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Preparation of porcelain tiles from Egyptian raw materials

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Abstract

A porcelain tile recipe was prepared from Egyptian raw materials. Ground powders were mixed and dry pressed at forming pressures ranging from 35 to 55 MPa then fired at temperatures in the range 1200–1230 °C (±2 °C) for soaking periods up to 20 min. The results showed that using a forming pressure of 45 MPa, and firing at 1220 °C for 20 min gave a dense body having a water absorption of 0.1%. Other physico-mechanical characteristics were determined and were found to match specifications.

Keywords: D. Porcelain; Tiles; Raw materials

1. Introduction

Porcelain tiles were introduced at the end of 1980s by Fiandre, one of the leading Italian porcelain tile manufacturers. Among the various types of ceramic floor and wall tile, porcelain tile is the product which in recent years has shown the greatest rate of increase (on a percent basis) in the amount produced, amount sold, and obviously amount used [1]. The American National standard Specifications for Ceramic Tile defines porcelain tiles as: dense, smooth, impervious (with water absorption of 0.5 percent or less), and stain resistant [2]. Their peculiar characteristics put them at the top of a class of available commercial products that can be used for both indoor and outdoor building applications. Since they achieve a very high level of combination between physico-mechanical properties and decorative quality, they are expected to replace the traditional ceramic floor tiles completely in the next few years.

In Egypt, there are several factories that produce ceramic wall and floor tiles, but there are only two factories producing porcelain tiles. They mainly use imported raw materials and a little portion of Egyptian raw materials. Consequently the aim of this work is to use Egyptian raw materials in order to produce a porcelain tile which satisfies the level required by the standards.

2. Experimental technique

2.1. Preparation of raw materials

From each raw material of the base composition, about 10 kg were provided in the form of lumps. They were crushed and ground. All powders were screened to pass 200 mesh. The fine powders were then mixed according to the designed proportions.

2.2. Preparation of the test samples

Discs of 2.5 cm diameter and 3 mm in thickness were pressed at different molding pressures in order to measure the physical properties. Rectangular prisms of 1×1×7 cm dimensions were pressed in order to measure the modulus of rupture whereas rectangular prisms of 5×5×2 cm dimensions were pressed in order to measure the abrasion resistance.

The pressed specimens were then dried for 24 h at 110 °C before firing. The dimensions of the pressed specimens were measured before and after firing in order to determine the firing shrinkage.

2.2.1. Forming pressures

The test specimens were pressed at five molding pressures: 35, 40, 45, 50, and 55 MPa in order to study the effect of molding pressure on the final properties.

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2.2.2. Firing of the test samples

All the specimens were fired according to the once-fast firing technique. The temperature of the samples was raised at a rate of 80 °C/min from room temperature to the maximum firing temperature. Four maximum firing temperatures were tested: 1200, 1210, 1220, and 1230 °C. The temperature sensitivity of the muffle furnace used was ± 2 °C. The maximum temperature was followed by a soaking time. Three soaking times were examined: 10, 15, and 20 min.

2.3. Investigation techniques

A full chemical analysis using X-ray fluorescence was applied to all raw materials. Their mineralogical compositions were studied using XRD. The plasticity was determined according to Pffeferkorn method. The thermal behaviour of the raw materials was studied using differential thermal analysis (DTA) and thermal gravimetric analysis (TGA).

The extent of vitrification was determined by measuring some physical properties such as linear shrinkage, water absorption, bulk density, apparent porosity, true porosity and closed porosity.

The microstructure of the fired samples was studied also using electron scanning microscopy analysis technique.

3. Raw materials

3.1. Choice of raw materials

In order to choose the suitable raw materials, the properties of the final product had to be taken into consideration.

The dense microstructure of the fired porcelain tiles is characterized by a very low porosity and a small pore size owing to the presence of small strongly interlocked crystals of mullite embedded in an amorphous glassy phase [3].

Mullite was first found to contribute to the strength of porcelain by strengthening the glassy phase with interlocked mullite and was known as the mullite hypothesis [4]. It was reported that both Young's modulus and shear modulus increased linearly with the total amount of mullite, implying that the strength of the whiteware would also increase in proportion to mullite content [5, 6].

