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Research Article

# THE EFFECT OF THERMAL POWER PLANT FLY ASH IN GRANITE BODY ON MICROSTRUCTURE AND TECHNICAL PROPERTIES

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### ABSTRACT

*In this study 2-5-10 wt.% thermal power plant ash was added to the granite body replace feldspar to use environmental waste in the ceramic body and to reduce production costs. Loss on ignitions, fired bending strengths, dry strength, fired shrinkages, water absorption and colorimeter degrees were measured. Microstructure was determined by scanning electron microscope (SEM), energy dispersive x-ray spectrometer (Edx) and x-ray diffractometer (XRD) measurements. After sintering, it was determined that 2 wt.% fly ash can be used in granite body. In sintered body, according to XRD phase analysis, amount of free quartz and mullite decreased and albite formation was observed. While the fired strength value is 399 kg/cm<sup>2</sup> in standard body, it is 315 kg/cm<sup>2</sup> in 2 wt.% fly ash added body. In the experiment with 2 wt.% ash additive, according to SEM images, the number and volume of large pores increased while small pores decreased. The ability of pressing of the sample with 10wt.% ash additive was negatively affected due to the excess amount of fine ash and so occur lamination and bloating.*

**Keywords:** Fly ash, granite, sintering, feldspar, microstructure.

## **1. INTRODUCTION**

Reuse of waste is very important for conservation of natural resources and a livable environment. In the ceramic industry, many solid wastes are used in ceramic bodies. Some of these are the use of granite cutting wastes in industrial porcelain bodies (Luz and Ribeiro, 2007), use of sewage waste in ceramic floor tiles (Amin et al., 2018), use of fired wall tile's scraps in floor tile body (Elmas, 2019), preparation of ceramic tiling from blast furnace slag (Ozturk and Gultekin, 2015), processing of unglazed ceramic tiles from blast furnace slag (Ozdemir and Yilmaz, 2006), effect of fly ash on the properties of ceramics from steel slag (Zong et al., 2018).

Coal-fired thermal power plant's fly ash waste with worldwide annual cause 600 million tons, 13 million tons in Turkey. (Türkiye'deki Uçucu Küllerin Sınıflandırılması ve Özellikleri dergisi, 2009). Many studies on the use of fly ash are available in the literature. The study made by Koçkal (2) was used fly ash replace feldspar according to 5-10-15-20 w.%. The usage of fly ash in big quantities at high temperature is seen bloating in the body. With increasing ash amount, fired shrinkage decreases. The fired strength value is increased up to 10% ash addition in 1190 °C firing. The water absorption value increases up to 10% in fly ash and then decreases. At the sintering temperature, the shrinkage rate decreases with increasing ash content. In the same study, when the amount of ash was increased up to 10%, the amount of water absorption in the body increased. Trnik et al. in a study that contain clay, grog and 20 wt.% of the ash, the elastic modulus of the sample was found to increase rapidly at increasing temperatures where fired shrinkage value is 24%. According to Figen et al. in the study on the use of ash in tile products with fired tile waste, it was found that the use of ash at a rate of 5 wt.% contributed positively to the physical and mechanical properties. Dry strength value and dry shrinkage value decrease with increasing amount of ash. In the addition of 5wt.% ash, the reduction in firing shrinkage in firing at 900-950-1000-1025 °C was found to be less than in the sample without ash. Dana et al. in a study made by kaolin-quartz and feldspar system, quartz was replaced with ash in 5-10-15 wt% and sintered at 1150 -1300 °C. An increase in the density of the ash-containing sample was observed at all temperatures. While the maximum bending strength was found to be 70.5 MPa at 15% ash content at 1300 °C, the fired strength of standard porcelain body is 61.1 MPa. In the XRD measurement, more mullite was observed in the ash containing sample. In SEM measurements, 25-50 micron quartz grains and secondary mullite needles were observed in the glassy matrix. In the study on the production of ceramic tiles with high alumina content from coal fly ash, it was found that the body, which contains 60% fly ash and 4% quartz, has a breaking strength of 51.28 MPa at 1200 °C ( Ji et al, 2016). In the same study, water absorption, apparent porosity and linear drawing values were found to exceed the desired value for porcelain tiles. In the study on producing ceramic wall tiles from blast furnace slag, the trial of 33% blast furnace slag has been of high fired strength (Ozturk and Gultekin, 2015). The addition of a high calcium glassy phase structure led to the growth of anortite crystals.

