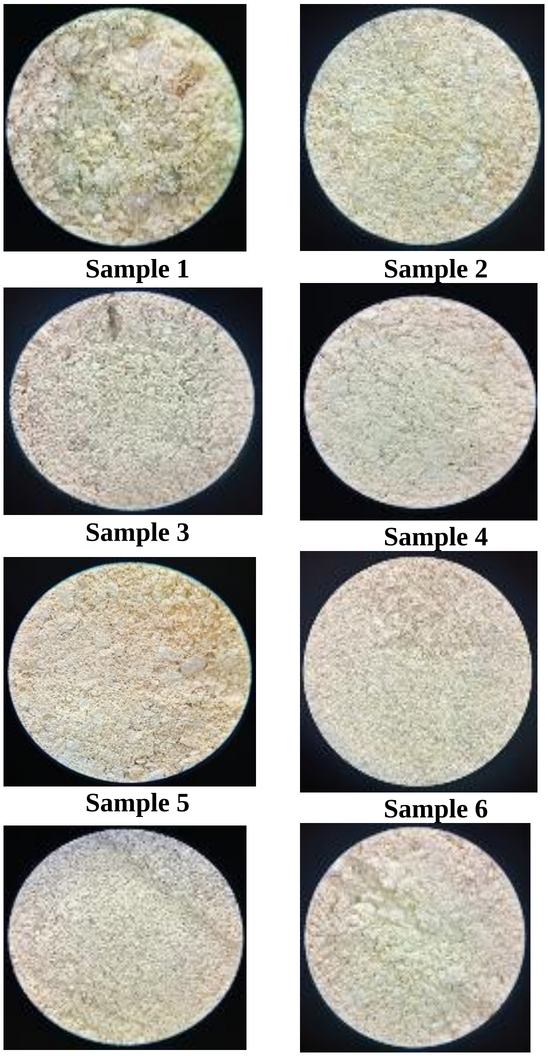
Site	Latitude (N)	Longitude (E)
1	25°55'44"N	14°04'36"E
2	25°20'00"N	11°00'00"E
3	26°58'52"N	13°19'12"E
4	27°56'39"N	14°45'07"E
5	27°49'33"N	13°12'25"E
6	27°59'11"N	14°36'11"E
7	27°54'59"N	14°27'27"E
8	27°04'03"N	14°43'55"E

Figure 1. Samples site locations

3. Results

3.1 Microscopic and descriptive observations

Microscopic examination provided qualitative information on particle appearance, color variation, visible grains, and texture. Figure 2 shows the images recorded for the eight samples. Most samples contained fine particles with visible light-colored grains or small crystals. These observations are useful as preliminary descriptors; however, mineral identification requires X-ray diffraction, FTIR, or SEM-EDS analysis rather than light microscopy alone.



Sample 1 Sample 2

Sample 3 Sample 4

Sample 5 Sample 6

Sample 7 Sample 8

Figure 2. Light-microscope images of the eight clay samples

3.2 Munsell color

The dry and wet colors of the clay samples varied across the study sites (Table 1). Light gray, white, pale yellow, pale red, olive, and very pale brown colors were recorded. These color differences may reflect variations in carbonates, iron oxides, organic matter, and redox conditions. For example, gray and light-gray colors may be associated with carbonate or salt accumulation, whereas red to yellow colors commonly suggest iron oxide staining (Elssaidi & Mohamed, 2020; Escadafal *et al*., 1989; Sirisathitkul & Sirisathitkul, 2025).The gray coloration detected in Samples 6 and 7 reflects pedogenic conditions of hyper-arid environments in southern Libya, where soil formation is by parent material rather than active weathering processes. Under extremely low rainfall and high evapotranspiration, chemical weathering and organic matter accumulation are minimal, resulting in low-chroma, high-value soils dominated by primary minerals and carbonate-rich fractions (Brady & Weil, 2017; FAO, 2001). Additionally, carbonate accumulation in Aridisols can coat soil particles and dilute pigment intensity, enhancing light gray appearances (McBratney *et al*., 2014). The consistency of color under both dry and wet conditions suggests lithogenic control with minimal pedogenic alteration, while the lack of sustained saturation prevents the development of hydromorphic features (Jenny, 1994).