3.1.1. Batch design considerations

1. A large amount of kaolinite is fundamental in mullite formation and, thus, in obtaining the desired mechanical properties of porcelain tiles [7].
2. A sufficient amount of feldspar is necessary to obtain the desired glassy phase (50–60% for floor tiles compositions) [8]. When the bodies are fired between cones 9 and 12, a vitrified body results with zero absorption.
3. As in all traditional ceramics, the presence of quartz is necessary in order to decrease shrinkage. It also reduces the body tendency to warp or distort during firing [9].
4. The presence of alumina is very useful in increasing the bending strength of the body. It was reported that as the amount of alumina in the melt increased the microhardness of the glassy phase increased and its brittleness decreased [10].
5. The addition of talc to porcelain tile bodies has also many advantages:
    - The positive effect of talc additions on the strength of aluminous porcelain was reported [11]. Small additions of talc have increased the mullite content of the samples. The addition up to 2% talc has no effect on mullite content, but it produces an increase in modulus of rupture [12]. Further additions of talc up to 5% increases the quantity of mullite [13].
    - Addition of 3.2% of talc to the batch of porcelain tile improved the bending resistance by 20%. It also improves the stain resistance of polished tiles [14].
    - Addition of talc to floor tile batch acts as an auxiliary fluxing agent [15]. The soda feldspar and talc eutectic reduces the vitrification temperature in rapid firing cycles [14].
6. Kaolins are characterized by low plasticity. Ball clays have a good plasticity but they contain considerable amounts and variety of impurities, which impart colour to the fired body. Bentonite was used in this work in order to give the required plasticity. Small additions (from 1 to 3%) of bentonite were added by several authors to ceramic bodies to enhance plasticity, green and dry strengths [16–18].
7. As a general rule, all the raw materials used in this work must be of high purity with regard to coloring oxides.

3.1.2. Proposed batch composition

In order to select the suitable amount of bentonite, three batch compositions containing three proportions of bentonite (1, 2, and 3%) were prepared. The plasticity of these batches was measured and compared with the plasticity of a batch, which is actually used in porcelain tiles factories. There was a matching between the plasticity of the batch containing 3% bentonite and that delivered by the factory.

Taking all the above considerations into account, the following recipe was suggested: 41% El Teeh clay, 41% soda feldspar, 5% talc, 5% quartz, 5% alumina, 3% bentonite.

3.2. Occurrence of raw materials

3.2.1. El Teeh clay

Clays beds in El Teeh area contain the major clay reserves in Sinai. They were estimated at about 88 million tons as reported by the Geological Survey and Mining Authority [19].

3.2.2. Soda feldspar

In Egypt, feldspar deposits are mainly located in Sinai and the Eastern Desert. Soda feldspar, which is used in this study, is present in Wadi El-Tarr region in south Sinai. The geological reserve is estimated to be more than 1.5 billion tons [20].

3.2.3. Quartz

The sand used in this work is delivered from El Zafarana region at the west to Suez Gulf. The sand in this area consists of a section with a thickness of about 100 m. The reserve estimated in only one mine is about 10 million m^3 [20].

3.2.4. Talc

In Egypt talc is located in many regions in the Eastern Desert. Talc used in this work is obtained from Hamata district in the Eastern Desert. It is white in colour and of high purity.

3.2.5. Bentonite

Bentonite occurs at many localities in Egypt. Bentonite used in this work is obtained from the Barkan area south of El Alamin. Studies have confirmed that the bentonite at this area is of good purity and has a montmorillonite content of about 83.7% which is higher than other locations [21].

3.2.6. Corundum

In Egypt, corundum (α-alumina) is available at Wadi Hafafit in Eastern Desert but it is still under investigation [20]. So, in this work, imported corundum was used.

3.3. Characterization of raw materials

The chemical compositions of the raw materials are determined using X-ray fluorescence technique. The results of XRF are given in Table 1 and the chemical analysis of the base composition was calculated and given in Table 2. The mineralogical compositions of raw materials were studied using X ray analysis. The obtained XRD are shown in Fig. 1.

