

Characterization and Reuse of Kiln Rollers Waste in the Manufacture of Ceramic Floor Tiles

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Abstract: Roller kilns used in the production of ceramic tiles are routinely ground to remove traces of contamination. The ground powder is usually discarded as useless waste. In a previous paper [1] the possibility of reusing this powder in refractory making was investigated. The present paper deals with the possibility of adding this powder without any treatment to standard wall tiles composition. XRD and XRF were used to identify the mineralogical and chemical composition of the powder. Powder compacts were prepared by uniaxial pressing at pressures ranging from 30 to 50 MPa. Firing was performed at temperatures ranging from 1200 to 1350°C for soaking periods ranging from 2 to 6 hours. It was proved that among the investigated parameters, firing temperature plays the leading role in assessing sintering characteristics, followed by soaking time. Forming pressure hardly affected sinterability. SEM micrographs were performed under different firing conditions to follow up the morphology of phases appearing. It was proved that adding up to 10% powder to ceramic floor tiles standard mix did not alter its final properties.

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1. Introduction:

In the past four decades roller kilns have become the main firing device in the manufacture of ceramic tiles as well as several other products such as flat dinnerware, small insulating refractories and other ware. They are characterized by their lower cost compared to the conventional tunnel kiln, the possibility of their use in fast firing processes and the uniform temperature distribution across its cross section owing to the reduced area of this section [2]. One of the environmental problems associated with the operation of such kilns is that its rollers need continuous surface grinding owing to the deposition of volatile salts particularly in the cooling zone of the kiln. In tile producing factories, the fine product obtained is produced at a rate of about 0.2 ton per 10⁴ m² tiles produced [3]. This product represents a health hazard since it is usually stored outdoors in piles. The continuous inhalation of the fine dust generated predisposes to silicosis [4].

In a previous paper, one of the authors of the present paper investigated the chemical and mineralogical analysis of this powder and found it to contain about 74% alumina with a balance of silica and alkali earth oxides. X-ray diffraction analysis showed the powder to consist of corundum, mullite, cordierite and spinel [1]. He found out that such powder could potentially be used in the refractory industry.

In the present paper, the sintering and the mechanical properties of the pressed powder are

investigated in view of using it as an additive in the ceramic tiles industry.

2. 2. Experimental

2.1 Raw material:

The raw material used was the product of grinding of ceramic rollers used in kilns for the production of tiles from PRIMA factory at Sadat City (60 km North of Cairo). Its particle size distribution has been previously determined by Ibrahim [1]. The particle size ranged from 40 to 150 µm with a specific surface area of 350 cm²/g. He also determined the true density of the powder and found it to be 2.94 g/cm³.

2.2 Sample preparation:

The as – received powder was dry pressed in a uniaxial hydraulic press under pressures ranging from 30 to 50 MPa in 1" diameter molds (2" molds were used for crushing strength tests and special tile shapes for MOR: 10x40x150 mm). The dry pressed samples were then fired for soaking times ranging from 0 to 6 hours in a muffle kiln at temperatures ranging from 1200 to 1350°C with 50° interval. Four specimens were tested each time and the average value of results calculated each time.

2.3 Tests performed:

The following tests were undergone on the fired specimens:

- The true and bulk density, water absorption and apparent porosity were determined using the hot test piece boiling water method [5].
- Cold crushing strength was tested by subjecting fired cylindrical samples of diameter = length = 2" to compressive loading [6]. The modulus of rupture was determined using the three point bending test [7].
- The Rockwell hardness of fired samples was determined according to ISO 674 – 1988 [8].
- X-Ray diffraction was used to assess the phases formed using Philips type PW 1373 X-Ray diffractometer.
- Scan electron Microscopy was used to follow the microstructure of fired samples. To this aim, a SEM apparatus (SEM; Model JSM-5410) was used.

3. Results and Discussion

3.1 Sintering characteristics:

Fig. 1 shows the effect of applied pressure on the porosity and the bulk density of green pellets. On raising the forming pressure from 30 to 50 MPa, the bulk density increase from about 1.79 g/cm³ to 1.89 g/cm³ corresponding to a reduction of porosity from 0.395 to 0.36.