In this study, instead of feldspar, 2-5-10 wt% coal thermal power plant fly ash was used; granite body prepared was fired under industrial rapid firing conditions. Physical properties such as dry strength, fired strength, water absorption, fired shrinkage were examined. At the same time, their effects on microstructure were determined by SEM images, XRD, Edx measurements. In this study, it is aimed to reduce the amount of feldspar by replacing feldspar with 5-10-15% of thermal power plant fly ash in the granite body in the fast firing furnace that makes continuous production and also to obtain positive physical and mechanical properties.

## 2. EXPERIMENTAL PROCEDURE

Kaolinite, albite, quartz, clay and coal thermal power plant ash were used in the formulation of the granite body. Chemical analyzes of raw materials are shown in Table 1. The mixture was grinded at a density of 1650 g / lt in wet grinding of 2 kg laboratory type cylindrical mills to be 3-4% above 63 microns. Prepared recipes are shown in Table 2. They were dried for 1 day at 110 °C and prepared to be pressed at 5-6% humidity. After that samples in dimensions 7x210x100 mm was pressed 380 kg/cm<sup>2</sup> in a laboratory-type press. Samples are fired at 1180<sup>0</sup>C in 63 min. industrial continuous kiln. Dwelling time at maximum sintering temperature is 3 min 20 s.

**Table 1.** Chemical analysis of used raw materials (wt.%)

Raw Materials	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SO <sub>3</sub>	L.I.
Clay	54	31.8	0.13	2.03	0.3	0.45	0.67	1.17	-	10.05
Kaolinite	51	34	0.15	1.20	0.20	0.25	0.75	0.25	-	12
Na-Feldspar	68.62	19.53	10.29	0.21	0.99	0.10	0.016	0.042	-	0.14
Quarz	99.35	0.12	0.05	0.06	0.05	0.05	0.06	0.01	-	0.2
Fly ash	18.3	10.02	-	0.15	32.67	1.06	4.6	0.6	18.72	8.05

L.I: Loss on ignition

**Tablo 2.** Prepared granite body formulations (wt.%)

	1	2	3	4
Kaolinite	25	25	25	25
Albite	30	28	25	20
Quarz	5	5	5	5
Clay	40	40	40	40
Fly Ash	0	2	5	10

The fired strength is made in 3 point gabrielli (italy) test machine. The formula used in the calculation is  $\sigma_f = 3 \times F \times L / 2 \times b \times h^2$

where  $\sigma_f$ , F, L, b and h respectively are fracture strength (N/mm<sup>2</sup>), breaking load (kg), distance between supporting bars (mm), tile width(mm), tile thickness(mm) .

The water absorption test (W) was performed according to the international standard (ASTM C 20) .

$$W(\%) = (W_s - W_d / W_s) \times 100$$

where W<sub>s</sub>, W<sub>d</sub> respectively wiped surface water saturated weight, dried weight.

The liner shrinkage is measured according to formula

$$L (\%) = (L_1 - L_2 / L_1) \times 100$$

where L<sub>1</sub> and L<sub>2</sub> in order of measured length of green and fired tile samples. Color measurements were measured on a fired body with a Minolta Chroma Meter. L, a, b values indicate lightness, redness and yellowness respectively. The phases in the ceramic body were measured with XRD Pan Analytic Empyron Series 45 Kv, K alpha. Microstructure photographs

were measured in SEM-JEOL JSM-7100 F and Edx in Oxford Instruments x-max quorum with 1 mbar / Pa, 10mA, Au / Pa (80-20%) coating.

## RESULTS AND DISCUSSION

### 3.1. Physical properties

There is no flow feature in 2-5-10 wt.% ash additions in slip with a density of 1650g/lit. Standard slip flows for 40 seconds in the fordcup. This flow characteristics cause problem of decharging of mill. The fired shrinkage of the body decreased in ash amount up to 5% and increased in ash amount up to 10%.

