

## Simultaneous Reuse of Three Types of Wastes in the Preparation of Ecofriendly Ceramic Floor Tiles

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### Abstract

Enormous amounts of wastes are produced annually from ceramic industries during its different production stages, a state of affairs which is associated with several environmental problems on land filling. Most of these wastes have not been properly utilized yet, so that implementing their use in the production of ceramic tiles as an alternative raw material will help in preserving natural resources, reducing the cost of production and hence producing an ecofriendly ceramic floor tile at lower cost. This paper discusses an adequate way of managing these wastes instead of their being disposed. Three types of wastes generated in ceramic tile manufacturing were reused in the tile production process: Waste sludge collected from different sections of the production line, fine powder collected from cyclones prior to tiles pressing and the powder obtained from periodically grinding the rollers of the kiln. The collected wastes were added in different ratios to a standard floor tile mix and factorial 2<sup>3</sup> design technique was used to investigate the effect of adding these wastes in different ratios on the properties of unfired and fired bodies. A suggested recipe was selected and three specimens were shaped in standard tile form, dried and fired at 1160°C for 15 minutes. Tests performed on these tiles reveal that they abide by standard requirements for ceramic tiles of water absorption lying in the range 3 – 6%.

**Keywords:** Waste Management, Recycling, Floor Tiles, Ceramic Industry, Factorial design

### INTRODUCTION

As a result of industrial and economic growth, recent years have witnessed rising social concern about the problem of waste management in general, and industrial waste and waste from the construction industry in particular. This problem is becoming ever more acute due to the growing quantity of industrial, construction and demolition waste generated. The need to manage these wastes has become one of the most pressing issues of our times, requiring specific actions aimed at preventing waste generation such as promotion of resource recovery systems (reuse, recycling and waste-to-energy systems) as a means of exploiting the resources contained within waste, which would otherwise be lost, thus reducing environmental impact [1].

A flagrant example of such state of affairs is the tile ceramic industry where diverse wastes are generated throughout the

manufacturing process. These wastes can be sometimes recycled to the raw mix equipment although this is rather the exception than the rule. Wastes such as ceramic sludge, partly or fully fired defective articles, powder generated from grinding of kiln rollers represent typical waste materials that are usually disposed by land filling. Besides economic losses, this represents a threat to environment particularly in case of sludge where the liquid diffuses across the soil affecting natural aquifers [2].

In this respect, Manfredini et al [3] has suggested to minimize the pollution due to ceramic sludge by rationalizing the addition of waste waters and sludge in tile production processes. The analytical and rheological results, obtained on the body slips used for "white gres" tile production in waste purified waters demonstrate that the addition of dried sludge's up to 5 % by weight makes the slip completely compatible with industrial requirements.

The possibility of the recycling of some solid wastes of ceramic industry in the preparation of ceramic tiles at the same factory was studied by El-Fadaly et al [4]. Cyclone dust, sludge, and filter dust were added to the base body in proportions ranging from 2.5 to 10 % weight content. The mixed powders were pressed at 225bar, consequently dried and fired in an industrial kiln at 1190°C in a 35 minutes cycle. The results showed that addition of cyclone dust improved the physico-mechanical properties of the base body, while sludge additions deteriorated these properties whereas filter dust had nearly no effect on properties. The phase analysis via XRD and SEM demonstrated that the proposed firing cycle is insufficient for the complete melting of soda feldspar and the formation of any mullite crystals.

Recently, García-Ten et al [5] investigated the possibility of substituting part of the main body mix of floor ceramic tiles by some of the wastes such as green scrap, fired scrap, dust from kiln cleaning filters, polishing sludge, glaze sludge and frit residues to produce zero waste ceramic tiles. Characterization of the composition indicates that it displays appropriate behavior in the different production process stages and exhibits the required properties for use as urban flooring.

