IBGM
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IRAQI BULLETIN OF GEOLOGY AND MINING
Global Impact Factor
0.744

Iraqi Bulletin of Geology and Mining *Vol.13, No.1, 2017* *p 79 – 97*

MINERALOGICAL AND INDUSTRIAL ASSESSMENT OF CLAY FROM PARTS OF THE FOLDED ZONE IN KURDISTAN REGION FOR CERAMIC PRODUCTION

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Received: 20/ 11/ 2016, Accepted: 11/ 05/ 2017
Key words: Briquettes, Ceramic, Clays, Mineralogy, Iraq

ABSTRACT

Clays are used in the ceramic industries largely because of their contribution to the molding and drying properties of the wares being produced; many clays, such as kaolinite and red clays, are used because of favorable behavior during firing or because they produce favorable properties in fired ware. To assure the most effective use of a clay, the ceramic technologist must meet the problems of purification, the improvement of the working properties of clays through additions of non-plastic materials or chemicals and their behavior during firing. Therefore this study addresses the problems of some clay deposits from parts of the folded zone in Kurdistan Region for ceramic industry. X-ray diffraction is used to establish the mineralogical composition. It is found that the main clay minerals of these samples are chlorite, smectite, mixed layer, illite, and kaolinite, in addition to the non-clay minerals such as quartz, calcite, dolomite and feldspar. The raw materials were ground and sieved; some of the raw materials were mixed in different proportions in order to prepare different mixtures. Three different mixtures were prepared using red clay sample, kaolin, granite, sandstone, and limestone. Briquettes were molded from clay alone (A) and from different mixtures (1B, 2C) using a semi-dry pressing method (10% moisture content and pressure 5.6 in/cm²) in a dimension 5 × 5 × 1 cm. The briquettes were fired at three different temperatures (1125, 1150 and 1175 °C) with at rate of heating of 30 C°/h. The evaluation tests, physical and mechanical properties as well as chemical compositions of the studied samples show that the fired briquettes made from those samples which contain considerable amount of Al_2O_3 and low content of CaO such as samples 1, 5 and 7 from Fatha Formation; 8 and 9 from Injana Formation give the best results for the production of floor tile, wall tile, facing tile, and clay brick industry. The results also, showed that the best mixture to adequate characteristics for ceramics industry are mixture 2C fired at 1175 °C.

التقييم المعدني والصناعي للطين من اجزاء من منطقة الطيات في إقليم كردستان لإنتاج السيراميك

تولة أحمد ميرزا و ريزان قادر فرج

المستخلص

يستخدم الطين في الصناعات الخزفية كثيرا وذلك بسبب قابليته في التشكيل والتجفيف والحرق حيث تتنج في النهاية الحزف: وتستخدم العديد من الأطيان في هذا المجال، مثل الكاؤلينايت والطين الأحمر، وذلك بسبب سلوك هذه الأطيان أثناء وبعد الحرق والتى تنتج خصائص ملائمة بعد الحرق. ولضمان الاستخدام الأكثر فعالية للطين، يجب على تقني السيراميك

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أن يعالج بالمشاكل التنقية، وتحسين خصائص الطين من خلال إضافة مواد غير بلاستيكية أو مواد كيميائية، وذلك لتحسين سلوك الطين أثناء التجفيف والحرق. لذلك فإن هذه الدراسة تتناول تقييم بعض الرواسب الطينية من اجزاء من منطقة اللطيات لصناعة السيراميك. وقد استخدمت الأشعة السينية الحائدة لمعرفة التركيب المعدني. حيث وجد أن المعادن الطينية الرئيسية لهذه النماذج هي الكلوريت، السمكتايت، الطبقة المختلطة، الإيلايت والكاولينايت، بالإضافة إلى المعادن غير الطينية مثل الكوارتز، الكالسيت، الدولوميت والفلسبار. تم طحن ونخل مواد الخام الى الأحجام المطلوبة ثم خلط المواد حسب نسب مختلفة من أجل تهيئة عدد من الخلطات.

تم تحضير ثلاثة خلطات مختلفة باستخدام (ترسبات طينية ذات اللون الأحمر من منطقة اللطيات في إقليم كردستان، وتم خلطه مع الكاؤولين، كرانايت، الحجر الرملي والحجر الجيري بنسب مختلفة) وتم تشكيل قوالب بابعاد 5 × 5 × 1 سم من الطين فقط (A) وخلطات مختلفة (1B، 2C). أستخدمت طريقة الكبس شبه الجاف (بأستخدام ١٠% من الرطوبة وبتسليط ضغط قدره 5.6 كنت/سم^{٢}) لتشكيل القوالب، ثم أحرقت تلك القوالب المكبوسة بفرن الكهربائي وبدرجات حرارة حرق ١١٢٥، ١١٥٠ و ١١٧٥ °م وفق برنامج حرق معين بحيث كان معدل الزيادة بدرجات الحرارة ٣٠ °م لكل ساعة. تم تحديد الفحوصات التقييمية التي تشمل الخواص الفيزيائية، الميكانيكية والكيميائية للعينات السيراميكية. حيث بينت نتائج هذه الفخوصات بأن القوالب التي تم إنتاجها من تلك العينات والتي تحتوي على كمية كبيرة من Al_2O_3 ومحتوى قليل من CaO مثل العينات 1 و 5 و 7 من تكوين الفتحة ونماذج 8 و 9 من تكوين إنجانة تعطي أفضل النتائج لإنتاج بلاط الأرضيات، بلاط الحائط، بلاط الواجهات، وصناعة الطابوق الطينى. وأظهرت النتائج أيضا أن أفضل خليط يعطى خصائص كافية لصناعة السيراميك هو خلطة 2C المحروقة في درجة الحرارة 1175 درجة مئوية.

INTRODUCTION

Clays are the main raw materials invested in the manufacturing of various ceramic products such as bricks, tiles, abrasives, pottery, porcelain refractory etc. The clays used in these products cover a wide range of chemical, physical and mineralogical properties. One thing that all must have in common, however, is that they must be easily accessible. In any given area, the accessibility of a deposit may prove almost as important as the basic requirements regarding chemical and physical properties.

According to Fakolujo *et al.* (2012) very few restrictions are placed on deposits considered for use in ceramic products. Probably the most important is that the clay is suitable for forming. Briefly listed below, in decreasing order of importance, are the specifications which, ideally, should be found in raw materials used in most structural clay products.

1) Plastic enough for shaping **2)** Dry without excessive cracking and warping **3)** Have low and wide vitrification range **4)** Low carbonate content **5)** Have a spread in particle size **6)** Contain non-clay constituents. The first two requirements are self-explanatory. The low and wide vitrification range is desirable for favorable unit firing cost and for ease of firing. The low carbonate content is desirable for two reasons: **(1)** the fluxing action of carbonates tends to shorten the firing range or cause localized flux spots and **(2)** CaO and MgO resulting from firing of common carbonate-bearing clays will hydrate rather easily with resulting disruption to the fired product. A spread in particle size tends to widen the vitrification range and also generally promotes a stronger texture than is found in a clay that has a narrow range of particle sizes. If the saleable product is one that depends in part upon fired color, then excessive amounts of iron compounds in the clay are harmful. This is of particular importance in only a few products such as face tile and, to a certain extent, face brick (Mohsen and El-maghraby, 2010).