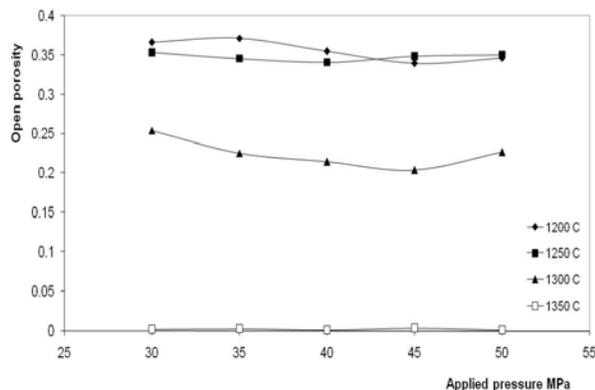


Fig.1: Effect of applied pressure and firing temperature on the minimum open porosity

As firing was performed for soaking periods ranging from 2 to 6 hours, the bulk density increased with firing temperature. Fig.2 shows the variation of bulk density with firing temperature and forming pressure for a soaking time of 2 h. As can be seen from the curve the forming pressure played little role in assessing the values of bulk density reached. This is the reason why it was thought sufficient to fit one single curve for all forming pressures involved. Increasing the firing temperature from 1200 to 1350°C had for effect to increase the bulk density from about 1.8 g/cm³ to 2.8 g/cm³. Increasing the soaking time to 6 hours did little to raise these values.

This means that the main factor controlling the variation in bulk density is the firing temperature. This was assessed by using the DATA ANALYSIS module (on EXCEL) to establish the following correlation table.

Table 1: Correlation table for fired density

	Pressure	Temp.	Time	Density
Pressure	1			
Temp.	0	1		
Time	0	0	1	
Density	0.029587	0.891457	0.03927	1

This table clearly indicates that the effect of either forming pressure or soaking time on the fired density is insignificant.

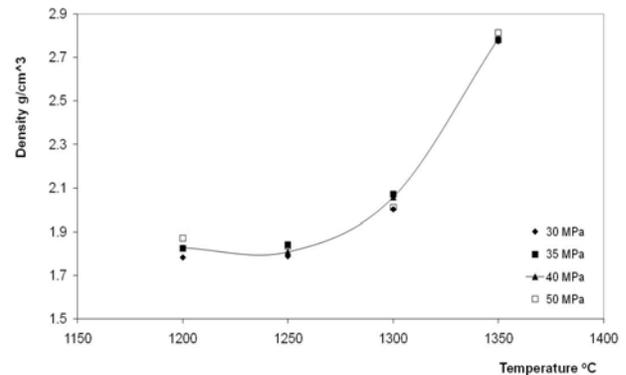


Fig.2: Effect of forming pressure and firing temperature on the bulk density of samples fired for 2 h

The maximum values of bulk density achieved at each firing temperature are plotted in Fig.3 which clearly shows that forming pressure did not affect such values and that below 1300°C there was no sensible densification. On firing at 1350°C there was an appreciable increase in density reaching about 96% of the theoretical density.

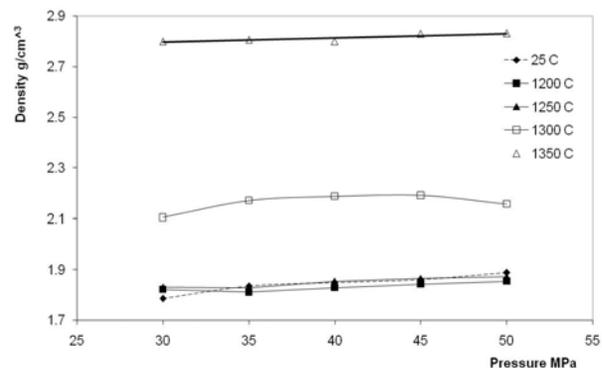


Fig.3: Effect of applied pressure and firing temperature on the maximum density

Fig.4, on the other hand, shows the variation in open, closed and total porosity of samples fired for 3 hours. Actually, all soaking times gave practically the same result. Here also, one curve was fitted through data collected at different forming pressures emphasizing the fact that forming pressure had a negligible effect on variation in porosity. These curves show that open porosity is almost zero on firing at 1350°C. At that temperature the pores are exclusively of the closed type, their level dropping to about 5%.