4. Results and discussion

4.1. Vitrification parameters

Tables 3–7 show the results of the vitrification parameters for all specimens pressed at the five selected molding pressures and fired at all the tested firing temperatures for the three examined soaking times.

Generally, from these tables, it is found that the minimum values of water absorption and apparent porosity are obtained at a temperature of 1220 °C and 20 min soaking time for the specimens pressed at 45, 50, and 55 MPa.

Since raising the molding pressure above 45 MPa did not cause any observed enhancement of the vitrification parameters, this pressure (45 MPa) was taken as the optimum molding pressure.

Table 2
Calculated chemical analysis of the base composition

Constituent	Base composition
SiO_2	57.03
TiO_2	0.81
Al_2O_3	27.73
Fe_2O_3	0.79
MgO	1.79
CaO	0.45
Na_2O	3.77
K_2O	0.15
L.O.I	7.08

Table 1
The chemical composition of raw materials

Constituent	El Teeh clay	Soda feldspar	Quartz	Talc	Bentonite	Alumina
SiO_2	45.47	70.09	99.00	63.57	50.24	0.25
TiO_2	1.56	0.36	0.03	<0.01	1.17	0.01
Al_2O_3	37.4	17.41	0.68	0.47	15.81	98.00
Fe_2O_3	0.9	0.47	0.05	0.21	6.93	0.05
MnO	0.01	0.01	0.01	0.02	0.02	0.01
MgO	<0.01	0.15	0.07	32.01	2.35	<0.01
CaO	0.17	0.82	0.07	0.28	0.73	0.08
Na_2O	<0.01	8.89	0.03	<0.01	2.6	0.88
K_2O	<0.01	0.29	0.05	<0.01	0.86	<0.01
P_2O_5	0.06	0.19	0.001	0.03	0.11	<0.01
L.O.I	14.22	1.00	0.15	4.89	18.77	0.44

