

# THE RELATIONSHIP BETWEEN BENDING STRENGTH AND POROSITY IN CERAMIC BRICK THAT CONTAINS VERMICULITE

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## ABSTRACT

This study focused on the characterization, product, and relationship between bending strength and porosity of ceramic bricks with added vermiculite. To achieve this goal, premade bodies containing 0%, 10%, and 20% calcined vermiculite by weight were mixed in to create the body compositions. Alumina ball manufactories homogenized fusions (60 rpm, 24 hours) were molded through uniaxial dry pressing (100 MPa). Additionally, the arranged samples were heated to a temperature of 800-1000 °C for a duration of 1 hour. Tests involving phase analysis (XRD), microstructure (SEM), mechanical properties (three point bending), and physical characteristics (Porosity, water absorption, shrinkage and bulk density) were carried out on the demitasse bodies that were manufactured. The data that was collected was displayed in graphs and tables, showing the results. As a result, the packages containing samples of vermiculite were effectively created, and the best parameters were easily displayed. The findings indicate that the quality of flawed packages improves as calcined vermiculite levels rise. Following the characterization results, the correlation between bending strength and porosity was investigated. Correlations, t-tests, and p-values were computed. The inclusion of vermiculite in ceramic bricks results in a significant negative correlation of -0.769 between their bending strength and porosity characteristics.

Keywords: Ceramic, Brick, Vermiculite, Bending Strength, Porosity, Correlation

## **1. INTRODUCTION**

Due to the abundance of clay in the world and the ease, simplicity and cost of brick production, bricks are widely used in many areas, especially as building materials. Due to the brick industry's ability to add different compounds during the mixing process, research has been conducted to highlight the effect of these additions on fired clay bricks. To improve energy efficiency, it is necessary to enhance the thermal insulation properties of bricks. One method for producing bricks involves adding pore-forming additives. Thermal decomposition and volatilization are used to form pore-forming materials, such as organic residues, wood sawdust, coal dust, polystyrene, papermaking sludge, powder limestone and heat-resistant porous materials in brick body have been widely used. In this sense, vermiculite is a material that can be utilized as a pore former. Sutcu (2015), Muñoz et al. (2014), Zhang et al. (2018), Monteiro & Fontes Vieira (2014), Muñoz et al. (2016), Zhang (2013), Effting et al. (2007), Aouba et al. (2016), Muñoz et al. (2020), Sarani et al. (2018), Achik et al. (2021), Elavarasan et al. (2021), Sutcu et al. (2015), Phonphuak et al. (2019).

'Vermiculite' is named after the Latin word 'Vermicularis', which means wormlike, and it is formed by the abrupt exposure of crystals to high temperature, resulting in curved, elongated, and twisted columns. One of the groups of phyllosilicate minerals, Vermiculite is a mica-like mineral that has a shiny flake. The expansion of vermiculite occurs at temperatures between 650-950 °C. The expansion process is known as exfoliation. The expansion and convection of vermiculite into loose, lightweight fragments, similar to "popcorn", is similar to that of "popcorn", and is separated by air entrainment when heated. The natural occurrence of vermiculite comprises hydrated Mg/Fe/Al silicate minerals (micalike), which form a laminate structure and come in a range of light brown/grey/gold colors. Vermiculite is also a very adaptable mineral with amazing qualities including low density, odorlessness, inertness, ease of handling, low thermal coefficient, strong thermal and absorption capacity, acoustic insulation, fire resistance, refractory nature, and non-combustion material. To exfoliate or expand its size, this material is mined, pulverized, and heated to over 300°C, ranging in size from 210 mm in diameter. Most applications require vermiculite to be exfoliated. Vermiculite's thermal conductivity, low density, and high melting temperature make it a viable option for fillers in the production of heat-insulating refractory materials. The material that is most commonly found in vermiculite is made up of Al<sub>2</sub>O<sub>3</sub> (10-13), SiO<sub>2</sub> (57%-42 wt%), Fe<sub>2</sub>O<sub>3</sub> (5-17), MgO (14-12), H<sub>2</sub>O (8-18), and FeO Suvorov & Skurikhin (2003), Anitha et al. (2023), Rashad (2016). Research findings suggest that vermiculite holds promise as a building material and can serve as an aggregate in lightweight concrete and plaster due to its exceptional thermal, fire, and sound insulation properties. Notably, vermiculite exhibits low specific gravity, high flame and heat resistance, and a significant ion exchange capacity, rendering it valuable in various sectors including construction, agriculture, animal husbandry, and wastewater treatment Onen et al. (2017), Spirina & Flerova (1975), Suvorov & Skurikhin (2002), Silva Jr et al. (2014). Vermiculite's potential use as a building material is a possibility given the studies' results. Due to its exceptional thermal, fire, and sound insulation properties, it is also suitable for use as an aggregate in lightweight concrete and plaster. According to Park and Ngayakamo, Kalalani vermiculite can be used as a raw ingredient in the production of porcelain insulators of high strength Ngayakamo & Park (2019).

Correlation analysis assesses the existence and strength of linear relationships between two variables, as indicated by the correlation coefficient. This coefficient ranges from 1 to -1, with negative values signifying a negative relationship, positive values indicating a positive relationship, and values nearing zero suggesting a weak relationship. Perfect correlation occurs when the coefficient is either 1 or -1. In the field of ceramics and materials science, correlation coefficient (r), along with t-tests and p-values, are commonly employed for various studies. These include investigating correlations between powder properties and sintering behaviours, phase transitions and luminescent properties, as well as crystalline phases and optical reflectance Prasada et al. (2010), Lee & Kim (2009), Park & Shin (2002). This study aims to assess the correlations between bending strength and porosity properties in ceramic brick doped with vermiculite. The correlation coefficient will quantify the degree and direction of the relationship between these quantitative variables, using the Pearson correlation coefficient due to the continuous nature of our data. Additionally, the significance of these correlations will be tested to determine their reliability and statistical validity.

