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Characterization of Porcelain Stoneware Tiles Based on Solid Ceramic Wastes

E. El-Fadaly

Environmental Studies Research Institute, El Sadat City University, El-Sadat City, El-Menofia, Egypt

Abstract: ***Porcelain stoneware tiles get a big role on the market. Considerable quantities of solid wastes are being generated from different industrial activities causing major environmental problems. An attempt to recycle these wastes in stoneware tile recipes was introduced to substitute the feldspar. Five formulations were studied on laboratory scale and on production simulation of tile making process, compared with standard one. Partially replaced of fired waste with feldspar improved; bulk density (2.35-2.42 g/cm^3), modulus of rupture reached to 50-70 MPa, resistance of deep abrasion (100-65mm^3) slight darkening of color (ΔE around 0.25-2) as well as acceptable coloring. The microstructure has a decisive influence on the mechanical and functional behavior of products. The fired ceramic waste resulting can be incorporated into porcelain stoneware tiles production as economic, technological and environmental correct solution.***

Keywords: stoneware tile, solid wastes, ceramics, porcelain, microstructure

1. Introduction

In the last decade, the growth rate of the global production of porcelain stoneware tiles increased more than other ceramic products; in fact, the excellent technical properties [1], together with the even more improved aesthetic appearance [2], gave porcelain stoneware a prominent role on the tile market [3]. Porcelain stoneware bodies consist mainly of a mixture of ball clay, feldspar and quartz- feldspathic sands, often containing also glass-ceramic frits and pigments. This kind of tiles is obtained by wet grinding, dry pressing, fast drying and fast single firing at maximum temperature around 1190-1230^{o}C. Finished products exhibit excellent technical properties, especially mechanical strength, resistance to deep abrasion, frost resistance as well as chemical and stain resistance [4-5]. Ceramic tile world production is 9515 million m^2, Africa 370 million m^2 and Egypt is 220 million m^2, in year 2010. While world saintaryware production is 275 million pieces and Egyptian production is about 30 million pieces/year [6]. Consumption of raw materials in ceramic tile production, in Egypt is about 5 million ton. The percent of expected solid wastes from these sectors is about 4%, 200,000 ton. So, Wastes produced from different industrial activates are becoming a big problem affected the economical conditions as well as environmental impact. Recent trends try to recycle these wastes and incorporate them in recipes of different ceramic products. These wastes compensate for the expected depletion of natural raw materials and minimize one of pollution problems.

The accumulation of granite and marble dust waste products of construction processing industries [7-9], as well as glassy frits, [10-11] blastfurance slag, fly ash, [12-16] and glass [17-18] , were already utilized in ceramic products. The waste based frits and soda lime float are used to produce glazed and unglazed porcelainized stoneware body improving water absorption and spot resistance [19]. Recycling of ceramic tile industry wastes as cyclone dust, filter dust and sludge of waste water treatment, in floor tiles recipes was recorded [20].

Fired ceramic wastes are a fully vitrified glazed product covered a glaze layer and contains mullite, glassy phase and residual quartz. Mullite is the main constituent of conventional ceramic products, characterized by its high strength and low coefficient of expansion. It has a significant role in the developed properties of traditional and advanced ceramics [21-23]. Glassy phases as well as glaze layer are considered as fluxing materials. Whereas residual quartz add to the content of quartz in the body formulation reducing the tendency to the pyroplastic deformation of the fired body [24-25]. The microstructure of the fired products is strictly dependent on the particular body composition and has a decisive influence on the mechanical and functional behavior of this class of products [1]. Incorporation of fired sanitary ware scrap in floor tile ceramic formulations was studied [26].

The aim of the present work is to incorporate the fired sanitary ware scrap as raw materials and a source of mullite; in porcelain tile bodies in an attempt to recycle the accumulated wastes thereby decreasing the environmental impact and reducing the production costs as well.

2. Material and Methods

Representative sample from crushed fired scrap was selected. Different mixes of ball clay, quartz, sodic feldspar 1mm, and fired scrap 1-3 mm, were formulated for the so called porcelain-stoneware tiles. Chemical and mineralogical constitutions of the raw materials were determined by XRF and XRD respectively, using Philips equipment.

