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Recycling Of The Mixture Resulted From Sanitary Ware Waste, Roller Kiln Waste And Ceramic Tiles Sludge Waste In The Manufacturing Of Ceramic Floor Tiles

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Abstract: ***Ceramic tiles industry is a big source for wastes that have a bad effect on the environment. The first source of waste is associated with roller kilns as it needs a periodic surface grinding to its rollers which results in a waste powder. The second type of waste is ceramic tiles sludge which produced from a water treatment plant. The third waste is sanitary ware waste which coming out from sanitary ware industry. The main aim of this paper is to investigate the ability of substituting part of the main body mixture of floor tiles by these three types of waste powder. Many Experiments were done on the raw materials. Rectangular tile specimens were moulded, dried and fired. Sintering parameters and mechanical properties were determined. The sample that consist of 2% Roller Kiln, 39% Ceramic tiles sludge, 15% sanitary ware waste, and 44% Floor tiles mixture has the optimum properties.***

Index Terms: ***ceramic sludge; roller kiln waste; sanitary ware; ceramic floor tiles; waste recycling.***

I. INTRODUCTION

The production of ceramics has been produced from the beginning of the history as in parts of the civilization in the world as in the Ancient Egypt, Greece also China. The raw materials for ceramic products are clay, sand, and feldspars. Clays give the main body of the product. Silica is the main glass forming raw material. Feldspars represent fluxing components since they melt at relatively low temperatures. The manufacturing of any ceramic products is done by crushing, grinding, screening and mixing of raw materials followed by ceramic body formation then drying and finally firing and glazing. Examples of traditional ceramics are white wares, refractory products, and glasses. Wall and floor tiles used for interior and exterior decoration belong to a class of ceramics known as white wares [1].

The roller kilns is the major firing device for the ceramic tiles. It has many advantages such as no kiln furniture is required, high flexibility of operation, low energy consumption, and uniform heat transfer. Roller kiln consists of a set of stationary parallel horizontal rollers spaced at a convenient distance and situated in a row along the length of the kiln. The distance between these rollers is determined by size as the product. Non–metallic rollers are usually manufactured by isostatic pressing [1]. The big environmental problem associated with roller kiln is that it needs a surface grinding periodically to the rollers of the kiln rollers which manufactured from high alumina [2, 3].

This hazardous waste was used in the production of alumino–silicates refractories on ground of the original composition of the rollers. The results showed that samples fired at 1350 °C are potential candidates for use as high alumina refractories [4]. Also, Youssef and Ghazal [3] proved that the roller kiln waste can be added up to 2% without any treatment to standard floor tiles composition without altering its final properties. On the other hand, Ahmed [5] found that the use of this powder depends on the firing temperature as it was used as shaped refractories on firing at 1300 °C for 6 hours and as porcelain tiles on firing at 1350 °C for 2 hrs. Roushdy et al. [6] proved that the roller kiln waste can be added up to 2% without any treatment to standard wall tiles composition without altering its final properties. Ceramic tiles sludge waste produced from washing all the equipment used in the ceramic tiles industry starting. To solve this problem, studying the technique of re using this waste must be taken in consider [7]. Andreola et al. [8] investigated the possibility of reusing the ceramic sludge produced during the purification of waste water resulted from the glazing unit in the manufacturing of floor or wall covering tiles. This researcher formed the tiles at firing temperature of 1000 °C instead of 1200°C. Nandi et al. [9] studied how it is possible to use ceramic sludge produced from treatment of waste water of ceramic tiles to get a single-fire ceramic tiles. He mixed 20-80 % by weight ceramic sludge and 12-48 % by weight recycled glass with dolomite and calcite as raw materials. Sanitary ware products that are not complied with the required standards are considered to be as waste and have a big bad effect on the environment. Some researchers studied the recycling of such waste to produce useful products. Tarhan et al. [10] studied the recycling of sanitary ware waste as a part of raw materials used in wall tiles production. He added up to 15 % powder to ceramic wall tiles standard mix He found that adding these waste results in increasing the bending strength of the resulted wall tiles.