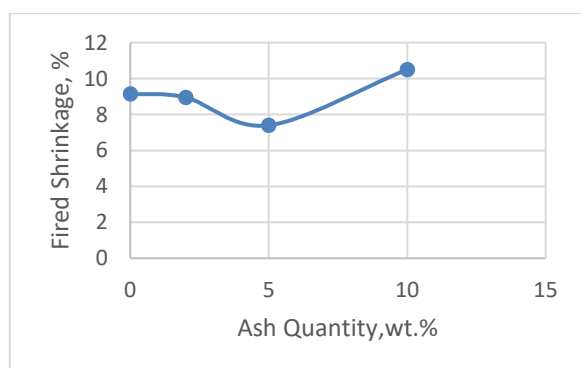
Water absorption value increases with increasing ash amount. It can be seen from the SEM analysis that the reason for this is due to the very fine grain size of ash, wide pore size distribution due to high carbon and volatile material content after sintering and the relatively increased open pores. High fired losses and short firing times are thought to cause this. The reason for the decrease in the water absorption value of Koçkal's study is that the fired loss value is as low as 0.77wt.%. In addition, amount of  $K_2O$  and  $Fe_2O_3$  that gives fluxing characteristics, amount of  $Al_2O_3$  and  $SiO_2$ , increase in  $Al_2O_3 / SiO_2$  ratio has an effect on the melting properties.  $Al_2O_3 / SiO_2$  ratio  $> 1/10$  show to non-melting or semi-melting behavior (Erciyes) . Ash's ratio  $Al_2O_3 / SiO_2$  is 0.58 that value is high that the reason of refractory properties.

Loss on ignition increases with increasing ash amount. This is due to carbon and other gas impurities in the ash. High fire loss is a negative feature for fast firing bodies (Fortuna, 2000).

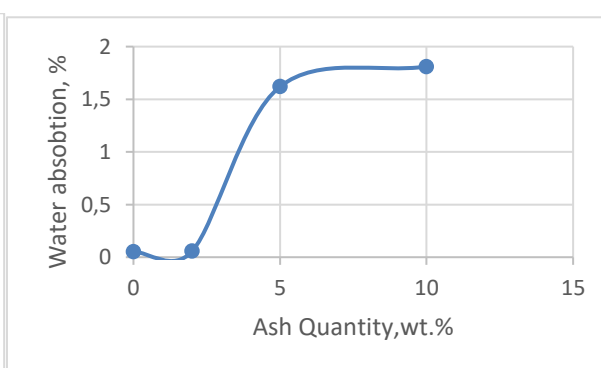
The fired strength value decreases with increasing ash amount. The reason for this is thought to be the large pore size distribution within the body that generate from high loss of igniton, insufficient flux properties of ash, also show similar characteristics of chamotte, high  $Al_2O_3 / SiO_2$  ratio that directly effect on melting , reduced mullite content and specially short dwelling time. The sample containing 10% ash showed swelling. Similarly in the study of Koçkal (2012) in place of 20 wt.% fly ash instead of feldspar at 1190 °C bloating was seen in the sample. The increase in pressing pressure increases the strength and makes the gas output difficult. Therefore, high ash content swelling was observed in the study of the Koçkal's study. The reason of increase of fired strength of Koçkal's study is high soaking time of firing schedule, high  $Fe_2O_3$  content that gives fluxing characteristics.

The dry strength value tends to decrease with increasing ash content (Figen et al., 2017), it is seen that dry strength value decreases with increasing amount of ash. This is due to the loss of compressibility due to the very fine ash grain size that cause lamination the body (Koçkal,2012).