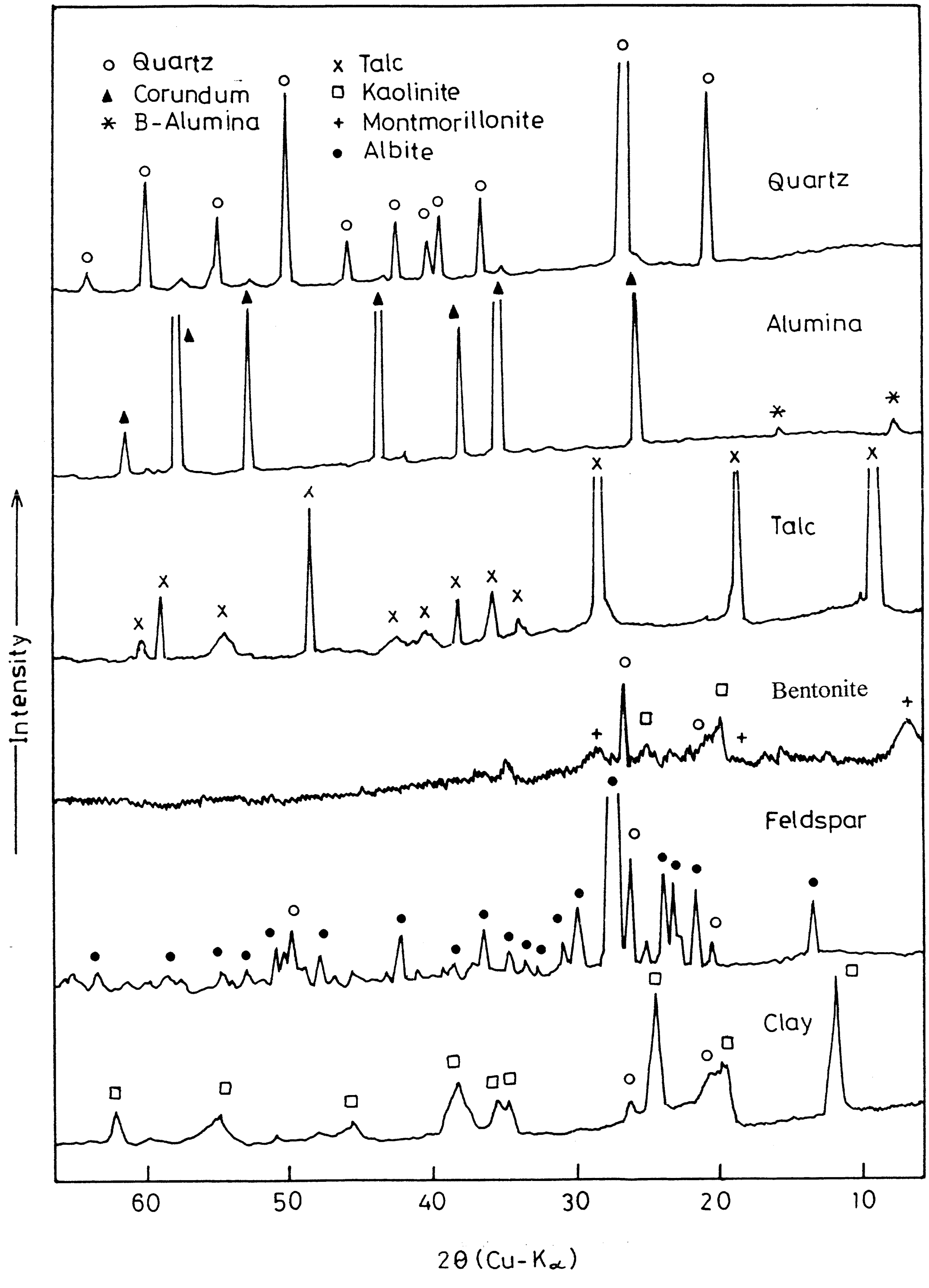

Fig. 1. XRD patterns of raw materials.

So the optimum conditions for the designed batch composition are 45 MPa molding pressure, 1220 °C firing temperature and 20 min soaking time.

It is obvious that as the peak firing temperature rises, water absorption, apparent porosity, and total porosity fall down while the bulk density, closed porosity and linear shrinkage increase. This pattern is inverted in all figures at a temperature of 1220 °C at which the water absorption and apparent porosity begin to increase again.

The sharp decrease in water absorption and apparent porosity from 1200 to 1220 °C is attributed to the large amount of liquid phase formed due to the high proportion of soda feldspar in the batch. This drop in apparent porosity is accompanied with a rise in the specimen's

Table 3
Effect of firing temperature on the vitrification parameters of specimens prepared at 35 MPa molding pressure and fired for 10, 15, and 20 min soaking time, respectively

Temperature (°C)	% W.A	% A.P	% B.D	% Shrinkage	% T.P	% C.P
(a) Soaking time: 10 min						
1200	11.7	23.8	2.05	1.0	23.8	0
1210	7.17	15.7	2.19	2.6	18.59	2.89
1220	4.02	9.37	2.33	3.9	13.38	4.01
1230	6.5	14.63	2.25	2.8	16.36	1.73
(b) Soaking time: 15 min						
1200	6	13.61	2.26	3.0	15.99	2.38
1210	3.35	7.94	2.37	4.1	11.9	3.96
1220	0.90	2.22	2.47	5.2	8.18	5.96
1230	3.11	7.40	2.38	4.6	11.52	4.12
(c) Soaking time: 20 min						
1200	2.3	5.52	2.40	4.6	10.78	5.26
1210	1.11	2.72	2.45	5.8	8.92	6.20
1220	0.16	0.4	2.50	6.7	7.06	6.66
1230	1.57	3.82	2.43	5.3	9.67	5.85

Table 4
Effect of firing temperature on the vitrification parameters of specimens prepared at 40 MPa molding pressure and fired for 10, 15, and 20 min soaking time, respectively