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II. EXPERIMENTAL WORK

A. Raw Materials

There are three types of raw materials used in this work as kindly supplied by Ceramica Venus Company, 10th of Ramadan city as follow:

1. Ceramic floor tiles basic mixtures. This floor tiles basic mixture was prepared from Egyptian raw materials as stated by the supplying company as shown in Table 1.
2. Sludge waste obtained from the water treatment unit.
3. Roller kiln waste obtained on surface grinding of the kiln rollers
4. Sanitary ware waste.

Table 1. Batch Composition of Floor Tiles Mixture

Raw Materials	Weight %
Kaolin Clay	1
Ball Clay	41
Bentonite	2
Feldspars	40.5
Sand	5.5
Green Tiles Scrap	7
Talc	3

B. Raw Materials Characteristics

The chemical analysis of fine ceramic waste was assessed using XRF. The equipment used was Wavelength Dispersive (WD–XRF) Sequential Spectrometer, installed at the central laboratories sector at the Egyptian mineral resources authority, the ministry of petroleum Center, Cairo.

The phase composition was obtained by XRD analysis using X–Ray Diffractometer apparatus (the central laboratories sector at the Egyptian mineral resources authority, the ministry of petroleum Center, Cairo).

On the other hand, thermal analysis was used to follow up possible thermal changes in the prepared mixtures (TGA and DTA), installed at the Center of accurate analysis, Cairo University, Cairo.

BT–2001 Laser Particle Size Analyzer was used to determine the particle size distribution of the fine waste and raw materials mixture according to ASTM D 422 [11]. It is present at the central laboratories sector at the Egyptian mineral resources authority, the ministry of petroleum Center, Cairo.

Finally, the density bottle method was used to determine the powder density was determined according to ASTM B 311 [12].

C. Preparation of Ceramic Tiles

The ceramic tiles sludge which produced from ceramic tiles factory is first dried at 110 ºC for 24 hours to remove its water content then crushed in jaw crusher and finally grinded in ball mill to get a final fine waste powder. The sanitary ware product which is not compatible with the required standards is considered to be waste. This waste product is crushed and grinded to produce powder which is named as sanitary ware waste powder. Prepared mixtures of waste powder were prepared using a roller kiln waste with percent 1% or 2% mixed with a ceramic sludge waste with percent in range from 0% to 50% with sanitary ware waste with percent in range from 0% to 15%. Using a 2^3 factorial design, eight mixtures were prepared. The floor tiles formation from the prepared mixtures was carried out as follow:

1. Rectangular ceramic tile specimens with dimensions 110.4 × 55.4 × 8 mm^3, were molded using dry pressing under pressure of 27 MPa.
2. Tile specimens were dried using a laboratory dryer on two steps. It dried for four hours at 70 °C, then for one hour at 110 °C.
3. Tile specimens were fired using the laboratory furnace, Protherm–electrical furnace model PLF 14015 at 1180 °C, for 15 min soaking time. Heating rates were chosen to be as close as possible to industrial conditions.

The single fast firing technique is used. The following steps show the firing schedule in a roller kiln:

1. The temperature is increased from room temperature to 600 °C quickly.
2. The temperature is increased gradually 600 °C to 700 °C, increasing 20 °C every 10 min, in order to provide slowly escape for combined water and prevent crack formation.
3. Fast increase the temperature from 700 °C to the required firing temperature and soaked for 15 min then stop the furnace.

D. Testing of Ceramic Tiles

According to ASTM C 326 [13] the percent linear firing shrinkage was determined, apparent porosity and percent water absorption were determined according to ASTM C 373 [14] and breaking strength and modulus of rupture according to ISO 10545 – 4 [15]. The micrographs of the optimum ceramic tiles were resulted using a scanning electron microscope (SEM). The SEM apparatus used is JEOL–JSM 6510 apparatus.

III. RESULTS AND DISCUSSION

A. Chemical composition of ceramic waste

The details of XRF analysis of the raw mix are shown in Table 2 and 3. The following conclusions the obtained results:

1. For ceramic sludge waste, silica is the main component rather than alumina. The loss on ignition has a reasonable value due to presence of limestone and organic matter in it.
2. For roller kiln waste, alumina is the main component rather than silica as the rollers in the kiln is high alumina type. Also an almost zero loss on ignition was observed.
3. For sanitary ware waste, silica is the main component rather than alumina. The loss on ignition has a reasonable value because

the waste due to presence of limestone in it.