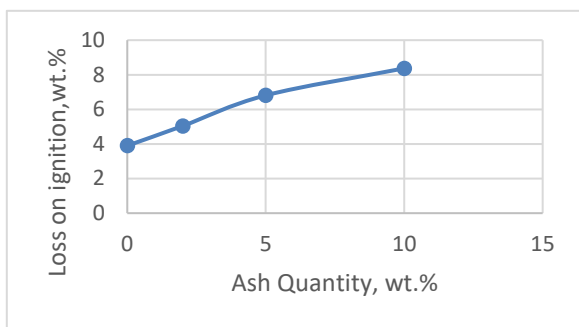
**Figure 1.** Fired shrinkage- ash wt.%



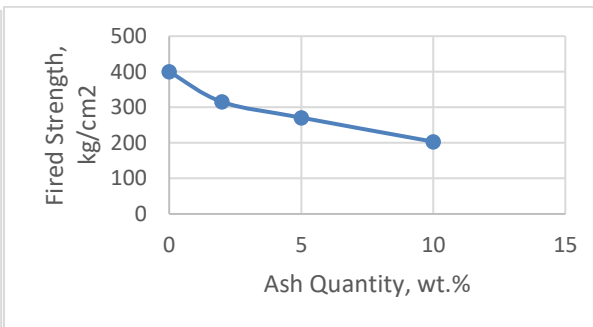
**Figure 2.** Water absorption-ash wt.%



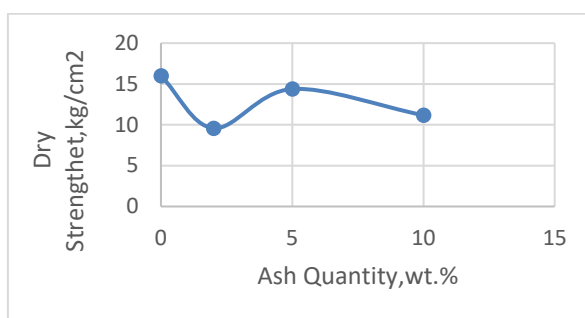
**Figure 3.** Loss of ignition-ash wt.%



**Figure 4.** Fired strength-ash wt.%



**Figure 5.** Dry strength-ash.wt.%



**Table 3.** Colorimeters values of samples

Sample	L	a	b
1(Std)	47.30	5.36	13.82
2	51.37	3.46	8.38
3	47.75	3.91	15.31
4	47.06	4.13	12.03

Std: Standard

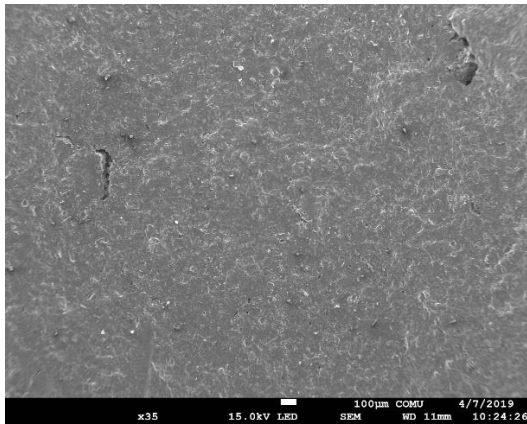
In the ash addition of 2%, the L value increased slightly, which means that lightness increased, a redness and b yellowness values decreased. In further ash additions, the L value is close to standard. The yellowness value b increases up to 5% in the samples containing ash, then decreases. The b value of the body with 2% ash added was significantly reduced.

### 3.2. Scanning Electron Microscopy Analysis (SEM)

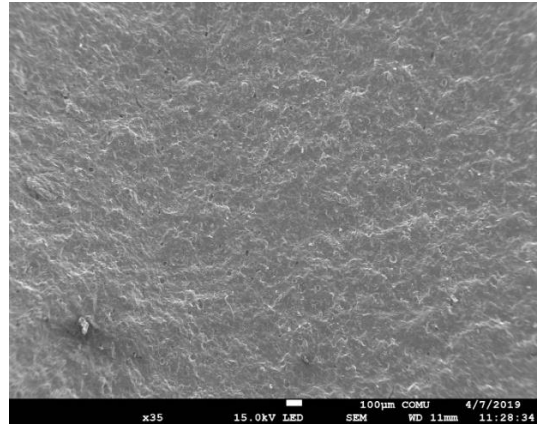
There is a decrease in the number and size of pores in the ash addition structure as seen from SEM analysis. While there are small pores and narrow pore size distribution in the study, Koçkal's study on the subject shows that although it has a low fired loss in ash doped body, larger pores and a larger pore size distribution are observed. This is due to the fact that the melting oxide compounds are high in the study of Koçkal and prevent the release of gas within the body.