Five formulations beside to the standard one, were suggested utilizing fired scrap in the proportion between 0-32 % partially substitution the feldspar content, the clay content form of 40 % ball clay and 6 % kaolin, as well as the silica in the form of quartz 7.5 % were kept constant.

All formulations were processed on the laboratory scale, simulating the industrial tile production process. The chemical composition of the different formulations is given in Table 2. The various proportions were accurately weighed, ground, and intimately wet mixed in a laboratory ball mill for 1-2 h, to pass 63μ sieve. Tile bodies were processed with the following dimensions 100*50*5 mm under a uniaxial and a

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pressure of press 350 kg/cm^2, dried and fired. The processed specimens were fired between 1050 and 1275°C. Physical properties in terms of firing shrinkage (ASTM C 326); water absorption, bulk density and apparent porosity were determined according to (ISO 10545-3). The main crystalline phases developed during firing were identified by XRD using a Philips PW 1700 spectrometer with copper Kα radiation and a Ni filter in the range of 2 θ between 4 and 60 °. The powders were pressed in the form of bars according to standard test specifications. Microstructure of the sintered bodies was observed by scanning electron microscope (Type X L30, Philips) with EDAX equipment. Specimens were polished, chemically etched using 20 % HF for 10 s, thoroughly washed, dried and gold sputtered. The modulus of rupture was measured using a bending test machine (model MOR /1-M/E) according to ISO 10545-4 1995. The degree of whiteness was tested by a tri-stimulus colorimeter (Model X 918 FL). Resistance to deep abrasion was determined according to ISO 10545-6 1995 using an abrasion tester (model AP/87).

3. Results and Discussion

The results of chemical analysis of the raw materials are shown in Table 1. Chemical analysis of kaolin and ball clay reveals their siliceous nature as evident from the content of SiO_2, 57 and 67%, respectively.

Table 1: Chemical analysis of raw materials as determined by (XRF)

Raw material	Clay	Kaolin	Soda-feldspar	Fired scrap	Silica sand
LOI	6.25	11.13	0.51	0.49	0.24
SiO2	67.01	56.82	66.83	69.48	99.24
Al2O3	20.39	27.27	19.07	23.70	0.12
Fe2O3	2.32	1.31	0.35	0.57	0.05
TiO2	1.28	2.16	0.35	1.17	0.04
CaO	0.39	0.28	0.58	0.35	0.07
MgO	0.33	0.09	0	0.04	0
K2O	1.82	0.12	0.23	2.10	0.1
Na2O	0.00	0.07	11.71	1.34	0.02
MnO2	0.01	0.02	0.03	0.32	0.02
P2O5	0.01	0.15	0.06	0.03	0
Cl -	0.03	0.04	0.05	0.03	0.02
SO3--	0.11	0.15	0.11	0	0.03
Total	99.97	99.81	99.88	99.62	99.95

Table 2: Chemical composition of formulated bodies

Raw material	M1	M2	M3	M4	M5	M6
SiO2	71.34	71.45	71.58	71.81	72.05	72.28
Al2O3	19.53	19.72	19.91	20.29	20.68	21.06
Fe2O3	1.25	1.26	1..27	1.28	1.30	1.32
TiO2	0.86	0.89	0.93	1.00	1.06	1.13
CaO	0.46	0.45	0.44	0.42	0.40	0.38
MgO	0.18	0.15	0.15	0.15	0.15	0.15
K2O	0.9	0.97	1.05	1.25	1.40	1.55
Na2O	5.45	5.10	4.66	3.93	3.00	2.15
RO	0.64	0.60	0.59	0.57	0.55	0.53
R2O	6.35	6.07	5.71	5.18	4.40	3.70

Table 3: Properties of fired tile bodies in lab kiln, 1200oC sucking time, 30 min

Property	M1	M2	M3	M4	M5	M6
Linear fired shrinkage, %.	8.5	8.11	7.32	7.56	7.53	7.35
Bulk density,gm/Cm^3.	2.35	2.39	2.39	2.40	2.42	2.40
Water absorption, %.	0	0.03	0.07	0.03	0.09	0.4
Modulus of rupture MPa.	48.8	49.5	62.4	52.5	70	60
Resist., deep abrasion, mm^3	100	85	77	72	69	65
l*	75	74.7	74.7	74.6	75.7	73.7
a*	3.00	3.4	2.8	2.8	3.0	2.6
b*	17.8	18.0	17.3	16.9	17.0	16.7
ΔE	-	0.34	0.73	1	1	1.8