From these qualifications, it is apparent that a wide variety of mineral assemblages will suffice. Even shales rich in relatively non plastic and impure illites and chlorites, if they contain enough non-clay minerals and are sufficiently plastic for shaping, are employed in ceramic products (Coroado *et al*., 2010).

Due to the increasing demand on ceramic products in Iraq which currently imports at a considerable cost, extensive research work has recently been directed towards the exploration and evaluation of local raw materials such as the work of Merza, 2002; Merza, 2004; Merza and Mohyaldin, 2005; Aqrawi, 2008; Hakeem, 1998; Hakeem, 2012; and Hakeem *et al.*, 2016. Clays occur in deposits of greatly varying nature; no two deposits have exactly the same clay and frequently different samples of clay from the same deposit differ (Fakolujo *et al.*, 2012). Clay of different types are widely distributed in Kurdistan, therefore this study will focus on evaluation of some raw clays at the Low Folded Zone from Kurdistan Region NE Iraq with some additive local raw materials such as sandstone, granite, limestone and kaolinite for manufacturing ceramic tile. The reserves of the studied clay samples and additive materials appear to be quite large based both on the extension of the outcropping areas and on lithostratigraphy. Therefore, there will be sufficient reserves to allow the feeding of several plants with clay raw materials for the production of construction ceramics.

GEOLOGY OF THE STUDIED AREA

The studied clays are collected from different formations and localities that are mainly Neogene succession and belong to the Low Folded Zone. Ten samples of clay have been taken from Fatha Formation (Middle Miocene), Injana Formation (Upper Miocene) and recent valley deposits from Garmian area. Some additive samples such as granite from the Mawat Ophiolite Complex (Cretaceous) from Daraban area, sandstone of Khabour Formation (Ordovician) from Kiasta Village, north of Amadyia and limestone of Sinjar Formation Bazian area respectively were taken. Duuekhla Kaolinitic Claystone of Ga'ara the Western Desert area was received from the Department of Ceramic-College of Fine Arts/ University of Sulaimaniyah.

Quaternary sediments are well developed in the Low Folded Zone, especially in the trough of wide synclines (Sissakian and Al-jibouri, 2012). The study area is characterized by the presence of recent deposits as a result of weathering and erosion of the surrounding rocks which has caused the formation of the clay deposits of yellow to light brown. The thickness of these deposits in the study area is about 4 – 15 m with lateral extension of 200 – 500 m. Three samples were collected from this deposit in Sarqalla and Qaratapa areas (samples 2, 3 and 4). The Fatha Formation shows large variation in its characteristics and thickness. In this study the Formation generally consists of reddish brown claystone, siltstone and sandstone as well as thin beds of limestone and gypsum (Fig.1). The thickness of the Fatha Formation changes in different localities of the studied area. Generaly, the thickness ranges between 3 – 8 m with lateral extension of 200 – 500 m. Four samples were taken from this formation from Darbandikhan town, Banikhellan, Zardalikaw and Sangaw villages (samples 1, 5, 6 and 7 respectively). Injana Formation of the studied area consists of clastic rocks generally including sandstone, siltstone and claystone with brown to red brown color. The thickness of the formation is variable. Mostly about 3 – 10 m with lateral extension of about 150 – 400 m. Three samples were collected from this formation at Shiwasur and Takya villages (samples 8, 9 and 10). Mawat ophiolite complex is one of the major Cretaceous ophiolite complexes in northeastern Iraq and is situated at about 30 Km northeast of Sulaimaniyah; it represents part of the Iraqi Zagros Suture Zone (Mirza and Ismail, 2007). The Igneous mass of Mawat ophiolite complex is composed mainly of basic, ultrabasic rocks with minor acidic intrusion of granite. Granite in Mawat ophiolite complex is white in color consisting mainly of quartz and alkali feldspar, muscovite with rare Ca-plagioclase. It appeares in different areas as discontinuous dykes within the ultrabasic rocks. The largest body is about 30 m by 15 m in dimensions which can be seen in the high mountain and the other bodies are smaller in size ranging from 0.5 m to 3 m which is the small intrusion hosted in a serpentinized ultramafic unit. In this study one sample was taken from the granite of Mawat ophiolite complex near Daraban village and was used as additive materials to achieve feldspar and quartz and represented by sample 11. The Khabour Formation comprises a thin-bedded, fine grained sandstone, quartzites and silty micaceous shale with olive-green to brown in color. The sandstone of Khabour Formation (sample 12) is characterized by yellowish red to reddish brown color and is relatively tough. The sandstone in north of Amadyia near Kiasta village (makes a major constituent besides the shale and mud rocks (Fig.1). Calcium carbonate is the principle flux in the ceramic industry and calcium is sometimes added in small amounts as a filler to reduce fired shrinkage and act as a whitener, hence the limestone of Sinjar Formation in the Bazian area was used for this purpose due to its high $CaCO_3$ content.