The present paper aims at investigating the possibility of substituting part of the main body mix of floor ceramic tiles by some wastes produced during manufacturing. The chosen wastes are: Cyclone dust waste, roller kiln grinding waste and ceramic sludge waste obtained from the water treatment unit.

## MATERIALS AND METHODS

### Raw Materials Characteristics

Four types of material were used, all of them kindly supplied by Ceramica Venus factory, 10<sup>th</sup> of Ramadan city:

- Ceramic floor tiles basic mixture, the composition of which is displayed in Table (1).
- Sludge waste obtained from the water treatment unit of the factory
- Cyclones dust waste.
- Roller kiln grinding waste.

**Table 1:** Raw mix tiles body composition

Percent	Kaolin Clay	Ball Clay	Bentonite	Feldspars	Sand	Green Tiles Scrap	Talc
Floor Tile Mix	1	41	2	40.5	5.5	7	3

The mineralogical composition of the four materials used was assessed using X-ray diffraction (Bruker D8 advanced computerized X-ray diffractometer apparatus with monochromatized Cu K $\alpha$  radiation, operated at 40 kV and 40 mA).

On the other hand, chemical composition was determined using X-ray fluorescence technique type. The used machine was Axios, Panalytical 2005, wavelength dispersive (WD-XRF) sequential spectrometer.

Thermal analyses (TGA – DTG – DTA) were performed in air on both materials using Netzsch STA 409 C/CD apparatus at a heating rate of 10°C/min.

The grain size distribution was determined according to the standard sieving procedure described by ASTM D 422 [6].

Finally, the powder densities of basic mixture of floor tiles (raw mix) and the selected wastes were measured using the standard Pycnometer method (density flask). This method is a very precise procedure for determining the density of powders, granules and dispersions that have poor flowability characteristic [7].

### Preparation of Samples

The samples were prepared by grinding the sludge waste using a laboratory ball mill fitted with alumina balls. Fine waste powder of cyclone dust and roller kiln waste was then added in predetermined levels. This mix was used to replace part of the basic mixture for floor tiles. A 2<sup>3</sup> factorial design was used to determine the proportions of wastes added. Eight mixtures were thus prepared besides three at the central of design were prepared [8]. These mixtures were mixed on dry basis for 10 minutes for each sample after which 5–7 % by weight water was added. The plasticity of the different blends was determined using the Pfefferkorn method [9].

Rectangular tile specimens of approximate dimensions (111 ×

57 × 7) mm<sup>3</sup> were molded by using an automatic laboratory hydraulic press, under uniaxial pressure of 35MPa. The samples were then dried in a muffle dryer for 8 hours at a temperature of 145°C.

Samples were subsequently fired in a laboratory muffle furnace following a programmed schedule that takes into account the evolution water from the dehydroxylation of kaolinite by fixing the temperature at 750°C for 30 min. The maximum temperature attained was 1160°C with a soaking time of 15 min to simulate fast firing conditions.

The following tests were performed to determine the characteristics of fired samples: Percent linear firing shrinkage [10], percent water absorption and apparent porosity [11] and breaking strength and modulus of rupture [12]. SEM was also used to provide micrographs of some chosen sections. The used SEM apparatus was of type JEOL–JSM 6510 apparatus at zoom magnification power = 2000 $\times$ .

## RESULTS AND DISCUSSION

### Analyses of Raw Materials

#### Chemical composition

XRF results for all raw materials are shown in Table (2).

As can be seen from that table, roller kiln waste contains a relatively high proportion of alumina. This is expected in view of the refractory nature of the rollers required to withstand temperatures exceeding 1150°C.