Table 1. Munsell color estimates of clay samples in dry and wet conditions.

Sample No.	Color in dry condition	Color in wet condition
Sample 1	5Y 7/1 Light gray	5Y 6/2 Light olive gray
Sample 2	5Y 7/2 Light gray	5Y 5/3 Olive
Sample 3	2.5Y 6/2 Pale red	5YR 5/1 Gray
Sample 4	5Y 8/1 White	5Y 7/1 Light gray
Sample 5	2.5Y 7/3 Pale yellow	2.5Y 6/4 Light yellowish brown
Sample 6	10YR 6/1 Gray	10YR 5/1 Red Gray
Sample 7	5Y 6/1 Gray	5Y 5/1 Gray
Sample 8	10YR 8/1 White	10YR 8/2 Very pale brown

3.3 Physicochemical properties

The physicochemical properties of the clay samples are summarized in Table 2. Moisture content was low in all samples, ranging from 0.09-1.06%. Sample 1 had the highest moisture content, whereas Samples 2, 6, 7, and 8 recorded the lowest value. These low values indicate that the samples were close to air-dry condition before analysis. Moisture content is important because changes in water content influence clay shrinkage, swelling, pore structure, and mechanical behavior (Malizia & Shakoor, 2018; Soinne *et al*., 2023). The clay samples of southern Libya exhibit significant spatial variability in their physicochemical properties ($p < 0.05$), reflecting the dominance of hyper-arid pedogenic conditions, lithological heterogeneity, and weak soil-forming processes. Despite their fine texture, the soils are largely controlled by evaporative concentration, limited leaching, and calcareous parent materials typical of Saharan environments (Brady & Weil, 2017).

The pH values ranged from 5.49-7.31. Sample 1 was near neutral to slightly alkaline, whereas Sample 7 was the most acidic. The remaining samples were slightly acidic to near neutral. Such pH variation may be linked to mineral composition, soluble salts, carbonate content, and surface charge behavior of clay minerals (Barrow & Hartemink, 2023). Soil pH showed highly significant differences among the studied clay samples ($p < 0.001$), This significant variability indicates substantial heterogeneity in the geochemical environment of the soils and suggests differences in carbonate distribution, exchangeable base composition, and parent material characteristics across the sampling sites. In arid and semi-arid soils, such variability is commonly associated with irregular accumulation of $CaCO_3$ and variations in base saturation resulting from weak leaching and localized pedogenic processes (Brady & Weil, 2017). The relatively low standard deviation (SD=0.551) compared with the range of values further indicates that. Electrical conductivity ranged from 0.20-5.27 $dS.m^{-1}$. Sample 2 showed the highest EC, indicating a greater concentration of soluble salts than the other samples. Samples 3 and 4 also had moderately elevated values, whereas Samples 6, 7, and 8 were comparatively low. Because EC primarily reflects soluble ionic content, the earlier interpretation that high EC was caused by iron oxides should be replaced with a salt-related explanation (Corwin & Yemoto, 2019; Qi & Wu, 2022). (EC) exhibited significant spatial variability among the studied clay samples ($p = 0.013$). This variation indicates heterogeneous salt distribution and localized salinity development within the study area. The relatively high standard deviation (SD=1.603) further reflects substantial differences in soluble salt accumulation among sampling sites. In arid clay soils, such variability is commonly attributed to evapoconcentration processes, where intense evaporation and limited leaching promote the accumulation of soluble salts in surface and subsurface horizons (FAO, 2018).