In line with the decreased porosity, the percent water absorption decreased with firing temperature and was practically unaffected by variations in forming pressure or soaking time. Fig.5 shows the variation of percent water absorption with firing temperature for 3 hours soaking. The values of percent water absorption dropped from about 19% on firing at 1200°C to near zero at 1350°C following closure of open pores.

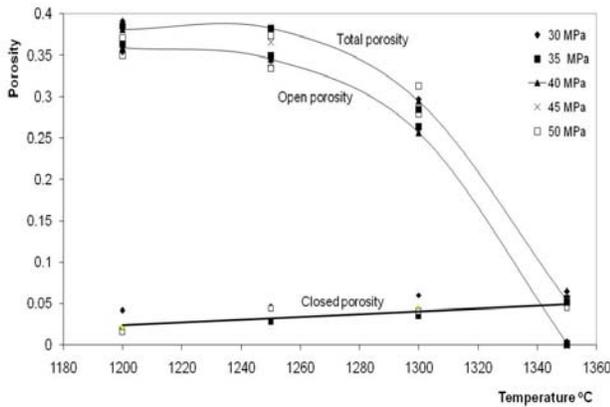


Fig.4: Effect of forming pressure and firing temperature on the total porosity of samples soaked for 3 hours

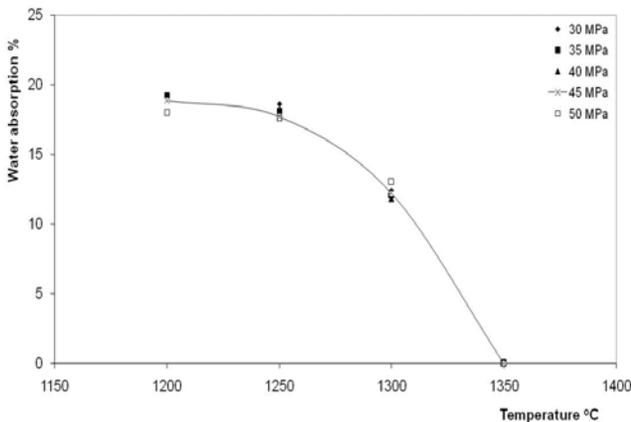


Fig.5: Effect of forming pressure and firing temperature on percent water absorption of samples fired for 3 h.

On the other hand, the linear firing shrinkage was more sensitive to variations in forming pressure and soaking time. As can be seen from Fig.6, the firing shrinkage at 1250°C increased linearly with soaking time as well as increasing with forming pressure. Fig.7 shows the maximum firing shrinkage obtained after 6 hours soaking as function of firing temperature. Forming pressure did not affect the maximum values of shrinkage achieved at each temperature. It will be noticed that unduly high values exceeding 20% were recorded for samples fired at 1350°C. As will be shown later this is due to the formation of a glassy phase causing liquid phase sintering.

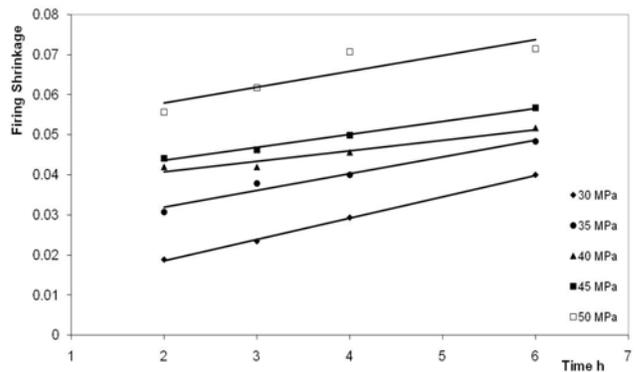


Fig.6: Firing shrinkage as function of time for samples fired at 1250 °C

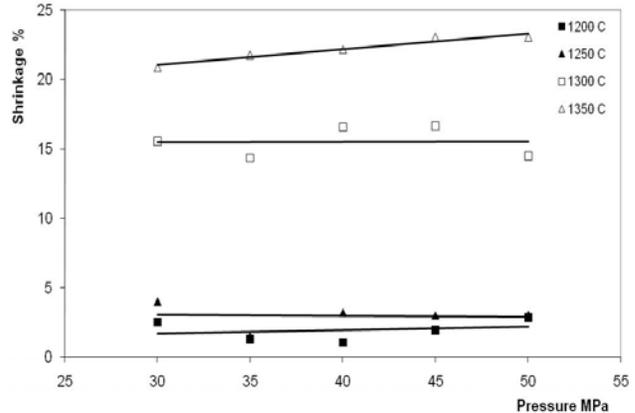


Fig.7: Maximum firing shrinkage as function of forming pressure and firing temperature