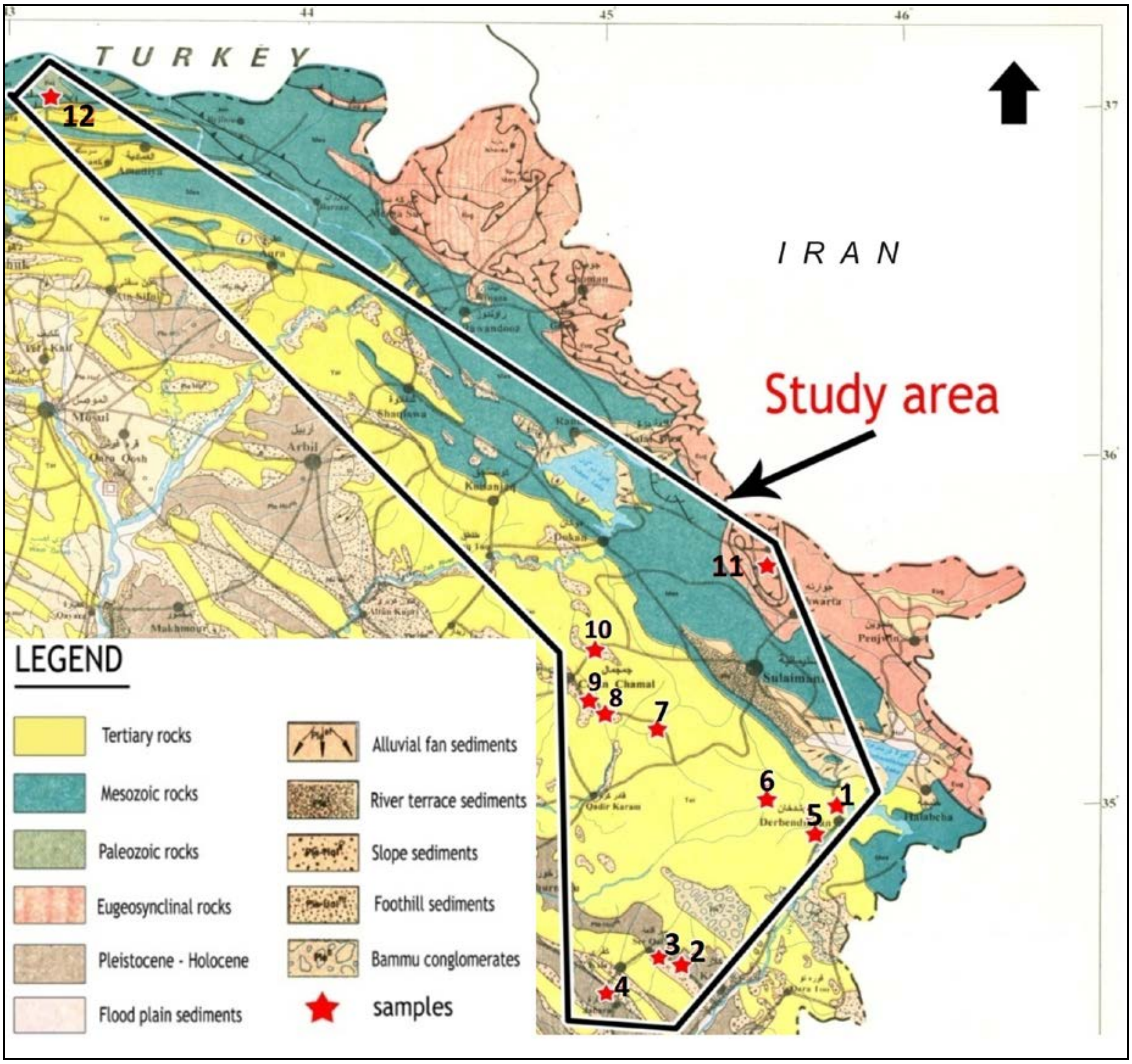

Fig.1: Quaternary Sediments Map of the studied area (after Barwary *et al*., 2002) with the location of the samples

METHODOLOGY

The clay raw materials were ground to fine size (powder) in order to get the desired microstructure. The clay samples as well as kaolin and limestone were powdered finer than 90 µm; the rock samples (granite and sandstone) were powdered to 63 µm.

Nine briquettes from each sample of clay alone were prepared and signed by A (Table 1). By the same procedure two types of mixtures were prepared from mixing each type of clay and additive materials which signed by mixture 1B and mixture 2C with proportions as in Table 1.

The samples are moisturized by adding 10% of water and formed by the semi-dry pressing method (5.6 kn/cm²) to produce the briquettes having dimensions 1 x 5 x 5 cm. They were formed at the College of Science-Department of Geology/ University of Sulaimaniyah. Pressed samples were dried in oven at temperature 105 °C for 24 hours, then fired in electric furnaces with special firing program and temperatures (1125, 1150 and 1175 °C) with rising temperature 30 °C/hour. The drying and firing processes were carried out at College of Fine Arts-Department of Ceramic/ University of Sulaimaniyah.

Table 1: Type and proportion of the raw materials used in the preparation of mixture 1B and mixture 2C

Type of mixture	Kaolin %	Red clay %	Sandstone %	Limestone %	Granite %
A	–	100	–	–	–
1B	35	37	15	3	10
2C	35	35	10	0	20

- **Tests Conducted**

Physical tests carried out on the studied raw materials include: grain size distribution by (Sieve and Hydrometer) at Sulaimaniyah Architecture Laboratory according to British standard (BS1377, 1967) and Atterberg Limits determined at Seko Engineering House/ Sulaimaniyah city, according to (ASTM, 1972) and (AASHTO, 2002; and AASHTO, 2004). Chemical analyses was carried out for all samples using X-Ray Fluorescence (XRF) in Washington State University. Mineralogical analysis was performed for nine samples (1, 3, 4, 5, 6, 7, 8, 9 and 10) using X-Ray Diffraction (XRD) in X-Ray Laboratories of Iraqi Geological Survey – Baghdad using standard work procedure, part 2 (Al-Janabi *et al*., 1992) and kaleseramik Fizik Laboratuvari (Turkey). A number of tests carried out on the studied ceramic briquettes include: Colour by visual inspection, linear firing shrinkage according to (ASTM, 1982), apparent porosity, water absorption and bulk density according to (ASTM, 1986) and compressive strength according to the (ASTM, 1969).

RESULT AND DISCUSSION

- **Raw materials**

Grain size distributions of the raw materials are shown in Table 2, On the basis of their granulometry, studied materials may be classified as clayey silt (Shepard, 1954). According to Winkler (1954) diagram (Figure 2A) samples 6, 7 and 2 are suitable for manufacturing bricks and perforate bricks respectively, samples1, 5 and 9 are suitable to be used as a thin walled hollow bricks, while samples 3, 4 and 10 can be used for manufacture of roofing tiles and light weight blocks, sample 8 is neither suitable for manufacturing any types of these ceramic tiles. In general the grain size analysis results revealed that most of the clay samples composed of high percentage of silt and low percentage of clay with minor proportion of sand (Table 2) as well as these causes the plasticity of most of the studied raw materials (Table 3). The plasticity is mainly caused by sheet-like structure of the clay minerals as well as by water which is both physically and chemically bound to these sheets; the sheets slide over each other (Bormans, 2004). The low plasticity of most of the studied samples are due to the high percentage of silt and low percentage of clay in addition to the persence of sand and this makes it to be more convenient for bricks industry using Bain and Highley (1978), diagram (Fig.2).

Mineralogical analysis of the clay samples using X-Ray Diffraction (XRD) technique was used to identify many types of clay and non-clay minerals on the basis of their diffraction pattern (Fig.3). Semi-quantitative analyses were used for calculating the percentage of clay minerals and non-clay minerals and the results as in Table 4. The results show that the main clay minerals present are chlorite, smectite, mixed layer illite-smectite, and kaolinite, in addition to the non-clay minerals such as quartz, calcite and feldspar.

Chemical analyses of raw materials indicate the presence of considerable amount of refractory oxides (SiO_2 and Al_2O_3) especially in the raw materials of Fatha Formation (sample no. 1, 5, 6, 7) and Injana Formations (samples no. 8, 9 and 10), (Table 5). The CaO acts as a flux that affects the formation of the liquid phase at a relatively low temperature and is used as one of the methods for controlling the firing shrinkage (Das *et al*., 2005). Some of clay samples especially those that taken from the recent valley deposits (Sample no. 2, 3 and 4) contain high percent of CaO, while the other samples contain moderate CaO content therefore CaO were added to mixture B for about 3% (Table 1). High proportion of two oxides (SiO_2 and Al_2O_3) referred to the facts that both oxides are the main components of the clay minerals and they have a decisive influence on the refractoriness and mechanical resistance of the final product (Medhioub *et al*., 2010). Silica is important for controlling the firing shrinkage as it lower linear shrinkage and increases the apparent porosity as well as provides the densification of the ceramic product (Aqrawi, 2008). Hence the Kaolin from Gaara Formation which is contain concidrable amount of SiO_2, Al_2O_3 and sandstone of Khabour Quartizite with high SiO_2 contenet (Table 5) were added to the prepared mixture (Table 1) to achive best densification of the ceramic tiles. The magnesium oxide (MgO) ranging between 2.48 – 4.13 % that refers to the clay minerals especially smectite and chlorite. According to (Medhioub *et al*., 2010) MgO acts as sintering promoters on the vitrification. The total coloring oxides ($Fe_2O_3 + TiO_2$) vary from 3.88 – 7.99 %. The Iron oxide (Fe_2O_3) is the main colorant in the clays, being responsible for the reddish color after firing (Mohsen and El-maghraby, 2010). The ($K_2O + Na_2O$) contenet in clay samples are moderate ranging between 1.57 – 4.35 %. The K_2O depends on illite content (Sousa and Holanda, 2005; and Mohsen and El-maghraby, 2010). These two oxides act as a flux and the flux controls the degree of vitrification of the ceramic body during firing and provides the densification of the fired products (Lewicka, 2010). According to the triangular diagram by Fabbri and Fiori (1985); and Fabbri and Dondi (1995). samples (1, 5, 6, 7, 8, 9 and 10) may be used as a feeding material for porous red tiles, while samples (2, 3, and 4) are unsuitable for stoneware and porous tiles uses (Fig.4) because the samples (2, 3 and 4) are high lime content (Table 5), this attributes to high carbonate content such as calcite. The oxides (P_2O_5, MnO and SO_3) are present in low proportion in the studied clay samples. loss On Ignition (L.O.I) ranges between 11.09 – 23.17 % (Table 5), the considerable amount of L.O.I is due to the decomposition of carbonate minerals and escaping of the CO_2 gas, as well as attributing to the molecular and adsorbed water present in and on the crystal structure of the clay minerals.

