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Assessment of some locally produced Egyptian ceramic wall tiles

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KEYWORDS

Ceramic wall;
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Modulus of rupture;
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Abstract Due to the fact that there are various types of wall tiles in the Egyptian market, of different qualities and prices, the aim of this research paper is to evaluate the properties and characteristics of tiles obtained from the local Egyptian market and attempt to identify the reasons for such differences. In other words, investigate the reasons why some tiles are more superior in quality than others. Test specimens were obtained from the local Egyptian market for fourteen factories. The mechanical properties (namely flexural strength) and physical properties (namely water absorption and apparent porosity) were measured, as they reflect the strength, the endurance during handling, as well as, the conditions to which the tiles are expected to be exposed to during use. The impact resistance and hardness were tested as well. Also, the phases (both major and minor) developed during the firing (maturing) step of manufacturing were identified. A correlation was made between the measured properties and the formed phases. The results showed that samples which yielded high physical and mechanical properties are those that constituted mainly the major phases, specifically, mullite and quartz, while those that contained impurities in the raw materials inevitably resulted in the development of minor phases, in addition to the major phases, had a negative effect on the measured properties. Consequently, it is feasible to obtain tiles that satisfy the requirements of ISO 13006 specification provided that the used raw materials are free (as much as possible) from impurities such as hematite, magnesium, and calcium.

Introduction

Many sources independently verify that the actual known history of clay-based tiles (and the known usage of wall and floor coverings) can be traced back as far as the fourth millennium BC (4000 BC) to Egypt.

Ceramic tiles are primarily hygiene products as this is evident from their varied usage from bathrooms, kitchens, to medical centers, labs, schools, public conveniences, and shopping malls [1]. Ceramic tiles (floor and wall) have proved to be one of the very fast growing heavy clay-based industries in the last three decades in Egypt. The experience and technological skills acquired during these decades encouraged several investors and manufacturers to enter this industry. The increasing and continuous demands, either locally or internationally, inspired several businessmen to lay down more investments

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Table 1 Summary of test results of water absorption, apparent porosity, apparent relative density and bulk density.

Factories	Absorption (%)	Apparent porosity (%)	Apparent relative density	Bulk density (gm/cm^3)
1	9.56	18.99	2.46	1.99
2	9.47	18.19	2.35	1.92
3	11.0	20.75	2.38	1.89
4	9.39	17.20	2.23	1.84
5	11.85	22.03	2.39	1.86
6	13.08	24.29	2.45	1.86
7	11.81	22.0	2.39	1.86
8	11.24	21.05	2.37	1.87
9	9.91	18.07	2.30	1.87
10	12.30	22.79	2.40	1.85
11	11.46	21.33	2.37	1.86
12	15.75	27.50	2.41	1.75
13	11.17	20.73	2.34	1.86
14	11.11	20.72	2.35	1.86

to establish ceramic tile factories. It is not expected now to find any of the newly founded towns in Egypt (and sometimes old ones) that do not encompass one of these factories. The greatest progress in the manufacturing of ceramic tiles was made in Africa, specifically the north of the continent, where production increased most notably in Egypt and Morocco. Egypt has doubled its manufacturing output in the last five years, reaching 200 million square meters in 2009. Egypt produces, consumes, and exports more tiles than any other African country. Use of tiles in Africa is concentrated in four countries; Egypt, Morocco, South Africa, and Algeria. Egypt, alone, is responsible for 38 percent of Africa's tile consumption with 180 million square meters of product purchased there in 2009. Also, Egypt is Africa's major exporter as it exported 23 million square meters in 2009 [2]. One of the major motivations of ceramic production in Egypt is the availability of the raw materials, as well as their low cost which results in making production of ceramic tiles a highly profitable business. The terms pottery, whitewares or triaxial ceramics include not only domestic tableware, but also wall and floor tiles, sanitary ware and electrical porcelain. The raw materials used in the pottery industry are mainly clays (backbone of ceramics), fillers (usually quartz sand), and fluxes (feldspars).

Clay supplies the workability of the green ceramic mixtures which simplifies the manufacture of clayware and also accounts for the dry strength of the unfired article and consequently assists in reducing handling losses. The properties of any particular clay are dependent on a number of factors including the type and amount of the used clay mineral, the particle size and size distribution of the clay and the other minerals present in the raw mineral mixture. The most important clay mineral for potters is Kaolinite which is seen by the electron microscope to crystallize into minute hexagonal plates. The ease with which these platey particles can slide over one another, particularly when lubricated with water, accounts for the plasticity, and the closeness with which they can pack accounting largely for the dry strength.