3.2 Mechanical properties:

3.2.1 Hardness:

This was determined using Rockwell scale. Fig.8 shows the variation of hardness with forming pressure and soaking time for samples fired at 1250°C. The effect of forming pressure is quite complicated since a maximum value appears at about

40 MPa forming pressure. The maximum values of hardness increase with soaking time from about 15 to 20 MPa. The reason for this behavior can be understood in the light of the dependence of surface hardness on pore formation. It is well known that a higher porosity favors low hardness so that on increasing the forming pressure, one expects hardness values to increase owing to decrease in porosity. However at high forming pressure air escape associated with pressure relief on specimens usually creates micro-cracks which may remain unhealed at relatively low firing temperature. These micro-cracks cause the observed decrease in hardness as the forming pressure exceeds 40 MPa.

Values of Rockwell hardness measured for samples fired at 1300°C attained higher maximum values reaching 65 MPa for samples fired for 6 hours. The results obtained at 1350°C were totally different from those obtained at the other two temperatures. At that temperature the values of hardness roughly increase with increased forming pressure while not showing a regular pattern as for variation with soaking time. The maximum values obtained are on the average lower than those obtained on firing at 1300°C. As will be shown later, this is probably due to the formation of a surface glassy layer which has a lower hardness than the solid material. However hardness constantly increased with forming pressure rather than showing a maximum at a certain forming pressure. This is due to the healing of micro-cracks owing to surface formation of a glassy phase on firing at such high temperature.

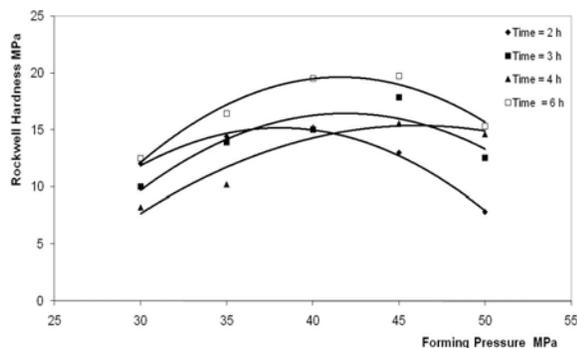


Fig.8: Effect of forming pressure and firing time on Rockwell Hardness for samples fired at 1250 °C

3.2.2 Cold Crushing Strength:

Following previous results of hardness, the cold crushing strength (CCS) was only determined for samples pressed at 40 MPa. Fig.9 shows a linear increase of CCS with both firing temperature and soaking time. The values of CCS ranged from 30 MPa for samples fired at 1250°C for 2 hours to 143 MPa for samples fired at 1350°C for 6 hours.

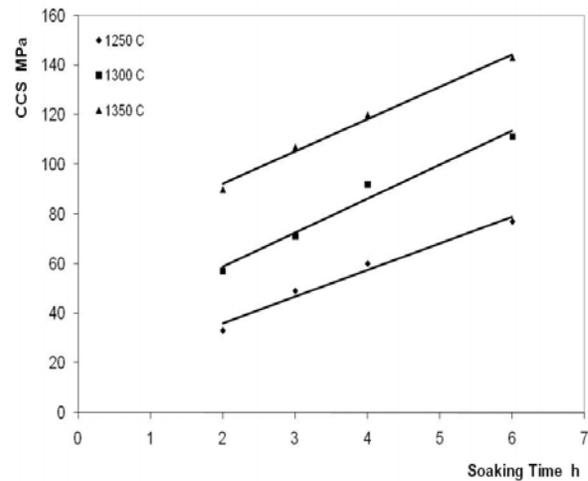


Fig.9: Effect of firing temperature and soaking time on the cold crushing strength of samples at forming pressure 40 MPa

3.2.3 Modulus of Rupture:

Fig.10 shows the results obtained on testing samples for MOR. This figure shows that as firing temperature is increased the MOR of samples highly increases. For example, for samples fired for 6 hours, the value increases from 14 to 63.5 MPa. As for the effect of soaking time, it becomes more apparent at high firing temperatures as evidenced by the increasing slopes of the lines obtained with increasing firing temperature.

