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Uçucu Külün Endüstriyel Porselen Karolarda Katkı Maddesi Olarak Geri Dönüşümü

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Anahtar kelimeler
"Geri Dönüşüm"
"Uçucu Kül"
"Porselen Karo"
"Atık Değerlendirme"
"Pişirme Kabiliyeti"

Öz

Uçucu kül, termoelektrik santrallerde kömürün yanması sonucu ortaya çıkan bir atık türüdür. Silika baskın bileşimi sayesinde uçucu kül, fayans, çimento ve beton gibi inşaat malzemelerinin üretiminde bir katkı maddesi olarak kullanılabilir. Bu çalışma kapsamında uçucu kül tozunun porselen karo bileşiminde uygulanarak geri dönüşümünün sağlanması amaçlandı. Bu bağlamda, AKSA Akrilik Şirketi'nden elde edilen CE sertifikalı uçucu kül tozlarının kimyasal bileşimi XRF analizi ile belirlendi. Uçucu külün yüksek Si02 (>%70) ve Al203 (~%15) içeriği, porselen karo bileşiminde kullanımın önünü açmıştır. Standart porselen karo kompozisyonunun Seger formülasyonları ile ilgili olarak, ağırlıkça %2.5, 5, 7.5, 10 ve %13.5 uçucu kül tozları içeren yeni bileşimler geliştirildi ve bir bulamaç halinde elde edildi. Hazırlanan örnekler 1175 ° C, 1185 ° C, 1195 ° C ve 1205 ° C'de şekillendirildi ve sinterlendi. Sinterlenmiş numunelerde oluşan fazları belirlemek için XRD analizi yapıldı. Numunelerde oluşan fazların dağılımını incelemek için numunelerin yüzeyine ve kesitlerine SEM analizi uygulandı. TS EN ISO 10545 standardına göre su emme ve üç noktalı bükme testleri yapıldı. Analizlere göre, uçucu kül içeren bileşimlerin, karşılaştırılan tüm parametrelerde standart bileşimlere benzer olduğu bulundu. %5 uçucu kül içeren ve 1205 ° C'de sinterlenen numunenin porselen karo mekanik dayanımı için geçerli olan TS EN ISO 10545 standardına ulaştığı belirlendi.

Recycling of Fly Ash as an Additive in Industrial Porcelain Tiles

Keywords
"Recycling"
"Fly Ash"
"Porcelain Tile"
"Waste Valorisation"
"Sintering Ability"

Abstract

Fly ash is a type of waste generated by the combustion of coal in thermoelectric power plants. Owing to its silica-dominant composition, fly ash can be used as an additive in the production of construction materials such as tiles, cement and concrete. Within the scope of this study, it was aimed to achieve the recycling of fly ash powder by implementing it in the porcelain tile composition. In this respect, chemical composition of CE certified fly ash powders acquired from AKSA Acrylic Company was determined by XRF analysis. High SiO_2 (> 70%) and Al_2O_3 (~15%) content of the fly ash paved the way for the utilization in porcelain tile composition. In respect with the Seger formulations of the standard porcelain tile composition, new compositions were developed including 2.5, 5, 7.5, 10 and 13.5 wt% fly ash powders and yielded as a slurry. Prepared samples were shaped and sintered at 1175 ° C, 1185 °C, 1195 °C and 1205 °C. XRD analysis was performed to determine the phases formed in sintered samples. SEM analysis was applied to the surface and cross-section of the samples to examine the distribution of the phases formed in the samples. Water absorption and three-point bending tests were performed according to TS EN ISO 10545 standard. In accordance with analyses, it was found that the compositions containing fly ash were similar to the standard compositions in all the parameters compared. Sample containing 5 wt'% fly ash and sintered at 1205°C sample was determined to have reached TS EN ISO 10545 standart which is valid for porcelain tile mechanical strength.

14-16 Ekim 2019 tarihleri arasında düzenlenen X. Uluslararası Katılımlı Seramik Kongresi'nde sunulan bildirilerden seçilen çalışmadır.