Fillers usually have a high melting temperature and are chemically resistant inorganic materials whose main function is to reduce the ceramic body's tendency to warp or distort when fired to temperatures which result in the formation of relatively large amounts of molten glass in the body. They also play an important role in determining the thermal expansion of the fired ceramic body. Free silica in the form of quartz, is by far the most common filler used in whitewares. In most sands and sandstones, quartz is the predominant mineral. However, many sand stones contain iron compounds that affect the color of the product. Most of the iron oxide associated with sand is present as a thin film covering each individual grain. It is possible to remove the bulk of this impurity by acid treatment, but this will increase the cost of the product.

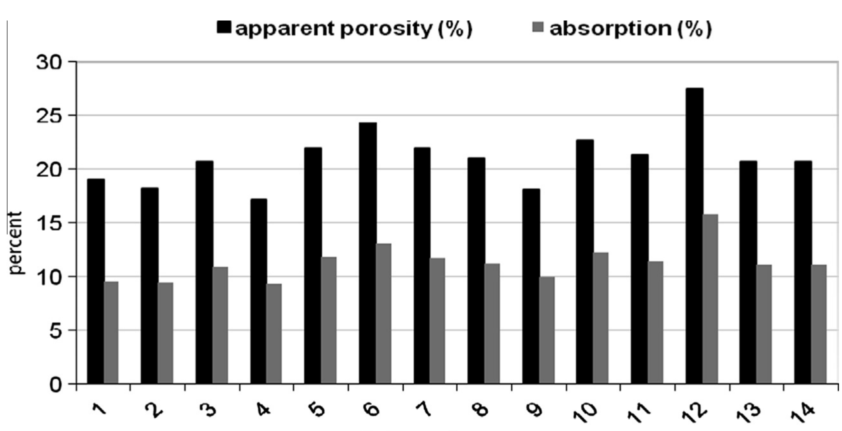

Fig. 1 Comparison between absorption and apparent porosity of factories for ceramic walls.

Table 2 Limits of breaking strength and modulus of rupture.

Water absorption (%)	Thickness (mm)	Breaking strength (N)	Modulus of rupture (N/mm^2)
$6 \leqslant E \leqslant 10$	(a) $\geqslant 7.5$	Not less than 800	Minimum average 18
	(b) < 7.5	Not less than 500	Individual minimum 16
$E > 10$	(a) $\geqslant 7.5$	Not less than 600	Minimum average 15
	(b) < 7.5	Not less than 200	Individual minimum 12

Table 3 Summary of test results of breaking strength, modulus of rupture, hardness, coefficient of restitution, and crazing.

Factories	Min. thickness (mm)	Breaking strength (N)	Modulus of rupture (N/mm^2)	Hardness (Moh's scale)	Coefficient of restitution	Crazing
1	7.0	748.18	17.47[a] 24.79[b]	5	0.57	None
2	7.5	677.47	17.34 18.42	5	0.26	None
3	7.5	568.71	14.53 15.94	4	0.66	None
4	7.5	718.98	14.54 18.88	4	0.62	None
5	< 7.5	636.53	20.63 17.50	4	0.51	None
6	> 7.5	690.73	16.16 16.70	4	0.35	None
7	9.0	706.62	12.48 13.64	4	0.39	None
8	8.0	763.36	16.88 19.36	4	0.29	None
9	8.5	664.56	13.47 13.61	4	0.46	None
10	> 7.5	655.48	14.32 18.04	5	0.37	None
11	7.5	738.88	19.0 19.56	5	0.45	None
12	> 7.5	528.25	13.14 13.96	5	0.31	None
13	8.0	788.43	18.85 20.72	5	0.30	None
14	< 7.5	805.16	22.25 22.43	4	0.38	None

[a] Individual minimum.
[b] Average.

Fluxes are constituents which melt when the ware is fired. The firing process results in the particles of other materials being drawn together so that the article contracts. When the ware cools, the glass solidifies and provides the bond which holds the mass together and this accounts for most of the fired strength of pottery ware. The temperature at which the individual fluxes start to melt is largely dependent on the total alkali content and on the particle size of the material. Mica, particularly in the extremely finely divided form in which it occurs in the clays, is one of the first of the glass- formers to melt. Feldspar is essentially a mixture of two feldspars, the minerals orthoclase and albite. The first is called Potash feldspar (high potassium content), while the second is called Soda feldspar (high sodium content).