#### Mineralogical analysis of raw materials

The mineralogical analysis of the roller grind waste has been previously investigated by Ibrahim [13] who found that the waste is solely constituted from alumina and mullite. As for the floor tile raw mix, its XRD pattern has been studied by Amin et al [14]. They found that the mix is mainly composed of the following phases: Quartz, albite, calcite and kaolinite. On the other hand, the phases constituting floor dust main are quartz and albite [15, 16]. As investigated by Amin et al [17], waste sludge is composed of a mixture of all ingredients constituting the raw mixes for wall and floor tiles namely, kaolinite (Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>.2H<sub>2</sub>O), quartz, microcline (K<sub>2</sub>O.Al<sub>2</sub>O<sub>3</sub>.6SiO<sub>2</sub>), calcite (CaCO<sub>3</sub>) and albite (Na<sub>2</sub>O.Al<sub>2</sub>O<sub>3</sub>.6SiO<sub>2</sub>).

**Table 2:** Chemical analysis of raw materials (weight %)

Main Constituents	Sludge Waste	Floor Dust Waste	Roller Kiln Waste	Floor Mix
SiO <sub>2</sub>	67.38	63.13	23.55	61.21
TiO <sub>2</sub>	0.65	0.98	0.33	0.83
AlO <sub>3</sub>	15.35	21.82	63.86	20.19
Fe <sub>2</sub> O <sub>3</sub> <sup>tot</sup>	4.25	3.66	1.16	4.78

Main Constituents	Sludge Waste	Floor Dust Waste	Roller Kiln Waste	Floor Mix
MgO	0.79	1.37	1.96	0.99
CaO	2.57	1.4	3.51	1.21
Na <sub>2</sub> O	2.75	2.96	0.8	2.72
K <sub>2</sub> O	1.43	1.52	0.48	1.21
ZrO <sub>2</sub>	0	0.48	3.45	0
ZnO	0	0.5	0.22	0
CuO	0	0.1	0.22	0
P <sub>2</sub> O <sub>5</sub>	0.19	0.2	0.17	0.21
SO <sub>3</sub>	0.14	0.4	0	0.28
Cl	0.03	0	0	0.10
Minor Oxides	0.276	1.1	0.18	0.286
LOI	4.18	5.26		5.99
TOTAL	99.986	99.62	99.89	100.006

Table 2 continued.

#### Screen analysis of raw materials

Following screen analyses performed on the dry raw materials, the median ( $D_{50}$ ) values were obtained, as indicated in Table (3).

Table 3: Median particle size of the raw materials

Powder	Floor mix	Floor dust	Roller kiln waste	Sludge
$D_{50}\mu\text{m}$	425	13.72	225	75

This table shows that cyclone floor dust is by far the finest fraction of the raw materials used whereas the floor mix is the coarsest.

#### Powder density

The powder densities of sludge waste, roller kiln waste, floor dust waste, and floor tiles mix were found to equal 2.35, 2.92, 2.37 and 2.49 g.cm<sup>-3</sup>, respectively. The elevated value of density of roller kiln waste is due to its high alumina content.

#### Properties of Unfired Floor Tiles Samples

##### Composition of chosen samples

Following the 2<sup>3</sup> factorial design procedure, the following mixes were selected including as previously mentioned three identical mixes at center of design.

Table 4: Mix compositions

% Sludge	% Dust	% Roller waste	Floor mix
0	0	0	100
30	0	0	70
0	5	0	95
30	5	0	65
0	0	2	98
30	0	2	68
0	5	2	93
30	5	2	63
15	2.5	1	81.5
15	2.5	1	81.5
15	2.5	1	81.5

#### Effect of wastes addition on mix plasticity

The simultaneous addition of the three wastes according to Table (4) affected the Pfefferkorn plasticity number as evidenced from the results illustrated in Table (5).

The extent to which each individual addition affects plasticity was obtained by determining the individual correlation coefficients between plasticity and the percent of each waste. The result is presented in Table (6).

It appears from that table that the effect of any of the three wastes on plasticity is rather low to moderate. The maximum negative effect is observed for roller kiln dust which affects the plasticity negatively while the two other wastes have positive effects. This is expected because of the non-plastic nature of that dust.

