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Vermikülit (Yıldızeli/Sivas) İlavesinin Vitrifiye Seramik Malzemelerin Özelliklerine Etkisinin Araştırılması

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ÖZ

Sıhhi gereç ürünleri bir banyo veya tuvalet içine yerleştirilmiş klozetler, lavabolar, ayaklıklar, pisuarlar ve küvetler gibi eşyaları tanımlamak için kullanılan bir terimdir. Çok parlak, leke tutmaz yüzeyleri hem banyo hem de mutfaklarda kullanım için idealdir. Hidromika grubundan olan bir mineral vermikülit, 300 °C'nin üzerinde ısıtıldığında genleştirilmiş vermiküllit oluşur. Genleştirilmiş vermikülit, benzersiz özellikleri, düşük kütle yoğunluğu, düşük termal iletkenliği, nispeten yüksek erime noktası, kimyasal kararlılığı, dayanıklılığı ve çevre güvenliği nedeniyle ısı yalıtım malzemeleri için dolgu maddesi olarak kullanılabilir. Bu çalışmada, Yıldızeli/Sivas bölgesinden elde edilen vermikülitin sıhhi gereç ürünlerinin bünye özelliklerine etkisi araştırılmıştır. Bu çalışmada kullanılan malzemeler kalsine vermikülit, kuvars, kil, kaolen ve feldspattır. Hazırlanan sıhhi gereç bünyeleri ağırlıkça %0, 10 ve 20, 1050 °C'de 1 saat kalsine edilmiş vermikülit ilave edilerek bünye kompozisyonları oluşturulmuştur. Numuneler kuru pres ile şekillendirildikten sonra sinterlenmiştir. Sinterlenen numunelere fiziksel ve mekanik testler uygulanmıştır. Vermikülit katkılı numunelerin özellikleri başarıyla geliştirilmiş ve optimum parametreler açıkça gösterilmiştir. Elde edilen sonuçlara göre vermikülit katkısı sonucu su emme ve porozite değerleri azalmış, bulk yoğunluk ve pişme küçülmesi değerleri artmıştır. Vermikülit katkısı sonucu mekanik olarak sertlikte ve 3 nokta eğme mukavemetinde de iyileşme gözlenmiştir.

Investigation of the Effect of Vermiculite (Yıldızeli/Sivas) Addition on the Properties of Sanitaryware Ceramic

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ABSTRACT

Sanitary ware products are a term used to describe items such as toilets, sinks, pedestals, urinals, and tubs built into a bathroom or toilet. Its highly glossy, stain-resistant surfaces are ideal for use in both bathrooms and kitchens. Expanded vermiculite is formed when a mineral from the hydromica group, vermiculite, is heated above 300 °C. Expanded vermiculite can be used as a filler for thermal insulation materials due to its unique properties, low bulk density, low thermal conductivity, relatively high melting point, chemical stability, durability, and environmental safety. In this study, the effect of vermiculite obtained from Yıldızeli/Sivas region on the structural properties of sanitary ware products was investigated. The materials used in this study are calcined vermiculite, quartz, clay, kaolin and feldspar. Body compositions were formed by adding 0, 10 and 20 weight percent vermiculite, calcined at 1050 °C for 1 hour, to the prepared sanitary ware bodies. The samples were sintered after being shaped with a dry press. Physical and mechanical tests were applied to the sintered samples. The properties of the vermiculite added samples have been successfully developed and the optimum parameters have been clearly demonstrated. According to the results obtained, water absorption and porosity values decreased, bulk density and firing shrinkage values increased as a result of the addition of vermiculite. As a result of the addition of vermiculite, an improvement was observed in mechanical hardness and 3-point bending strength.