**Figure 6.1.a,b,c,d,e** Sem images of fracture surface of standard body

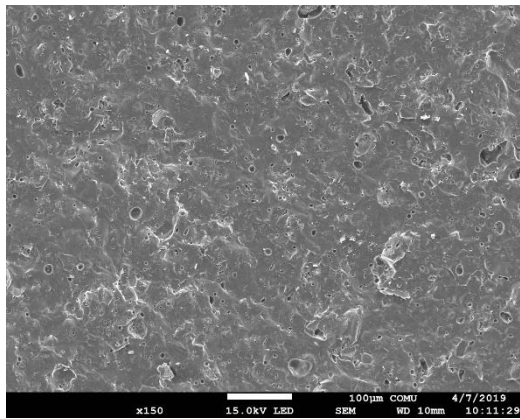
**Figure 6.2.a,b,c,d,e** Sem images of fracture surface of 2.wt% ash added granite body



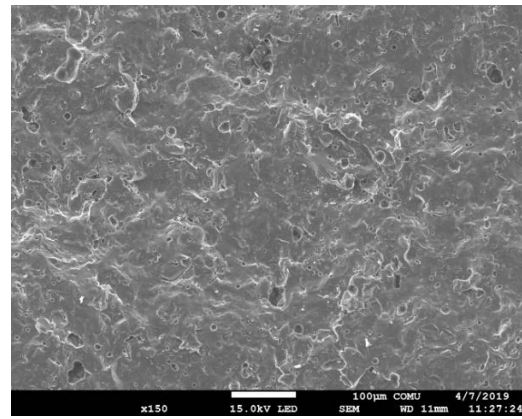
**a**



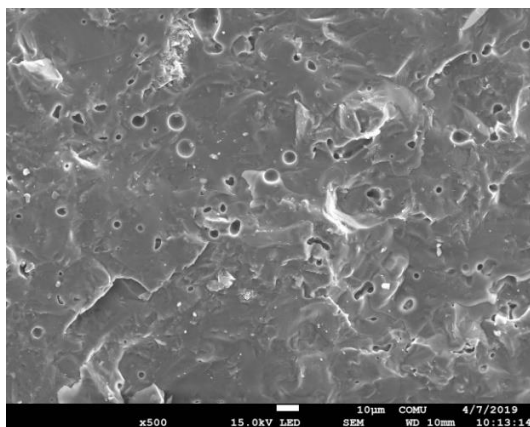
**a**



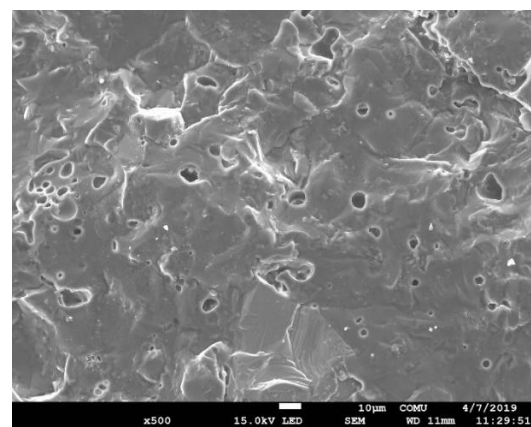
**b**



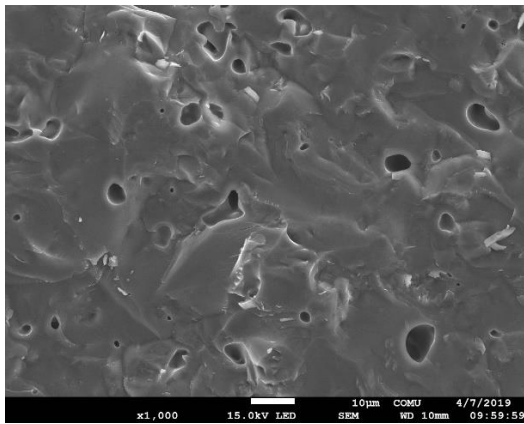
**b**



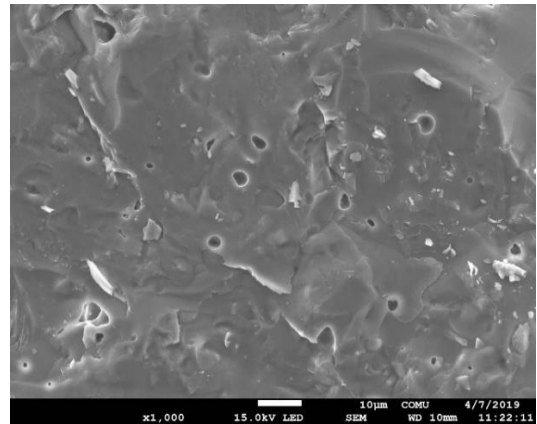
**c**



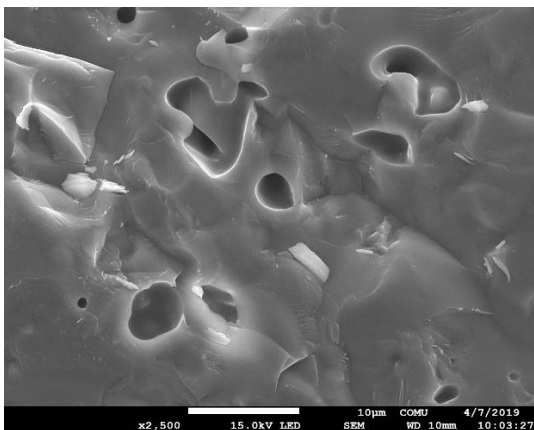
**c**



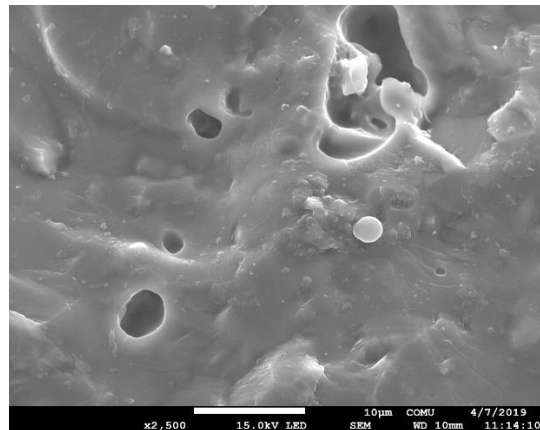