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Table 2. Chemical Analysis of Ceramic Sludge and Roller Kiln

Main constituents	Sludge waste	Roller kiln waste
SiO_2	54.35	10.69
Al_2O_3	21.32	83.75
Fe_2O_3	4.67	1.61
TiO_2	0.85	0.21
ZrO_2	< 0.01	2.35
MnO	0.12	< 0.01
SO_3	0.12	0.07
MgO	0.11	0.22
CaO	5.99	0.25
Na_2O	2.88	0.02
K_2O	1.4	0.03
Cl	0.05	0.06
P_2O_5	0.17	0.03
LOI	7.7	0.42

Table 3. Chemical Analysis of Sanitary Ware and Floor Mix

Main constituents	Sanitary ware waste	Floor mix
SiO_2	66.29	61.21
Al_2O_3	22.38	0.83
Fe_2O_3	0.76	20.19
TiO_2	0.33	4.78
ZrO_2	0.3	0.99
MnO	< 0.01	1.21
SO_3	0.08	2.72
MgO	0.93	1.21
CaO	2.13	0.21
Na_2O	3.28	0.28
K_2O	1.72	0.10
Cl	0.05	0.286
P_2O_5	0.1	5.99
LOI	1.39	0

B. Mineralogical of ceramic waste

The XRD pattern of floor tiles mixture as shown in Fig 1., showed it to be constituted from the following phases: Quartz (SiO_2), Albite Na (Al Si_3 O_8), and Kaolinite ($Al_2Si_2O_5(OH)_4$). The main phase is quartz [16]. The XRD pattern of ceramic sludge waste as shown in Fig 2., showed it to be constituted from the following phases: Quartz (SiO_2), Calcite ($CaCO_3$), Albite ($Na_{0.98}Ca_{0.02}$) ($Al_{1.02}Si_{2.98}O_8$), and Kaolinite ($Al_2Si_2O_5(OH)_4$). The main phase is quartz. As expected, ceramic sludge is composed of a mixture of all ingredients constituting the raw mixes for wall and floor tiles. The XRD pattern of roller kiln waste as shown in Fig 3., showed it to be constituted from the following phases: Mullite ($Al_{2.3}Si_7O_{4.85}$), Corundum (Al_2O_3), Anorthite ($CaAl_2Si_3O_8$), and Gehlenite ($Ca_2Al(Al\ Si)O_7$). The main phase is Corundum. The results are expected as the roller kiln waste resulted from surface grinding of kiln rollers which is fabricated from high alumina material [6]. The XRD pattern of sanitary ware waste as shown in Fig 4., showed it to be constituted from the following phases: Quartz (SiO_2), Mullite ($Al_{2.3}Si_7O_{4.85}$), Calcite ($CaCO_3$), Albite Na ($AlSi_3O_8$), and Orthoclase K (Al, Fe)Si_2O_8. The main phase is quartz. It reveals the presence of quartz, soda and potash feldspars and mullite. The presence of this latter is expected since some of the original bodies which constitute the scrap were fired well above the temperature of formation of mullite (1200ºC)