Table 2: Grain Size Distribution of raw materials

Sample no.	**Sand %**	**Silt %**	**Clay %**
1	2.19	62.93	34.88
2	1.41	77.52	21.07
3	1.66	69.86	28.48
4	2.27	73.97	23.76
5	3.83	63.88	32.29
6	4.82	77.37	17.81
7	6.14	72.62	21.24
8	41.57	49.33	9.1
9	4.81	69.12	26.07
10	18.07	59.09	22.84

Table 3: Atterberg Limits for the raw materials

Sample.No.	**Liquid limit %**	**Plastic limit %**	**Plasticity index**
1	29.85	21.33	8.52
2	28.65	20.3	8.35
3	47.92	31.85	16.07
4	28.65	19.3	9.35
5	27.4	18.2	9.2
6	29.03	21.3	7.73
7	25.2	18.95	6.25
8	21.85	17.73	4.12
9	25.7	17.02	8.68
10	26.8	19.02	7.78

Table 4: Semi-quantitative analysis (percentage) of clay minerals and non-clay minerals in the raw materials

Sample. No.	**Clay minerals %**					**Non-clay minerals %**				
	Chlorite	**Mixed Layer**	**Smectite**	**Kaolinite**	**Illite**	**Quartz**	**Calcite**	**Feldspar**	**Dolomite**	**Hematite**
1	**	****	*	***	----	*****	***	**	----	----
3	****	****	**	**	----	****	*****	*	----	----
4	***	****	**	****	----	*****	****	*	----	----
5	***	-----	**	***	****	*****	***	**	----	----
6	****	-----	**	***	**	****	*****	*	----	----
7	**	****	*	***	----	*****	***	*	----	----
8	***	**	*	****	----	****	***	**	----	----
9	***	-----	*	****	****	*****	***	**	----	----
10	****	***	**	***	----	*****	***	**	----	----

Note: ***** Prevalent, **** Available, *** Few, ** Scarce, * Rare

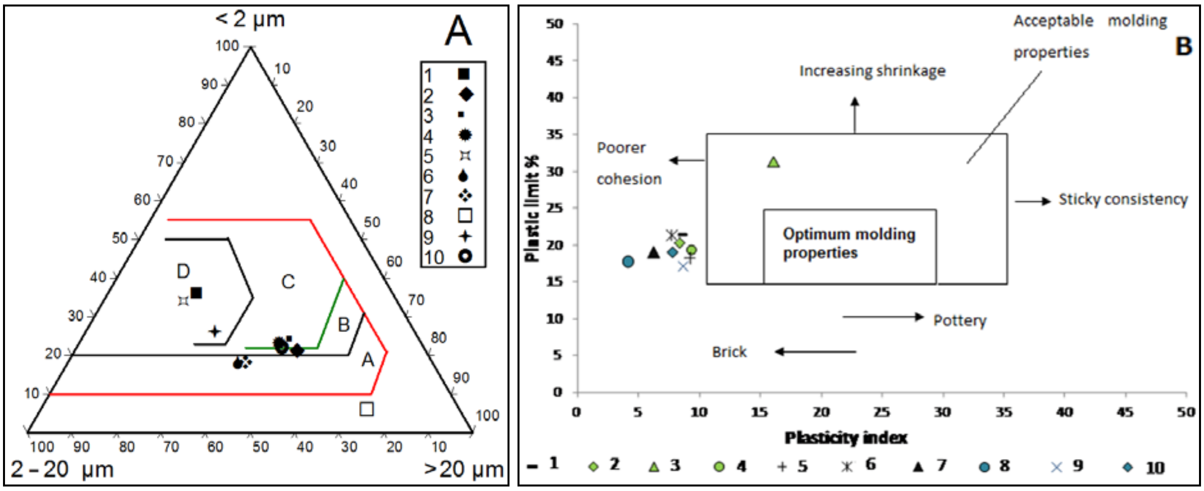

Fig.2: **A)** Winkler diagram (Winkler, 1954) for the technological classification of bodies for structural clay products and plots of the studied samples. **(A)** A- Vertically perforated bricks B- roofing tiles and light blocks. C and D- thin-walled hollow bricks and blocks, **B)** Clay workability chart (after Bain and Highley, 1978) and plots of the studied samples