It is important to consider first the effect of heat on the individual minerals constituting the clay-ware mixture. The changes which take place are both chemical and physical, and result in the disappearance of some minerals and the creation of new ones. Between 450 and 500 °, Kaolinite starts to decompose. The reaction is accompanied by an expansion so that the ware at this stage is larger than when it entered the kiln. At about 980 °, a sudden evolution of heat accompanies the complete breakdown of the structure with the formation of the minerals, mullite and quartz. The temperature at which the individual fluxes start to melt is largely dependent on the total alkali content and on the particle size of the material.

The firing temperature of all pottery bodies is several hundred degrees below the melting temperature of the filler. However, the filler may react chemically with glass if the particles are extremely small or the firing temperature is high and prolonged, or it may be converted into a different crystal form with different physical properties. Silica in particular can exist in a variety of crystal forms which differ in density and thermal expansion characteristics from one another.

During the vitrification stage, fluxes start to melt and the ware shrinks. The glass formed provides the bond which gives the fired article its strength, and as the amount of glass

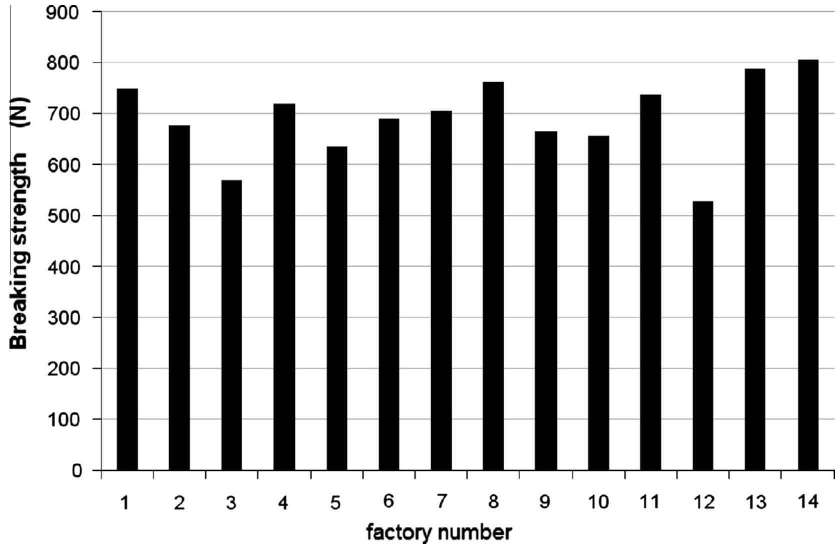

Fig. 2 Breaking strength results for different factories for ceramic walls.

increases, so the porosity of the ware decreases and since porosity is an important property the degree of firing is controlled to produce the value required [3].

Das et al. [4] characterized and evaluated the influence of five different clays of Indian sources on wall tile compositions. The results showed that the compositions containing a higher amount of quartzitic clays possessed lower shrinkage (< 1.0%) in the temperature range of 1050–1150 °. Bodies containing originally higher amount of Kaolinitic clays showed lowest water absorption and highest strength due to better densification. XRD studies conducted on fired tile specimens (1150 °) showed the formation of anorthite and quartz as major crystalline phases and monticellite and mullite as minor phases. Probably, there was a need to raise the firing temperature to increase the amount of the mullite phase.

Method of approach

Due to the unavailability of data regarding the used raw materials (constituents and nature), the manufacturing processes, and the curing methods employed as the tested specimens were purchased from the local market, the mechanical properties (namely flexural strength) and physical properties (namely water absorption and apparent porosity) were the essential criteria for the evaluation. Modulus of rupture is a mechanical characteristic to which reference is usually made concerning the quality control of ceramic tiles. MOR is utilized for two reasons: firstly, to determine the correctness of the production process in relation to having obtained the desired degree of compactness and consistency. Secondly, to determine whether or not the tile is capable to support the loads which will be imposed on it [5]. Also, flexural strength depends on the body composition and dimensions and morphology of the flaws. The behavior of the specimens can be explained taking into account the different microstructures developed during firing [6]. Physical and chemical characteristics of any tile depend on its porosity. Consequently, water absorption was selected as one of the classification parameters in the EN Standards 5. The significance of determining the water absorption of the ceramic tiles is based on the value of water absorption, the limits of breaking strength, and modulus of rupture of the specimens are specified according to ISO 13006 [7] and ESS 3168 [8,9]. The thickness of the specimens is also taken into account when determining the limits of breaking strength and modulus of rupture. In other words, both water absorption and thickness of the specimen are the two criteria that determine the limits of breaking strength and modulus of rupture of the specimens. Acceptance criteria for commercially produced tiles may be subdivided into three categories: facial and structural soundness, dimensional characteristics, and physical properties. The latter is considered to be directly related to the properties of tile materials. Consequently, the tile bodies, regardless of their applications need to satisfy the two most important requirements, water absorption and breaking strength (reflecting tensile strength) [10].