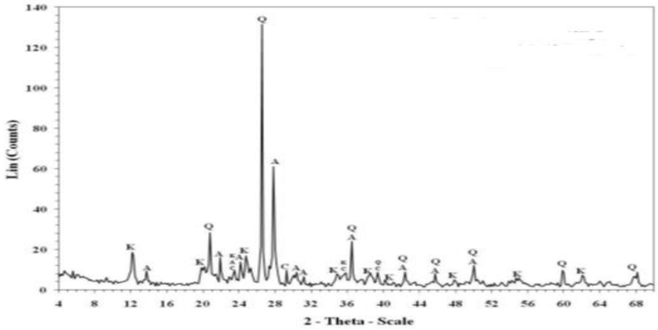

Fig. 1. XRD Pattern of Floor Mix

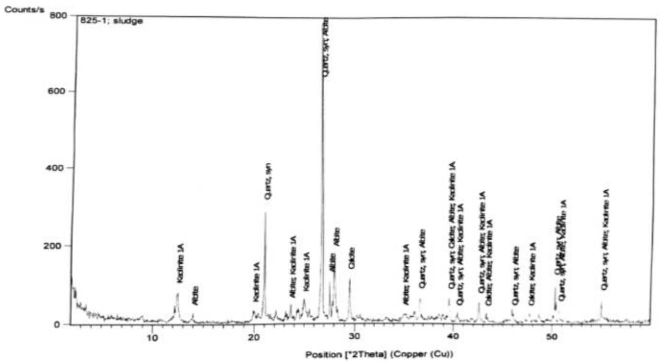

Fig. 2. XRD Pattern of Ceramic Sludge Waste

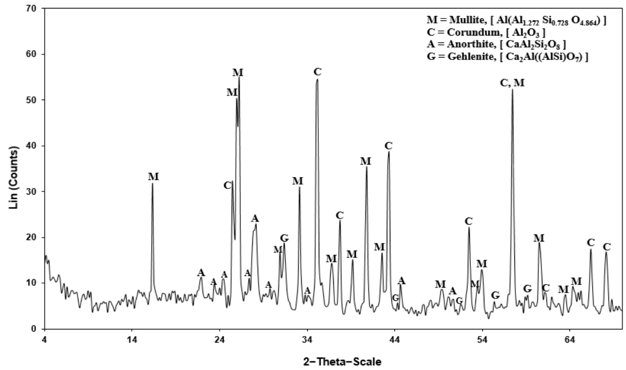

Fig. 3. XRD Pattern of Roller Kiln Waste

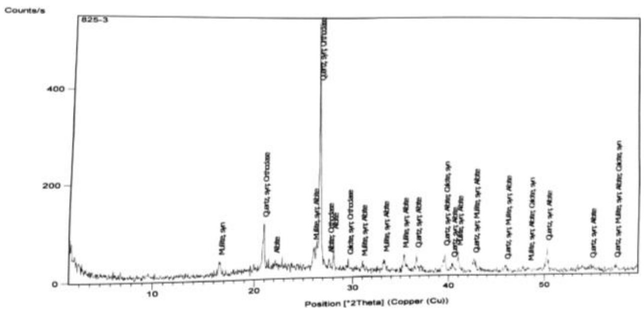

Fig. 4. XRD Pattern of Ceramic Sanitary Ware Waste

C. Thermal analysis of ceramic waste

Combined TGA – DTA chart for floor tile mixes is shown in Fig 5 and 6. The first weight loss is because of physical water removal.

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Then there is an exothermic because of the organic impurities oxidation that ends at about 400ºC. Then after that there is an endothermic peak because of lattice water of clays that ends at about 525ºC and practically completed at about 650ºC on TGA. Also owing to the presence of limestone another peak can be observed at about 720ºC [16].

Combined TGA – DTA chart for roller kiln waste is shown in Fig 7 and 8. There is some very small losses in temperature range between the 110–170 °C because of moisture content losses, after that there is a exothermic peak because of organic impurities oxidation. The weight increase on TGA can be neglected because it rises from 4.1 mg at 300 °C to 4.35 mg at 1000 °C because of a misshaping of the base line [6].

Fig 9 and 10. Show the thermal analyses (DTA and TGA) pattern for sanitary ware waste. There is some very small losses in temperature range between the 110–170 °C because of moisture content losses and after that change in weight up to 1000 °C is negligible.