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1. Introduction

Sanitaryware is used in bathroom or restroom as example toilet, washbasin, pedestal, bidet, urinal. Sanitary ware mostly consists of clay, kaolin, feldspar and quartz but also minor amounts of other raw materials. The high-gloss, stainproof surface is perfect for bathroom and kitchen applications. The cast slip is formed of the aforementioned basic ingredients and cast to produce a green body into plaster molds. It is demonstrated that ceramic sanitarywares are financially savvy and valuable for the since a long time ago run. Sanitarywares can withstand in excess of 400 kg load and astounding protection from synthetic assaults. Sanitaryware things can be handily cleaned on account of its lustrous surface properties. Artistic materials are generally utilized in an extraordinary assortment of modern applications going from the creation of sterile products to superior exhibitions mechanical components. Due to their high mechanical performance, chemical/thermal stability and post-glass impermeability, both the manufacturing and finished products have been employed with success. Various pore forming materials used in sanitary body have been widely utilized for thermostat decomposition and volatilizing (for example wood-sawdust, polystyrene, organic wastes, coal dust, powder-calk and sludge) and thermal pore materials (for instance pumice, diatomite and perlite). Vermiculite is a substance which may be utilized in this way as a pore former (Sasipriya et al., 2013; López and Rodríguez, 2015; Knies et al., 2016; İssi et al., 2017; Tarhan and Aydin, 2017; Kurama and Sari, 2019; Martini et al., 2021).

The name "vermiculite" is gotten from the Latin – vermicularis (twisted) due to the twisted, draw out and wound fragments made when the valuable stones are startlingly introduced to high temperatures. Vermiculite is a mica-like mineral with a shimmering drop which is one person from the phyllosilicate bundle. Vermiculite develops when warmed up to 650–950 ℃. Exactly when vermiculite warmed, it develops, like "popcorn", by around 8–30 times its special size and converts into a free, lightweight part that detached through air entrainment. Vermiculite is a usual collection of hydrated silicates Al-Fe/Mg (mica-like) with a light-brown/dark/gold shading structure. Vermiculites are, moreover, a very flexible mineral with excellent characteristics like low size, odourlessness, inactivity, ease of care, low warm coefficient, high acoustic and warm protection, limits for assimilation, non-burning material and a resistant nature. It's rock like material that is mined, crushed and warmed to more than 300 ℃ at a distance of 210 mm to remove or extend in size. Vermiculite is required in its exfoliated state in the majority of applications. Vermiculite is a very promising material to use as a filler in the production of heat insulating refractory material with low density and thermal conductivity and a high melting temperature. Substances consist largely of SiO_2 (37-42 wt.%), MgO (14-12 wt.%) and FeO (1-3 wt.%) and FeO (5-13 wt.%), Fe_2O_3 (5-17 wt.%), H_2O (8-18 wt.%), respectively. Due to the findings of the investigations, it appears possible to utilize vermiculite as a construction material. Due to its outstanding thermal, fire and sound insulation qualities, it may also be utilized as an aggregate in lightweight concrete and plaster. According to Ngayakamo and Park (2009), Kalalani vermiculite may be used to make high-strength porcelain insulators as a raw material (Spirina and Flerova, 1975; Suvorov and Skurikhin, 2002; Rashad, 2016; Önen et al., 2017; Ngayakamo and Park, 2019; Tapik et al., 2019).

The present paper is focused on sanitaryware, and vermiculite added sanitaryware samples. Then, the effect of vermiculite additive on the properties (shrinkage, water absorption, bulk density, porosity, 3-point bending strength, hardness, macro images, phase analyses and microstructure) of sanitaryware was investigated. This is the first study to examine the addition of vermiculite to the sanitary ware recipe. The results regarding the possibility of vermiculite as a raw material or additive for sanitary ware have been presented to the literature.