In order to conduct the assessment, it was necessary to identify the internal structure of the tiles. The identification was conducted using X-ray diffraction analyses (XRD) to specify the various phases and minerals that were developed during the firing process. Consequently, determining a correlation between the measured properties and internal developed constituents.

Experimental procedure

The ceramic samples used in this study were glazed wall tiles obtained from the Egyptian local market. Each sample consisted of twenty tiles and represented a factory.

Three tests were carried out on the samples according to both the ESS (Egyptian Standard Specifications) 293 and ISO (International Organization for Standardization) 10,545 as the methods of the three tests according to both standards are the same. The three tests are as follows: determination of water absorption, apparent porosity, apparent relative density and bulk density, determination of modulus of rupture and breaking strength, and finally determination of crazing resistance for glazed tiles.

The determination of impact resistance was carried out according to ISO 10545-5, while Moh's hardness test was performed according to ESS293-1993.

The test method for the determination of water absorption, apparent porosity, apparent relative density and bulk density was carried out according to ISO 10545-3 [11] and ESS 293-1 [12]. The test was carried out as follows: the tiles were placed vertically, with no contact between them, in a vacuum chamber. Pressure was evacuated to (10 ± 1) kPa and maintained for 30 min. Then, while maintaining the vacuum, sufficient water was admitted slowly to cover the tiles by 5 cm. The vacuum was released and the tiles were allowed to remain submerged for 15 min. Tiles were placed on a flat surface and lightly dried in turn. Immediately after this procedure, tiles were weighed and the results were recorded to the same accuracy as for the dry state. After impregnation under vacuum of the test specimens, the mass of each specimen was determined while suspended in water. Weighing was carried out by placing the specimen in a wire loop halter, or basket that is suspended from one arm of the balance. Before actually weighing, the scale was counterbalanced with the wire loop in place and immersed in water to the same depth as is used when the specimens are in place. According to IS0 10545-3 and ESS 293-1, there are two methods to determine the water absorption: Boiling method and the vacuum method. In this study, specimens were tested according to the vacuum method. Apparent porosity, relative density and bulk density were also determined for the tested samples in order to meet the requirements of both standards (ESS and ISO), respectively.

Water absorption, apparent porosity, apparent relative density, and bulk density:

M_1 is the mass of the dry tile.
M_2 is the mass of the tile impregnated by immersion under vacuum;
M_3 is the mass of the suspended tile impregnated by immersion under vacuum;
V is the external volume calculated using the following equation: $(m_2 - m_3)$.

Water absorption = $((m_2 - m_1)/m_1)*100$.
Apparent porosity = $((m_2 - m_1)/V)*100$.
Apparent relative density = $m_1/(m_1 - m_3)$.
Bulk density = m_1/V.

The determination of modulus of rupture and breaking strength was according to ESS 293-10 [13] and ISO10545-4 [14]. Breaking strength is defined as the force obtained by multiplying the breaking load by the ratio (span between support rods)/(width of the test specimen). It should be noted that the breaking strength is expressed in newtons. Seven test specimens were tested for each sample.

The breaking strength S, is calculated by means of the equation

$$S = (FL)/b \quad (1)$$

where, F is the breaking load, in newtons; L is the span, in millimeters, between the support rods; b is the width of the test specimen, in millimeters.

The modulus of rupture R, expressed in newtons/square millimeter, is calculated by means of the equation

$$R = (3S)/(2h^2) \quad (2)$$

where h is the minimum thickness of the test specimen, in millimeters, measured after the test along the broken edge.

The resistance to impact for tested glazed tiles was carried out according to ISO 10545-5. The test is carried out by testing five pieces in dimensions 75 * 75 mm cut from five tiles. The test samples consisted of test specimens fixed to mature concrete blocks by means of a rigid epoxide resin. The ball-release apparatus is adjusted by means of a leveling screw so that the steel bar is vertical. The test sample was placed under the electromagnet so that the steel ball released from the electromagnet falls onto the center of the test unit clamped in position. A steel ball is then dropped from a fixed height onto the test specimen and is allowed to bounce. The average coefficient of restitution is then calculated using ISO 10545-5 [15].

