

Geological, Mineralogical and Physical Properties of Aswan Kaolinitic Clays, Egypt: Implications for Industrial Applications

Hossam K. Sharaka^{1*}, Hatem M. El-Desoky²,
Mohamed W. Abd El-Moghny², Nabil A. Abdel Hafez², Sayed A. Abuelleban¹

¹ Egyptian Mineral Resources Authority, 3 Salah Salem Rd., Abbassiya, P.O. Box 11517, Cairo, Egypt

² Al-Azhar University, Faculty of Science, Geology Department, Nasser City, P.O. Box 11884, Cairo, Egypt

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Abstract

The upper Cretaceous succession from Northeast Aswan-Egypt consists of the Nubia group (Abu Aggag, Timsah, and Umm Barmil formations). This work is aimed at characterization and evaluation of refractory properties of some selected clay deposits in Northeast Aswan, Egypt to ascertain whether its suitability as a refractory material and for relevant application in Egyptian manufacturing industries. The ceramic properties depend on the clay mineral contents and the degree of crystallinity. The chemical and mineralogical composition have indicated that iron and titanium oxides (hematite and anatase) are considered accessory (coloring) minerals. No emissions of HCl and SO₂ to the atmosphere were noticed through the treatment process of these clays that indicates it is environmentally safe. The primary physical properties (e.g. apparent porosity, water absorption, apparent specific gravity, and bulk density) were used to evaluate the product quality and to determine the industrial applications of the different refractory products.

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1. Introduction

The exposed sedimentary succession in the studied area belongs to upper-Cretaceous sediments and can be subdivided into three rock units depending on the stratigraphic position. These units are related to the Nubian Sandstone (Group) Facies, namely Abu Aggag, Timsah, and Um Barmil formations. The three formations are separated by major unconformities (Fawwaz et al., 2017). Attia (1955) classified the Nubian Sandstone Succession into three groups, from base to top as follows: Group (I), Group (II), and Group (III). El-Naggar (1970) followed the classification of Attia (1955) and gives the group's formal names, Abu Aggag, Timsah, and Um Barmil formations, respectively.

The area is occupied generally by a clastic sequence underlain by the basement complex. Klitzsch (1986) subdivided the upper Cretaceous sedimentary sequence in the study area into three units: the basal Abu Aggag Formation, the Timsah Formation, and the uppermost Umm Barmil Formation. The first group (Abu Aggag Formation) is uncomfortably overlain the Precambrian basement rocks. The second group (Timsah Formation), uncomfortably overlies the Abu Aggag Formation and underlies the Um Barmil Formation. This Formation contains a considerable amount of economic clays and oolitic-iron ore oxides. The last group (Um Barmil Formation), uncomfortably overlain the Timsah Formation and is composed mainly of sandstone of continental origin and covered partially by Quaternary deposits. The structural pattern of the studied area is dominated by normal faulting that is related to the extensional forces affecting the studied area.

Kaolinitic clays are considered to be the most important raw materials for ceramics, refractories, paper, paints, pharmacy, etc. (Grim, 1962; Jepson, 1984; Murray 1999, 2000; Harvay and Lagaly, 2006; Christidis, 2011). Refractory materials are those that are physically and chemically capable of resisting high temperatures. High-quality refractory materials resist high-temperature fluctuations between 1000 °C and 1500 °C and are also good as thermal and electrical insulators (Ameh and Obasi, 2009; Umaru et al., 2012). Ball clay has a temperature of deformation (melting) at 1670 °C, whereas after firing became denser and vitreous (Hosterman, 1984; Wilson, 1998). The primary raw materials (e.g. kaolinite, chromite, and magnesite) are used for the production of various refractory products (Umaru et al., 2012). Aluminosilicate sources (e.g. kaolinite mineral) and magnesite refractory products are the main types used in the Nigerian manufacturing industries (Omowumi, 2001).

El-Desoky et al. (2019) studied the Cretaceous and Oligocene clay materials of the Bahariya Oasis for the first time to use in the ceramic industry, especially vitrified clay pipes for sewers. They assigned that these raw materials are more appropriate for manufacturing the clay pipes because their clay materials are mainly kaolinite that needs the lowest water content during formation and this means low thermal energy in the drying process. Baioumy and Ismail (2014) assumed the suitability of the Aswan ball clays as good quality and environmentally friendly raw materials for ceramic and refractory industries. Farouk et al. (2020) study the rock units which contain high amounts of kaolinitic materials of the Cretaceous age in the application for sewage

* Corresponding author e-mail: hossam.sharaka_2014@yahoo.com

clay pipes. They reported that the technological properties of the processed samples showed positive results for all requirements with the Naqb El Sillim Formation; meanwhile, the Malha and Abu Aggag formations showed some negative results in water absorption and chemical resistance.

The present paper aims to study the geology of an area extending around Wadi Abu Aggag and Wadi Abu Subeira (Figure 1) Northeast Aswan, and located between latitudes 24° 05' - 24° 15' N and longitudes 32° 50' to 33° 00' E. Also, the occurrence, refractory properties, and suitability of Aswan ball clay to be used in industry will be presented in this work.