#### Effect of wastes addition on drying shrinkage

Upon simultaneously adding the three wastes to the basic floor mixture, it was found that the drying shrinkage did not take place uniformly in the three dimensions. That is why it was thought preferable to substitute the volume shrinkage (VDS) for the linear one. The results obtained are summarized in Table (5) together with the corresponding correlation table (Table 6).

It appears from the results that the effect of any of the additions is generally low to moderate with floor dust positively affecting the values of shrinkage while sludge hardly affecting it at all. These results are more or less compatible with those of plasticity as an increased plasticity is usually accompanied with more shrinkage.

**Table 5:** Effect of wastes addition on unfired mixes

% Sludge	% Dust	% Roller waste	% Floor mix	Plasticity No.	% Drying volume shrinkage	Green MOR MPa
0	0	0	100	17.9	0.788	0.2351
30	0	0	70	18.5	0.614	0.16205
0	5	0	95	19.2	1.176	0.41023
30	5	0	65	21.5	0.856	0.12403
0	0	2	98	19	0.148	0.20574
30	0	2	68	18	0.614	0.12709
0	5	2	93	17	0.440	0.25313
30	5	2	63	18.5	1.067	0.10114
15	2.5	1	81.5	17.5	0.356	0.2255
15	2.5	1	81.5	17.5	0.356	0.2255
15	2.5	1	81.5	17.5	0.365	0.2255

**Effect of wastes addition on green strength**

The green strength of tiles is not a standard requirement. However, a high MOR will ensure less broken green tiles on conveying. The effect of wastes additions on MOR are also illustrated in Tables (5, 6).

As can be seen from the correlation table, sludge addition is the most influential factor on green strength as it contributes negatively to diminishing this strength. The same effect is observed on adding roller dust but to a lesser extent. This is presumably due to the presence of large amounts of non-plastic feldspar and quartz in sludge as well as the non-plastic nature of roller kiln dust. A slight positive inference is observed for fine dust because of its extreme fineness.

**Table 6:** Correlation coefficients for unfired mixes

	Sludge	Dust	Roller
<b>Plasticity</b>	0.306	0.252	-0.414
<b>% VDS</b>	0.150	0.425	-0.350
<b>MOR</b>	-0.779	0.209	-0.323

**Properties of Fired Floor Tiles Samples**

Because of the importance of assessing the effect of wastes addition on final tiles properties, it was necessary to deduce expressions correlating the fired properties to the mix composition. The corresponding equations following a two-level first order model for three variables take the following general form:

$$y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{23}X_2X_3 + a_{31}X_3X_1 + a_{12}a_{31}X_1X_2X_3 \quad (1)$$

Where,  $X_1$ ,  $X_2$  and  $X_3$  are dimensionless coded variables defined in general as follows:

$$X_i = \frac{Z_i - Z_0}{\Delta Z} \quad (2)$$

Where  $Z_i$  is the actual value of the variable,  $Z_0$  the value at center of design:

$$Z_0 = \frac{Z_{max} + Z_{min}}{2} \quad (3)$$

And  $\Delta Z$  defined as:

$$\Delta Z = \frac{Z_{max} - Z_{min}}{2} \quad (4)$$

This way, the minimum value of the variable will be coded as -1 whereas the maximum value will be coded as +1.

Table (7) shows the selected levels of addition of each waste together with its corresponding coded value.

As the fired properties of samples were determined, the results were obtained that are displayed in Table (8).