The hardness test was carried out using Moh's scale in accordance with ESS 293-1993 [16]. A minimum of three test

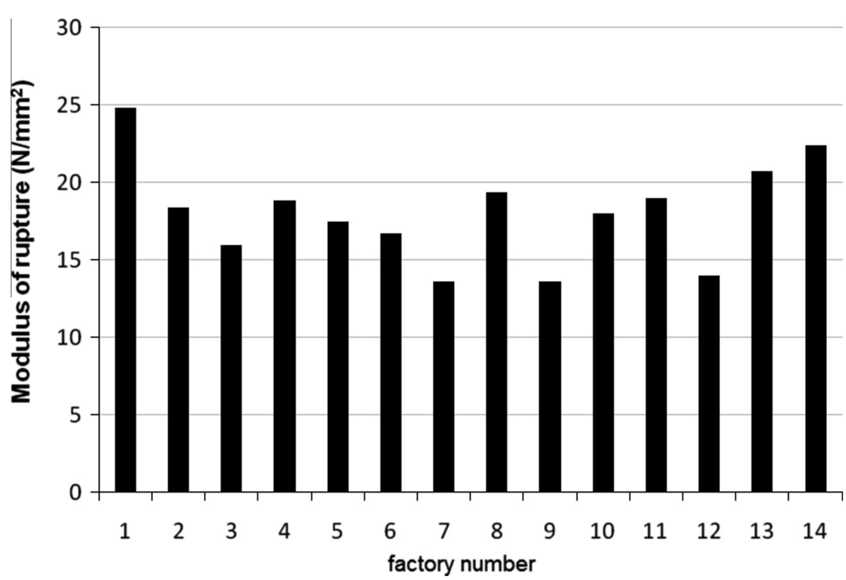

Fig. 3 Modulus of rupture results for different factories for ceramic walls.

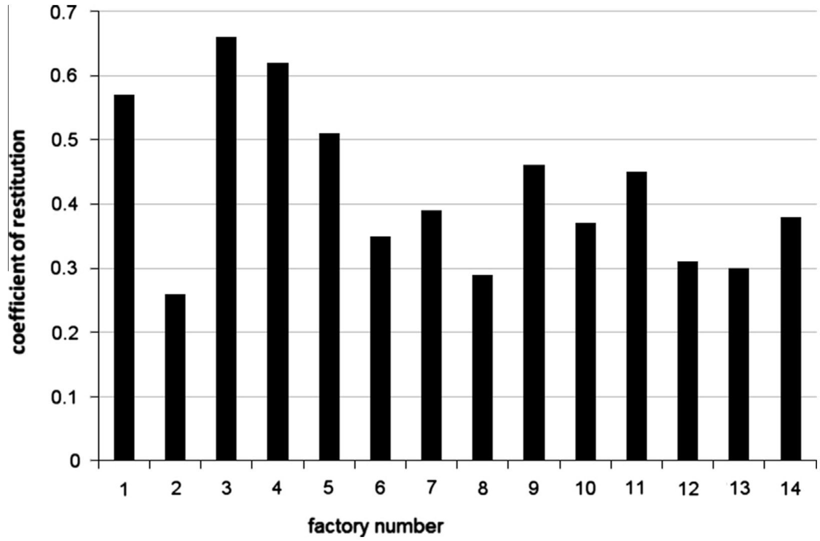

Fig. 4 Coefficient of restitution results for different factories for ceramic walls.

tiles must be used, for each type. The mineral of highest Moh's hardness that results in no more than one scratch is recorded. In case of tiles of variable hardness, the lowest value of Moh's hardness is recorded.

The resistance to crazing for glazed tiles was carried out according to ISO 10545-11 [17]. Five whole tiles were tested. The test specimens were placed in the autoclave in such a way that there was air space between them. The pressure in the autoclave was raised gradually for a period of 1 h until it is in the range of 480–520 kPa, and temperature in the range of 158–160 °C. Then the steam source is turned off and pressure is allowed to fall as rapidly as possible to atmospheric. The specimens are then allowed to cool in the autoclave for 0.5 h. The specimens were brought to laboratory atmosphere, and then placed singly on a flat surface and allowed to cool for another 0.5 h. A stain such as 1% aqueous solution of methylene blue containing a small quantity of wetting agent is then brushed and later wiped off after 1 min with a damp cloth. The specimens are then examined for crazes.