2. Materials and Methods

Calcined vermiculite, quartz, clay, kaolin, and feldspar ceramic raw materials were used in this investigation. An acceptable recipe for sanitarywares used in the ceramic sector was created based on the results of the chemical analysis. In an electric furnace, raw vermiculite acquired from Organic Mining in the Yıldzeli/Sivas region was calcined in 1 hour at 1050 ℃. Sanitaryware compositions were made in the ternary equilibrium diagram kaolin-feldspar-quartz (Ece Banyo and Refsan) according to the required recipe from the raw material. Table 1 shows the results of XRF (Thermo ARL) analyses of raw materials. All mixtures were combined wet in alumina ball mills at 60 rpm rotation speed for 24 hours at the ratios listed in Table 2. To create the body compositions, calcined vermiculite was added to Sanitaryware bodies in percentages of 0, 10, and 20 by weight. S20V1100 (S: Sanitaryware; xx% vermiculite addition; T: firing temperature) was the code for the samples. The homogenized mixtures were dried and shaped into 10x30x70 mm dimensions under 100 MPa pressure by uniaxial dry pressing. 5 samples were prepared for each experimental test. The prepared pressed samples were then fired for 1 hour at 1050 ℃, 1100 ℃, and 1150 ℃. The sanitaryware bodies were then tested for microstructure (SEM), phase analysis (XRD), thermal test (DTA/TG), mechanical (hardness, 3-point bending), and physical properties (percent shrinkage, water absorption, porosity, and density).

Table 1. The chemical composition of the raw. calcined vermiculite and sanitaryware powders.

%w	Raw vermiculite	Calcined vermiculite	Feldspar	Kaolin	Clay	Quartz
SiO_2	36.90	40.61	68.60	46.50	51.26	97.67
Al_2O_3	17.70	19.48	17.72	28.91	19.87	0.92
TiO_2	2.18	2.40	0.25	0.15	1.13	0.00
Fe_2O_3	11.20	12.31	0.18	1.58	6.26	0.22
CaO	3.54	3.90	1.48	0.62	0.39	1.02
MgO	16.40	18.05	0.80	0.52	0.61	0.00
Na_2O	0.15	0.17	10.60	0.22	0.15	0.00
K_2O	2.64	2.91	0.16	1.17	2.60	0.00
MnO	0.15	0.17	0.02	0.03	0.00	0.00
LOI	9.14	0.00	0.19	20.30	17.73	0.17

Table 2. Codes and ratios of prepared sanitaryware- vermiculite mixtures.

	Vermiculite % w.	Clay % w.	Kaolin % w.	Feldspar % w.	Quartz % w.
S00V	---	20	25	35	20
S10V	10	18	22.5	31.5	18
S20V	20	16	20	28	16

A digital calliper was used to measure and determine the percent shrinkage of sintered samples. The Archimedes concept was also used to calculate density, porosity, and water absorption tests (ASTMC373-88). The samples were tested for 3-point bending strength on a mechanical tester with a load sensitivity of 1 N and a power of 5 kN. For each test item, five measurements were taken, and the average result was used to determine the test piece's strength. To assess the processes occurring in the body, DTA / TG analysis of samples up to 1100 ℃ temperature was performed using a 10 ℃ / min heating rate. The samples' colour analysis was done with the naked eye in visible light. The changes in colours have been detected as increasing/decreasing darkening and colour differences as additive ratios and firing temperatures increase. Following the 400, 800, 1200, and 2000 grid sanding processes, the samples were polished on a velvet baze with a 1 m diamond solution. On an Alfred Amsler & Co brand Vickers tester, diagonal field marks were made on polished samples using a square pyramidal diamond tip with a vertex angle of 136° under a pressure of 1 kg and 2 kg. Five measurements were conducted to calculate the hardness values, and the results were averaged (Çitak and Boyraz, 2014; Önen and Boyraz, 2014; Sacli et al., 2015; Boyraz and Akkus, 2021). The materials were analysed using a Panalytical X'Pert Powder X-ray diffraction (XRD) Analyser in the 40 to 70 2-theta range. The Pananalytical X'Pert High Score application was used to determine the phase analysis of XRD patterns. Mira3XMU FE-SEM was used to examine the samples using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX) (Tescan, Czech Republic). The information gathered was presented in graphs and tables along with comments.