Particle density ranged from 1.88-2.31 $g.cm^{-3}$ with a mean value of 2.115 $g.cm^{-3}$. The highest values were recorded in Samples 3 and 6, while the lowest value was recorded in Sample 2. Particle density is relevant for estimating porosity and understanding the mineral contribution to soil behavior (Horneck *et al.*, 2011). Particle density (ρs) showed a very significant difference across the clay samples ($p < 0.001$). The relatively high particle density figures indicate compact soil conditions linked to low organic matter levels, poor aggregation, and restricted biological activity, typical of arid and hyper-arid clay soils. The noted variability shows diversity in particle arrangement and mineral makeup across sampling locations. The shrinkage/expansion coefficient ranged from 1.81-6.59%. Sample 2 recorded the highest value, suggesting greater volume change during drying, while Sample 1 recorded the lowest value. Shrinkage and swelling are controlled by clay mineralogy, moisture change, pore structure, and the type of exchangeable cations present (Cáceres *et al.*, 2021; Cerato & Lutenegger, 2006). Shrink–swell behavior also showed highly significant differences ($p < 0.001$), suggesting marked variability in clay mineralogy and clay activity. The higher expansion values observed in some samples likely indicate the presence of expandable 2:1 clay minerals, particularly smectites, which possess high water adsorption and swelling capacities. In arid environments, these physical properties strongly influence soil consistency, porosity, and water movement dynamics (Brady & Weil, 2017; Sumner, 2000).

Sodium displayed an uneven distribution trend across the analyzed clay samples, with several showing no detection (ND) and others having low concentrations. This varied situation suggests localized and initial salinization rather than the formation of complete sodic soil conditions. The restricted occurrence of Na^+ indicates that salt accumulation processes vary spatially and are affected by microenvironmental factors like drainage conditions, evaporation rates, and capillary water movement. In dry and extremely dry regions, sodium build-up typically occurs due to evapoconcentration and the upward movement of saline solutions, especially where leaching is limited (FAO, 1985; FAO, 2018). Although Na^+ is present in certain samples, the exchange complex is primarily influenced by Ca^{2+} and Mg^{2+}, suggesting that calcium–magnesium systems continue to govern soil exchange processes. This dominance is crucial for preserving relative structural stability, since high sodium saturation often encourages clay dispersion, aggregate disintegration, and a decline in soil physical properties

The Ca + Mg concentration ranged from 0.18-0.34 $meq.L^{-1}$, with the highest values in Samples 7 and 8 and the lowest value in Sample 4. Calcium was highest in Samples 1 and 7, whereas magnesium was highest in Sample 8. Sodium was not detected in Samples 1, 2, 5, 6, 7, and 8 but was present in Samples 3 and 4. Potassium ranged from 0.07-0.57 $meq.L^{-1}$, with the maximum value recorded in Sample 4. These cations are relevant because negatively charged clay surfaces can attract and exchange cations without destroying the basic clay mineral structure (Bergaya & Lagaly, 2013; Kumari & Mohan, 2021). Magnesium concentration also exhibited noticeable variation, suggesting mineralogical heterogeneity among the clay deposits. In contrast, potassium concentrations remained relatively low, reflecting limited mineral weathering and low nutrient replenishment under arid climatic conditions. The restricted release of K^+ from primary silicate minerals is commonly associated with weak pedogenic alteration and minimal chemical weathering in desert soils. Furthermore, the low variability and concentrations of exchangeable potassium may indicate reduced clay–organic interactions due to the scarcity of organic matter and biological activity in the studied soils. The concentration of Ca+Mg exhibited considerable variation across the examined clay samples, especially for Ca^{2+}, Mg^{2+}, and K^{+} ($p < 0.05$). Calcium and magnesium were the primary exchangeable cations, highlighting the significant impact of calcareous parent materials and the limited leaching conditions common in arid and hyper-arid regions. The dominance of Ca^{2+} in the exchange complex indicates the common presence of carbonate minerals in Saharan sediments, which help sustain base-rich soil conditions and moderate buffering capacity (Brady & Weil, 2017)