The used XRD apparatus is an X'Pert PRO PW3040/60 (PANalytical) diffractometer equipped with monochromatic Cu-K_α radiation source. The test was run at 40 kV and 40 mA. A continuous mode was used for collecting data in the 2θ range from 5° to 60° at a scanning speed of 2°/m. X-ray diffraction (XRD) was conducted on five fine powder samples (each sample was from a different factory), at both the un-

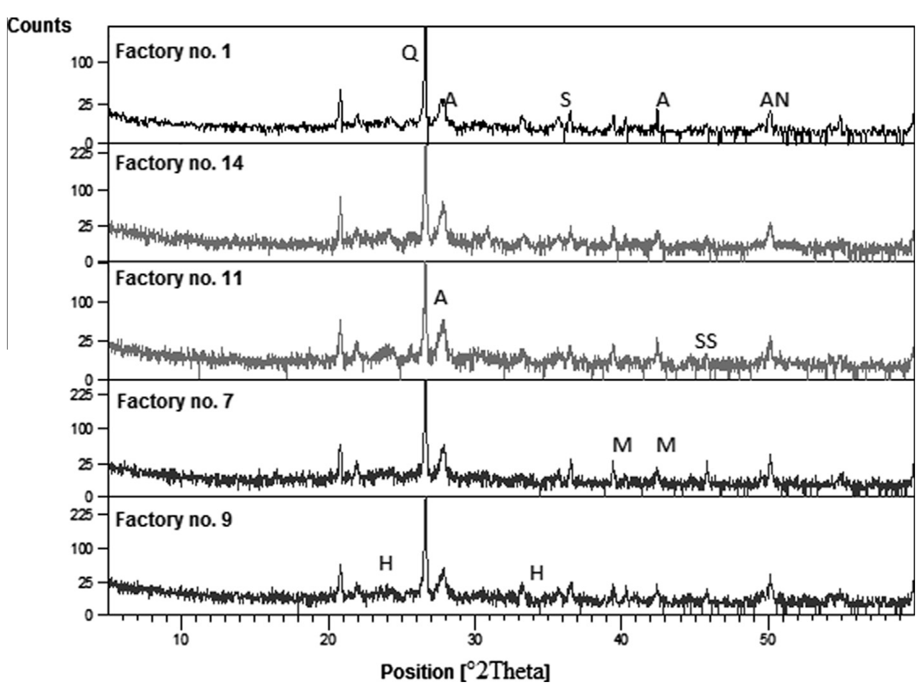

Fig. 5a XRD of ceramic tiles edge.

glazed edge and core of each sample, to examine the homogeneity of the formed phases during the firing process. The samples were selected based on the flexural strength results as the five samples gave different results regarding flexural strength. The five samples were from factory numbers 1, 14, 11, 7, and 9, respectively.

Test results and analysis

Water absorption, apparent porosity, apparent relative density and bulk density

The obtained test results of water absorption, apparent porosity, apparent relative density and bulk density are shown in Table 1. The results show that water absorption of the ceramic samples obtained from the chosen factories ranged from 9.39% to 15.75%. It should be noted that the vacuum method fills almost all the open pores.

The apparent porosity is the relationship of the volume of the open pores of the test specimen to its exterior volume. The apparent porosity results ranged from 17.2% to 27.5% for the samples from the chosen factories. The apparent relative density of the impervious portion of the test specimen ranged from 2.3 to 2.46. The bulk density of a specimen is the quotient of its dry mass divided by the exterior volume, including pores. The obtained results ranged from 1.75 to 1.99 g/cm^3.

Fig. 1 shows comparison between water absorption and apparent porosity with respect to the fourteen factories. The ratio between water absorption and apparent porosity was approximately in the range of 0.5

Breaking strength and modulus of rupture

As mentioned earlier, the limits of breaking strength and modulus of rupture depend on both water absorption and thickness of the specimen.

Table 2 shows the limits of breaking strength and modulus of rupture for dry-pressed ceramic tiles according to ISO13006 and ESS3168 respectively.

