# CHARACTERIZATION OF CERAMIC SLUDGE AND RECYCLING IN CERAMIC TILE PRODUCTION

# Alper Ö. Yaşar, Kağan Kayacı

## Kaleseramik R&D Center, Kaleseramik Çanakkale Kalebodur Seramik San. A.S. Çan, Çanakkale, Turkey

#### ABSTRACT

In this study, ceramic sludge was identified, analyzed, and then the usability of this waste in a range of 2-8 wt% in the prepared formulation was investigated. The ceramic sludge was characterized by X-ray fluorescence (XRF) spectroscopy, X-ray diffraction (XRD) spectroscopy, and a sieve analysis, respectively. Then, the density of the prepared formulation was determined. The linear shrinkage, water absorption and color values (L, a, b) of the prepared ceramic tile were determined and the standard and new formulations were compared. It was determined that the ceramic sludge comprised approximately 80% SiO2 and Al2O3 and its particle size was below 32  $\mu$ m (91.27%). Linear shrinkage and water absorption values of the ceramic sludge were found to be between 8.10-8.46 and 0,00-0,40, respectively. The L value of new formulations containing ceramic sludge was found to be slightly higher than that of the standard formulation.

## **1. INTRODUCTION**

In the 21st century, the efficient use of raw materials and energy has gained importance due to the limited natural resources and increased demand for these [1]. Furthermore, it is known that the wastes produced because of the use of some industrial raw materials have negative effects on the environment [2]. Waste recycling has become even more important in this century due to the factors mentioned above. The use of industrial wastes as a raw material provides both waste disposal and reduction of raw materials use [3]. Thus, materials with high added value can be produced by recycling waste. Certain sludge wastes also appear in the ceramic tile production sector. Recently, waste materials such as drilling mud, boron waste, fly ash and sludge waste have been investigated for use in tile formulations together with traditional raw materials [3-6]. In this study, the reuse of ceramic sludge from the wastewater treatment plant of a local tile company was investigated in commercial ceramic tile formulations.

### 2. MATERIALS AND METHOD

### 2.1. MATERIALS

Clay, kaolin and ceramic sludge were used as raw materials for the production tile. Sodium tripolyphosphate and sodium silicate were used as electrolyte in preparing the ceramic slurry.

### 2.2. METHOD

The formulations were prepared by using sufficient amounts of the raw materials such as clay, kaolin and the ceramic sludge, on a lab scale. Amounts of 2, 4, 6 and 8 wt% ceramic sludge were added to the standard formulation used in a ceramic tile company. The compositions prepared with the raw materials were wet milled, the slurry was dried, followed by powder granulation, pressing and drying, after which the samples were sintered. Sintering took place in 35 minutes from room temperature to a peak temperature of 1180°C [1,3]. The formulations containing 2, 4, 6 and 8 wt% ceramic sludge, standard formulation and ceramic sludge formulation were referenced F2, F4, F6, F8, STD and CS, respectively.

### 2.3. CHARACTERIZATION

The prepared ceramic slurries were characterized by X-ray fluorescence (XRF) spectroscopy (PANalytical Axios), X-ray diffraction (XRD) spectroscopy (PANalytical X'Pert Pro MPD) diffractometer, and particle sizes (Sieve set, +150  $\mu$ m, +106  $\mu$ m, +90  $\mu$ m, +63  $\mu$ m, +45  $\mu$ m and +32  $\mu$ m). The physical and color properties of the formulations, such as water absorption, linear shrinkage, and the chromatic coordinates (Konica Minolta Chroma meter CR-400), were also determined).

### 3. RESULTS AND DISSCUSSION

The ceramic sludge was analyzed XRF, XRD and particle size and then, the formulations containing 2, 4, 6 and 8 wt% ceramic sludge were prepared with the raw materials such as clay and kaolin on a lab scale. Shrinkage, water absorption and color values (L, a, b) of these formulations were investigated. The chemical composition of waste ceramic sludge was analyzed by XRF for seven days and the obtained results were given in Table 1. It is clearly seen that the main oxides were SiO2 (62.36% ± 1.29) and Al2O3 (17.36% ± 0.73) in comparison with TiO2 (0.71% ± 0.10), Fe2O3 (1.44% ± 0.14), CaO (4.21% ± 0.77), MgO (1.18% ± 0.17), Na2O (2.44% ± 0.20), K2O (2.19% ± 0.11), ZrO2 (1.91% ± 0.48) and ZnO (1.04% ± 0.27).

| Oxides                         | Mass wt%        | Oxides | Mass wt%        |  |
|--------------------------------|-----------------|--------|-----------------|--|
| LOI                            | 4,72 ± 0,42     | MgO    | $1,18 \pm 0,17$ |  |
| SiO <sub>2</sub>               | 62,36 ± 1,29    | Na2O   | $2,44 \pm 0,20$ |  |
| Al <sub>2</sub> O <sub>3</sub> | 17,36 ± 0,73    | K2O    | $2,19 \pm 0,11$ |  |
| TiO <sub>2</sub>               | $0,71 \pm 0,10$ | ZrO2   | $1,91 \pm 0,48$ |  |
| Fe <sub>2</sub> O <sub>3</sub> | $1,44 \pm 0,14$ | ZnO    | $1,04 \pm 0,27$ |  |

LOI: Los son ignition

The percent of variability values for the mass wt% of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> as the main oxides were calculated, these being 2.10% and 4.21%, respectively. It may be clearly seen that the waste ceramic sludge was stable and can be used in the preparation of floor tile formulations in certain proportions. The XRD pattern of the ceramic sludge is shown in Figure 1. The ceramic sludge consisted of albite (30.4%), quartz (24.8%), muscovite (23.7%), kaolinite (17.1%), calcite (1.9%), zircon (1.2%), and magnesite (0.9%). It is clear that most of the ceramic sludge was composed of albite, quartz, muscovite and kaolin (96%).