1. Introduction

Economic growth in the world, technological development, industrialization, urbanization and population growth bring about consumption and waste to grow in quantity.(Altınışık, 2014) Recycling, which is one of the most important steps of waste direction recyclable wastes are converted into secondary raw materials by various physical or chemical methods and included in the production process.(Kazan et al., 2015) For this reason, recycling focuses on environmental maintainability depending upon the use of secondary raw materials and improves the economic system.(Geissdoerfer, Savaget, Bocken, & Jan, 2017)

Fly ash is a very small grain material that is found in thermoelectric power plants.(Ahmaruzzaman, 2010) (Guzmán-Carrillo, Pérez, Aguilar Reyes, & Romero, 2017)(Chakraborty, Maiti, & Pathak, 2009). They are glassy and mostly spherical particles ranging from 0.5 to 200 µm. Their specific surface area is around 2800 - 3800 cm2 / gr.(Görhan, Kahraman, Başpınar, & Demir, 2009). Because of fly ash has a heterogeneous structure, it contains many compounds. Essential components that contain fly ash; (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3) and calcium oxide (CaO), the amounts of which vary according to the type of fly ash.(Guzmán-Carrillo et al., 2017)(Olgun, Erdogan, Ayhan, & Zeybek, 2005)(Ahmaruzzaman, 2010)(Zimmer & Bergmann, 2007) According to the ASTM678 classification, there are two category of fly ash, namely class F and class C. The Class F is characterised by high amounts of total silica and alumina (70 wt% min), while Class C has a high lime percentage (20 wt% max). Both its physical and chemical properties depend on the type of coal used and combustion conditions.(Guzmán-Carrillo et al., 2017).(Görhan et al., 2009) Because of its fine and amorphous mineralogy, high (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3) content and amorphous structure, fly ash is a pozzolanic material, which is like a fine-grained natural pozzolan. Fly ash combines with calcium hydroxide $Ca(OH)_2$, silicon dioxide (SiO_2) and water to form calcium silicate (Ca_2SiO_3) in the structure and acquires binding properties in the structure. Because of its pozzolanic character and its chemical compositions almost identical to traditional ceramic raw materials, fly ash used in wall tiles, ground tiles, cement and porcelain production.(Görhan et al., 2009)

Traditional ceramics are products obtained by mixing and shaping inorganic raw materials such as clay, kaolin, feldspar, quartz, calcite, dolomite incorporating metal oxides at specific ratios and roasting at high temperatures.(Int Kyn. 1) Different types of ceramics can be produced having various physical and chemical characteristic for use in a wide range of usage areas such as washbasins, toilet bowls, reservoirs, bricks and tiles.(Cicek, Karadagli, & Duman, 2018). The production of 1 m2 of ceramic tile necessitates the use of 20 kg of raw materials, on average.(Zimmer & Bergmann, 2007). The size of the world ceramic tableware and decorative goods sector is about 15 billion US dollars.(Int Kyn 1) The consumption of world porcelain products is developing by approximately 5% per year on average.(Int Kyn 1)(Int Kyn 2) World tile production, in 2016, has exceeded the mark of 13 billion square meters and achieved 13.056 billion square meters. World tile consumption b(+ 5%) in 2016.(Int Kyn.3) (Int Kyn. 2). The increase in the consumption of traditional ceramic products together with the increasing population has occasioned the production capacity of these products to increase year by year and in this case resulted in the increase of the amount of raw materials used.

The use of fly ash in ceramic structure provides serious advantages in terms of the use of no cost raw materials, the conservation of natural resources and the assessment of waste.(Ferreira, Ribeiro, & Ottosen, 2003) A number of methods have been developed for the recycling of fly ash in the cement industry and the glass-ceramic industry.(Monteiro, Mota, Lima, & Regina, 2015). It is aimed to reduce the need for ceramic raw materials and to prevent the accumulation of fly ash wastes by using the fly ash in ceramic structure. The ratio of the raw materials in the traditional compositions used in traditional ceramic production has been reduced and fly ash has been used. The main aim of this study is to utilize the puzzolanic feature of fly ash, activating the fly ash binding property and increasing the strength of the structure as well as contributing to the fly ash recycling.