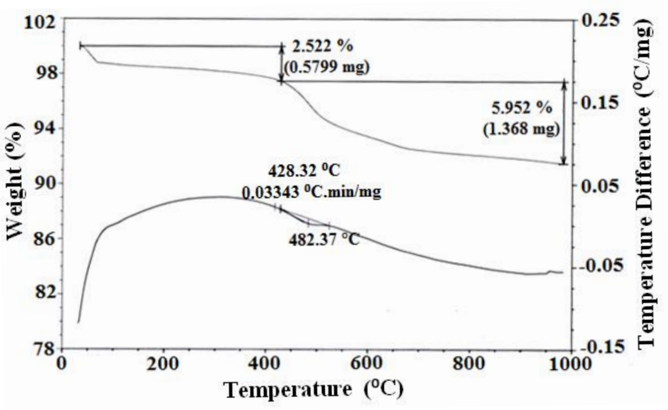

Fig. 5. DTA and TGA Pattern of Floor Mix

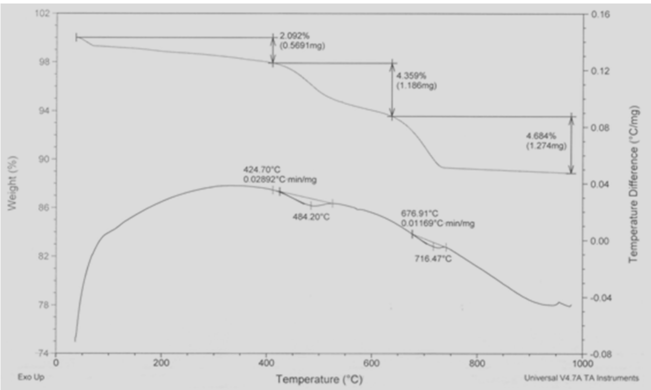

Fig. 6. DTA and TGA Pattern of Ceramic Sludge

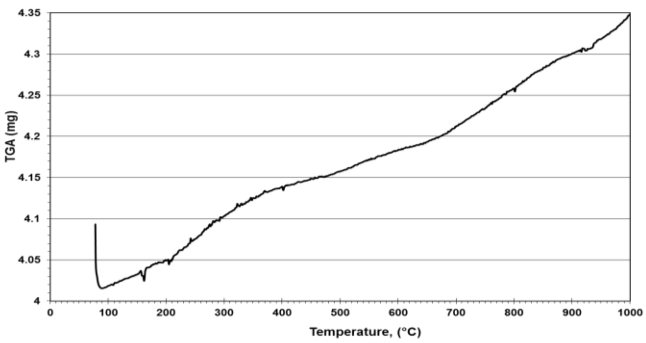

Fig. 7. TGA Pattern of Roller Kiln Waste

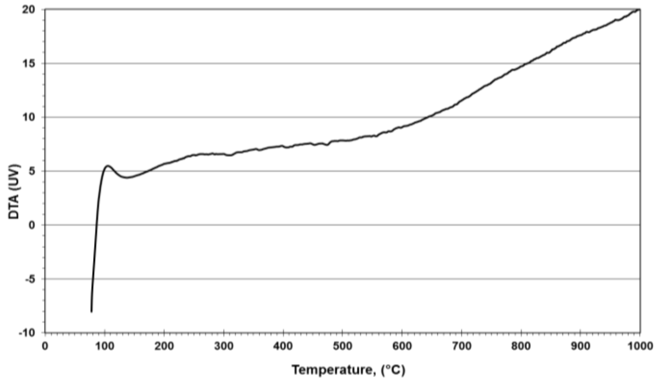

Fig. 8. DTA Pattern of Roller Kiln Waste

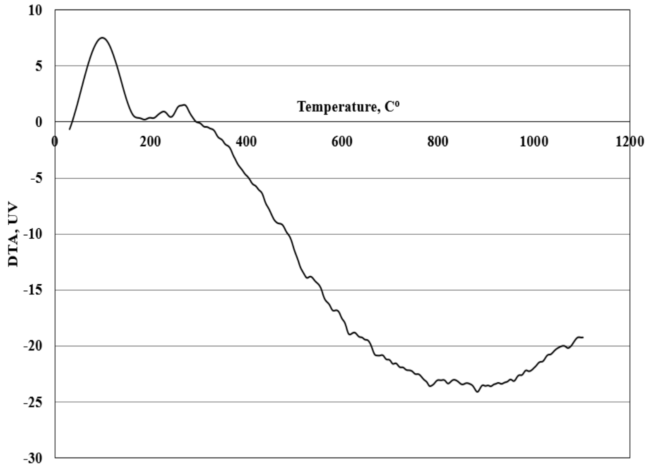

Fig. 9. DTA Pattern of Sanitary Ware

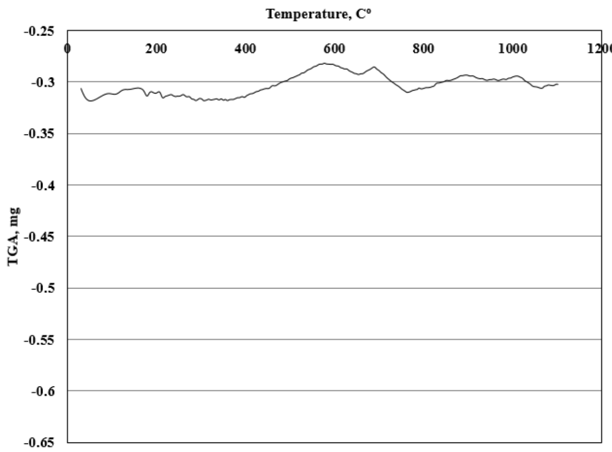

Fig. 10. TGA Pattern of Sanitary Ware

D. Screen analysis of raw materials

Fig. 11. shows the cumulative screen analysis of ceramic roller kiln waste, sludge waste, and sanitary ware waste compared with ceramic floor tiles. The mean particle size of any type of waste powder is much lower than that of floor tiles mix.

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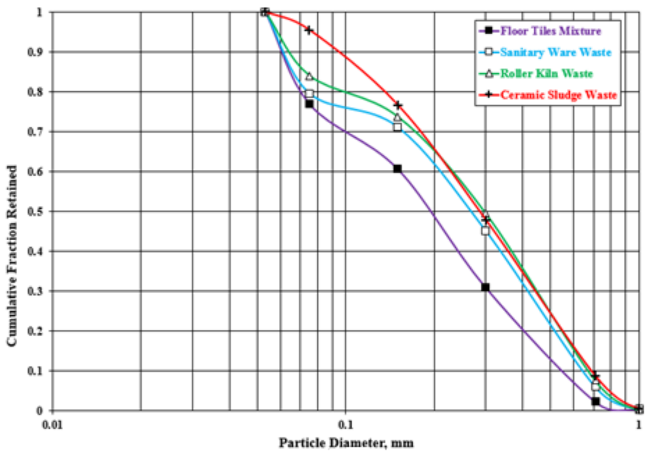

Fig. 11: Cumulative analysis of floor tiles mixture and all types of waste powder

E. Green Breaking 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 illustrated in Table 4. Values of MOR are higher than the recommended figure of 1 MPa.

F. Linear firing shrinkage

Table 5 summarizes the effect of wastes addition on the linear firing shrinkage results. These results show that shrinkage decreases upon adding the roller kiln waste and ceramic sludge waste while an increase in sanitary ware waste will increase this shrinkage. Floor tiles contain large amount of feldspar which is the source of liquid phase sintering that is responsible for linear firing shrinkage. The addition of ceramic sludge waste will replace the feldspar which will lead to decreasing the firing shrinkage. The increase in the amount of roller kiln waste added has an effect to decrease the firing shrinkage. It is believed that the addition of the high alumina