**Table 7:** Levels of waste addition

% Sludge		% Dust		% Roller waste	
Value S	Coded $X_1$	Value D	Coded $X_2$	Value R	Coded $X_3$
0	-1	0	-1	0	-1
15	0	2.5	0	1	0
30	+1	5	+1	2	+1

**Table 8:** Properties of fired samples

% Sludge	% Dust	% Roller waste	Floor mix	% LFS	% WA	Breaking strength N	MOR MPa
<b>0</b>	0	0	100	5.263	1.652	917.8	32.584
<b>30</b>	0	0	70	4.211	5.433	646.1	20.066
<b>0</b>	5	0	95	5.965	2.024	1007.5	39.955
<b>30</b>	5	0	65	4.912	3.163	1143.9	34.523

% Sludge	% Dust	% Roller waste	Floor mix	% LFS	% WA	Breaking strength N	MOR MPa
0	0	2	98	5.351	2.223	1044.7	37.089
30	0	2	68	3.772	7.638	592.1	18.657
0	5	2	93	5.263	2.185	1151.8	45.681
30	5	2	63	4.561	5.448	699.2	23.719
15	2.5	1	81.5	5.877	3.508	877.5	29.766
15	2.5	1	81.5	5.351	2.631	978.6	32.703
15	2.5	1	81.5	5.439	17.5	858.5	27.352

Table 8 continued

**Effect of wastes addition on linear firing shrinkage (LFS)**

As opposed to drying shrinkage, the linear firing shrinkage was found to be isotropic to a great extent. On simultaneously adding different wastes according to the preset scheme, a relatively high shrinkage was observed, particularly for samples containing high amounts of floor mix. The reason is associated with the elevated percentage of feldspar usually present in floor tiles that can reach 50%. The presence of a high amount of feldspar promotes formation of a liquid phase which in turns favors a high firing shrinkage. On the other hand, the effect of the two other additions is modest compared to that of sludge (Table 9). Actually, it was not possible to find a simple first order model to account for the variability of LFS as function of the three variable percentages of wastes. Since the LSF is not among the main variables specified by standards, this point was not pursued.

**Effect of wastes addition on percent water absorption (WA)**

The results of percent water absorption obtained for the different mixes including three replicate runs at center of design are displayed in Table (7). Except for one mix these mixes can be considered to correspond to the tiles category with  $3 < \% WA < 6\%$ .

Table (9) depicts the correlation table between percent water absorption and each of the three wastes levels. Here also, the percent sludge plays the most important role in assessing water absorption while the role of any of the two other additions is relatively modest.

It is interesting to note that the correlations coefficients of WA to different additions go in opposite direction to those of LSF, an expected outcome as the presence of elevated levels of liquid phase will tend to decrease water absorption. This is emphasized in Fig (1) where a decreasing trend was obtained on plotting these two variables.

Water absorption being a decisive factor in assessing the classification of floor tiles, a correlation equation was obtained following the method described by Lazic´ [8]. The three results at center of design allow for the elimination of statistically insignificant terms. The coded equation obtained was:

$$\%WA = 3.7212 + 1.7X_1 + 0.6527X_3 - 0.6X_1.X_2 \quad (5)$$

Which, when transformed to actual variables took the form:

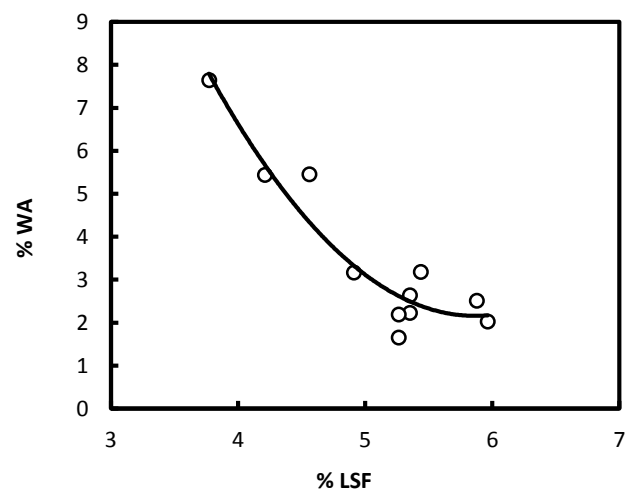
$$\%WA = 0.7685 + 0.1533S + 0.24D + 0.6527R - 0.016S.D \quad (6)$$

The *F* – test was applied to validate the above correlation. The calculated value of *F* = 5.01 while the critical value at significance level 0.05 was 19.24, confirming the validity of that correlation.