Table 4-a: Properties of colored (blue) tile bodies fired in production kiln, 1210oC/ firing cycle 55min

Property	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**
Linear fired shrinkage,%.	9.6	9.3	9.5	10	9	8.6
Bulk density,gm/Cm^3.	2.45	2.45	2.45	2.46	2.46	2.45
Water absorption,%.	0.04	0.06	0.06	0.1	0.09	0.07
Apparent porosity,%.	0.098	0.15	0..15	0.25	0.22	0.17
l*	47.8	48.2	48.3	49.4	49.5	49.9
a*	-5.00	-5.10	-5.00	-5.10	-5.40	-5.50
b*	-19.4	-16.2	-17.6	-16.6	-14.4	-12.2
ΔE	-	0.63	1.01	2.27	4.67	6.87

Table 4-b: Properties of colored (black) tile bodies fired in production kiln, 1210^{o}C/ firing cycle 55min.

Property	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**
Linear fired shrinkage, %.	8.8	9.2	9.4	10	8.7	8.4
Bulk density,gm/Cm^3.	2.45	2.44	2.43	2.44	2.44	2.42
Water absorption,%.	0.07	0.1	0.08	0.09	0.13	0.33
Apparent porosity,%.	0.17	0.24	0.2	0..22	0.32	0.8
l*	29.8	34.5	35.0	35.9	37.7	39.0
a*	+1.8	+1.5	+1.7	+1.4	+1.0	+1.2
b*	+0.4	+0.6	+0.8	+0.4	+0.4	-1.2
ΔE	-	4.7	5.27	6.19	7.94	9.34

Table 4-c: Properties of colored (orange) tile bodies fired in production kiln, 1210^{o}C/ firing cycle 55min.

Property	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**
Linear fired shrinkage, %.	8.9	9.4	9.7	9.1	8.9	8.4
Bulk density,gm/Cm^3.	2.4	2.41	2.43	2.44	2.43	2.41
Water absorption,%.	0.4	0.3	0.21	0.1	0.3	0.60
Apparent porosity,%.	0.96	0.72	0.51	0.24	0.72	1.44
l*	66.77	65.70	67.07	67.11	67.42	67.72
a*	+15.4	+15.2	+15.2	+14.5	+14.0	+13.2
b*	+39.16	+38.3	+38.5	+37.0	+34.6	+33.2
ΔE	-	0.34	0.76	2.36	4.85	6.42

Table 4-d: Properties of colored (red) tile bodies fired in production kiln, 1210^{o}C/ firing cycle 55min

Property	M1	M2	M3	M4	M5	M6
Linear fired shrinkage,%.	8.9	9.2	9.4	8.8	8	7.8
Bulk density,gm/Cm^3.	2.38	2.39	2.41	2.41	2.40	2.38
Water absorption,%.	0.3	0.3	0.08	0.1	0.44	0.73
Apparent porosity,%.	0.71	0.72	0.19	0.24	1.05	1.73
l*	47.44	47.67	48.11	49.06	50.97	51.94
a*	+19.8	+19.7	+19.9	+20.6	+19.5	+18.6
b*	+17.4	+17.6	+17.5	+17.2	+16.6	+17.2
ΔE	-	0.29	0.68	1.81	3.64	4.66