*Figure 1.* XRD pattern of the ceramic sludge (A: Albite, C: Calcite, K: Kaolinite, M: Muscovite, Ma: Magnesite, Q: Quartz, Z: Zircon)

Table 1. Chemical composition of the waste ceramic sludge determined by XRF.

The particle size of the ceramic sludge was determined as 0.58% for +150  $\mu$ m, 0.54% for +106  $\mu$ m, 0.53% for +90  $\mu$ m, 1.26% for +63  $\mu$ m, 2.48% for +45  $\mu$ m, 3.34% for +32  $\mu$ m and 91.27% for below 32  $\mu$ m in the sieve analysis. It is clear that the particle size of the ceramic sludge was mostly below 32  $\mu$ m (91.27%). The density of prepared formulations (2-8 wt% CS) was measured approximately in range of 1707-1719 g/L and these values are almost the same as those of the standard formulation (1707 g/L). The linear shrinkage, water absorption and color results (L, a, b) of the prepared formulations are summarized in Table 2. It was found that their shrinkage and water absorption values ranged between 0,00-0,40 and 8.10-8.46, respectively.

|            |   | STD   | CS    | F2    | F4    | F6    | F8    |
|------------|---|-------|-------|-------|-------|-------|-------|
| Shrinkage  |   | 8,10  | 6,40  | 8,46  | 8,30  | 8,29  | 8,34  |
| Water abs. |   | 0,22  | 0,04  | 0,18  | 0,00  | 0,09  | 0,40  |
| L          | % | 45,17 | 62,94 | 46,28 | 45,96 | 46,93 | 46,44 |
| а          |   | 5,38  | 1,12  | 5,11  | 5,08  | 5,44  | 4,78  |
| b          |   | 12,67 | 10,62 | 12,97 | 12,48 | 13,07 | 12,29 |

Table 2. Shrinkage, water absorption and color results (L, a, b) of the prepared formulations

As a result, the shrinkage and water absorption results of the formulations (F2, F4, F6 and F8) containing ceramic sludge (2-8 wt%) were almost same as those of the STD formulation.

## 4. CONCLUSIONS

The ceramic sludge was characterized by XRF, XRD and sieve set and the formulations containing 2, 4, 6 and 8 wt% ceramic sludge were prepared on a lab scale. The shrinkage, water absorption and color values L, a, b) of the prepared ceramic tile were determined and the standard and new formulations were compared. It was found that the ceramic sludge consisted mainly of SiO2 and Al2O3 (about 80%) according to the XRF result and its particle size was below 32  $\mu$ m (91.27%) according to the sieve set tests. The shrinkage and water absorption values of the test pieces prepared using ceramic sludge (2-8 wt%) were calculated between 0,00-0,40 and 8.10-8.46, respectively. The L value of the formulations containing ceramic sludge (2-8 wt%) was found to be slightly higher than that of the standard formulation. The study shows that ceramic sludge from the wastewater treatment plant can be reused in commercial ceramic tile formulations in certain proportions (2-8 wt%).

#### 5. AGRADECIMIENTOS

The authors would like to thank Kaleseramik for this scientific research.

#### 6. **REFERENCES**

- M. C. Casagrande, A. P. Novaes De Oliveira, D. Hotza, A. Oliveira Da Silva, L. R. Alexandre, Recycling of Solid Wastes in The Production of Ceramic Floor Tiles, 2002, Qualicer, pos39-46.
- [2] E. Rambaldi, L. Valeriani, L. Grandi, C. Beneventi, M.C. Bignozzi, High-Recycling Content Porcelain Stoneware Tiles: From Industrial Production to Product Certification, Qualicer'18.
- [3] A. Eker, Ş. Türk, K. Kayacı, Recycling of Green Process Waste in Porcelain Tile, Qualicer'14.
- [4] M. Kavaklı, Seramik Sanayi Atık sularının Özellikleri, Arıtılması ve Kontrolü, IV. Seramik Kongresi Bildiriler Kitabı (Ed: S. Turan, F. Kara, ve E. Pütün), Türk Seramik Derneği Yayınları No: 20, Eskişehir, 111-118, 1998.
- [5] S. Kurama, A. Kara, H. Kurama, The Effect of Boron Waste in Phase and Microstructural Development of a Terracotta Body During Firing, Journal of European Ceramic Society, 24, 3177-3185, 2005.
- [6] A. Olgun, Y. Erdoğan, Y. Ayhan, B. Zeybek, Development of Ceramic Tiles from Coal Fly Ash and Tincal Ore Waste, Ceramics International, 31, 153-158, 2005.