**d**



**d**



**e**

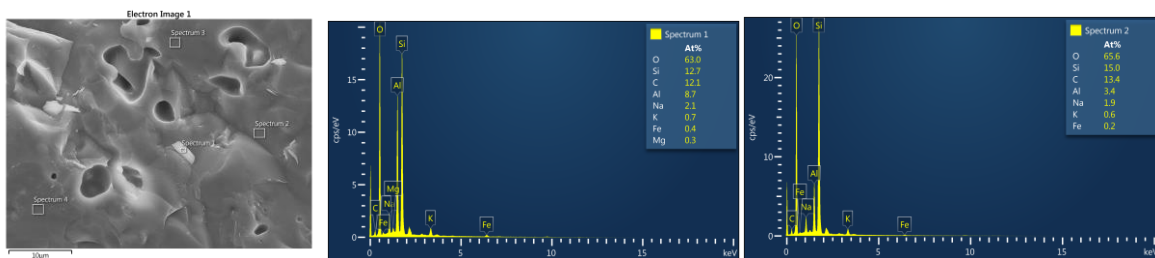


**e**

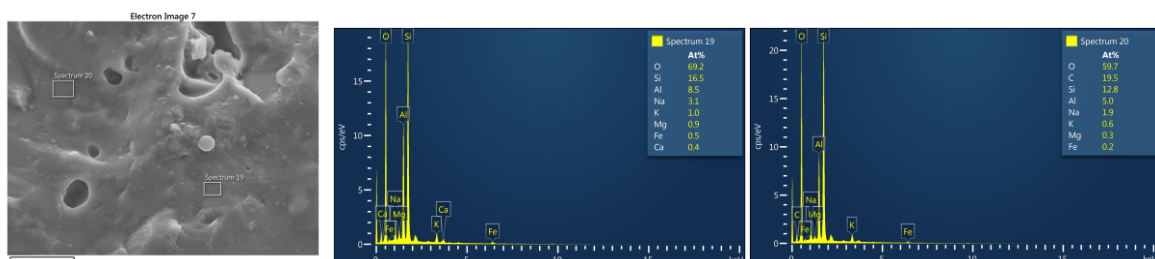
### 3.3. Energy Dispersive X ray Spectroscopy (Edx) Analysis

In the edx analysis of the ash-containing body, components such as C and Ca are seen in the body. At the same time, it has been observed that carbon-containing impurities are in particulate form due to lack of good mixing and grinding. The varying amounts of elements at different magnifications indicate that there is no homogeneous mixture.