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The percent of Al_2O_3 is relatively low about 20.27, 20.39 % and corresponding LOI, 11.13, 6.23% respectively. Also, they contain relatively high proportion of impurities in the form of coloring oxides Fe_2O_3 and TiO_2, 3.6 % and alkalis 1.82%. The albite content in the raw feldspar is about 98.9%. Fired sanitary ware scrap(FSS) , ceramic waste is highly siliceous; silica content is about 70% while the alumina is relatively low 24% and contains appreciable amounts of fluxing oxides in the form of alkalis. Results of XRD of these materials are mainly kaolinite in clay and kaolin; albitite with traces of quartz in albite, silica sand is mainly quartz. XRD of FSS is formed mainly of mullite and quartz relics, as shown in Fig.3. The chemical composition of all formulations lie in the same range with slight variation in the content of the total alkalis; Na_2O and K_2O**,** that varies with the content of scrap and albite in the batch. The results of water absorption in Fig 1 show that temperature of 1175^oC is the appropriate maturing temperature of all formulations. Also, the percent of water absorption increases with the content of fired solid wastes added. Addition of solid wastes up to 16 % lowers the water absorption about 1%. Results of bulk density and strength were also affected by the content of fired solid wastes. Properties of fired bodies at the selected firing temperature are tabulated in Table 3. The shrinkage increases with firing temperature for all compositions up to maturation then slightly reversed due to beginning of internal deformation, decreases with fired solid wastes additions. Bulk density values increase with temperature until reaching maturation state; where the scrap additions decrease the compressibility as well as too viscosity of SiO_2 –rich melts of the rejects. The bulk density of matured bodies increases with increasing fired waste addition. This may be attributed to increase of mullite with fired waste increase as well as decreasing of glassy phase; where glassy phase always has lower density than the corresponding mixture of crystalline phases.

The water absorption is observed in Fig 1 with firing temperatures 1125-1175 ^oC. It decreases with increasing temperature where the lower value at 8% fired waste addition and less than 1% at 1175^oC. While the lowest value at 1200 is zero with no addition of scrap; although WA% increases (0-0.4%) with scrap addition (0-32%) but it still within standard range according to ISO13006BIa (E≤0.5). This may be explained by decreasing of (RO+R_2O) % by increasing of scrap leading to less glassy phase as well as too viscosity of the SiO_2-rich melts which confirmed by its XRD.

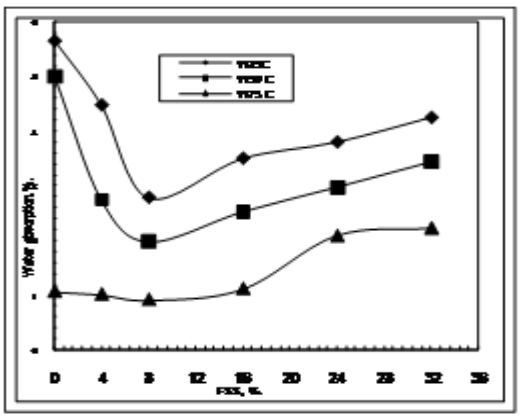


Figure 1: Relation between water absorption and percent of solid waste at different temperatures

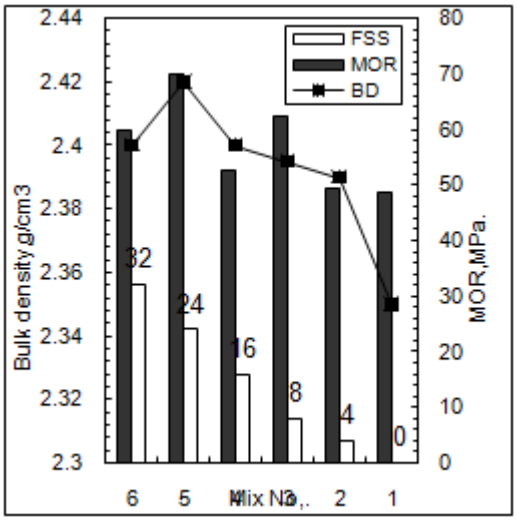


Figure 2: Relation between fried solid wastes (%), and MOR, MPa, and bulk density/cm^3, of matured bodies

Fig (2) illustrates the modulus or rapture of all the compositions at maturation temperatures. The strength of all bodies developed from 1150^oC increasing with FSS addition these may be attributed to crystalline nature of product which contains more crystalline phase. MOR of matured bodies improved reached to 50-70 MPa. The resistance of deep abrasion of matured bodies is related to MOR where average volume removed (mm^3) ranged from 100 to 65.