The results of breaking strength and modulus of rupture are shown in Table 3. The analysis of test results showed that factories 2, 8, 11, 13 did not satisfy the requirement of the breaking strength but met the requirements of the modulus of rupture. Factories numbers 3, 4, 6, 7, 9, 10 and, 12 did not meet the requirements of both breaking strength and modulus of rupture. Factories numbers 5, 6, 7, met the requirements of modulus of rupture. Factories numbers 1, 5 (the MOR was 3% less than required by minimum average) and 14 satisfied the requirements of both breaking strength and modulus of rupture.

Figs. 2 and 3 show the values of the breaking strength and modulus of rupture for different factories.

Hardness

Moh's scale is a geological scale of minerals to rate scratch resistance which is a mechanical characteristic of the tile surface and subjectively assigns a "Moh's" number to the glazed surface. The strength of the glaze is determined by its hardness. The test results are given in Table 3. The hardness of all tested specimens was either 4 (Fluorite) or 5 (Apatite).

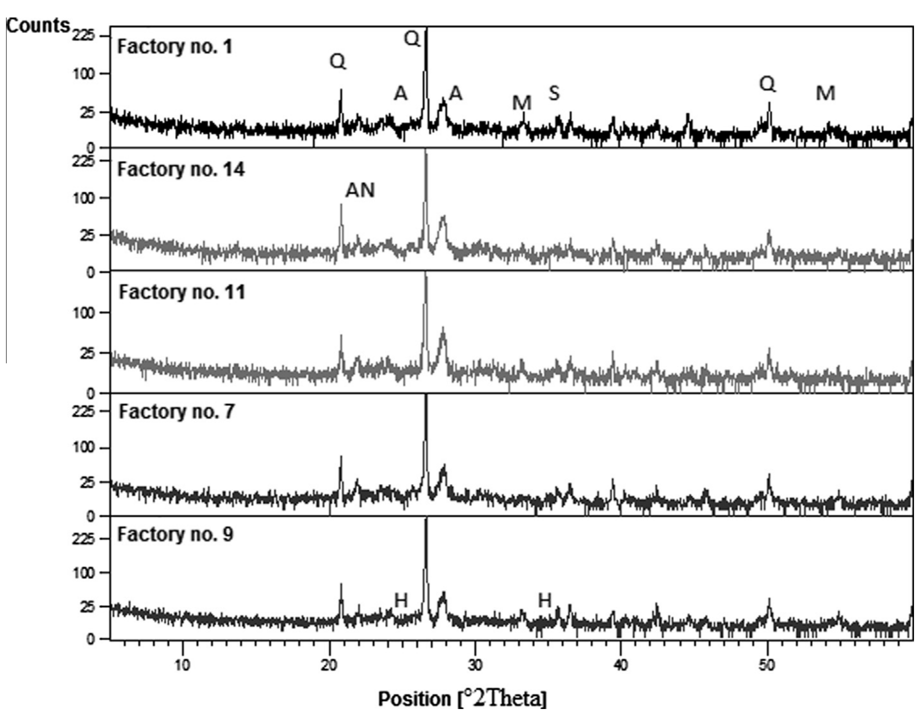

Fig. 5b XRD of ceramic tile core. Q: Quartz, A: Albite, S: Spinel, AN: Andradite, SS: Spinel, syn, H: Hematite, M: Mullite.

Coefficient of restitution

The results of coefficient of restitution are shown in Table 3. Fig. 4 shows the coefficient of restitution for different factories. The results were in the range of 0.26–0.66. An informative annex in ISO 13006 indicates that the normal requirement for light duty an installation is a coefficient of restitution of 0.55. For heavier duty applications a higher value would be required. The results show that only three factories had a coefficient of restitution greater than 0.55.

Crazing

The test results are shown in Table 3. The obtained results show that none of the tested specimens crazed.

X-ray diffraction analyses (XRD)

When comparing the XRD patterns that were conducted on the five selected tiles (from the different factories) at both the edges and core of each tile, the results showed limited differences regarding the formed phases pertaining to edges and cores respectively. The XRD patterns for both the edge and core of tested specimens are shown in Figs. 5a and 5b, respectively. This reflects that during the firing treatment stage, the temperature was uniform over the kiln.

Traditionally, tiles, whether floor or wall tiles, are members of the triaxial family. The main constituents when fired are mullite and quartz (filler material at the beginning) which are held together by the formed glass (initiated from the melting of feldspar). Due to the presence of impurities in some raw materials, attached minor phases inevitably develop. Due to the fact that the ceramic systems are non-equilibrium systems, different phases develop, with each phase having its own structure, composition, and properties, as the heating temperature varies both in time duration and degree. Consequently, it becomes difficult to predict the exact final phases of the end product. During cooling, the behavior of the different phases varies in its recrystallization behavior resulting in flaws, porosities, and dislocation, and mismatching due to different thermal contractions of different compounds. Generally, the presence of undesired phases, worsens the mechanical properties.