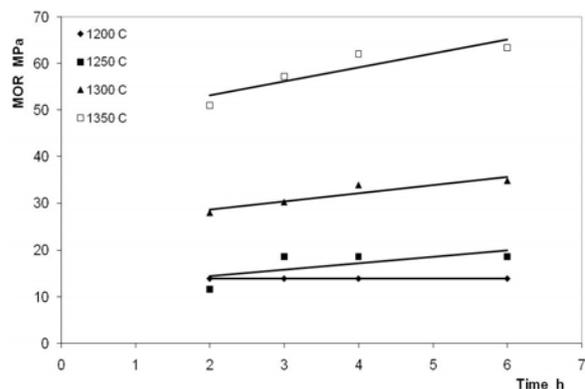


Fig.10: Effect of firing temperature and soaking time on the MOR of samples at forming pressure 40 MPa

3.3 Microstructure:

SEM micrographs and XRD diffractograms have been obtained to follow up the phases appearing on firing and their impact on the physico-mechanical properties discussed earlier.

Since it was earlier established that forming pressure and soaking time do not play a major role in the densification process all chosen specimens were those pressed at 40 MPa and fired for 6 hours.

The XRD pattern for the specimen fired at 1200°C did not differ much from that of raw powder [1]. The main lines were those of corundum and mullite with occasional lines of magnesium aluminate spinel and cordierite. The SEM for such a sample is shown in Fig.11. As can be seen from this photo neat primary mullite crystals appear (dark phase), a slightly lighter dark phase (probably cordierite) with scattered corundum crystals (white phase). In addition there appears an appreciable proportion of porosity. This is in accordance with previous findings (Fig.4). SEM micrographs obtained for samples fired at 1250 and 1300°C did not differ much except for the reduced amount of pores.

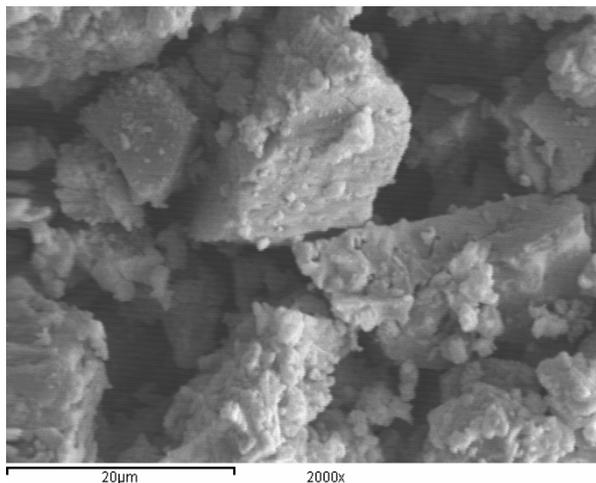


Fig.11: SEM micrograph of specimen fired at 1250 °C

When a specimen fired at 1350°C for 6 hours was XRD investigated, the pattern obtained was totally different from those obtained on firing at lower temperatures. First mullite lines completely disappeared while lines of quartz appeared for the first time. Other phases appearing are mainly cordierite, magnesium aluminate spinel, and soda – lime feldspar with few lines of corundum. The interpretation of such change can be understood in view of the SEM micrograph shown in Fig.12. This micrograph shows two important features: First, there is an almost total elimination of porosity and second, a continuous matrix covers the surface of the sample reminiscent of the presence of a glassy layer. This latter is the cooling product of a liquid phase. The presence of a liquid phase explains the disappearance of primary mullite crystals due their dissolution in the liquid as well as the subsequent precipitation of the silica phase.