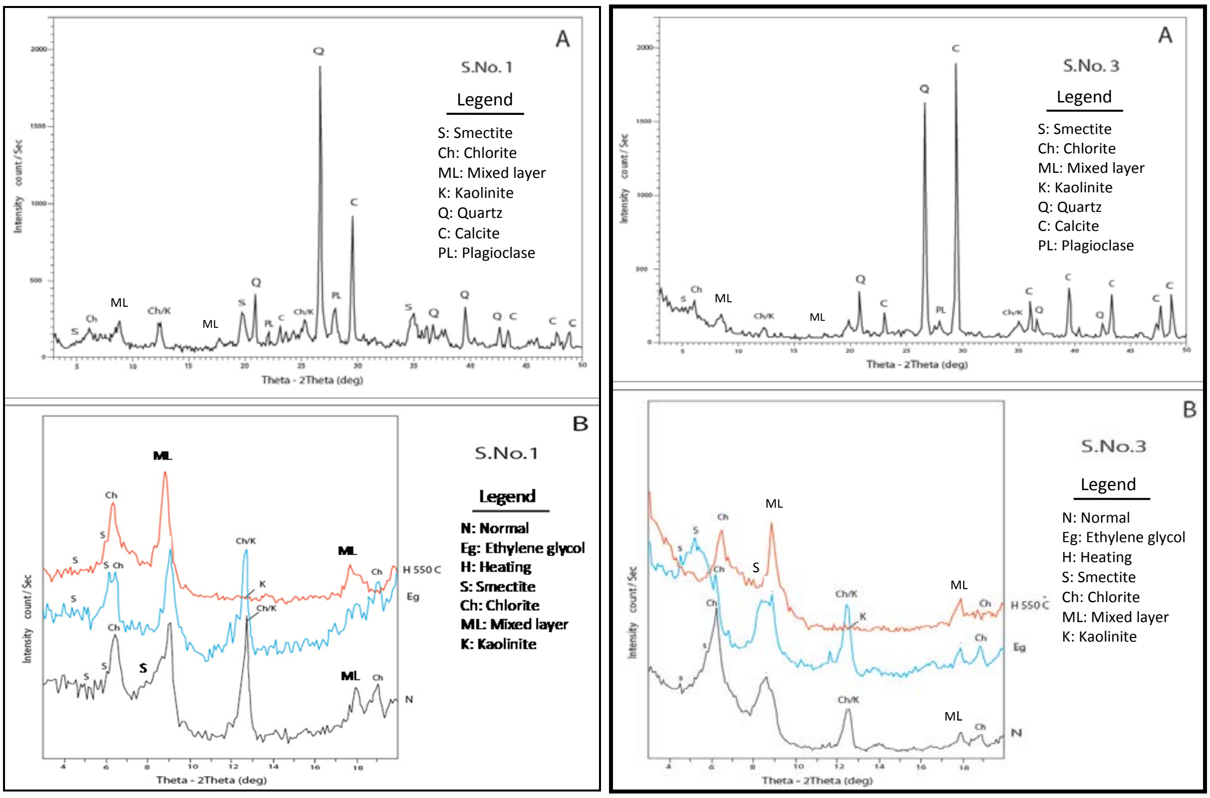

Fig.3: Some of X-Ray Diffraction pattern for sample no.1 from Fatha Formation and Sample no. 3 from recentvalley deposit. **A)** Bulk sample **B)** Oriented sample

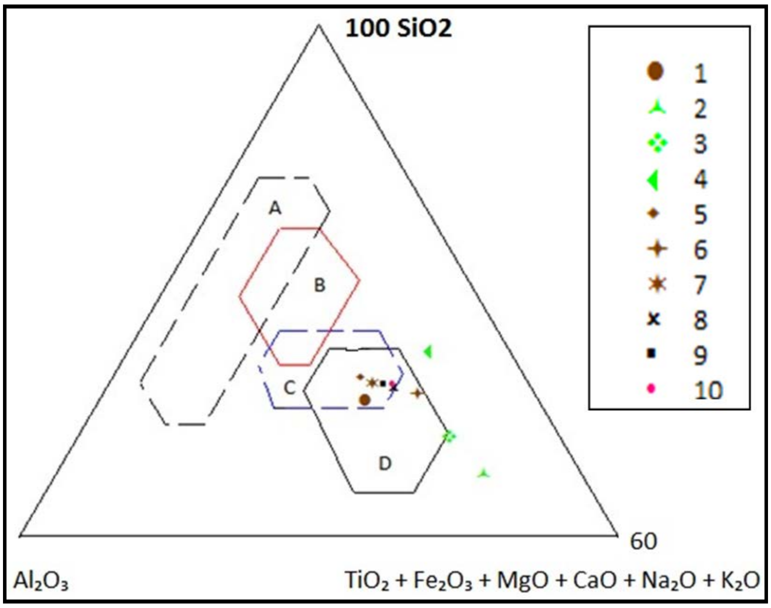

Fig.4: Clay chemical composition domains for preparing stoneware tiles {white (A) and red (B) bodies} and porous tiles (C and D) for the studied clay samples by (Fabbri and Fiori, 1985; and Fabbri and Dondi, 1995)

Table 5: Chemical analysis of the raw materials

Samples / Oxides %	Clay samples										Additive materials			
	1	2	3	4	5	6	7	8	9	10	Granite. S.No.11	Sandstone S. No. 12	Limestone S.No.13	Kaolin S.No.14
SiO_2	48.68	35.03	39.58	50.16	51.54	45.17	49.72	48.60	48.56	48.08	75.74	74.39	0.49	48.45
TiO_2	0.836	0.52	0.59	0.453	0.755	0.531	0.706	0.665	0.68	0.621	0.69	0.68	-----	1.304
Al_2O_3	15.29	7.36	8.57	6.83	14.58	9.43	13.56	11.78	12.49	11.58	13.37	11	0.63	29.13
Fe_2O_3	7.15	3.36	3.95	3.06	6.18	4.19	5.20	4.92	5.10	4.93	0.24	4.47	0.24	1.76
MnO	0.085	0.07	0.1	0.078	0.080	0.102	0.077	0.096	0.104	0.085	----	----	----	0.014
MgO	3.46	3.28	2.86	2.48	4.13	2.81	3.18	3.77	3.36	2.74	0.77	2.10	----	0.76
CaO	7.62	22.8	19.9	16.80	6.83	15.80	10.02	11.71	10.96	13.04	0.28	0.86	55.37	2.61
Na_2O	0.88	0.87	0.30	0.59	1.30	0.38	1.18	1.47	1.17	1.00	4.73	2.08	----	0.22
K_2O	3.47	1.15	1.27	1.15	2.89	1.51	2.52	1.99	2.06	1.53	3.37	2.09	----	0.59
P_2O_5	0.173	0.116	0.117	0.130	0.173	0.104	0.139	0.153	0.128	0.107	----	----	----	0.043
SO_3	0.02	0.65	0.01	0.03	0.01	0.00	0.03	0.02	0.03	0.00	----	----	----	0.05
L.O.I	12.52	23.17	21.88	17.53	11.09	18.84	12.61	13.80	14.15	15.68	0.6	2.24	42.81	13.99
Total	100	98.4	99.1	99.29	99.54	98.89	98.96	98.97	98.78	99.41	99.79	99.91	99.54	98.93
$Fe_2O_3 + TiO_2$	7.99	3.88	4.54	3.51	6.94	4.72	5.91	5.59	5.78	5.55				
$K_2O + Na_2O$	4.35	2.02	1.57	1.74	4.19	1.89	3.7	3.46	3.23	2.53				
$Fe_2O_3 + MgO + CaO + K_2O + Na_2O$	22.6	31.5	28.3	24.1	21.3	24.7	22.1	23.9	22.7	23.2				

- **Fired Briquettes**

– Color, Linear firing shrinkage, apparent porosity, water absorption, and bulk density: The color of the studied ceramic briquettes prepared from the studied raw materials was determined by visual inspection. The briquettes before firing were viewed in different colors which are very dark brown, dark and light brown, reddish brown, pinkish gray, strong pale brown, whitish and yellowish brown and progressively changed to reddish brown, dark and light reddish brown, yellowish brown, yellowish white, light brown after firing. According to Bonnet and Gallard (2007), the color of ceramic product depends essentially on the CaO, Al_2O_3, SiO_2, and Fe_2O_3 proportions. The change in color of the studied briquettes during firing is caused essentially by the reactions that take place between the raw material components, and the most important reactions are oxidation, calcination, and silica formation (Hakeem *et al.*, 2016). In the case of presence of iron, the final colour will be changed during firing; the Fe^{+3} ions dissolve in the new mineral phases and confer a yellow colouring to it. Hematite (Fe_2O_3) gives a brown-red color. The abundance of CaO in the raw materials and addition of limestone to the mixture is favorable to bleaching the original color and evolution of coloring towards yellow, due to reaction with iron oxides (Merza, 1997; and Hakeem *et al.*, 2016).