Water-extractable phosphorus varied strongly, from 0.97-87.6 mg.Kg^{-1}. Sample 5 recorded the highest value, followed by Sample 8 and Sample 1, whereas Sample 3 recorded the lowest value. This variation suggests differences in soluble phosphorus availability, adsorption capacity, mineral composition, or associated impurities among the samples, (Schneider & Skarbøvik, 2022). Water phosphorous showed significant variation across the examined clay samples, though the differences were only marginally non-significant at the 5% probability threshold (p = 0.057). Sample 5 exhibited the highest phosphorus concentration, with Samples 8 and 1 following, while Sample 3 had the lowest level. The comparatively high standard deviation (SD=32.350) reveals significant variability in the distribution of soluble phosphorus across different sampling sites. This variation may be linked to variations in phosphorus adsorption capacity, clay mineral makeup, carbonate levels, and the existence of related mineral impurities

Table 2. Physicochemical properties of the clay samples.

Sample	W (%)	pH	EC (Ds.m^{-1})	ρs (g.cm^{-3})	Shrinkage/ expansion (%)	Ca+Mg (meq.L^{-1})	Ca (meq.L^{-1})	Mg (meq.L^{-1})	Na (meq.L^{-1})	K (meq.L^{-1})	P-water (meq.L^{-1})
1	1.06	7.31	1.66	2.00	1.81	0.24	0.20	0.04	ND	0.35	26.5
2	0.09	6.47	5.27	1.88	6.59	0.24	0.18	0.06	ND	0.14	2.1
3	0.20	6.41	2.58	2.31	2.08	0.20	0.16	0.04	0.26	0.21	0.97
4	0.18	6.00	2.28	2.14	2.60	0.18	0.08	0.10	0.26	0.57	2.02
5	0.87	6.37	1.50	2.14	3.85	0.24	0.12	0.12	ND	0.07	87.6
6	0.09	5.79	0.64	2.31	4.32	0.30	0.12	0.18	ND	0.07	14.1
7	0.09	5.49	0.74	2.14	4.68	0.34	0.20	0.14	ND	0.07	11.5
8	0.09	6.00	0.20	2.00	2.48	0.34	0.12	0.22	ND	0.14	63.5
mean	***0.334***	***6.230***	***1.859***	***2.115***	***3.551***	***0.263***	***0.148***	***0.113***	***0.260***	***0.203***	***26.036***
SD	***0.395***	***0.551***	***1.603***	***0.151***	***1.623***	***0.058***	***0.044***	***0.066***	***0.000***	***0.176***	***32.350***
p-value	***0.0480***	***<0.001***	***0.0130***	***<0.001***	***<0.001***	***<0.001***	***<0.001***	***0.0020***	***N/A***	***0.0140***	***0.0570***

Among the studied clay samples exhibited considerable P-water variability, although the differences were marginally non-significant at the 5% probability level (p = 0.057). Sample 5 recorded the highest phosphorus concentration, followed by Samples 8 and 1, whereas Sample 3 showed the lowest value. The relatively high standard deviation (SD=32.350) indicates substantial heterogeneity in soluble phosphorus distribution among sampling locations. These variabilities may be attributed to differences in phosphorus adsorption capacity, clay mineral composition, carbonate content, and the presence of associated mineral impurities. In arid and calcareous soils, phosphorus availability is strongly influenced by precipitation and adsorption reactions with calcium, carbonate and clay surfaces, which can either immobilize or locally concentrate soluble phosphorus forms (Brady & Weil, 2017). Furthermore, mineralogical heterogeneity among the clay deposits may influence phosphorus retention behavior and contribute to the observed spatial variation in extractable phosphorus concentrations. Available phosphorus shows significant variability, indicating a strong influence of carbonate chemistry and adsorption–precipitation processes common in calcareous desert soils, where P availability is typically limited.

In general, the findings indicate that clay soils in southern Libya are weak in terms of pedogenesis, dominated by calcium, and exhibit high heterogeneity. Soil characteristics are mainly influenced by arid-region processes instead of advanced pedogenic evolution, with sodium activity reflecting solely initial, localized salinity formation.