2. Materials and Methods

In this study, 4 different porcelain, wall and floor tile compositions, including varying amounts of fly ash added, were developed to investigate the structural effects of fly ash addition. Same fly ash was employed in each prepared tile composition. The amounts of fly ash used vary for each composition, so the amount used of other raw materials is reduced accordingly. Entering raw material amounts were recalculated respecting the Seger formulations of the standard compositions, in other words, total oxide amounts and ratios of the fly-ash including compositions were modificated similar to same of the standard composition. Used fly ash was obtained from Aksa acrylic company. AKSA Acrylic's Flesh Fly Ash waste has granted with CE certification, thus making it the only example in Turkey. The chemical composition of the fly ash was determined by X-ray fluorescence (XRF) analysis.

2.1 Preparation of porcelain ceramic tiles

Chemical analysis of fly ash resulted in 22,69% of the total amount of Al2O3 and 60,65% of SiO2. The silica acts as a binder at the same time providing fire resistance. The alumina provides heat stability in the structure and prevents cracks that may occur as a result of expansion. Alumina and Silica form the basic raw materials in the traditional ceramic structure and show similarity with fly ash in this sense.

In order to prepare the porcelain tiles, all the raw materials to be used in the structure were firstly dried for 24 hours in an oven operating at 100 ° C. The dried raw materials were weighed at certain ratios and a dry powder mixture of 400 g in total was formed and mixed. Water and deflocculant were introduced to the slurry to make dry slurry transferred to the ceramic bowl. Finally, alumina balls with a total weight of 354 g were added to the ceramic bowl to reduce the particle size of the slurry below 70 µm. The formed sample was milled for 13 minutes and turned into ceramic slurry. A pycnometer (TQC VF2097) was used to measure the density and the result was compared to the standard value for ceramic tiles (1648 - 1782 gr / lt). It was understood that the measured amount of density and grain size were at the desired level, as a result of the analyzes made. The resulting slurry was then subjected to deflocculant addition until it had the desired viscosity and thixotropic properties. In this rheological study, deflocculant was added until the slurry had sufficient fluidity. After each addition, the slurry was mixed at 740 rpm using a mixer (IKA RW 20 Digital) for 5 minutes, and homogenous distribution of the added deflocculant was achieved. After each mixing operation, the viscosity was measured using a viscometer (Brookfield Dial Reading). Since the standard porcelain tile should have a viscosity range of 4 to 6 Poise, the addition of deflocculant continued until this range was reached.

Porcelain tiles are more resistant than floor and wall tiles in terms of raw material types and proportions and sintering temperature. A higher sintering temperature (1200-1300 °C) reduces porosity in the structure. Porcelain tiles have low impurity content and high feldspar content. The feldspar forms vitreous phase with quartz in the sintering process. The amount of porosity in the structure is reduced by the glassy phase formed. Low porosity tends to reduce water absorption rate. According to American Material Testing and Materials (ASTM) C373 standard, the water absorption rate of porcelain tile is 0.5%. Porcelain tiles prepared using fly ash waste are similar to the standard composition in terms of microstructure and phase distribution. For this reason, the standard raw material quantities in the prepared porcelain tile prescriptions were removed at certain ratios and fly ash waste was added. By changing the amount of fly ash without altering the properties of porcelain tiles, 4 new compositions were prepared and the maximum added fly ash content was determined as 13.5%.