refractory waste would have effect to limit or even suppress the formation of a liquid phase. This would explain the drop in firing shrinkage observed on adding such waste. Firing shrinkage is known to increase with increased sintering and verification. The linear firing shrinkage increases with the addition of sanitary ware as it contains soda and potash feldspars so the amount of feldspar increases which promotes liquid phase sintering which increase the values of linear firing shrinkage.

The actual equation related all waste and linear firing shrinkage using Design expert program is the following

LFS=6.84487 + 0.030149A – 0.038910B – 0.314357C (1)

Table 5. Effect of Wastes Addition on Linear Firing Shrinkage

Percentage				Linear Shrinkage based on length, %
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	
100	0	0	0	6.85239385
85	15	0	0	7.14054054
50	0	50	0	4.92522522
35	15	50	0	5.47477477
98	0	0	2	6.313
83	15	0	2	6.72061206
48	0	50	2	4.14041404
33	15	50	2	4.70405405
66.5	7.5	25	1	5.94414414

G. Loss on ignition

Table 6 summarizes the effect of wastes addition on loss on ignition results. As can be expected the addition of the roller kiln waste decrease the total loss on ignition since the waste does not contain any decomposable material. The results showed that the addition of the ceramic sludge waste increase the total loss on ignition. The loss on ignition has a reasonable value because of the presence of limestone and organic matter in it. The addition of the sanitary waste decreases the total loss on ignition since the waste does not contain any decomposable material.The actual equation related all waste and loss on ignition using Design expert program is the following

Table 4. Effect of wastes addition on green MOR

LOI=7.21986–0.058395A+0.013887B – 0.181672 C (2)