Temperature (°C)	% W.A	% A.P	% B.D	% Shrinkage	% T.P	% C.P
(a) Soaking time: 10 min						
1200	11.0	22.88	2.08	1.2	22.68	0
1210	6.50	14.63	2.25	2.8	16.36	1.73
1220	3.50	8.27	2.36	4.0	12.27	4.0
1230	6.14	13.88	2.26	2.9	15.99	2.11
(b) Soaking time: 15 min						
1200	5.15	11.69	2.27	3.2	15.61	3.92
1210	3.01	7.41	2.39	4.4	11.15	3.74
1220	0.64	1.59	2.49	5.6	7.44	5.85
1230	2.97	7.09	2.39	4.8	11.15	4.06
(c) Soaking time: 20 min						
1200	2.11	5.10	2.40	4.6	10.78	5.68
1210	0.90	2.22	2.47	6.0	8.18	5.96
1220	0.14	0.35	2.51	6.8	6.69	6.31
1230	1.46	3.57	2.45	5.4	8.92	5.35

closed porosity. This results from the progressive incommunication of the pores that takes place on increasing the glassy phase proportion [22,23]. For all examined molding pressures and at any soaking time, the apparent porosity reaches a minimum value and the closed porosity reaches a maximum value at temperature of 1220 °C. At higher temperatures the pressure of the trapped gases within the pores rises, opposing the progressive densification of the material [24]. So, after yielding a minimum value of apparent porosity, bloating occurs causing an increase in apparent porosity. It is clear that it is not possible to attain the required low water absorption by raising the firing temperature only, but the soaking time must be increased to 20 min and the molding pressure to 45 MPa. At this point, the total porosity is almost equal to the closed porosity.

4.2. XRD results

Fig. 2 represents the X-ray results of specimens fired at 1220 °C for three different soaking times. Both specimens fired for 10 and 15 min soaking times reveal the presence of mullite, quartz, corundum and albite. While in the specimen fired for 20 min, the peaks related to albite have completely disappeared. This means that the firing temperature and soaking time lower than these conditions are not sufficient for the complete melting of feldspar. This is confirmed by the results of vitrification

Table 5
Effect of firing temperature on the vitrification parameters of specimens prepared at 45 MPa molding pressure and fired for 10, 15, and 20 min soaking time, respectively

Temperature (°C)	% W.A	% A.P	% B.D	% Shrinkage	% T.P	% C.P
(a) Soaking time: 10 min						
1200	11.0	22.88	2.08	1.2	22.68	0
1210	6.47	14.56	2.25	2.8	16.36	1.80
1220	3.40	8.06	2.37	4.1	11.9	3.84
1230	6.11	13.81	2.26	2.9	15.99	2.18
(b) Soaking time: 15 min						
1200	5.11	11.60	2.27	3.2	15.61	4.01
1210	3.0	7.17	2.39	4.6	11.15	3.98
1220	0.62	1.54	2.49	5.6	7.44	5.90
1230	2.81	6.72	2.39	4.9	11.15	4.43
(c) Soaking time: 20 min						
1200	2.10	5.03	2.40	4.6	10.78	5.75
1210	0.91	2.25	2.47	6.0	8.18	5.97
1220	0.10	0.25	2.51	6.8	6.69	6.44
1230	1.44	3.53	2.45	5.4	8.92	5.39

Table 6
Effect of firing temperature on the vitrification parameters of specimens prepared at 50 MPa molding pressure and fired for 10, 15, and 20 min soaking time, respectively

Temperature (°C)	% W.A	% A.P	% B.D	% Shrinkage	% T.P	% C.P
(a) Soaking time: 10 min						
1200	10.60	22.26	2.10	1.2	21.93	0
1210	6.40	14.40	2.25	2.8	16.36	1.96
1220	3.32	7.87	2.37	4.1	11.9	4.03
1230	5.81	13.13	2.26	3.0	15.99	2.86
(b) Soaking time: 15 min						
1200	4.92	12.27	2.29	3.2	14.87	3.6
1210	2.88	6.88	2.39	4.9	11.15	4.27
1220	0.60	1.5	2.50	5.6	7.06	6.10
1230	2.73	6.53	2.39	4.9	11.15	4.62
(c) Soaking time: 20 min						
1200	2.0	4.90	2.45	4.6	8.92	4.02
1210	0.86	2.13	2.48	6.2	7.81	5.68
1220	0.10	0.25	2.51	6.8	6.69	6.44
1230	1.38	3.40	2.46	5.4	8.55	5.15

parameters where the specimen fired at these conditions shows the best vitrification behaviour.