3. Results and Discussion

3.1 Rheological Study of Compositions

The oxide compositions and alkali ratios according to the content and structure of the compounds of the standard porcelain tile structure were obtained from the seger table.(Dondi, Ercolani, Melandri, Mingazzini, & Marsigli, 1999) The type and ratios of the compounds that make up the fly ash were determined as a result of XRF analysis and were compared with the standard porcelain tile composition. As a result of the composition analysis, it was found that the quantity of basic elements of SiO_2 and $Al2O_3$ that form fly ash was similar to the standard porcelain tile composition. When preparing porcelain tile with fly ash addition, it is very important to maintain the oxide and alkali content of the standard porcelain tile composition so as to be able to create the porcelain tile without changing its properties. At this point, using the seger table, a congruence was formed regarding the oxide and alkali ratios between the standard porcelain tile composition and the fly ash added porcelain tile composition. Sodium Silicate-Na_2O_3 deflocculant with 37% concentration was used in order to conduct the rheological studies on the prepared fly ash added porcelain tile slurry. During the rheological study, different amounts of deflocculant inlet was added order to keep the viscosity range of the liquid ceramic sludges with varying fly ash ratios within the standard porcelain tile viscosity range. The amount of deflocculant added is very important for the sludge to be brought to suspension.(Delavi, Noni jr, & Hotza, 2013) The addition rate to increase the viscosity value and its completion before reversing the suspension state is critical in terms of achieving the desired thixotropy and formation of the appropriate casting conditions. (Klüger, 2017). As a result of the rheological studies, it was observed that the porcelain tile sludge with varying amounts of fly ash was kept within the range of 4-6 Poise which was the desired viscosity range in the standard porcelain tile slurry.(Bernardin Adriano, Casagrande, Mariana, & Riella,2006). This showed that the type of deflocculant used in the porcelain tile composition with fly ash addition was suitable for the studies because the desired viscosity range was achieved. The resulting low viscosity increased the flowability of the glassy phase within the structure and ensured that the pores in the structure were closed and that the closed pores were spherical in form. The results of Scanning Electron Microscopy (SEM) analysis were included in the study to confirm this hypothesis. (Gil, Chiva, Cerisuelo, Carda, & Chemistry, 2006).

3.2 Phase and Microstructural Analysis

After rheological studies, the samples were shaped by unidirectional pressing. The fly ash added porcelain tile composition was sintered at 1240°C temperature after being shaped by pressing. Sintered samples were transformed into powder form for XRD analysis.

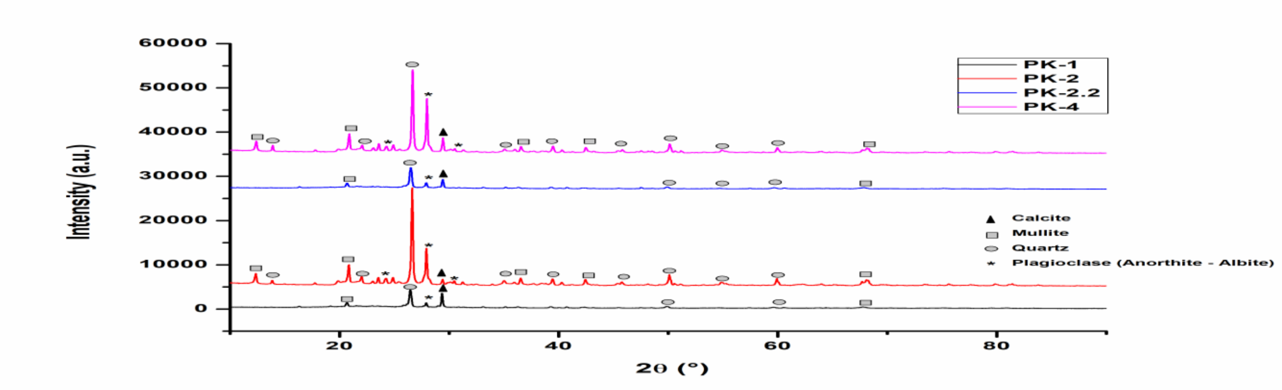

Fig. 1. X-Ray Diffraction patterns of fly ash-including compositions.