3. Results and Discussion

DTA/TG graph of the sanitaryware sample shown in Figure 1, it is seen that free water has moved away in the temperature range of 25-200 ℃. The loss measured in this temperature range is about 0.2% by weight. Hygroscopic water flows away from the body in the range of 200-400 ℃ and the loss was measured as 0.2% by weight. The loss seen in the TG curve in the 400-600 ℃ range is due to the loss of crystal water by the clay and kaolin in the sample, respectively, and the weight loss was measured at 4.6% in this range. The reason for the endothermic peak seen in the DTA curve in the range of 500-600 ℃ is due to the fact that the water in the kaolinite structure moves away from the structure and turns into meta kaolinite form (Djangang et al., 2007). There was 6% weight loss in the sample in total. The second peak observed at 980 ℃ belongs to the structure formed by mullite crystallization (Romero et al., 2006). After 1000 ℃, there is no weight loss and no DTA peak in the system.

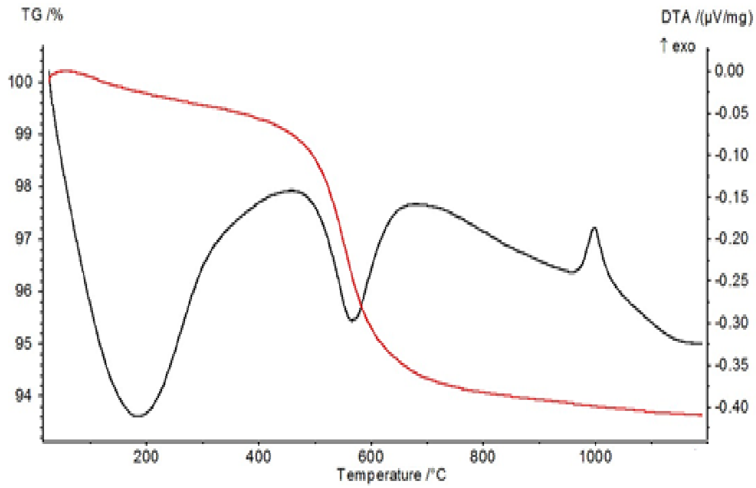


Figure 1. DTA-TG curve of the non-additive sanitaryware body used in experimental studies.

Table 3 shows the findings of physical measures (water absorption, bulk density, porosity and shrinkage). The results showed that as the sintering temperature was raised, the bulk density, shrinkage, and bending strength values increased. As the sintering temperature was raised, the porosity and water absorption values dropped.

According to Table 3, the addition of vermiculite decreased the water absorption and porosity values of the vitrified body. It also increased firing shrinkage and bulk density values. It is thought that this situation arises from the glassy structures formed by the melting of alkali oxides such as potassium oxide and sodium oxide in the chemical composition of vermiculite. It is seen in the results that the sintering is better than the unadded body as a result of the melting of alkali oxides at low temperatures (Tapik et al., 2019). The increase in sample values continued with the increase of sintering temperature. It has been observed that the transformation of the mullite phase at high temperatures also increases the strength values of the body (Romero et al., 2006). At the same time, as a result of the addition of vermiculite, magnesium silicate-based ringwoodite phase was also crystallized in the added samples.

Table 3. Physical and mechanical test results of sanitaryware and vermiculite added sanitaryware samples.

Sample codes	Water Absorption	Bulk density	Porosity	Shrinkage	Bending Strength	Hardness
	%	gr/cm^3	%	%	MPa	Hv
S0V1050	14.91	1.91	30.70	0.42	4.79	213.00
S0V1100	14.31	2.06	27.26	2.10	9.35	250.00
S0V1150	3.94	2.20	8.90	6.52	24.70	420.00
S10V1050	13.46	1.96	26.34	1.40	13.15	257.00
S10V1100	8.09	2.14	17.27	3.70	29.51	285.00
S10V1150	0.42	2.26	0.27	6.95	36.58	445.00
S20V1050	13.33	1.99	26.30	1.57	14.98	269.00
S20V1100	7.19	2.17	15.61	4.80	35.56	325.00
S20V1150	0.31	2.45	0.69	7.25	46.20	460.00

When the colour analysis results of the samples are examined in Figure 2, the brownish colour increased from light to dark with increasing vermiculite. Similarly, the colour became darker with the increase of sintering temperature. It is thought that this situation is caused by the amount of iron oxide in the chemical composition of vermiculite.