3.4 Thermal mass-loss behavior

The drying graphs are presented in Figure 3. The minor mass loss under 200 °C signifies the elimination of loosely bound and free water linked to the outer surfaces and pore areas of clay particles. Comparable low-temperature dehydration patterns have been extensively documented in clay minerals and are regarded as indicative of the removal of physically adsorbed moisture during thermal processing (Derkowski & Kuligiewicz, 2023). Significant mass losses were observed between 400 and 800 °C, likely linked to various thermally driven processes, such as the dehydroxylation of clay minerals, oxidation of organic material, and breakdown of carbonate-containing phases. Dehydroxylation is a key thermal reaction in clay minerals, involving the release of inherently bound hydroxyl groups from minerals like kaolinite, illite, and smectite when heated (Cheng *et al*., 2021; Derkowski & Kuligiewicz, 2023). Minerals that contain carbonates, especially calcite and dolomite, can also lead to mass loss at high temperatures due to thermal decomposition and the release of CO_2 (Huang *et al*., 2024). Comparable thermal changes have been reported in recent investigations of calcined clays and ceramic base materials (Hanein *et al*., 2022). Nonetheless, since complementary mineralogical analyses like X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), thermogravimetric derivative analysis (DTG), or carbonate quantification were not performed, these thermal events must be viewed with caution. Thermal analysis by itself is inadequate for the conclusive identification of the mineral phases accountable for the detected mass losses, particularly in diverse natural clay systems (Derkowski & Kuligiewicz, 2023)

According to the recorded mass data, the overall mass loss at 800 °C varied between about 8.8% and 15.7%. Sample 8 demonstrated the greatest total mass loss, with Sample 4 next, while Samples 3 and 7 revealed the lowest figures. This amendment is crucial because the previous analysis erroneously claimed that Sample 8 exhibited the least weight loss. The differences in thermal mass loss observed in the analyzed samples probably indicate variations in mineral composition, hydroxyl-containing phases, carbonate levels, and organic material concentration. Comparable connections between thermal properties and clay makeup have been observed in recent studies of natural clays and ceramic substances (Plevová & Vaculíková, 2024; Wersin *et al*., 2024). As a result, the drying data reveal significant compositional diversity among the examined samples and highlight the necessity of thorough mineralogical analysis prior to suggesting industrial or ceramic uses.

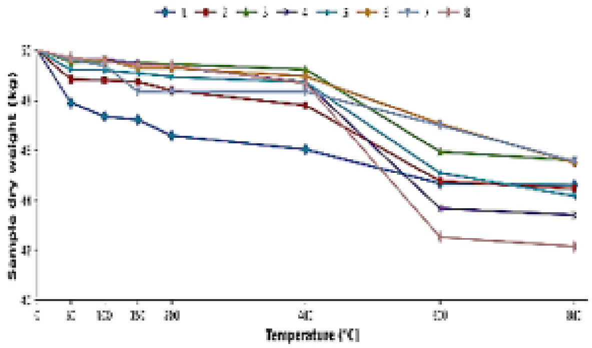


Figure 3. Drying curves of the clay samples based on mass loss from 0 to 800 °C.

Conclusions

Eight clay samples from southern Libya showed measurable variation in color, texture, moisture content, pH, electrical conductivity, particle density, shrinkage/expansion coefficient, soluble and exchangeable cations, water-extractable phosphorus, and thermal mass-loss behavior. The samples were generally low in moisture and ranged from slightly acidic to near neutral in pH. Sample 2 showed the highest electrical conductivity and shrinkage/expansion coefficient, while Sample 5 showed the highest water-extractable phosphorus. Drying curves indicated low free-water content and notable high-temperature mass losses, with Sample 8 showing the highest total mass loss by 800 °C. Overall, the results provide preliminary evidence that southern Libyan clay is a potentially useful natural resource. However, future work should include mineralogical characterization by XRD, microchemical analysis by SEM-EDS, particle-size distribution, plasticity index, cation exchange capacity, carbonate/organic matter determination, and performance testing for the intended environmental or industrial application.

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Conflict of interest: The authors declare that there are no conflicts of interest

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