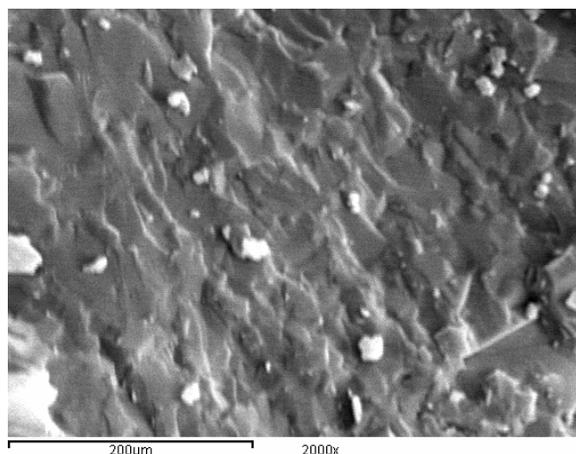


Fig.12: SEM micrograph of specimen fired at 1350 °C

3.4 Application to the ceramic tile industry:

This powder consists mainly of the same oxides present in the final fired ceramic body of wall tiles, namely, SiO₂, Al₂O₃, CaO, Fe₂O₃ and MgO. Although the chemical analysis of fired wall tile bodies differs from that of the powder, it was thought possible to incorporate the powder as filler in small proportions of the tile green body. This trial has been performed in the Prima factory in Sadat City where four tiles were prepared in which the raw roller powder was incorporated. Table (2) shows the body composition.

Table 2: Body composition for wall tiles

Material	Kaolin Clay	Ball Clay	Quartz Sand	Potash Feldspar	Calcium Carbonate	Roller Powder
%	10	30	20	20	10	10

Standard production procedure was followed in the factory and glazed tiles were fired at about 1150°C for a total cycle of 50 minutes. These tiles were then tested for percent water absorption and MOR. The following table shows the results.

Table 3: Comparison between actual tiles and Egyptian standard [9]

Property	% W.A.	MOR MPa
Actual tiles	15.5 %	14.5
ES standard	< 17 %	> 12

On the other hand, a common type of floor tiles produced under the brand name "Porcelain tiles" is actually rather a stoneware product. This is since the formal definition of porcelain requires it to have a near zero porosity while stoneware tiles can have

porosities up to 9%. Because of the development of liquid phase, it was thought possible to use the powder pressed at 40 MPa and fired at 1350°C for 2 hours as a cheap type of "porcelain tiles". A comparison between commercial tiles and the actual

bodies was made. These commercial floor tiles are produced by pressing at 40 MPa and firing at 1320°C for 1 hour. The comparison is shown in the following table.

Table 4: Comparison between floor tiles from the mix and commercial tiles [10]

Property	Water Absorption %	Total Porosity	Bulk Density g/cm ³	Closed Porosity %	Linear Shrinkage %	MOR MPa
Trial tiles	0.6	6 %	2.7	5.3 %	22	52
Commercial tiles [10]	< 3	< 9 %	2.4	5.4 %	< 7	> 35

As can be seen from this comparison, except for the large linear shrinkage displayed by the trial tiles, all other properties are superior in case of trial tiles. Further investigation will be necessary to limit the degree of shrinkage for such type of tiles.

4. Conclusions

Roller kiln grinding powder was obtained from a ceramic tile factory and its physico – mechanical properties determined. The following results were obtained:

- Increasing the forming pressure increased the bulk density of green samples while decreasing their porosities.
- The temperature of firing was practically the sole factor that affected the sintering properties of pressed powder: An increase in firing temperature from 1200 to 1350°C had for effect to raise considerably the bulk density and to decrease the open porosity to almost zero level with consequent decrease in percent water absorption. The effect of variations in forming pressure or time of soaking was insignificant in this respect.
- The percent linear shrinkage was affected by the three variables reaching unduly high values on firing at 1350°C (over 20%), because of the formation of a surface glassy phase.
- The measured values of Rockwell hardness showed a marked increase on raising the firing temperature from 1200 to 1300°C displaying a maximum value at about 40 MPa forming pressure. Values of hardness determined for samples fired at 1350°C showed a decrease below the previous values owing to the formation of a soft surface glassy phase.
- Both cold crushing strength and modulus of rupture increased with increased firing temperature and soaking time.
- It was possible to prepare sample tiles containing 10% powder added to a standard wall tiles mix.

The properties of these tiles were comparable to those of the commercial tiles.

- The properties of tiles fired at 1350°C for 2 hours were comparable to the locally produced "porcelain tiles" except for their higher firing shrinkage.

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