Figure 2. Macro images of non-additive and vermiculite additive sanitaryware samples.

Figure 3 shows the XRD pattern of the sanitaryware starting powders utilized in the investigation. Quartz, albite, annite, kaolinite, mullite, phlogopite and ringwoodite phases can be seen in the XRD results. Figure 3 illustrates the XRD pattern of starting powders with the code S00V and chemical compositions listed in Table 1 and 2. The pattern revealed (96-900-9667) quartz, (96-900-1632) albite, (96-101-1046) kaolinite and (96-900-2314) annite phases. The oxide components of the observed phases match the clay, kaolin, feldspar and quartz raw materials contained in the sample recipe when compared to the XRF data. The peaks of (96-900-1622) Mullite, (96-900-1632) Albite and (96-900-9667). Quartz phases can be found in the XRD patterns of S0V1050 and S0V1150 samples. Primary mullite crystals started to form in the sample coded S00V1050. When the sample coded S00V1150 was examined, it was determined that the mullite crystals increased with the increase in the intensity and visible peaks of the mullite peaks. XRD patterns of vermiculite added samples are given in Figure 3. In the sample coded S20V1050 (96-900-1622) Mullite, (96-900-1632) Albite, (96-900-9667) Quartz (96-901-0171) Phlogopite and (96-901-5248) ringwoodite phases were detected. In the sample coded S20V1150, 4 phases other than Phlogopite are seen. When the S20V1050 and S20V1150 samples are compared, the same phases are seen in both samples, and the peak intensities of mullite and ringwoodite increased with the temperature increase to 1150 ℃. Again. there is a decrease in albite peaks in the sample coded S20V1150, which is due to the formation of a liquid phase by albite. The presence of ringwoodite peaks is due to the addition of vermiculite mineral. Considering the prescriptions of the S-coded samples, the glassy phase is more common due to the excess amount of feldspar. It is thought that the crystallization of the ringwoodite (magnesium silicate) phase in the structure is caused by the alkalis and iron oxide passing into the glassy structure and crystallized as magnesium silicate. Phlogopite and ringwoodite phases were crystallized in vitrified structure as a result of vermiculite addition. This is due to the presence of MgO in the chemical content of vermiculite.