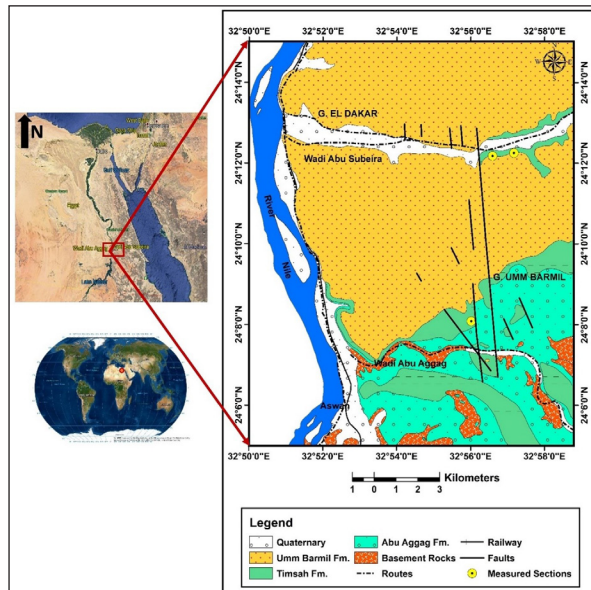


Figure 1. Landsat image and Geological map showing the studied sections at Wadi Abu Aggag (AG) and Wadi Abu Sobeira (AS) Northeast Aswan, Egypt (modified after Attia, 1955)

2. Geologic Setting

The area of study is mainly built up of Precambrian (igneous and metamorphic) rocks at the base and sandstones and clays of the Nubian Sandstones Series (Upper Cretaceous) in the upper part. The igneous rocks are composed of diorites, granodiorites, and red granites (Aswan granite), while the metamorphic rocks are mainly schists with subordinate gneisses. Nevertheless, the sedimentary sequence in the studied areas is represented by three units: the basal Abu Aggag, the Timsah, and the uppermost Um Barmil formations.

The clay-bearing formation is made up of clayey sandstone alternating with clay, siltstone, and iron-rich beds. The clastic beds are related to the Timsah Formation. Clays are varicolored (pale to dark grey, light grey, reddish grey, brownish grey, and yellowish-grey), massive to laminated, moderately hard, fragmented with some detrital quartz, and iron oxide minerals (Figures 2 and 3). Abundant and/or minor amounts of carbonaceous materials, with some gypsum veinlets, increase upwards. The sandstone is greenish-white, grey, reddish grey, brownish yellow, fine to coarse-grained, cross-bedded, ripple-marked, and indurated very hard. The total thickness of the middle group ranges between 20 meters to 45 meters.

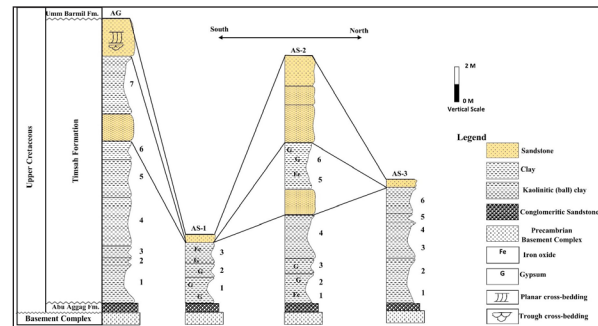


Figure 2. General lithostratigraphic columnar sections of Abu Aggag (AG), and Abu Subeira (AS-1, 2, and 3) at Northeast Aswan, Egypt.

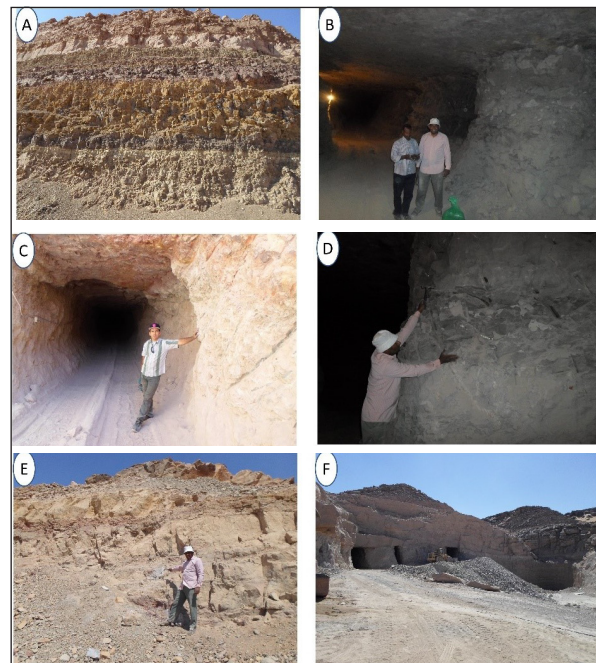


Figure 3. Field photographs showing Aswan kaolinitic clays; A) varicolored kaolinitic clays at Wadi Abu Aggag; B) room and pillar method for raw material extraction; C) the passage for underground mining; D) pale to dark grey bed with plant remains; E) sandstone cap-rock of the upper part of the Timsah Formation at Wadi Abu Subeira; and F) operating interface at Wadi Abu Subeira.

2.1. Abu Aggag Formation:

The Abu Aggag Formation was first introduced by El-Naggar (1971) to describe the sedimentary rocks unit that overlies the basement rocks and underlies the Timsah Formation at Wadi Abu Aggag northeast Aswan. It is distributed underneath the Timsah Formation and occasionally without overburden, especially in the areas close to the basement complex. The most characteristic features are conglomerate, pebble-bands, and kaolinitic sandstone with paleosol in the lower part. Abu Aggag Formation is composed of basal conglomerate, kaolinitic beds, clays, sandstones, sandy clay to clayey sandstone. The thickness of this unit ranges from a few meters to about 50 meters. The total thickness depends on the elevation of the Precambrian rocks upon which the deposition of the sandstone series has taken place. Attia (1955) gave a Cenomanian age to this Formation, while El-Naggar (1970) gave a Turonian age.

2.2. Timsah Formation

The Timsah Formation was named firstly by Attia (1955) for the siltstone and fine-grained sandstone with thick claystone intercalations exposed at Gebel Timsah, Northeast Aswan. It uncomfortably overlies the Abu Aggag Formation and uncomfortably underlies Um Barmil Formation. Stratigraphically, Timsah Formation lies between the fluvial cycles of the Abu Aggag Formation at the base and the Umm Barmil Formation at the top (Fawwaz et al., 2017). Attia (1955) assigned a Senonian age to this rock unit, while El-Naggar (1970) assigned a Coniacian-Santonian age. The total thickness ranges between 15 to 45 meters. The Timsah Formation has a relatively large reserve (~ 10 million metric tons) and the production rate is approximately 5000 metric tons per month.