According to Mesbah et al. [18], as heating proceeds, a spinel-type phase crystallizes within the metakaolinite, beginning above 900 °. Experimentally, it is well known that at about 1100–1200 ° spinel is lost and mullite crystals begin to develop. Some peaks of spinel and mullite phases start to develop and co-exist at 1000 °. Both the intensity of these peaks (in the XRD) and the crystallinity of mullite are slightly increased by firing at 1100 °. Mullite is poorly-crystalline in the firing range 1000–1100 °. With increasing firing to 1200 °, the spinel-type phase completely disappears, and the mullite phase becomes very pronounced and it is the dominant crystalline phase at this temperature.

The XRD analyses in this research indicated that the major phases were quartz (SiO_2), mullite ($3Al_2O_3 \cdot 2SiO_2$), and albite ($NaAlSi_3O_8$) within the glassy phase, in the five tile types. In addition, smaller quantities of spinel ($LiFe_5O_8$), Andradite ($Ca_3Fe_2 \cdot (SiO_4)_3$ and spinel, syn. ($MgAl_2O_4$) were also present. Hematite (Fe_2O_3) was present only in factory No. 7 tile type. The presence of albite in the five types of tiles indicates that not all the albite has been melted. However, the melted amount was partially sufficient to hold the formed compounds together. It was also observed that the amount of mullite is limited denoting that the firing temperature might have been in the range of 1000 and 1100 °. The attached minerals such as spinel and andradite are attributed to some side reactions of the impurities present in the raw materials. The limited amounts of attached materials (most synthetic) are due to the reaction of impurities and main constituent materials. The presence of iron containing minerals denotes that Aswan clay which usually contains iron oxide in the range of 5–7% was used in the original batches. Also, the presence of compounds that contain magnesium or calcium, may possibly indicate that dolomite (Ca–Mg Carbonate) may have been added, which, in turn, resulted in mineralization process in the solid state during the firing process. The XRD graphs for both the edge and core of tested specimens are shown in Figs. 5a and 5b, respectively.

As mentioned earlier, flexural strength depends on composition and morphology of produced flaws during firing. Thus, the difference in the flexural strength values between specimens may possibly be attributed to the presence of impurities in the raw materials, which, in turn, resulted in undesired phases that had negative effects on flexural strength. Hematite was observed in the investigated factory No. 7 tile, and may possibly have caused the observed low modulus of rupture.

Also, apparent porosity reveals the presence of entrapped air from gases due to its entrapment during the cooling stage leading to different pores (open and closed).Another possible explanation concerning the obtained modulus of rupture and apparent porosity may be attributed to the particle size of the used quartz. Quartz particle size that was used by the different factories was not available to the researcher. Thus, there is a possibility that different factories may have used various particle sizes of quartz, which, in turn, affected both flexural strength and apparent porosity. This explanation is in agreement with the findings of previous research works. Amoros et al. [19] examined the effect of quartz particle size in raw material customarily used for the manufacture of porous single-fired wall tiles on the characteristics of the green tiles and on the thermal and mechanical properties of the fired tiles. Quartz particle size was varied while the quantity and particle size of the other raw materials were kept constant. The resulting fired microstructure was then characterized and tile thermal and mechanical properties were determined. The obtained results confirm that the green and fired properties of porous single-fired wall tiles may be considerably enhanced, while holding low shrinkage and high porosity, compatible with low moisture expansion, by reducing quartz particle size and appropriately adjusting the pressure and firing temperature. This should enable thin and/or large-sized porous wall tiles to be manufactured, without curvatures, and with a higher breaking load than that required by the specifications.

Conclusions

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

The samples which yielded physical and mechanical properties that met the requirements of ISO 13006 specification, are those that are members of the triaxial family. In other words, their bodies contain mainly mullite and quartz which are held together by the formed glass.

The samples which yielded physical and mechanical properties that did not satisfy the requirements of ISO 13006 specification, are those which contained impurities in the raw materials. Consequently, minor phases were inevitably developed, (in addition, to the major phases) and had a negative effect on the measured properties.

It is feasible to obtain tiles that meet the conditions of the specification, provided that the used raw materials are free (as much as possible) from impurities such as hematite, magnesium, and calcium.

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