Table 6. Effect of Wastes Addition on Loss on Ignition

Percentage				Green MOR
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	
100	0	0	0	2.2434
85	15	0	0	1.3675
50	0	50	0	1.8161
35	15	50	0	1.3514
98	0	0	2	2.2682
83	15	0	2	1.8211
48	0	50	2	2.2753
33	15	50	2	1.4043
66.5	7.5	25	1	1.3648

Percentage				Loss on ignition, %
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	
100	0	0	0	7.109597
85	15	0	0	6.346715803
50	0	50	0	8.108713197

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35	15	50	0	6.951295723
98	0	0	2	6.7093
83	15	0	2	6.235308161
48	0	50	2	7.613875
33	15	50	2	6.504464745
66.5	7.5	25	1	7.287271594

H. Water absorption

These results are presented in Table 7. The resulted tiles can be categorized using the values of water absorption and Egyptian standards [17] into two categories: tiles of low water absorption, 0.5% • E • 3 % and tiles of medium water absorption, 3 % • E • 10 %. The behavior of water absorption is in harmony with firing shrinkage. It increases with the increase in both ceramic sludge waste and roller kiln waste percent. The addition of ceramic sludge waste on the expense of feldspars will tend to decreasing firing shrinkage so as to increase the porosity to increase the values of water absorption. The increase in the amount of roller kiln waste added has an effect to decrease the firing shrinkage. It is believed that the addition of the high alumina refractory waste would have effect to limit or even suppress the formation of a liquid phase so as to increase the porosity to increase the values of water absorption. The linear firing shrinkage increases with the addition of sanitary ware as it contains soda and potash feldspars so the amount of feldspar increases which promotes liquid phase sintering which increase the values of linear firing shrinkage so as to decrease the porosity and water absorption. The actual equation related all waste and water absorption using Design expert program is the following

$$WA=0.966462–0.034517A+0.064859B+0.306355C \qquad (3)$$

Table 7. Effect of Wastes Addition on Percent Water Absorption

Percentage				
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	Percent Water Absorption
100	0	0	0	1.511133212
85	15	0	0	1.097966442
50	0	50	0	3.57822761
35	15	50	0	3.128317686
98	0	0	2	0.9159
83	15	0	2	0.530753192
48	0	50	2	5.571320414
33	15	50	2	4.748510901
66.5	7.5	25	1	1.170339585

I. Apparent porosity

These results are presented in Table 8. Porosity is not a standard requirement for floor tiles. However, it is often necessary to ensure that floor tiles in particular have limited porosity to reduce the possibility of liquid infiltration.

The actual equation related all waste and open porosity using Design expert program is the following

$$P=1.35068–0.048239A+0.090635B+0.428146C \qquad (4)$$

Table 8. Effect of Wastes Addition on Percent Water Absorption

Percentage				
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	Percent Open Porosity
100	0	0	0	2.111881672
85	15	0	0	1.53446115
50	0	50	0	5.000745961
35	15	50	0	4.371975107
98	0	0	2	1.2800145
83	15	0	2	0.741753229
48	0	50	2	7.786189449
33	15	50	2	6.636273401
66.5	7.5	25	1	1.635606114

J. Fired Breaking Strength

Table 9 shows the behavior of the firing bending strength. The values of bending strength decreases with the increase in ceramic tiles sludge and roller kiln waste. The behavior of breaking strength has the same behavior and reasons as the linear firing shrinkage. When the linear shrinkage increases the porosity decreases and then the strength increases. The actual equation related all waste and breaking strength using Design expert program is the following

$$BS=944.00547+7.52916A–7.22706B–23.6221C \qquad (5)$$

Table 9. Effect of Wastes Addition on Percent Breaking Strength

Percentage				
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	Breaking Strength
100	0	0	0	920.9185883
85	15	0	0	1050.009381
50	0	50	0	598.153
35	15	50	0	710.1104972
98	0	0	2	936.35
83	15	0	2	1000.13037

48	0	50	2	503.4065934
33	15	50	2	650.3274336
66.5	7.5	25	1	859.85

Fired Modulus of rupture (MOR)

Table 10 shows the behavior of MOR. The values of MOR of the tested samples were naturally related to their strengths so that there was a strong similarity between the two cases.