The Timsah Formation at Wadi Abu Aggag (AG) and Wadi Abu Subeira (AS) (Figures 1-3) is composed of sandstone in the upper part and pale to dark grey, brownish to yellowish-grey, massive to laminated, and moderately hard clays at the base. The sandstone is greyish to greenish-white, grey, reddish-grey, brownish-yellow, and yellowish to pale-grey, fine to coarse-grained, trough and cross-bedded, and very hard to indurate. Clays characterized by varicolored ranges from (brownish to yellowish-grey, reddish-brown, violet, and whitish to pale-grey), massive, compacted, jointed, with some gypsum veinlets and iron oxide patches at Wadi Abu Aggag (Figures 2 and 3A). On the other hand, clays in Wadi Abu Subeira are light to dark grey, massive, compacted, soft soap, with gypsum veinlets which increased upward, and some planets remain (Figures 2 and 3B, D).

2.3. Um Barmil (Nubia) Formation

Russeger (1837) introduced the term "Nubia Sandstone" for these un-fossiliferous sandstones underlying the Cretaceous beds of southern Egypt and northern Nubia. Youssef (1949) was the first to describe the claystone and shales in the upper part of the Nubia Sandstone, but he did not separate these rocks as a formation. Nubia (Um Barmil) Formation uncomfortably overlies Timsah Formation and conformably underlies the Quseir Formation to the north of the study area. It is composed of sandstone, clayey sandstone, and gritty sandstone, clay, and kaolinitic beds alternated with sandstone and grey clay (Fawwaz et al., 2017). It attains a thickness of about 60 meters. El Naggar (1971) gave Santonian-Campanian age to this Formation.

3. Materials and Methods

3.1. Materials

Four litho-stratigraphic sections exposed at the studied area, one from Wadi Abu Aggag (AG) and three from Wadi Abu Subeira (AS1, 2, and 3) were fields described, measured, and sampled (Twenty-two samples). The mineralogical & chemical composition and thermal behavior of the studied clay samples were accomplished.

3.2. Methods of investigation

The four selected representative claystone samples were examined using an X-ray Diffraction analysis (XRD) automated powder diffractometer system of Philips type PanALytica equipment model X-Pert-PRO with Ni-filter, Cu-K alpha radiation ($\lambda=1.542\text{\AA}$) at 45 kV, 35 mA, and

normal scanning speed 0.03o/sec. The chemical composition of the major oxides (Twenty-two samples) was determined by using the X-ray Fluorescence (XRF) technique using a Philips model Pw/2404 at 30 Kwt. Both XRD and XRF analyses were carried out at the central laboratory sector of the Egyptian Geological Survey.

Four composite samples were examined by Differential Thermal (DTA) and Thermo-gravimetric (TGA) analyses at Housing and Building National Research Center (HBRC) Cairo, Egypt, using a computerized DT.50 thermal analyzer (Shimadzu Co., Kyoto, Japan). The heating rate was 20°C/min.; the heating temperature was up to 1000°C under a nitrogen atmosphere (30 ml/min).

The primary physical properties (e.g. apparent porosity, water absorption, apparent specific gravity, and bulk density) for four composite claystone samples were examined according to (ASTM-C20). The shrinkage test was carried out according to (ASTM-C326). The mechanical properties of these samples were determined by using a modulus of rupture test (MOR) according to (ASTM-C99 and C120). These properties are widely used in the evaluation of product quality and the determining of industrial applications of the different refractory products. The mechanical properties were carried out at the Sweillem Vitrified Clay Pipes Com., Egypt.

4. Results

4.1. Mineralogical and chemical results

The XRD results have revealed that the studied samples are mineralogically composed of quartz, kaolinite, and dolomite as well as iron oxide and anatase as accessory (coloring) minerals (Figure 4). The studied samples were plotted on a ternary diagram of $[\text{SiO}_2/\text{Al}_2\text{O}_3]/(\text{Fe}+\text{Ti}+\text{Mg}+\text{Ca}+\text{K}+\text{Na})$ can be classified as silico-aluminous clays (Figure 5).

The results of the chemical analysis have revealed high concentration values of SiO_2 and Al_2O_3 (with average values of 59.17% and 23.35%, respectively). TiO_2 and Fe_2O_3 average values are 1.97% and 5.67%, respectively (Table 1). Loss on ignition values ranges from 3.92% to 13.38% (with an average value of 7.99 wt. %). Aswan ball clay contains a low concentration of Cl and SO_3 (0.01 and 0.08, respectively). The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios range from 1.72 to 4.09, whereas the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratios range from 0.24 to 0.58. Besides, $\text{Al}_2\text{O}_3/\text{Fe}_2\text{O}_3$ ratios range from 1.22 to 15.98 (Table 1).

4.2. DTA and TGA results

The rate of firing curve changes according to the points of decomposition and loss on ignition (Földvári, 2011). These can be noted by using differential thermal (DTA) and thermogravimetric (TGA) analyses that are very important techniques to study the thermal behavior of clays for the refractory industry. The studied samples are characterized by the initial endothermic peak at around 100°C that appears as a small peak in all the studied samples. A second endothermic reaction peak appears at a range between 120 to 150 °C as a weak peak only in the samples of Wadi Abu Subeira (Figure 6).