Next, the method of steepest descent was applied to select the mix that would yield the lowest water absorption [18]. In this method, selected steps of variation are chosen, according to certain rules, for each independent variable. The dependent variable (in the present case Water Absorption) is then calculated each time starting from the center of design until a minimum value is obtained. Calculations are shown in Table (10) that indicates that a minimum value of water absorption was obtained on using 9% sludge, 2.5% dust, 0.93% roller kiln dust and the balance floor mix. When such mixture was prepared and water absorption determined, a value of 3.1% was obtained, a figure fairly close to the calculated value of 2.74%.

**Table 9:** Correlation coefficients for fired tiles properties

(a)	Sludge	Dust	Roller
% LFS	-0.729	0.350	-0.223
%WA	0.818	-0.248	0.314
B.S.	-0.611	0.468	-0.133
MOR	-0.785	0.477	-0.02



**Figure 1:** Correlating WA to LSF

**Table 10:** Optimization of WA

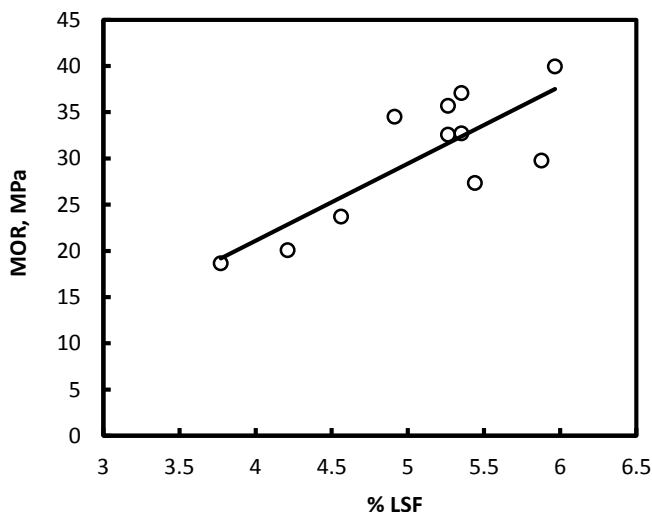
	S	D	R	% WA
	6	2.5	0.901	2.998
<b>Minimum</b>	<b>9</b>	<b>2.5</b>	<b>0.934</b>	<b>2.738</b>
	12	2.5	0.967	3.359
<b>Center</b>	15	2.5	1	3.721
	18	2.5	1.033	4.082

**Effect of wastes addition on mechanical strength**

As per standard specifications, two requirements have to be met for the mechanical properties of floor tiles: the breaking “strength” (BS N) and the bending strength (or Modulus of Rupture MOR MPa).

According to ISO13006 [19], the breaking strength for floor tiles of thickness less than 7.5 mm and of water absorption < 6% should exceed 600 N whereas the minimum MOR = 22 MPa. For a percent water absorption < 3%, the corresponding figures are 700 N and 30 MPa respectively.

Experimental data are displayed in Table (8) whereas the correlation coefficients of both parameters are shown in Table (9). Here also, there is a clear direct dependence of strength on firing shrinkage and an inverse correlation with water absorption. Fig (2) showing a plot between MOR and LSF accentuates these findings.



**Figure 2:** Correlating MOR to LSF

On the other hand, the regression equation obtained correlating Breaking Strength and MOR to the percent additions of wastes, took the following form, after transforming the coded equation to actual variables:

$$BS = 804 - 2.254 S + 40.1 D + 96.24 R - 6.416 S.R \quad (7)$$

$$MOR = 34.371 - 0.496S + 1.774D \quad (8)$$

The values of Breaking Strengths reported in Table (9) exceed

the standard figure of 600 N except for one mix composition. On the other hand, the minimum prerequisite of 22 N for MOR was obtained in all mixes.