As a result of XRD analysis, four different phases were observed in the structure, namely Quartz, Mullite, Anorthite and Albite. Using the Seger table, the oxide and alkali ratios were calculated and this allowed the fly ash, which was added to replace the raw materials removed from the standard composition, to not cause any changes in the internal structure. Therefore, these four different phases of the standard porcelain tile composition were also seen in the porcelain tile composition prepared with the addition of fly ash. (Jorge Martín-Márquez, Rincón, & Romero, 2010) Due to the high SiO_2 (60%) component, the raw materials with high clay content added to the structure resulted in the formation of the Quartz phase as expected. The 23% Alumina present in the structure combined with the 60% SiO_2 and with the effect of high sintering temperature, formed the mullite phase. (Avcıata, 2003)(Sarkar & Mallick, 2018). The Na coming from the sodium oxide (Na_2O) in the prescription and Ca coming from calcium oxide (CaO) formed the plagioclase phase in the structure, which is the common name of the anorthite and albite phases. Of these phases, Quartz provided heat resistance in the structure and allowed the porcelain tile to resist high temperatures due to its covalent bonds in the internal structure.(J. Martín-Márquez, Rincón, & Romero, 2008) Mullite, on the other hand, causes an increase in the strength of the structure due to the interlocking caused by its needle-like structure. Albite, being a member of the feldspar family, melts before the other phases during sintering and enables the sintering of the liquid phase. In order to investigate the internal structure distribution of the phases seen in XRD analysis and to perform microstructure analysis to establish a basis for XRD analysis results, porcelain tile sample microstructures of sintered fly ash additions were examined under a scanning electron microscope (SEM) by sectioning. During SEM analysis, it was carefully examined the samples from their section instead of their surfaces in order to be able to fully understand the internal structure and to determine the pores in the structure.

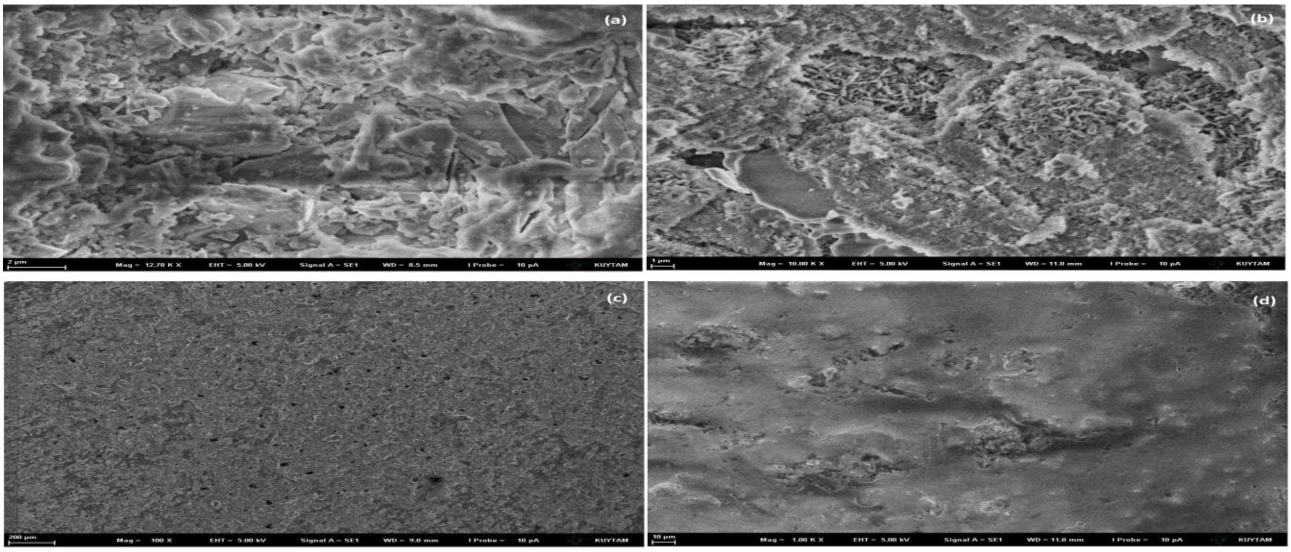

Fig. 2. SEM images from the cross-sections of PK-1 (a), PK-2.2 (b), PK-4 (c), PK-2 (d)