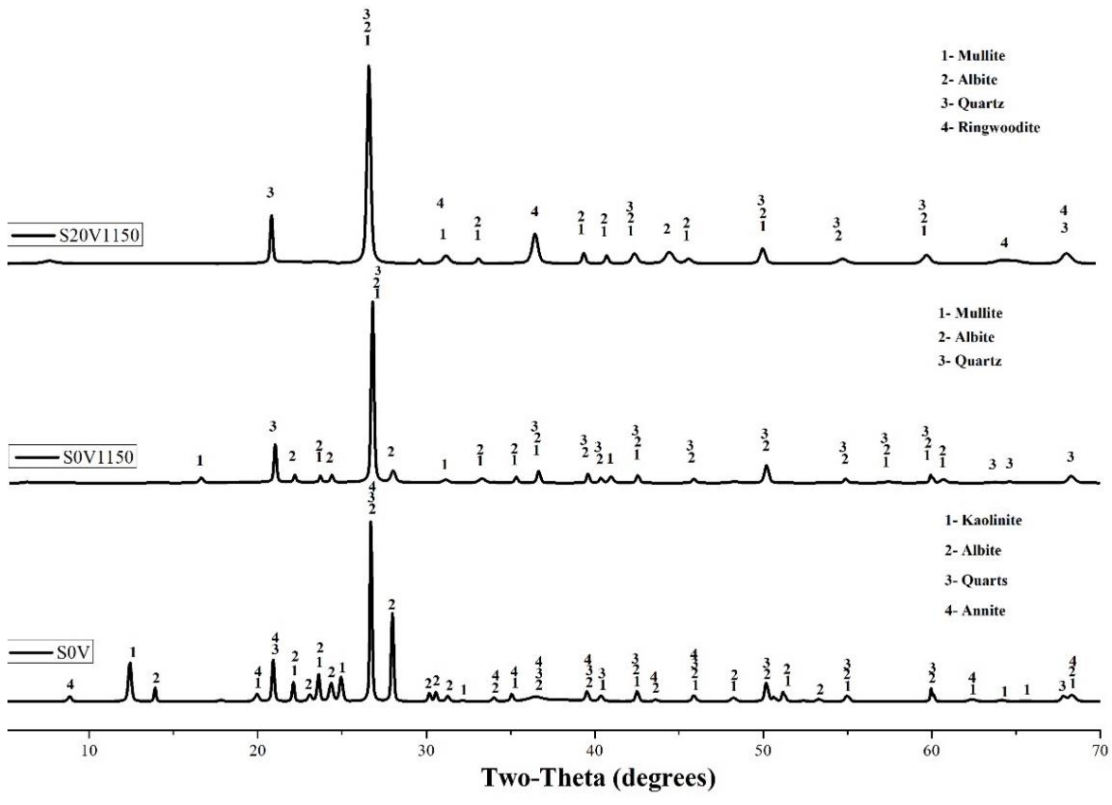
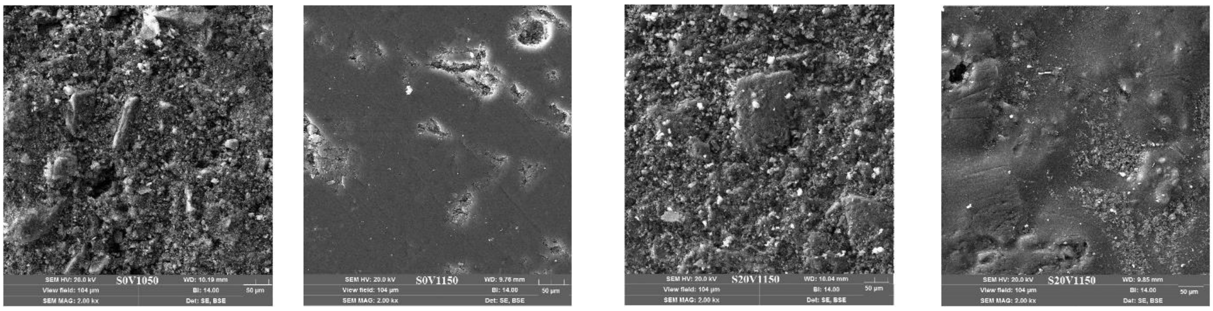


Figure 3. XRD patterns of non-sintered sample, S00V1150 and S20V1150 samples.

Electron microscope images of sanitary ware samples are given in Figure 4. Looking at the SEM photographs, it was observed that increasing firing temperature and increasing vermiculite additives increased the glassing in the structure and reduced the visible micro porosities. This situation was associated with the high amount of feldspar in the prescriptions of sanitary ware samples. Physical test results are also compatible with this situation. In Figure 4, the results of the EDS analysis performed on the sample coded S20V1150 are given. Elemental analyses were made over the A general map area, B and C points. For a general EDS analysis, area A was chosen. Points B and C were chosen based on the tonal difference in the colour, which is thought to be due to the density difference. When the results obtained from the general area are examined, it is seen that the sample is compatible with the recipe. B and C point analyses also match with the XRD pattern results of the S20V1150 coded sample.