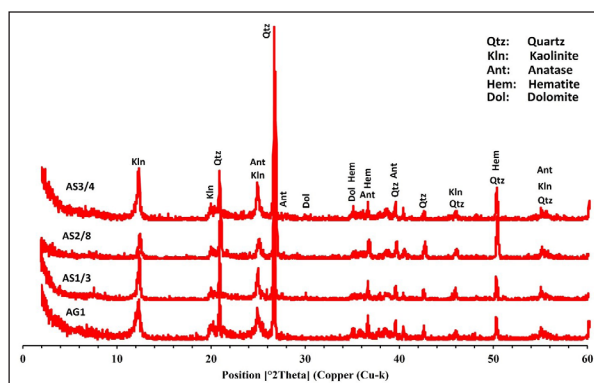


Figure 4. Representative XRD patterns of clay raw materials at Abu Aggag (AG) and Abu Subeira (AS1, 2, and 3) sections of the Upper Cretaceous kaolinitic clay at Northeast Aswan, Egypt. Note the mineral abbreviations of rock-forming minerals taken from (Kretz, 1983; Whitney and Evan, 2010).

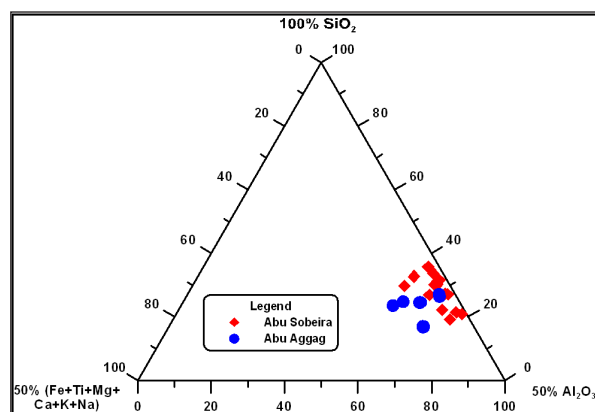


Figure 5. Ternary diagram of $[\text{SiO}_2/\text{Al}_2\text{O}_3/(\text{Fe}+\text{Ti}+\text{Mn}+\text{K}+\text{Ca}+\text{Na})]$ illustrated the chemical composition for the studied samples at Abu Aggag (AG) and Abu Subeira (AS) sections at Northeast Aswan, Egypt (after Garcia-Valles et al., 2020).

Furthermore, another endothermic reaction peak appears at 340 °C and appears small in the Abu Aggag sample. The second large symmetric major endothermic reaction peaks appear at around 520-580 °C in all the studied samples. An exothermic reaction peak appears at about 1000 °C. The

dehydration curve indicates weight (humidity) loss below 300°C with an average loss value of 0.28%, while the lost weight as a result of the dehydroxylation reaction between 500 to 600°C of kaolinite gives an average value of 3.69%.

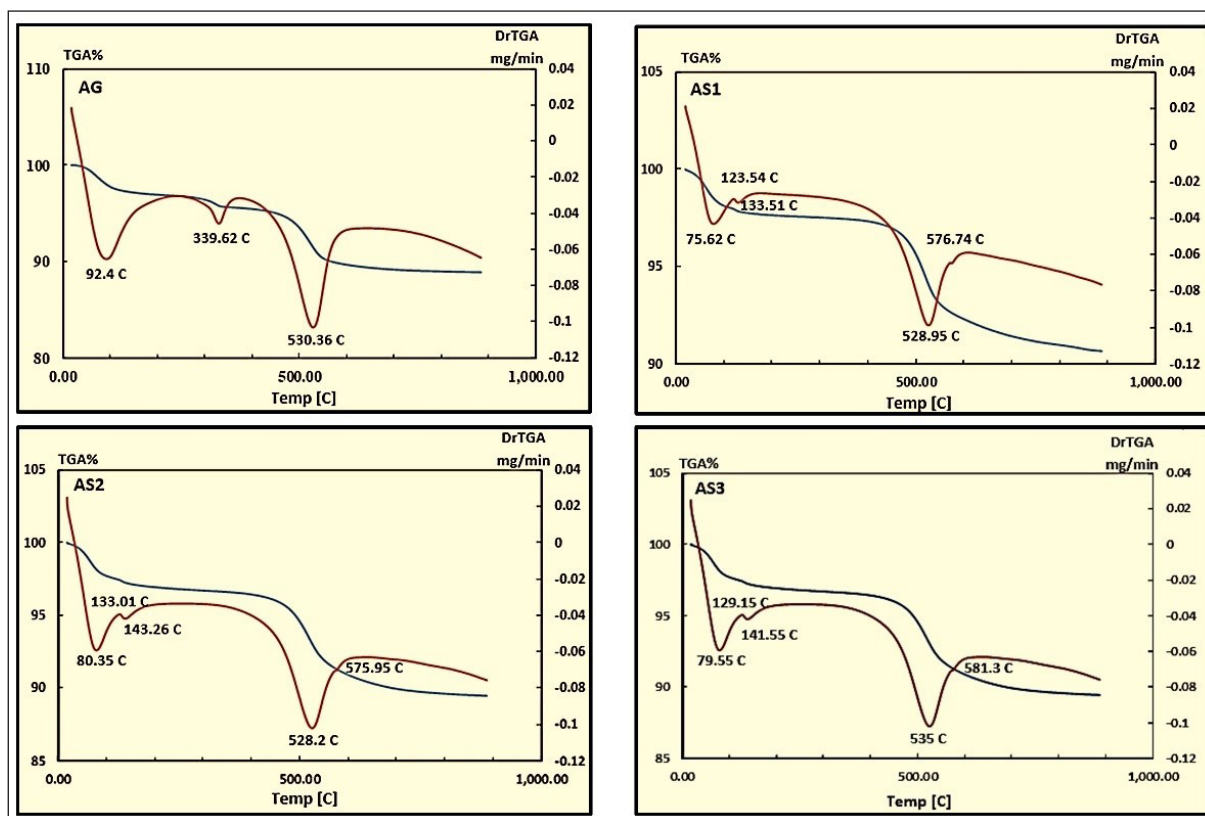


Figure 6. Differential thermal (DTA) and thermogravimetric (TG) for kaolinitic clay (Composite) samples at Wadi Abu Aggag (AG) and Wadi Abu Subeira (AS1, 2, and 3), northeast Aswan, Egypt.