**Figure 7.** Edx analysis of standard granite body

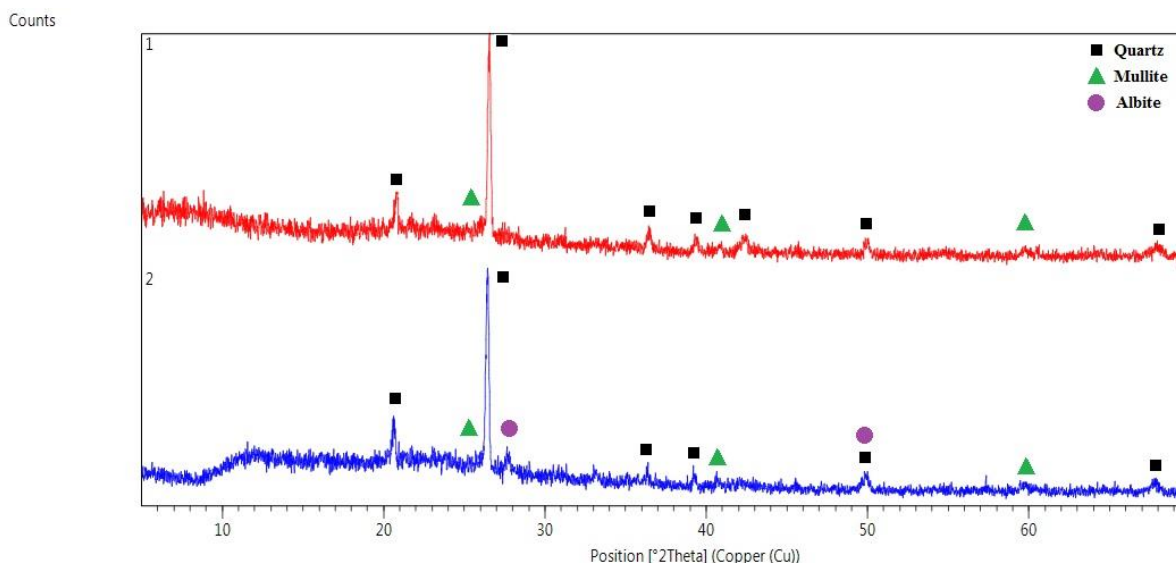


**Figure 8.** Edx analysis of 2wt.% ash added granite body



### 3.4. X-ray Diffraction Pattern (XRD) Analysis

Figure 9. XRD analyzes of standard (1) and 2 wt.% ash added granite body (2)



While the standard body (1) contained 77.2% quartz and 22.8% mullite, the amount of with 2% ash added (2) was 41.5% quartz, mullite 12.2% and albite 46.3%. CaO content changed the activity, triggered quartz transformation (Richerson, 1992); thereby increasing quartz solubility. This changing chemical activity reduced mullitization reactions and triggered albite formation. Luo et al.(2017) study at 1150 °C, it was observed that albite was formed in addition to mullite and quartz in ash-tempered ceramic structure. In the study of Koçkal (2012), quartz, cristobalite, hematite and plagioclases (albite, anorthite) were observed at 1190 °C in ceramic with thermal power plant ash. Viscosity and chemical reactivity of the liquid phase affect the mullitization reactions.

### 4. CONCLUSION

2% thermal power plant ash was found to be usable in granite structure. Acceptable physical properties were obtained for the standard body. L, lightness value increased slightly in 2% sample and red and blue value decreased. When the microstructure was examined, quartz and mullite were 77.2% and 22.8% respectively in the standard structure, whereas quartz and mullite amount in the 2% ash doped sample was 41.5% and 12.2%, respectively. Albite was observed in 46.3% of the sample with the same ash addition, albite formation reactions were triggered. This shows that chemical reactivity of the liquid phase change significantly. Chemical analysis of ash, SiO<sub>2</sub>/ Al<sub>2</sub>O<sub>3</sub> ratio, amount of melting oxide such as Na<sub>2</sub>O and Fe<sub>2</sub>O<sub>3</sub> and gas output quantity affect ash properties, usage area and amount of ash.



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