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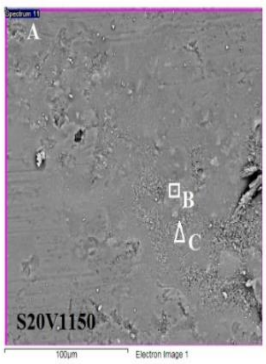

Element	Weight%	Atomic%	Element	Weight%	Atomic%	Element	Weight%	Atomic%
O K	50,38	64,33	O K	52,67	71,11	O K	43,23	57,86
Na K	2,19	1,95	Na K	1,40	1,32	Na K	1,93	1,80
Mg K	2,14	1,80	Mg K	3,19	2,83	Mg K	4,46	3,93
Al K	10,86	8,22	Al K	7,88	6,30	Al K	13,64	10,83
Si K	29,81	21,68	Si K	10,13	7,79	Si K	28,97	22,09
K K	1,34	0,70	K K	0,43	0,24	K K	1,99	1,09
Ca K	0,66	0,34	Ca K	0,27	0,14	Ca K	0,98	0,52
Ti K	0,49	0,21	Ti K	15,09	6,80	Ti K	0,71	0,32
Fe K	2,12	0,78	Fe K	8,94	3,46	Fe K	4,09	1,57

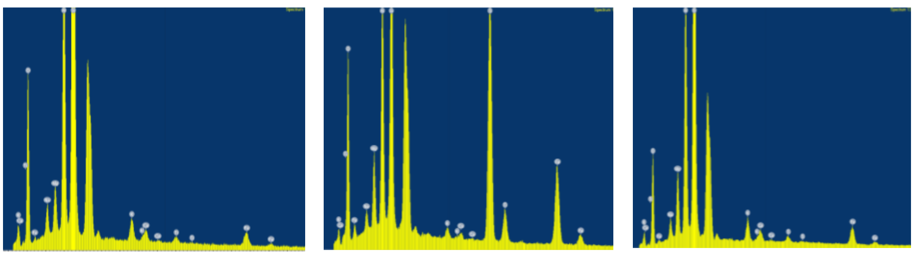

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Figure 4. SEM images of sintered samples at 1050 °C and 1150 °C. a) S0V1050 b) S0V1150 c) S20V1050 d) S20V1150. EDX analyses %20 vermiculite additive sanitaryware samples sintered 1150°C. e) image of EDS area and f) area of A EDS g) B point EDS and h) C point EDS.

4. Conclusion

The use of vermiculite in the production of sanitaryware was explored in this study. DTA/TG graph of the sanitaryware sample; removal of free water at 25-200 ℃, hygroscopic water at 200-400 ℃, crystal water at 400-600 ℃, conversion to meta kaolinite form at 500-600 ℃ and the formation Mullite phase at 980-1000 ℃. The results of physical measurements; to become increased in bulk density, shrinkage and bending strength values with increasing sintering temperature but porosity and water absorption values decreased. The physical results also show the same effect in vermiculite additive specimens. As the amount of vermiculite and sintering temperature increased in vitrified samples, the hardness of the samples increased. The brownish colour of sintered samples increased from light to dark with rising vermiculite and sintering temperatures. Quartz, albite, annite, kaolinite, mullite, phlogopite and ringwoodite phases were determined in the XRD patterns of raw and sintered at 1150 ℃. With the addition of vermiculite, magnesium silicate-based ringwoodite phase crystallized in the vitrified body. At the same time, it can be said that alkali oxides such as potassium oxide and sodium oxide in the chemical composition of vermiculite form a glassy phase as a result of vermiculite additive, and that a slope that deviates from linearity in the XRD patterns in the range of 20-30 theta. It is seen in the physical test results that the formed glassy phase reduces the porosity, decreases the water absorption values, and increases the bulk density. Looking at the SEM photographs, it was observed that increasing firing temperature and increasing vermiculite additives increased the glassy phase in the structure and reduced the visible micro porosities. This situation was associated with the high amount of feldspar in the prescriptions of sanitary ware samples. Physical test results are also compatible with this situation. The results of EDX Elemental analyses and XRD patterns are compatible with the recipe of sanitarywares. The C point EDS analysis is in agreement with the chemical content of the mullite phase obtained in the XRD results.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Şaban Tapik: Conceptualization, Methodology, Software. **Umut Önen:** Data curation, Writing – original draft. **Tahsin Boyraz:** Writing – review & editing.

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