The composition corresponding to minimum water absorption in Table (10) (9% sludge, 2.5% dust and about 1% roller kiln dust) was investigated for mechanical strength. The average values of strength for three specimens were as follows, compared to the corresponding values calculated by equations (7) and (8).

**Table 11:** Experimental and calculated values of strength for optimum composition

Breaking Strength N		Modulus of Rupture MPa	
Experimental	Calculated	Experimental	Calculated
866	922	34.3	34.4

These values exceed the minimum requisites required by standards for floor tiles of thickness < 7.5 mm.

**Industrial Assessment of Results**

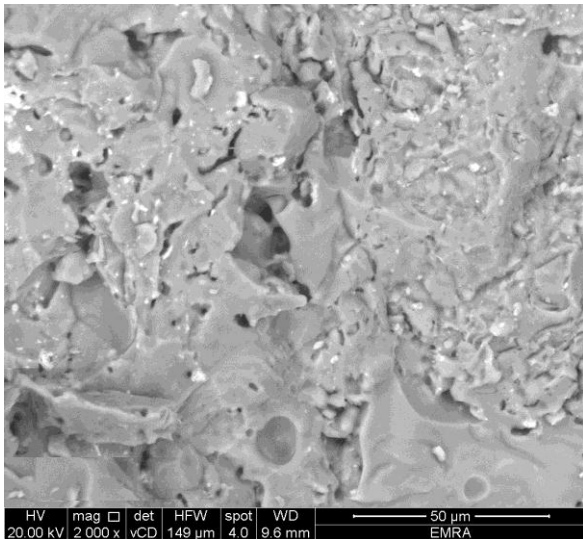
In order to assess the viability of the results obtained, three floor tiles specimens with commercial dimensions of (250 × 250 × 7) mm<sup>3</sup> were prepared using the former optimum recipe of 9% sludge, 2.5% dust, 1% roller kiln dust and the balance floor mix. These were fired at 1170°C in a roller kiln in a 50 minutes firing run along with tiles produced by the factory. The results obtained are shown in Table (12). Although these values abide by ISO13006 [19], they are generally poorer than those obtained on lab scale (Table 11), a consequence of the smaller dimensions of the later compared to the former [20].

**Table 12:** Results on commercial scale tiles

% Water Absorption		Breaking Strength N		MOR MPa	
Value	Standard	Value	Standard	Value	Standard
3.1	3 – 6	710	> 600	24.3	> 22

**SEM Results for Fired Samples**

In order to assess the previously obtained results, a specimen of one of the three kiln fired tiles was examined by SEM at magnification 2000×. The micrograph shown in Fig (3) reveals extensive formation of liquid phase, a result compatible with the low water absorption observed.



**Figure 3:** Scan Electron Microscope at (2000×) magnification of the selected specimen

## CONCLUSIONS

Three wastes from the ceramic tiles industry were added to standard floor mix in predetermined levels according to a factorial design  $2^3$  scheme. These wastes are ceramic sludge, cyclone dust and the fine powder obtained on grinding kiln rollers. The samples were formed by dust pressing under a uniaxial pressure of 35 MPa, dried for 8 hours at 145°C then subsequently fired for 15 minutes at 1160°C to simulate industrial conditions.

Green and fired properties were determined and the steepest descent technique used to choose a mix of minimum water absorption. Mechanical properties were measured (Breaking Strength and MOR) for the selected recipe and the results fairly matching to those obtained from the statistical models. In all cases Standards for strength were satisfied.

Three tiles of commercial dimensions ( $250 \times 250 \times 7 \text{ mm}^3$ ) were prepared and fired in an industrial roller kiln at 1170°C. Although the values of strength were lower than those obtained for the smaller laboratory specimens they still abided by standard values.

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