According to the analysis results, 4 different phases were encountered in the microstructure during XRD analysis. In accordance with the obtained microstructure images, it can be stated that the SEM analysis results confirm the XRD analysis results. In addition, it was observed that the porosities present in the examined microstructures were filled by the crystal forms inside the structure. This preserved theporosity in the structure. Preservation of porosity ratio causes effects such as increase in density of porcelain structure, positive change in thermal conductivity, decrease in water absorption rate and increase in the specific heat of the material. On the other hand, closing of the pores will cause a reduction in the shock resistance of the material. (Hua et al., 2016)

3.3 Mechanical Properties

The effects of the porosity rate, the sintering temperature of the samples and the fly ash content on the strength of the material were investigated. The fact that porcelain tiles are baked at higher temperatures than other types of tiles, and that the prepared compositions vary according to wall and floor tiles are factors that raise the mechanical properties of the porcelain tile to the next level. Within the scope of this study, 4 samples with different fly ash ratios (2.75% - 5% - 5%- 13%, respectively) were subjected to water absorption and 3 - point bending strength tests in order to determine the mechanical and physical properties of the porcelain tiles. The test results are given in the table below.

Table. 1. Mechanical properties of fly ash containing compositions.

According to the results given in the table, the samples with the same sintering temperatures had different values due to varying fly ash ratios and varying grinding times. According to these results; The PK-1 sample with a 2.75% fly ash ratio has the smallest grain size among the other samples due to the grinding time of 30 minutes. The decrease in the particle size makes the PK-1 sample have the highest surface area. Excessive surface area energy of the sample during sintering increases the fracture toughness by increasing the interactions between the grains. (Eren & Kurama, 2012) Decrease in the grinding times and respective increase in the fly ash ratios added to the prescription resulted in a decrease in strength. Although PK-2 and PK-2.2 samples contained the same amount of fly ash (5%), it was observed that the strength of the PK-2.2 sample was found to be lower than that of PK-2. The difference in strength seen between these two samples, which are similar in terms of the composition and sintering temperatures, can be explained by the duration of grinding. The PK-2.2 sample of that was grinded for 22 minutes had a grain size larger than the PK-2 sample that was grinded for 28 min. This situation reduced the inter-grain interaction within PK-2.2 during sintering and lead to a decrease in strength. The PK-4 sample contained 13.5% of fly ash and the grinding time was set to 23 minutes, increasing the ratio of raw material removed from the prescription and increasing the grain size, and consequently the desired mechanical properties in the PK-4 sample could not be achieved. In the evaluation of porcelain tiles, another test that is as important as the mechanical analysis is the water absorption test which is a type of physical analysis. In this test that aims to determine the amount of porosity in the cooked sample, the permissible water absorption value in porcelain tiles should be below 0.5%. Sintering of the porcelain tile at higher temperatures compared to the floor tile or wall tile ensures that the pores in the inner structure are closed and this decreases the water absorption amounts of porcelain tiles. (Vieira et al., 2017) Although the sintering temperatures and heating rates of the porcelain tile compositions were the same, varying results in water absorption values were encountered. The reason for this situation can be said to be due their different grinding durations. The fact that their grinding times were not the same resulted in a change in the grain size distribution and this resulted in a difference in the amount of porosity in the internal structure. When the results of the analysis are evaluated, it is seen that the results for all 4 samples were under the desired value, below 0.5%, signifying that the test was successfully conducted.

4. Conclusion

In the study, porcelain tiles which used in our lives were obtained via fly ash wastes to achieve various purposes. The first of these is that recyling of fly ash. Latter, worked to reduce of the amount of raw materials used. Thirdly, it was requested that the porcelain tile with additive material has the same mechanical properties as the standard porcelain tile. Used waste materials provided from AKSA Acrylic Company. The developed compositions containing fly ash wastes were prepared and sintered respecting existing production methods and the industrial standards. Water absorption values (0.1%) and mechanical strength (36.5 N/mm2) were within admissible acceptable industrial standards.

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