The obtained results of the linear firing shrinkage test in the case of clay alone A (Table 6) increase with increasing the firing temperature from (1125 to 1175 °C) in most of the studied ceramic briquettes, this is attributed to dehydration and decay of carbonate material , escape of CO_2 at high temperatures and starting vitrification which leads to filling the pores with glass melt that causes decreasing the porosity and increasing the bulk density and then volume shrinkage (Gonzalez *et al.*, 1992). The sample 2 and 4 from recent valley deposits were exposed to negative shrinkage (elongation) in their length (Table 6) due to low content of Al_2O_3 and high content of SiO_2 and CaO as well as the bloating ability for clays (Tables 4) and growing of new mineral phases also contribute to the expansion of ceramic briquettes.

The linear shrinkage of fired briquettes prepared from mixture 1B compared to those briquettes made of clay alone (A) the results show that samples 1, 3, 5, 7, 8, 9 and 10 show decrease in linear shrinkage at all firing temperatures and some of the studied ceramic briquettes (samples 4 and 6) slightly increased in linear shrinkage at all firing temperature, and sample 2 at 1125 and 1150 °C (Table 6).

The presence of quartz mineral in the studied clay samples is one of the causes that lead to expansion the ceramic body, quartz mineral expands with increasing the firing temperature due to phase transformation of quartz mineral (Budnikov, 1964; and Bonnet and Gaillard, 2007). Also liberating of CO_2 as a result of carbonate decomposition prevents the convergent of the grain from each other (Hakeem, 1998; and Hakeem, 2012).

Results of apparent porosity, water absorption and bulk density in case of clay alone (Tables 7, 8 and 9) revealed that apparent porosity and water absorption for most of the studied ceramic briquettes decrease and bulk density increased with increasing firing temperatures from 1125 to 1175 °C this is owing to vitrification and decomposition of minerals content of raw materials and crystallization of new silicate minerals filling the pores present between the particles during the firing process (Dondi *et al.*, 1992; Gonzalez *et al.*, 1992; and Merza, 2002). Above the temperature 1150 °C apparent porosity and water absorption increase and bulk density decrease for both samples (6 and 10) while samples (1 and 5) changed to glassier material at 1175 °C (Tables 7, 8 and 9) these results depending on the type of component of the fired materials (chemical composition), their particle size, the firing temperature and the duration of firing and time of maturity (Grim, 1968; Ryan, 1978, and Ryan, 1999). According to Shreve and Brink (1977) the calcite decomposition at firing temperatures 800 °C to 1000 °C leaves the pores in the ceramic body by releasing the CO_2 gas, as well as the bloating ability of clays which accompanied with water molecules existing are another factors that increase the apparent porosity and water absorption, and in contrast will decrease the bulk density.

In mixture 1B and mixture 2C when compared to the case of clay alone A it is noted that the samples 2, 3, 4 and 6 decreased in porosity and water absorption and increased in bulk density (Tables 7, 8 and 9) because these samples are high carbonate content (Table 5) due to CO_2 liberation during the carbonate decomposition causes the formation of the new pores (Jain, 1980; Gonzalez *et al*., 1992; and Bill *et al*., 1992), this causes the formation of high porosity and water absorption and low bulk density in the case of clay alone A but due to the addition of additive materials (sandstone, granite , kaolin and lime) the above reaction is modified; silica reacts with each CaO to form new crystalline phases at high temperature which are alumino-silicate of calcium, it is available in the samples with high carbonate content that filled certain ratio of pores between particles (Gonzalez *et al*., 1990), as well as kaolinite mineral causes an increase in shrinkage and feldspar acts as a flux that accelerates the vitrification, altogether decreasing the apparent porosity and water absorption and increasing the bulk density. The samples 1, 5, 7, 8 and 9 in mixture 1B and mixture 2C show increase in apparent porosity and water absorption capacity and decrease in bulk density compared to the case of clay alone A, (Table 7, 8 and 9) because these samples are low carbonate content and generally rich in silica (Table 5) which formed nearly low porosity in case of clay alone A, through the addition of additive materials the percentage of bonding materials (clays) decrease and quartz mineral increased in the mixture, that causes decrease the linear shrinkage (Budnikov, 1964; and Bonnet and Gaillard, 2007). That causes decrease in the linear shrinkage and relatively increase the apparent porosity and water absorption as well decrease the bulk density (Tables 7, 8 and 9). Sample 10 increased in apparent porosity and water absorption value and decreased in bulk density in both mixing at the temperature of 1150 °C (Tables 7, 8 and 9) which may be referred to the crystallization of new silicate minerals resulted in expansion of the materials (Gonzalez *et al*., 1992). The compressive strength values (Table 10) progressively increased at firing temperatures 1125 °C to 1175 °C. The reasons for the increase of compressive strength are attributed to the cohesion between the particles of the raw material by a glass liquid formed during firing due to sintering and vitrification processes (Boch and Leriche, 2007).

Generally, most of the studied ceramic briquettes in mixture 1B and mixture 2C show higher compressive strength than in the case of clay alone A (Table 10). This attributed to the addition of additive materials (kaolin, sandstone and granite) as a source of silica, alumina and fluxing oxide especially silica because the increase in silica causes the formation of the largest amount of glass phases during the firing that connects the granules with each other. Silica has the medium positive relationship with the compressive strength, with increase the SiO_2 compressive strength increase but to a certain limit (maximum 50%) and more than this percentage in the mixture causes the tiles be more vulnerable to poor bonding materials (clays) (Jassim and Dabby, 2013). This can be noted from the samples 1, 4, 6 and 9 in mixture C which show relatively low compressive strength than in case of clay alone A.

Table 6: Results of Linear firing shrinkage for the studied ceramic briquettes

Sample no.	**Linear firing shrinkage %**								
	Clay alone A			**Mixture B**			**Mixture C**		
	1125 °C	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
1	6.94	9.91	*	1.37	1.19	2.77	1.56	1.74	2.15
2	- 1.78	- 2.13	1.58	0.29	0.69	0.87	0.2	0.1	0.58
3	1.78	1.38	3.3	1.07	0.97	1.92	0.97	1.06	2.13
4	- 1.36	- 0.98	0.39	0.3	0.19	1.16	0.1	0.19	0.68
5	3.15	7.62	*	1.36	0.98	3.79	1.44	1.19	1.88
6	0.68	0.1	1.47	0.89	1.18	2.44	0.68	0.49	0.88
7	1.16	1.27	2.56	0.58	0.49	0.29	0.87	0.68	1.36
8	0.69	1.65	7.17	0.49	0.78	0.49	0.39	0.99	1.07
9	0.58	1.77	3.7	0.58	0.69	1.37	0.58	0.78	1.94
10	0.59	0.9	0.97	0.49	0.2	0.88	0.29	0.78	0.9

Note: * melted

Table 7: Results of Apparent porosity of the studied ceramic briquettes

Sample no.	**Apparent porosity %**								
	Clay alone A			**Mixture B**			**Mixture C**		
	1125 °C	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
1	4.66	13.08	*	31.51	32.79	27.99	31.31	30.26	30.6
2	47.83	48.61	43.4	39.48	38.52	37.83	38.45	37.57	37.76
3	48.2	48.26	45.44	41.31	40.68	38.84	39.09	39.38	39.21
4	44.41	42.1	42.8	39.35	38.26	38.91	36.78	37.69	37.6
5	25.26	17.28	*	31.66	32.77	27.39	29.63	31.5	31.94
6	37.76	37.74	40.23	37.7	36.89	34.31	34.47	36.3	35.11
7	31.84	29.99	29.04	32.87	34.01	35.43	32.63	32.25	32.54
8	33.13	30.95	21.44	33.64	33.59	32.95	32.42	32.87	32.56
9	29.97	27.2	25.33	32.59	34.29	31.42	32.35	33.19	31.86
10	35.71	32.12	35.29	34.51	35.4	35.41	32.34	36.16	33.14