XRD of two samples (M1&M5) of formulations that are studied in this paper are shown in Fig (3-5). Quartz and mullite are observed to be present as major phases in all as well as traces of corundum in M5 or albite in M1. The quartz is caused by presence of fired scrap which is siliceous nature and un-dissolved quartz which results from raw materials in recipes. XRD analysis revealed the presence of fired waste which considered as its seed as well as transformation of kaolinite of clay. The alkaline and alkaline earth partially contributed to the formation of crystalline phase, beside its participation in the formation of liquid phase. SEM micrographs of the fired ceramic waste as raw material are shown in Fig. (6). It had a dense microstructure with low residual pore presence as shown in general view of microstructure(100µm).

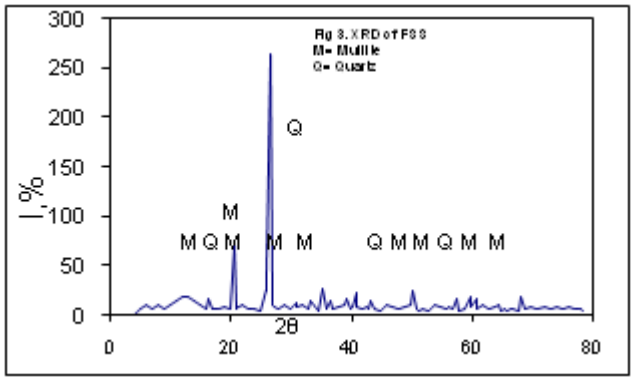


Figure 3: XRD of fired sanitary ware scrap.(FSS)

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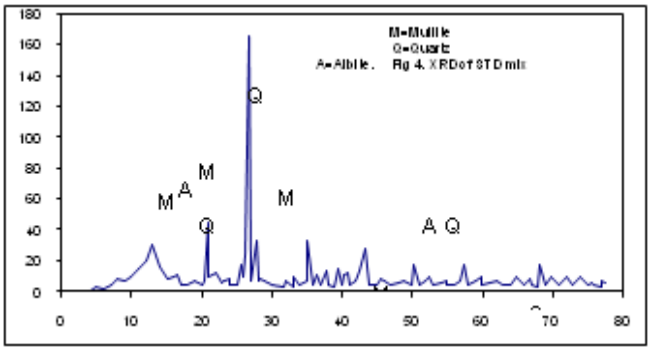


Figure 4: XRD of standard porcelain body (mix 1)

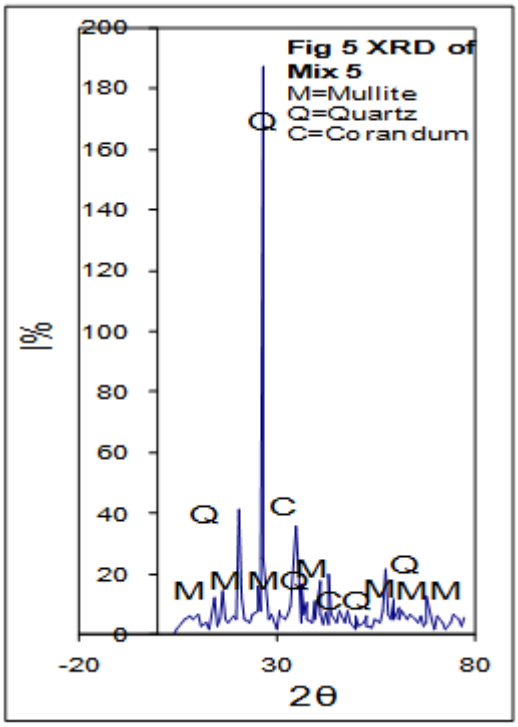


Figure 5: XRD of mix 5. (mixed feldspar and FSS)

Mullite crystals derived from the pure clay agglomerate relicts are cubical and are referred to as primary mullite since they form at the lowest temperature [27]. Secondary mullite result from the feldspar-rich melts forms later in the firing process as shown in Fig (7). Elongated needle-shaped mullite is shown in SEM (Fig 8) and EDAX (Fig 9) of it embedded in glassy phase. Fig (10) shows the EDAX of glassy phase area in fired waste which is Na, K aluminum silicate based on K and Na feldspar. Sanitary ware is classified as porcelain. The general micro structural features of commercial porcelains are coarse quartz grains surrounded by solution rims of almost pure silica glass embedded in a much finer matrix system. The matrix is predominantly glass of varying composition being aluminosilicate in clay relict regions which also contain fine, cubical, primary mullite crystals and alkali (Na, K) aluminosilicate in flux penetrated regions which also contain larger, elongated secondary mullite [28-30]. This microstructure is a direct result clearly suitability for using solid waste as source of mullite and raw material in porcelain tile.