The actual equation related all waste and MOR using Design expert program is the following

$$MOR=53.05468+0.2782A-0.586653B-2.10530C \quad (6)$$

Table 10. Effect of Wastes Addition on MOR

Percentage				MOR
Main Body	Sanitary (A)	Ceramic Tiles Sludge (B)	Roller Kiln Waste (C)	
100	0	0	0	52.825
85	15	0	0	53.551
50	0	50	0	21.129
35	15	50	0	34.392
98	0	0	2	52.529
83	15	0	2	53.237
48	0	50	2	18.648
33	15	50	2	20.642
66.5	7.5	25	1	42.072

IV. POSSIBILITY OF WASTE RECYCLING INTO FLOOR TILE BODIES

According to the previous properties that were measured, it was found that the sample with the composition (2% Roller Kiln, 39% Ceramic tiles sludge, 15% sanitary ware waste, and 44% Floor tiles mixture) has the optimum properties with respect to all experiments. The composition of optimum sample was determined using design expert program that was licensed by British University in Egypt at 2018. 3 specimens with the same optimum ceramic tile composition were prepared and fired then tested for percent water absorption, breaking strength and MOR. The results are shown in Table 17 compared with the standard values [17].

In order to prove the previously obtained results, a specimen represent the optimum sample was examined by SEM at magnification 1000×. Fig. 12. Shows the high level of liquid phase formation which results in the low water absorption observed.

Table 17.Properties of Optimum Samples

Property	Optimum Sample	Standard Sample
Composition of Optimum Ceramic Tiles	2% Roller kiln 39% Ceramic tiles sludge 15% Sanitary ware 44% Floor tile mixture	
% Water absorption	2.891	0.5%-3%
Breaking Strength N	726.148	> 700
MOR MPa	32	> 30

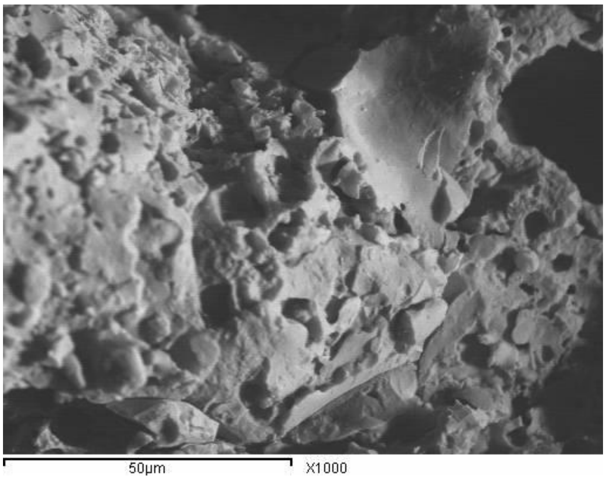

Fig. 12. SEM Micrographs (1000x) for the Optimum Sample

V. CONCLUSION

Ceramic tiles industry produced many types of wastes that are routinely discarded and accumulates within the plant premises as stock piles. This represents an extremely high ecological hazard as such powder if inhaled for long periods can lead to serious lung problems such as silicosis. The roller kilns are periodically needed to grind its rollers which results in a big environmental problem. The second type of waste is ceramic tiles sludge which produced from a water treatment plant in the ceramic factory. The third waste is sanitary ware waste which coming out from sanitary ware industry. The main aim of this thesis is to investigate the possibility of substituting part of the main body mix of floor tiles by these three types of waste powder. The experimental program includes performing XRF, XRD, DTA and TGA of raw materials. Rectangular tile specimens of dimensions 110.4 × 55.4 × 8 mm^3 were molded by dry pressing under uniaxial pressure of 27 MPa then dried overnight at 120 ºC. Tile samples were fired at for 15 min at temperature of 1180 ºC. Linear firing shrinkage, loss on ignition, water absorption, mechanical properties, and apparent porosity were determined and compared to ISO standards. According to the previous properties that were measured, it was found that the sample with the composition (2% Roller Kiln, 39% Ceramic tiles sludge, 15% sanitary ware waste, and 44% Floor tiles mixture) has the optimum properties with respect to all experiments.

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The composition of optimum sample was determined using design expert program that was licensed by British University in Egypt at 2018.

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