Note: * melted

Table 8: Results water absorption of the studied ceramic briquettes

Sample no.	**Water absorption %**								
	Clay alone A			**Mixture B**			**Mixture C**		
	1125 °C	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
1	2.48	7.66	*	22.39	23.82	19.18	22.47	20.93	22.03
2	42.84	44.95	37.57	31.36	30.94	30.89	30.07	29.73	30.01
3	43.45	43.46	38.75	33.53	32.44	31.84	30.8	30.78	31.33
4	38.39	35.93	36.03	30.6	30.53	30.25	28.3	29.46	28.05
5	16.7	10.98	*	23.96	24.25	19.05	20.79	22.66	22.32
6	29.25	32.09	31.7	30.02	28.99	25.64	25.62	27.63	26.14
7	22.55	20.97	20.22	24.07	24.99	26.27	24.21	23.69	23.49
8	24.07	22.7	13.44	24.68	23.84	24.21	23.92	24.62	24.38
9	21.89	19.46	16.9	23.99	24.75	22.76	24.33	25.23	23.61
10	27.84	23.71	26.71	25.61	26.37	26.09	24.15	27.56	25.39

Note: * melted

Table 9: Results bulk density of the studied ceramic briquettes

Sample no.	**Bulk density gm/cm³**								
	Clay alone A			**Mixture B**			**Mixture C**		
	1125 °C	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
1	1.88	1.71	*	1.41	1.38	1.46	1.39	1.45	1.39
2	1.12	1.08	1.16	1.26	1.24	1.22	1.28	1.26	1.26
3	1.11	1.11	1.17	1.23	1.25	1.22	1.27	1.28	1.25
4	1.16	1.17	1.19	1.29	1.25	1.29	1.3	1.28	1.34
5	1.51	1.57	*	1.32	1.35	1.44	1.43	1.39	1.43
6	1.29	1.18	1.27	1.26	1.27	1.34	1.35	1.31	1.34
7	1.41	1.43	1.44	1.37	1.36	1.35	1.35	1.36	1.39
8	1.38	1.36	1.59	1.36	1.41	1.36	1.36	1.34	1.34
9	1.37	1.4	1.5	1.36	1.39	1.38	1.33	1.32	1.35
10	1.28	1.35	1.32	1.35	1.34	1.36	1.34	1.31	1.31

Note: * melted

Table 10: Results of the compressive strength for the studied ceramic briquettes

Sample no.	**Compressive strength psi**								
	Clay alone A			**Mixture B**			**Mixture C**		
	1125 °C	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
1	1826.85	2081.44	*	2148.72	2409.86	2663.75	1664.27	1895.55	2034.79
2	759.67	909.3	984.26	1007.72	1409.25	1440.26	1401	1433.14	1448.51
3	924.52	1136.45	1173.43	1893.55	1933.95	2172.62	1231.74	1443.1	1486.06
4	1198.46	1240.99	1482.36	1610.94	2179.02	2428.92	1024.65	1131.89	1165.46
5	1213.25	1435.42	*	1714.77	1849.75	2002.65	1192.06	1544.94	1574.38
6	970.6	1937.65	2436.32	1120.8	1975.05	2544.27	1413.52	1469.98	1514.5
7	1161.48	1313.38	1350.79	1171.15	1348.94	1362.31	1292.76	1428.88	1449.07
8	1291.48	1362.45	1692.72	1455.76	1641.23	1823.57	1623.74	1719.74	1754.59
9	1266.16	1794.99	1881.04	1438.83	1586.33	1967.09	1272.56	1528.16	1694.28
10	1273.84	1317.22	1363.73	1187.36	1971.07	2198.64	1494.87	1434.57	1537.12

Note: * melted

SUITABILITY ACCORDING ASTM STANDARDS

- **Wall Tiles**

Laboratory tests showed that samples (1 and 5) at firing temperature 1125 and 1150 °C and samples (8 and 9) at 1175 °C are suitable for End Construction Wall Tile and Side Construction Wall Tile in grade LBX while samples (7, 8, 9 and 10) at firing temperatures 1125 and 1150 °C and samples (7 and 10) at firing temperature 1175 °C are suitable for both End Construction Tile and Side Construction Tile in grade LB (Table 11).

In mixture 1B it is revealed that the samples 1 and 5 fired at temperature 1175 °C are suitable for end construction and side construction load-bearing wall tiles in grade LBX. While the samples 1, 5, 7, 8, 9 and 10 fired at temperature 1125 °C ,1150 °C and samples 6, 7, 8, 9 and 10 fired at temperature 1175°C are suitable for end construction and side construction load-bearing wall tiles of grade LB (Table 11).

In mixture 2C the samples 1, 5, 6, 7, 8, 9 and 10 at all firing temperatures and sample 4 at temperature 1175 °C are suitable for end construction and side construction load-bearing wall tiles in grade LB (Table 11).

Table 11: ASTM standard specification for structural clay load-bearing wall tile (End Construction Tile and Side Construction Tile) C56 – 71 (1981), and ASTM standard specification for structural clay facing tiles (End Construction Tile) C212 (1981) with distribution of the studied samples in the case of clay alone A, mixture 1B and mixture 2C, at different firing temperatures 1125 °C, 1150 °C and 1175 °C

Grade	**Maximum water absorption by 1-h boiling percent**	**Minimum Compressive strength psi**		**Distribution of briquettes on these grades**								
				Clay alone A			**Mixture 1B**			**Mixture 2C**		
		End Construction Tile	**Side Construction tile**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**	**1125 °C**	**1150 °C**	**1175 °C**
LBX	19	1000	500	1, 5	1, 5	8, 9			1, 5			
LB	28	700	500	7, 8, 9, 10	7, 8, 9, 10	7, 10	1, 5, 7, 8, 9, 10	1, 5, 7, 8, 9, 10	6, 7, 8, 9, 10	1, 5, 6, 7, 8, 9, 10	1, 5, 6, 7, 8, 9, 10	1, 4, 5, 6, 7, 8, 9, 10
Class	**Minimum Compressive strength psi End Construction Tile**			1, 4, 5, 7, 8, 9, 10	3, 4, 5, 6, 7, 8, 9, 10	3, 4, 7, 8, 9, 10	2, 3, 4, 5, 6, 7, 8, 9, 10	2, 3, 5, 6, 7, 8, 9, 10	2, 7, 8, 9	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	2, 3, 4, 5, 6, 7, 8, 9, 10
Standard specification for structural clay facing tiles (End Construction Tile) C212 (1981)												
Standard	1000				1	6	1	1, 4	1, 3, 4, 5, 6, 10			1
Special duty	2000		1	6	1	1, 4	1, 3, 4, 5, 6, 10			1		

- **Facing Tiles**

Table 11 shows the distribution of the studied samples on these classes of end construction tile, in case of clay alone A it reveals that the samples (1, 4, 5, 7, 8, 9 and 10), (3, 4, 5, 6, 7, 8, 9 and 10) and (3, 4, 7, 8, 9 and 10) fired at temperatures 1125 °C, 1150 °C and 1175 °C respectively are suitable for end construction tile of class standard while sample 1 fired at temperature 1150 °C and sample 6 fired at temperature 1175 °C are suitable for end construction tile of special duty (Table 11).