Colorability of porcelain tile based on fired waste is studied. The same formulations M1-M6 were colored by using three pigments, blue, orange and black 2% from each one alone, as well as natural one, 8% red color. The previous procedure was followed except firing was completed in a production kiln at 1210^{o}C for a cycle 55 min. The chromaticity l*, a*, b* measured and ΔE are calculated as well as other properties of the fired bodies. Moreover, a fired solid waste causes a slight darkening of color (ΔE around 0.25-1 for 4-16 % addition; ΔE around 1-2% for 16-32 addition). This may attributed to increase of summation of coloring oxides as TiO_2 and Fe_2O_3 in mixes blended with scrap. This variation consists mainly in a decrease of lightness (l^*), while the difference in a^* and b^* parameters are limited to a general change more Table (4a-d).

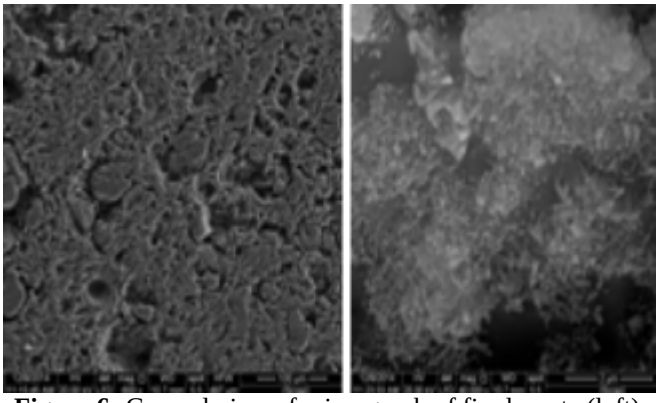


Figure 6: General view of micrograph of fired waste (left)
Figure 7: Fine grains mixed with cubical and small elongated needles of mullite (primary and secondary and tertiary mullite) embedded in glassy phase (right).

The studied products exhibit a phase composition made of a glassy phase prevailing over residual quartz and feldspars, new formed mullite in the standard mix and added mullite from fired waste in blended mix. The chemical composition of the vitreous phase changes from sample to sample; consequently, the viscosity of the liquid phase at higher temperatures and sintering kinetics vary upon the body formulation.

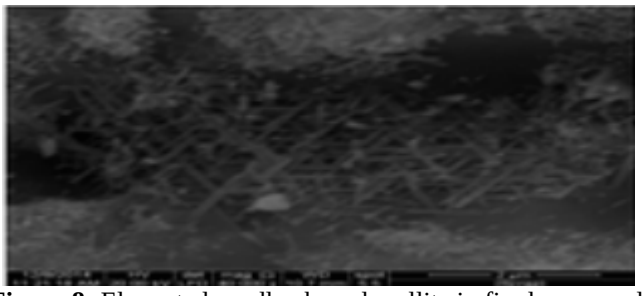


Figure 8: Elongated needle-shaped mullite in fired scrap and residual quartz(white crystals)