Table 1. Chemical composition of the studied clays compared with other studied clay samples

Sections	S. No.	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Cl	SO ₃	LOI	Sum	Kaolinite content	Si/Al ratio	Al/Si ratio	Al/Fe ratio
Abu Subeira	AS1-1	62.36	1.49	24.27	2.36	0.01	0.69	0.11	0.01	0.93	0.01	0.01	0.21	7.52	99.98	52.84	2.57	0.39	10.28
	AS1-2	65.61	1.90	25.05	1.89	0.01	0.54	0.05	0.01	0.67	0.01	0.01	0.17	3.92	99.84	54.54	2.62	0.38	13.25
	AS1-3	54.02	2.61	28.71	2.34	0.01	0.27	0.53	0.01	0.03	0.10	0.01	0.30	10.50	99.44	62.51	1.88	0.53	12.27
	AS2-1	68.67	1.76	17.36	4.95	0.01	1.05	0.06	0.01	0.76	0.03	0.01	0.02	5.05	99.74	37.80	3.96	0.25	3.51
	AS2-2	64.65	1.81	19.93	5.41	0.01	0.95	0.06	0.01	0.73	0.03	0.01	0.06	6.07	99.73	43.39	3.24	0.31	3.68
	AS2-3	71.35	1.44	19.02	1.19	0.01	1.15	0.03	0.01	0.65	0.03	0.01	0.36	4.55	99.8	41.41	3.75	0.27	15.98
	AS2-4	70.12	1.68	19.75	2.35	0.01	0.68	0.08	0.01	0.65	0.03	0.01	0.04	4.47	99.88	43.00	3.55	0.28	8.40
	AS2-5	55.25	1.49	22.56	5.98	0.01	2.42	1.48	0.36	0.88	0.01	0.01	0.04	9.25	99.74	49.12	2.45	0.41	3.77
	AS2-6	51.00	1.49	23.22	2.91	0.01	2.68	4.30	0.01	0.67	0.01	0.01	0.04	13.38	99.73	50.56	2.20	0.46	7.98
	AS3-1	51.33	1.46	28.48	7.27	0.01	0.19	0.02	0.01	0.11	0.01	0.01	0.03	10.81	99.74	62.01	1.80	0.55	3.92
	AS3-2	58.53	1.68	26.65	2.88	0.01	0.42	0.10	0.01	0.61	0.01	0.01	0.11	8.72	99.74	58.02	2.20	0.46	9.25
	AS3-3	58.04	1.69	26.73	3.67	0.01	0.29	0.07	0.01	0.36	0.01	0.01	0.04	8.80	99.73	58.20	2.17	0.46	7.28
	AS3-4	53.12	1.63	30.96	2.70	0.01	0.06	0.04	0.01	0.08	0.01	0.01	0.03	11.18	99.84	67.41	1.72	0.58	11.47
	AS3-5	58.75	2.03	28.01	1.82	0.01	0.05	0.13	0.01	0.35	0.01	0.01	0.02	8.70	99.9	60.98	2.10	0.48	15.39
Abu Aggag	AS3-6	51.74	1.94	26.70	11.19	0.01	0.05	0.01	0.01	0.27	0.01	0.01	0.02	7.77	99.73	58.13	1.94	0.52	2.39
	AG1	60.22	2.12	23.87	3.92	0.01	0.45	0.16	0.01	0.10	0.02	0.01	0.04	8.79	99.72	51.97	2.52	0.40	6.09
	AG2	61.19	1.93	23.46	4.14	0.01	0.62	0.06	0.01	0.24	0.31	0.01	0.05	7.70	99.73	51.08	2.61	0.38	5.67
	AG3	52.06	2.47	21.54	13.33	0.01	0.90	0.13	0.01	0.54	0.01	0.01	0.01	8.73	99.75	46.90	2.42	0.41	1.62
	AG4	50.26	2.43	20.49	16.35	0.01	0.83	0.13	0.01	0.41	0.01	0.01	0.01	8.80	99.75	44.61	2.45	0.41	1.25
	AG5	62.83	1.88	15.35	12.55	0.01	2.11	0.04	0.01	0.12	0.01	0.01	0.08	4.73	99.73	33.42	4.09	0.24	1.22
	AG6	55.87	2.56	21.22	9.50	0.01	1.12	0.30	0.01	0.92	0.07	0.01	0.02	8.12	99.73	46.20	2.63	0.38	2.23
Average	AG7	55.76	3.40	21.15	9.30	0.01	0.75	0.47	0.01	0.26	0.18	0.01	0.04	8.38	99.72	46.05	2.64	0.38	2.27
	Farouk et al., 2020	58.76	1.94	23.39	5.82	0.01	0.83	0.38	0.03	0.47	0.04	0.01	0.08	7.99	99.76	50.93	2.61	0.41	6.78
	Brazil (São Simão) ball clay	52.88	1.96	24.04	7.82	0.22	0.64	0.68	0.3	1.4	0.14	0.01	0.2	9.74	99.39	52.34	2.2	0.46	3.08
		54.42	0.68	27.87	2.6	0.01	1.22	1.1	0.01	2.2	0.01	0.01	0.01	9.85	99.99	60.68	1.95	0.51	10.72

4.3. Physical properties results after thermal treatment

The water absorption, bulk density, apparent porosity, linear shrinkage, and modulus of rupture are considered as the main physical properties were determined after the thermal treatment of the studied samples as follows:

4.3.1. Bulk density

The bulk density gave values ranging from 1.72 to 1.91 gm/cm³ (with an average value of 1.82 gm/cm³). This value lies within the range of the standard Fireclay (Table 2; and Figure 7a).

4.3.2. Shrinkage

The firing shrinkage results range from 0.00 to 3.6% (with an average value of 1.8%). This value is less than the recommended range of the standard samples of 7% - 10% for fired clays (Table 2; and Figure 7a).