In mixture 1B, it reveals that the samples (2, 3, 4, 5, 6, 7, 8, 9 and 10), (2, 3, 5, 6, 7, 8, 9 and 10) and (2, 7, 8 and 9) fired at temperatures 1125 °C, 1150 °C and 1175 °C respectively are suitable for end construction tile of standard class. While the samples (1 and 4) and (1, 3, 4, 5, 6 and 10) fired at temperatures 1125 °C, 1150 °C and 1175 °C respectively are suitable for end construction tile of special duty class (Table 11).

In mixture 2C it is noted that the samples 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 fired at temperatures 1125 °C and 1150 °C as well as samples 2, 3, 4, 5, 6, 7, 8, 9 and 10 at temperature 1175 °C are suitable for end construction tile of standard class; sample 1 at temperature 1175 °C shows suitability for end construction tile of special duty (Table 11).

- **Floor Tiles**

Table 12 shows the suitability of the studied samples for end construction floor tiles and side construction floor tiles and their grades. In the case of clay alone A the samples (1, 5 and 9) and (8 and 9) fired at 1125 °C, 1150 °C and 1175 °C respectively are suitable for end construction floor tile in grade FT2 (Table 12).

In mixture 1B it has been noted that the sample 1 fired at temperature 1150 °C and 1175 °C are suitable for end construction floor tile of grade FT1. So the samples (1) fired at temperatures 1125 °C and (5, 8 and 9) at all firing temperatures are suitable for end construction floor tile in grade FT2 (Table 12).

In mixture 2C the samples (1, 8 and 10), (1, 5, 7, 8 and 9) and (1, 5, 7, 8, 9 and 10) fired at temperature 1125 °C, 1150 °C and 1175 °C respectively are suitable for end construction floor tiles in grade FT2.

In case of clay alone A the samples 7, 8 and 9 at all firing temperatures, samples 1 and 5 at 1125 °C, 1150 °C and sample 10 at 1150 °C are suitable for side construction floor tile in grade FT1 (Table 12).

In mixture 1B the samples 1, 5, 8 and 9 at all firing temperatures and sample 7 at 1125 °C and 1150 °C are suitable for side construction floor tile in grade FT1 (Table 12).

In mixture 2C it is revealed that the samples (1, 5, 7, 8, 9 and 10), (1, 5, 7 and 8) and (1, 5, 7, 8 and 9) fired at temperatures 1125 °C, 1150 °C and 1175 °C respectively are suitable for side construction floor tile in grade FT1 (Table 12).

Table 12 shows the distribution of the studied samples for side construction tile of different classes, in case of clay alone A it is noted that the samples 2, 3 and 6 fired at temperature 1125 °C as well as the samples 2 fired at temperature 1150 °C, 1175 °C are suitable for side construction tile of class standard. While the samples (1, 4, 5, 7, 8, 9 and 10), (1, 3, 4, 5, 6, 7, 8, 9 and 10) and (3, 4, 6, 7, 8, 9 and 10) fired at temperatures 1125 °C, 1150 °C and 1175 °C respectively are suitable for side construction tile of special duty (Table 12).

In mixture 1B and mixture 2C it is noted that the samples 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 at all firing temperatures are suitable for side construction tile of special duty class (Table 12).

Table 12: ASTM standard specification for structural clay floor tiles (End Construction Floor Tile and Side Construction Floor Tile), C57 (1978), and ASTM standard specification for structural clay facing tiles (Side Construction Tile) C212 (1981), with distribution of the studied samples in case of clay alone A, mixture 1B and mixture 2C at different firing temperatures 1125 °C, 1150 °C and 1175 °C

Grade	**Maximum water absorption by 1-h boiling percent**	**Minimum Compressive strength psi**		**End Construction Tile**									**Side Construction Tile**								
				Clay alone A			**Mixture 1B**			**Mixture 2C**			**Clay alone A**			**Mixture 1B**			**Mixture 2C**		
		End Construction Tile	**Side Construction Tile**	**1125°C**	**1150°C**	**1175°C**	**1125°C**	**1150°C**	**1175°C**	**1125°C**	**1150°C**	**1175°C**	**1125°C**	**1150°C**	**1175°C**	**1125°C**	**1150°C**	**1175°C**	**1125°C**	**1150°C**	**1175°C**
FT1	25	2250	1100					1	1				1, 5, 7, 8, 9	1, 5, 7, 8, 9, 10	7, 8, 9	1, 5, 7, 8, 9	1, 5, 7, 8, 9	1, 5, 8, 9	1, 5, 7, 8, 9, 10	1, 5, 7, 8	1, 5, 7, 8, 9
FT2	25	1400	850	1	1, 5, 9	8, 9	1, 5, 8 , 9	5, 8, 9	5, 8, 9	1, 8, 10	1, 5, 7, 8, 9	1, 5, 7, 8, 9, 10									
ASTM standard specification for structural clay facing tiles (Side Construction Tile) C212 (1981)																					
Standard	500												2, 3, 6	2	2						
Special duty	1000												1, 4, 5, 7, 8, 9, 10	1, 3, 4, 5, 6, 7, 8, 9, 10	3, 4, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10	1, 2, 3, 4, 5, 6, 7, 8, 9, 10

CONCLUSIONS

Based on the experimental results obtained from this study, the following conclusions can be drawn as follows:

Three different firing temperatures (1125 °C, 1150 °C and 1175 °C) were selected for firing the prepared briquettes depending on experimental study on clay samples for withstanding high temperatures. The results show that most of clay samples withstand to 1200 °C. It has been noted that the physical properties of the studied ceramic briquettes are affected by the percentage of refractory oxides (silica and alumina) and percentage of fluxing oxides, especially the percentage of calcium oxide (present in high proportion in some of the studied samples). The results of the physical and mechanical properties of the studied ceramic briquettes compared with the American Society for Testing and Materials (ASTM) and Iraqi Standard (IQS), they show that some of the studied clay samples are suitable for the production of floor tile, wall tile, facing tile and clay brick industry in case of using clay alone and in different types of mixture (1B and 2C) fired at different firing temperatures.

The samples which yielded physical and mechanical properties that met the requirements of ASTM standard specification are samples 1, 5, 7 from Fatha Formation; and 8 and 9 from Injana Formation; gave the best results for production of floor tile, wall tile, facing tile and clay brick industry at different firing temperatures and different mixing ratio of materials.

The samples which yielded physical and mechanical properties that did not satisfy the requirements of ASTM standard specification for the production of floor tile, wall tile, facing tile and clay brick industry, are those which contained impurities in the raw materials especially those with high percentage of CaO and had a negative effect on the measured properties.

ACKNOWLEDGMENTS

The authors wish to thank Mrs. A'alia Tofiq, administrator of the Sulaimaniyah Architecture Laboratory for her advice and assistance in determining some physical tests. Also grateful thanks to Mr. Sami, Mr. Jawdat Ali Sharif and Mr. Dana Nasraddin at Seko Engineering House for their agreement to doing some laboratory works in Seko Engineering House.

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