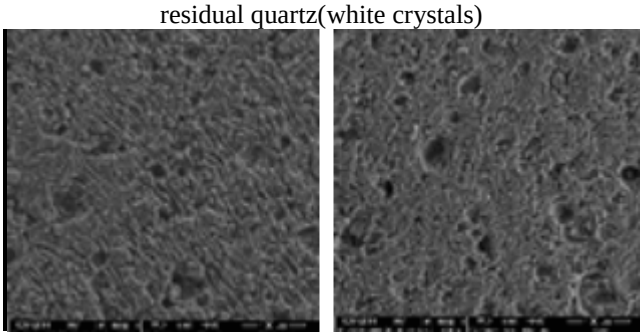
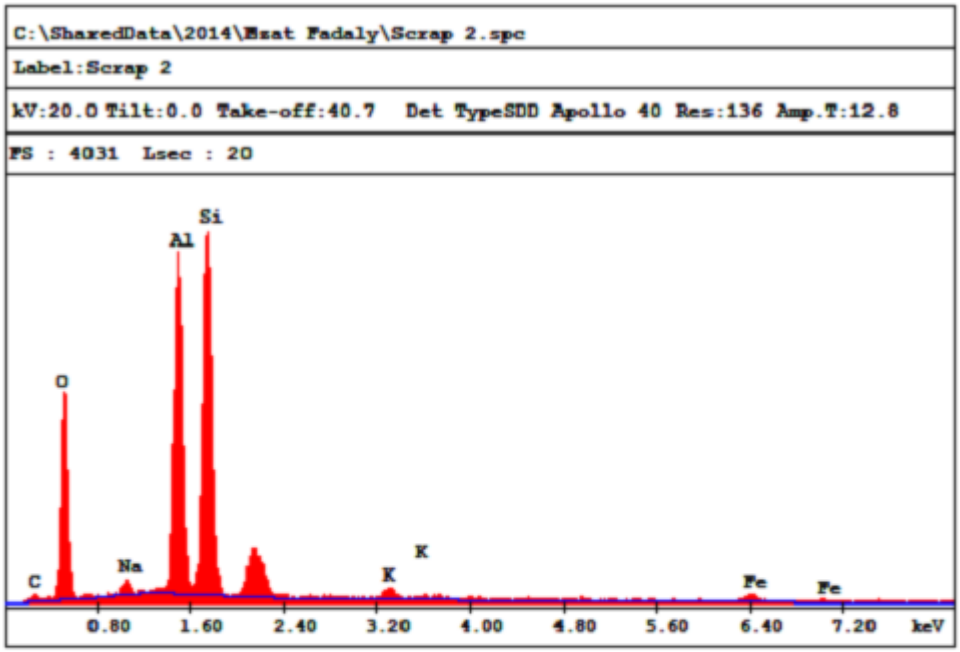


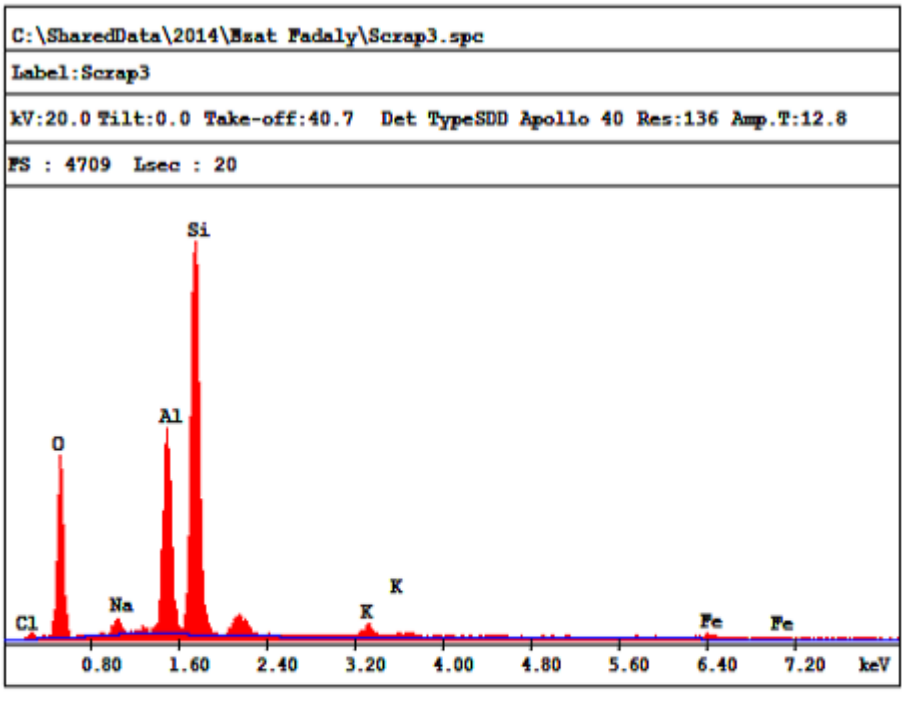
Figure 11: SEM, General view of standard, mix1 (left) and mix5 (right) fired at maturation.