4.3. Modulus of rupture

For the base composition body, only the specimen fired at optimum condition was tested. The average modulus of rupture was found to be 77 MPa, which is a high value compared to the obtained values of commercial products (from 50 to 57 MPa) [25,26]. This comparison is only tentative since the size of the specimens in different experiments affect the strength [27]. This high value may be attributed to the use of alumina in the base body composition as alumina is often used as a reinforcer. It was reported that dispersed particles increase the fracture energy by the interaction between dispersed particles and propagating crack fronts [28]. Also in the designed batch in this work, only 5% quartz is added. The α–β quartz transformation usually causes microcracks. So avoiding large amounts of quartz was advantageous.

4.4. Microstructure

Figs. 3 and 4 represent a general view of two specimens. The first was fired at the optimum firing conditions (i.e 1220 °C, 20 min) where the second was fired at 1230 °C for 20 min soaking time. It is obvious that the first specimen contains a small amount of pores, while the second reveals an increase in pores number and volume due to bloating. Fig. 5 shows the presence of well crystalline secondary mullite and quartz grains in the vitrified specimen.

Table 7
Effect of firing temperature on the vitrification parameters of specimens prepared at 55 MPa molding pressure and fired for 10, 15, and 20 min soaking time, respectively

Temperature (°C)	% W.A	% A.P	% B.D	% Shrinkage	% T.P	% C.P
(a) Soaking time: 10 min						
1200	10.5	22.05	2.10	1.3	21.93	0
1210	6.31	14.26	2.26	2.9	15.99	1.73
1220	3.21	7.64	2.38	4.1	11.52	3.88
1230	5.53	12.55	2.27	3.0	15.61	3.06
(b) Soaking time: 15 min						
1200	4.77	11.02	2.31	3.3	14.13	3.11
1210	2.85	6.84	2.40	4.9	10.78	3.94
1220	0.55	1.38	2.51	5.6	6.69	5.31
1230	2.70	6.45	2.39	4.9	11.15	4.70
(c) Soaking time: 20 min						
1200	1.96	4.80	2.45	4.6	8.92	4.12
1210	0.81	2.02	2.49	6.2	7.44	5.42
1220	0.10	0.25	2.51	6.8	6.69	6.44
1230	1.36	3.37	2.48	5.4	7.81	4.44

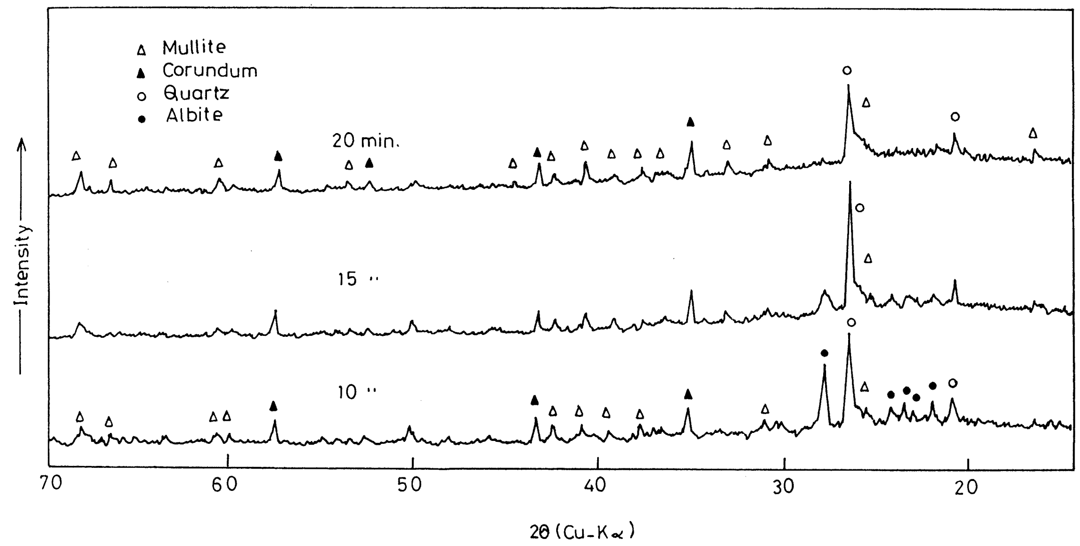

Fig. 2. XRD patterns of specimens fired at 1220 °C for different soaking times.