Smectite effects

The smectite content strongly affects the rheological behavior of the clay dispersion. Smectite has particle sizes lower than kaolinitic minerals, and when added, it increases the surface area of the solid material. Therefore, the presence of smectite minerals significantly increases

viscosity. Besides, in the absence of good quality ball clay, small smectite contents may even contribute to stabilizing the system. However, the amount of smectite cannot be too high or it can compromise the productive process (Laursen et al., 2019).

4.3.3. Water absorption

The water absorption values range from 8.28 to 14.75% (with an average of 11.52%). This value is slightly near the range of the standard Fireclay (Table 2; and Figure 7b).

4.3.4. Apparent porosity

The obtained results gave an apparent porosity value that ranges from 13.33 to 22.86% (with an average value of 18.09%) as indicated in (Table 2; and Figure 7b). This value is also less than the required value (20% - 30 for calcined clay (Gilchrist 1977).

4.3.5. Modulus of rupture

The average values of the modulus of rupture (MOR) range from 2.56 to 9.54 MN/m² (with an average value of 6.05 MN/m²). These values are less than the obtained value of the standard fireclay values of MOR range from 2 - 4 MN/m² and (Table 2; and Figure 7a-b).

Table 2. Comparison of the determined properties of investigated clay with some established refractory standards

Physical properties of fire clay	Calcined Temperature	The current study (Northeast Aswan)	Nigerian Ozanagogo clay	Standard Fireclay
Firing Shrinkage (%)	800 – 1200 C°	0.00 – 3.60	1.01	7 - 10
Bulk density (gm/cm ³)		1.72 – 1.91	2.14	1.71 - 2.1
Apparent porosity (%)		13.33 – 22.86	20.4	20 - 30
Water absorption (%)		8.28 – 14.75	-----	12 - 20
Modulus of rupture (MN/m ²)		2.56 – 9.54	8.28	2 - 4

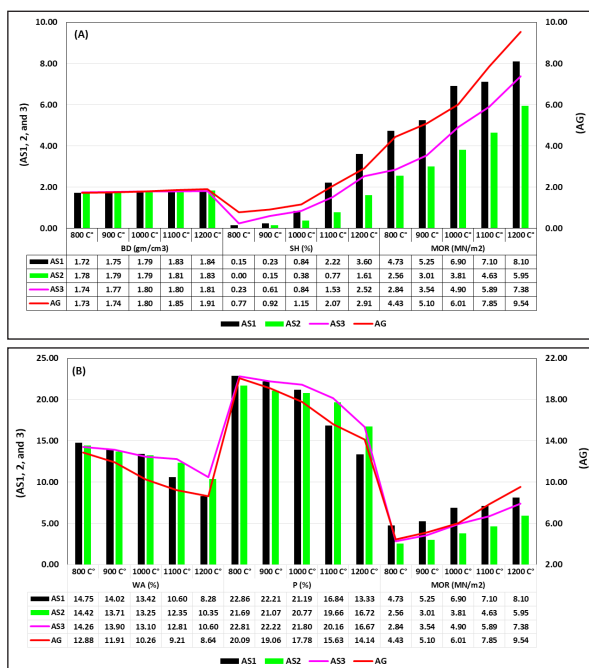


Figure 7. Illustrated the primary physical properties: A) Bulk density (BD) and Shrinkage (SH) against modulus of rupture (MOR); B) Porosity (P) and water absorption (WA) versus MOR.

5. Discussions

Plastic (Ball) clay is a term used to describe light to dark grey, fine-grained, high plasticity sedimentary kaolinitic clays. The name “ball clay” is related to an early method of mining, when traditional hand tools were used to extract the clay in rough cube shapes. Ball clay in the study area is extracted by the room and pillar method (Figure 3b) using mechanical equipment, of which the most popular are hydraulic excavators.

It was cut into 15 to 17 kg cubes and during transport, the corners of the cubes were knocked through handling and storage these cubes became rounded off leaving “balls”. It is also, sometimes referred to as plastic clay (Andreola et al., 2009). Ball clay is formed by chemical weathering of parent rocks (aluminosilicate feldspars-rich source rocks), to give kaolinite. After that, paleo-rivers and streams transport and deposit kaolinite mixed with other minerals and the carbonaceous matter (plant remains (Phlebopteris sp) Masoud et al. (2013).

The presence of iron oxides indicates a surficial oxidation process. The variation in thickness of the studied kaolinitic clays may be related to their sedimentary supply, the nature of the sedimentary basin, and the relief of the Precambrian basement rocks (Figure 2). SiO₂ and Al₂O₃ are the main

oxides in the studied samples (Table 1) that reflect their mineralogical composition. The XRD results have revealed that the high silica content is related to the high amount of quartz of terrigenous origin. Nevertheless, the silica content (50.26–71.35 wt. %) falls within the standard range of 46–62 % that is required for the production of good refractory materials as reported by Yami and Umaru (2007) from the Nigerian clays.

Furthermore, these values lie within the range of the German requirements (55–70 wt. %) and slightly near to the Belgian requirements (60–81 wt. %) (European Commission Ceramic Manufacturing Industry 2007). The degree of kaolinization and the amount of Al_2O_3 content depends upon the intensity of the chemical weathering. Meanwhile, the Al_2O_3 content of the studied samples (15.35–30.96 wt. %) is very close to the Belgian (15–27 wt. %) requirements, and less than the German requirements (20–35 wt. %) (European Commission Ceramic Manufacturing Industry 2007). The chemical composition of analyzed samples revealed that these samples belong to silico-aluminous clays (Figure 5) and are similar to Kaolinitic Clays from Terra Alta (Catalonia, Spain).

The Si/Al ratio (Table 1) is higher than the pure kaolinite (1.17) (Felix, 1977) and is attributed to the presence of high quartz content in the argillaceous sandstone. Plummer and Romary (1967); Ogbukagu (1980) suggested that the Si/Al geochemical ratio ranges from (1.6 to 2.6) for plastic fireclay, (2.4 to 4.0) for ball clay, and (3.5 to 6.7) for siliceous fireclay. This indicates the studied clay samples are close to Brazil (São Simão) ball clay. On the other hand, Al/Si ratio is lower than the value of 0.85 assigned for the ideal kaolin introduced by Schroeder et al., 2004; Ga'miz et al., 2005.