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Element	Wt %	At %	K-Ratio	Z	A	F
C K	4.26	7.61	0.0052	1.0534	0.1159	1.0004
O K	33.73	45.24	0.1061	1.0357	0.3037	1.0007
NaK	1.44	1.34	0.0069	0.9693	0.4894	1.0068
AlK	24.11	19.18	0.1812	0.9644	0.7705	1.0111
SiK	32.58	24.90	0.2079	0.9925	0.6429	1.0002
K K	1.46	0.80	0.0120	0.9415	0.8705	1.0007
FeK	2.43	0.93	0.0212	0.8755	0.9969	1.0000
Total	100.00	100.00				

Figure 9: EDAX of Elongated needle-shaped mullite embedded in glassy phase (middle area in fig 8)(up)



Element	Wt %	At %	K-Ratio	Z	A	F
O K	35.40	49.14	0.1134	1.0355	0.3091	1.0007
NaK	1.73	1.67	0.0083	0.9690	0.4900	1.0069
AlK	17.74	14.60	0.1334	0.9642	0.7681	1.0156
SiK	41.55	32.85	0.2856	0.9923	0.6925	1.0003
K K	1.84	1.05	0.0149	0.9414	0.8603	1.0005
FeK	1.73	0.69	0.0150	0.8755	0.9947	1.0000
Total	100.00	100.00				

Figure 10: EDAX of black zone (glassy phase) in Fig 8 right up needle shaped (dawn).

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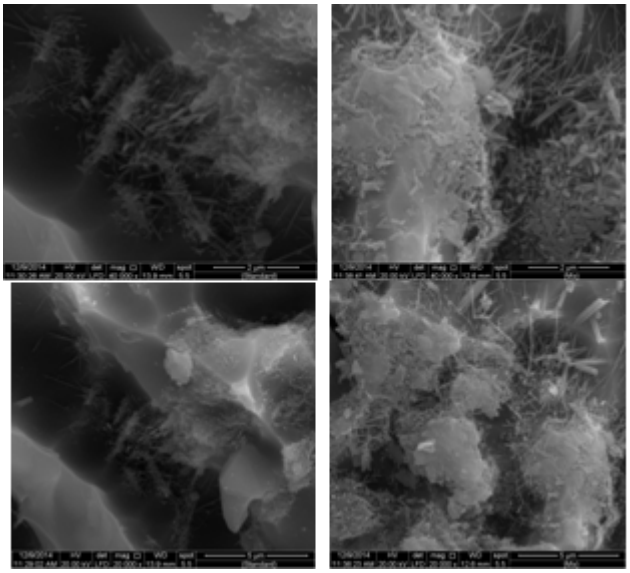


Figure 12: SEM of standard mix1 (left) and blended one mix5 (right) which explain crystalline nature of suggested mix compared with standard one

Porcelain stoneware tiles are characterized by very low water absorption, though a residual closed porosity is present in significant amounts as approved from general view of microstructure at 100µ (Fig 11)leading to a different compactness and microstructure. Products exhibit excellent mechanical properties: 50–70MPa of bending strength, with a clear dependence of these properties on porosity and phase composition. Mullite and crystalline net tend to increase the mechanical performances, through a predominant mechanism of matrix reinforcement, while quartz and glassy phase play an opposite role. This is approved as shown in Fig (12). So the strength of body containing scrap as source of crystalline phases is higher than the standard.

4. Conclusions

The fired scrap resulting from the sainataryware production can be incorporated into porcelain stoneware production tiles as economic, technological and environmental correct solution. It presents partially replaced with feldspar in uncolored porcelain tile recipes till 25% with slight darkening of color; while 0-8 % addition is better in case of coloring. In conclusion, the addition of fired sainataryware scrap to porcelain stoneware tiles reduces the sintering interval and helps to obtain better mechanical properties.

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Author Profile



Dr. Ezzat Ahmed Mohamed El-Fadaly, received both his B.Sc. in Chemistry (1991) and his M.Sc. (1996) in Physical and Inorganic Chemistry from the Faculty of Science, Zagazig University (Egypt). He earned his Ph.D. from Cairo University in 2005. Since then he has been working as a Lecturer for Applied Inorganic Chemistry and consultant for the environmental studies and ceramic industry in the Environmental Studies and Research Institute of El-Sadat City University, Minufiya (Egypt).

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