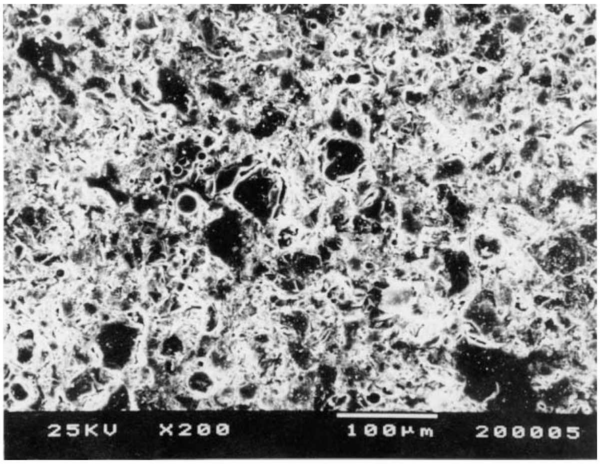

Fig. 3. SEM photomicrograph of the vitrified specimen fired at 1220 °C for 20 min soaking time (×200).

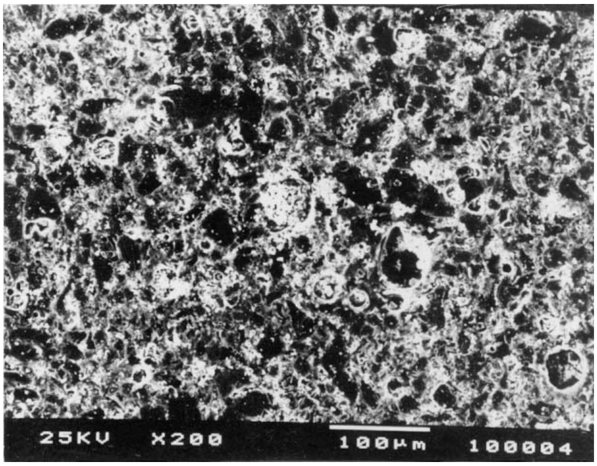

Fig. 4. SEM photomicrograph of a specimen fired at 1230 °C for 20 min soaking time (×200).

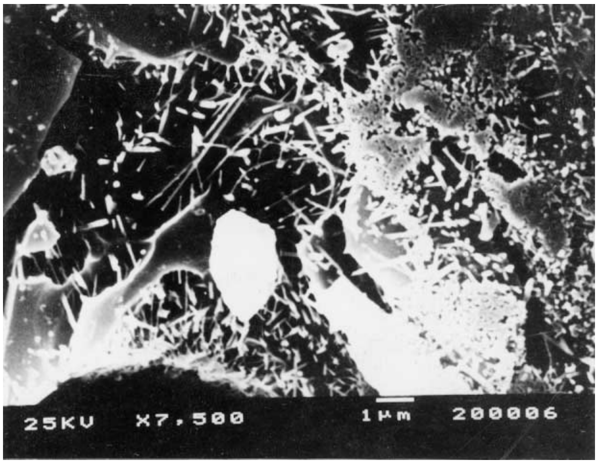

Fig. 5. SEM photomicrograph of the vitrified specimen fired at 1200°C for 20 min soaking time showing the presence of secondary mullite and quartz grains (×7500).

5. Conclusion

It was suitable to prepare porcelain tiles from a recipe containing local Egyptian raw materials up to 95%. It was concluded that the optimum molding pressure for the suggested composition was 45 MPa. Raising the molding pressure above this value did not enhance the vitrification properties. The optimum firing conditions are 1220 °C firing temperature and 20 min soaking time. These conditions are necessary to obtain the required low water absorption (0.1%). Raising the firing temperature to 1230 °C deteriorate the vitrification parameters due to bloating. A very fast rate of firing (80 °C/min) was suitable for the suggested composition. Specimens fired at the optimum conditions gave very high value of modulus of rupture compared to the value specified by the standard.

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