$\text{Al}_2\text{O}_3/\text{Fe}_2\text{O}_3$ ratio defines the possible uses for clays in ceramic technology. If it > 5.5 , it is considered alumina-rich, if it < 5.5 , they are iron-rich and can be used in the manufacture of building materials (e.g. bricks, tiles, etc.) Garcia-Valles et al. (2020). In the present study, the Al/Fe ratio (Table 1) ranges from 1.22 to 15.98 (with an average value of 6.78). The majority of the Abu Sobeira (AS) samples have an Al/Fe ratio > 5.5 , whereas, all samples of Abu Aggag (AG) have an Al/Fe ratio < 5.5 , except two samples (AG1 and 2) consists of Al/Fe ratios > 5.5 . This indicates that the studied samples of Abu Sobeira are useful for manufacturing refractory ceramics and are considered alumina rich than Abu Aggag samples.

Titanium oxides (e.g. Anatase) have an average value of 1.94% (Table 1). It is higher than Brazil (São Simão) ball clay of (0.68 wt. %) and average shale (0.82 wt. %) reported by Condie (1993). Also, it matches with data published by (Baïoumy and Ismail, 2014) and Farouk et al. (2020). Ti occurred as disseminated or colloidal within the clays as discrete minerals (Degens, 1965). Anatase is the main titanium mineral identified by XRD (Figure 4). Furthermore, during the weathering and deposition process of kaolinite, Ti oxides are detrital but can be adsorbed within the kaolinite structure (Murray and Keller, 1993). In the present work, it is present as adsorbed colloidal of TiO_2 and Fe_2O_3 .

Fe_2O_3 (higher than 3%) is a direct measure of the iron

mineral contents such as hematite as revealed by XRD (Figure 4); the iron content is responsible for generating the reddish color of samples after the firing process. The iron oxide contents range from (1.19 to 16.35 wt. %) is similar to the German requirements (1.0–10 wt. %) and higher than the maximum limit values of the Belgian requirements (1.0–7.4 wt. %) (European Commission ceramic manufacturing industry 2007). Clays used in the production of porcelain tile must have $\text{Fe}_2\text{O}_3 < 1\%$, while ball clays used for the production of light-firing bodies such as sanitary ware is overall accepted at a limit of 3% of Fe_2O_3 (Table 3) Laursen et al. (2019). The dark-coloring after the firing process and the decrease of firing temperature are related to the presence of an iron oxide higher than 3% (Tables 1 and 3). The decreasing firing temperature is related to the development of a large amount of liquid phase during firing. Besides, a great number of coating compounds are required when iron oxides are present in the ceramic industrial technology by high concentration (Laursen et al., 2019).

A high amount of iron oxide leads to deformation of the ceramic structure, incorporation of iron ions in the glassy phase and/or in the crystalline phases, and the development of dark-firing bodies (Jiang et al., 2017). Furthermore, the temperature required for alteration of soft clay body into a long-lasting refractory body depends on the presence of alkali elements (e.g. K, Na, and Ca) in the raw materials. The depletion and low flow agents of these elements as indicated in (Table 1) reflect that these clays require high temperatures for densification and the formation of glassy phases for the ceramification process.

Small amounts added as fluxing agents (alkaline elements) are adequate to greatly reduce the firing temperature without causing high pyro-plastic deformation of the bodies (Melchiades et al., 2011). The low content of CaO and MgO oxides is related to the low content of carbonate minerals such as dolomite (Tables 1; Figure 4). Nevertheless, neither calcite nor gypsum was detected in the XRD pattern, which suggests a micro-environmental heterogeneity of the kaolinitic clay (Masoud et al., 2013).

The contained Magnesium oxide ranges from 0.05 to 2.68 % in some selected clay samples. MgO is usually used as insulators in industrial cables and as primary refractory materials for laboratory crucibles and as a principal fireproofing ingredient in construction materials (Umaru et al., 2012). LOI reflects the mass loss during firing due to dehydroxylation of clay minerals and the elimination of organic matter. Ball clays usually have a high percentage of organic matter that can be colloidal, non-colloidal, or both (Laursen et al., 2019). The LOI of the studied clay (Table 1) was close to that of the traditional ball clay from Brazil, São Simão clay, which may indicate a lower amount of clay material or organic matter in these clays. The studied clays have mineralogical compositions close to the data of ball clays (Murray, 2007).

Thermal analysis of the studied samples (Figure 6) has a very low content of carbonaceous materials as revealed by XRF analysis where the samples have a low content of calcium and magnesium oxides that makes these samples safe in the thermal treatment during drying and firing

processes. The thermal behavior results (DTA and TGA) match the results of Grim, 1968; Todor, 1976; Smykatz-Kloss, 1974a; Kulp and Trites, 1951; Bray et al., 1998; Kallai, 2013; Laursen et al., 2019).

Bulk Density (BD) of calcined clay depends upon the specific gravity of the raw material, method of manufacturing, and degree of burning (Gilchrist, 1977). With increasing temperature, the density and strength of the calcined clay increase, while its water absorption and apparent porosity decrease. This indicates that the BD has a positive relationship with the firing temperature (Figure 7a) and is due to the increased consolidation between particles in the body (Alexander et al., 2013). The BD values of the studied samples are slightly similar to the typical bulk density for fireclay (Table 2), which ranges from 1.71 to 2.1 gm/cm³ as suggested by Gilchrist (1977).

Generally, a fireclay must have a firing shrinkage (FS) lower than 8% to retain good mechanical performance (Weng et al., 2003; Abdeen and Shihada, 2017). The firing shrinkage increases with increasing firing temperature and the formation of a glassy phase (Figure 7a). However, the low value obtained in this study (Table 2) could be attributed to thermal stability that serves as an anti-shrinkage agent (Misra, 1975).

The water absorption (WA) indicates the amount of moisture that may be absorbed and could be subjected to destructive freezing in outdoor structures. Furthermore, the apparent porosity (AP) is a measure of the amount of open pore space in the fired samples, relative to its bulk volume, and is expressed as a percent. Less than 22% of water absorption and apparent porosity is considered promising for slow-calcined materials. This should be within the limit of 12 to 20% (Table 2) according to the BS-5628: Part 1:2005 and Indonesian Standard SNI 15-2094-2000 (O'Neill and Barnes 1979; Abdeen and Shihada, 2017; Fernando, 2017).

The WA and AP have a negative relationship with firing temperature, while there is a reverse relationship between water absorption and shrinkage (Figure 7a-b); there is also a positive relationship between water absorption and porosity (Figure 7b). These results could be an indication of the relative resistance to damage during freezing and thawing. The modulus of rupture (MOR) is a measure of the fracture strength of materials against the forces of breakage or crack due to the application of a certain amount of pressure, the distance over which the force was applied, and the width and thickness of the sample (Alexander et al. 2013; Abdeen and Shihada, 2017).

The MOR values are considered slightly similar to the standard values as the calcined clay (strength ranges from 2 to 4 MN/m²) is believed to have performed well (ASTM Part 17/2005) (Table 2; and Figure 7a-b). This is related to the combined effect of both clay and organic matter content that allowed them to achieve higher strength values. The maximum means of dry density of 1.91 Mg.m³ was recorded and approximately satisfies the requirements of the minimum density of 2.0 Mg.m³ of the British Standard No. 3921 (1985) for calcined clay which stated the results of all the fired clay samples satisfy the recommended apparent porosity

and water absorption values of 22.86% according to the Indonesian Standard (SNI) 15-2094-2000 (20%) and 14.75% according to the British Standard No. 5628: Part 1 (2005) (12%). The maximum compressive and flexural strength of calcined clay (9.54 MN.m²) were recorded and are higher than the British Standard No. 3921-1985 (5 MN.m²).

The ceramic properties depend on the clay mineral content and the crystallinity of kaolinite (Leonelli et al., 2001; Andreola et al., 2002). Based on the thermal analyses (thermogravimetric peaks), it is possible to determine the crystallinity degree of kaolinite (e.g. extremely disordered <530 °C, strongly disordered 530-555 °C, slightly disordered 555-575 °C, and well-ordered >575 °C) which introduced by Smykatz-Kloss (1974b).

In the present study, the raw material is characterized by a low-ordered kaolinite structure which can be noted by DTA analyses from the large endothermic peak around 530 °C. Also, it's characterized by high plasticity and an abundance of clay fractions (less than 2 µm) reported by Baioumy and Ismael (2014) and matches the international standard for the ceramic industry. Also, Fe and Ti oxides are considered as the main impurities elements in the raw materials. These oxides affect the whiteness of the raw materials and adversely affect their quality that limited their applications. The beneficiation method to remove these impurities depends on the type, content, and distribution of the Ti and Fe oxides. However, the white-firing properties of the ceramics industry can be determined by the amounts of coloring/fluxing oxides within the clay. The quality parameters depend on Al₂O₃/SiO₂ ratio, Fe₂O₃, and TiO₂ contents.

For enhancing the whiteness and hence upgrading the quality of the deposit, the contaminated oxides could be removed by bleaching, magnetic separation, or flotation for use in paper coating and filling, and in higher-grade ceramics (Abdel-Khalek et al., 1998; Abdel-Khalek, 1999; Murray, 2007). Tableware and Sanitary ware typically include 30% ball clay to provide high plasticity, workability, bonding properties, and good white-fired color. Raw materials and processing conditions are the main controlling quality factors for the ceramics industry. The studied "Ball clays" are used in many different industries and have a wide range of colors related to their sedimentary origin and are considered as a vital material in the ceramic and refractory industry. The nature of the aluminosilicate resources (e.g. Kaolinite) gives a very high whiteness after firing, but it is brittle and weak when it is used alone and must be mixed with ball clay to produce a workable, malleable, and more plasticity raw material.

Table 3. Standard properties of the potential use for the kaolinitic clays in ceramics technology (after Laursen et al., 2019).

Ceramic Properties	Standard
Iron oxide content (w/w)	<3.0%
Loss on ignition (w/w)	>8%
Mass loss 200-400 °C (organic matter, w/w)	<2.0%
Amount of smectite type mineral (w/w)	<5%
Particle equivalent diameter <2 µm	>40%
Plasticity index	>12%
Modulus of rupture of the green body (MPa)	>1.5

6. Conclusions

In the present study, the selected clay materials from northeast Aswan to realize their suitability for the ceramic and refractories industry:

1) The upper Cretaceous kaolinitic clay of the Timsah Formation consists mainly of kaolinite, and quartz, while anatase, dolomite, and hematite are considered accessory minerals. The source rocks of these clays are considered to be the weathering products of the parent's Precambrian basement rocks.

2) The thermal (DTA and TGA) results indicate a very low content of carbonaceous materials, which makes the deposits safe in the thermal treatment during drying and firing processes.

3) The Abu Sobeira clay samples are more suitable for application in ceramic and refractory industries as a result of high kaolinite content that needs low thermal energy in the firing process.

4) Aswan ball clay indicated no emissions of HCl and SO₂ gases through the firing process and is considered friendly to the environment.

5) Primary physical properties of the processed samples fall within the range of the standard requirements; while, the shrinkage showed some negative results and was less than the standard requirements.

6) The application of Aswan deposit is restricted to the fabrication of tiles, bricks, and Sanitary ware. It could be used in the ceramic industry, which plays a primary role to impart plasticity or aiding rheological stability during the shaping processes.

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