## 2. MATERIALS AND METHODS

The properties of brick bodies were studied through the use of vermiculite obtained from the Organic Mining region in Sivas, Turkey. The body compositions were formed by adding ready-made brick bodies (Corum Brick, Turkey) and calcined vermiculite %0 - 20 by weight. An electric furnace with a temperature of 1050 degrees C was used to calcine the raw vermiculite for one hour. Brick bodies that were already made were dried and turned into powder. XRF (Thermo ARL) analysis results for calcined and raw vermiculite, as well as ceramic brick, are presented in Table 1. B10V900 was the code for the samples, which included B: Brick; 10V: 10% vermiculite addition and 900: 900 °C firing temperature. The mixtures were wet mixed in alumina ball mills at 60 rpm for 24 hours and dried in the oven at 110 degrees for 24 hours. The powder mixtures, which were dried and made ready for shaping, were shaped into 10x30x70 mm dimensions by uniaxial dry pressing under 100 MPa pressure. After preparation, the press samples were fired at 800-1000 degrees Celsius for an hour. The produced brick samples were tested for mechanical, physical properties, microstructure, and phase analysis.

A digital calliper was used to measure and calculate the shrinkage percentage of the sintered samples. Archimedes' principle (ASTMC373-88) was used to calculate density, porosity, and water absorption tests. The samples were tested for bending strength at three points using a mechanical tester with a load sensing of 1 N and a power of 5 kN. The strength values of the samples were determined by the average results of five measurements taken for each sample. Using the naked eve, the color analysis of the samples was evaluated under visible light. Color changes can be identified as either increasing or decreasing darkening and color differences depending on the increasing additive ratios and firing temperatures. The samples were scanned with Mira3XMU FE-SEM (Tescan, Czech Republic) and the energy dispersive spectrum (EDX) analysis were performed as well. Graphs and tables were used to present the obtained data, and comments were made. A Panalytical X'Pert Powder X-ray diffraction (XRD) analyzer used for X-ray evaluation was employed to analyze the samples in terms of 40 to 700 Angstrom. The Pananalytical X'Pert High Score program was used to determine the phases of the XRD patterns. Onen et al. (2017), Spirina & Flerova (1975), Silva Jr et al. (2014).

#### Table 1

Table 1 The Chemical Makeup of Vermiculite and Brick Powders that have been Raw and Calcined

%w	<b>Calcined vermiculite</b>	- Raw vermiculite	Brick
TiO <sub>2</sub>	2,4	2,18	1,10
CaO	3,9	3,54	7,35
SiO <sub>2</sub>	40,61	36,9	46,60
Al <sub>2</sub> O <sub>3</sub>	19,48	17,7	15,30
MgO	18,05	16,4	6,54
CaO	3,9	3,54	7,35
Fe <sub>2</sub> O <sub>3</sub>	12,31	11,2	10,17

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K <sub>2</sub> O	2,91	2,64	2,59
Na <sub>2</sub> O	0,17	0,15	1,21
MnO	0,17	0,15	-
LOI	-	9,14	9,21

The Pearson correlation coefficient was employed for correlation analyses, and subsequently, a t-test was conducted to assess whether the correlation coefficient significantly deviated from zero. The significance of the correlation was determined by calculating a p-value based on the t-distribution table.

## **3. RESULTS AND DISCUSSIONS**

Table 2, Figure 1 and Figure 2 presents the outcomes of physical and mechanical assessments, including water absorption, bulk density, porosity, and shrinkage. It was observed that as the sintering temperature increased, there was a rise in shrinkage, bending strength and bulk density values, while water absorption and porosity values decreased. These trends were consistent across samples with vermiculite additions as well.

#### Table 2

Table 2 Test Results of Samples with and without Vermiculite Additives						
Samples	Bulk density	Water Absorption	Shrinkage	Bending Strength	Porosity	
	gr/cm <sup>3</sup>	%	%	MPa	%	
B00V800	1,55	25,06	5,51	9,05	39,17	
B10V800	1,59	24,04	5,55	9,24	36,52	
B20V800	1,61	20,44	5,62	9,51	35,52	
B00V900	1,58	24,29	6,02	10,52	38,25	
B10V900	1,60	23,61	6,41	11,02	35,01	
B20V900	1,62	19,89	6,82	11,55	33,82	
B00V1000	1,61	23,48	6,52	13,82	36,15	
B10V1000	1,62	22,40	7,35	14,83	33,56	
B20V1000	1,63	19,63	7,75	15,96	30,52	

Between Figure 1 and Table 2, the results of physical measurements are presented. As the sintering temperature increases, the results show an increase in, shrinkage, bulk density and bending strength values. The sintering temperature increased, leading to a decrease in porosity and water absorption values. Vermiculite added samples also experienced increases and decreases.

The test results showed that there was an increase in the % shrinkage with the increase of vermiculite addition and sintering temperature. While this increase was 5.51% in pure brick samples fired at 800 degrees, it was measured as 6.522% in samples fired at 1000 degrees. The shrinkage in samples with 20% vermiculite added was found to be 5.62% in bricks fired at 800 degrees and 7.75% in bricks fired at 1000 degrees. Porosity results decreased with increasing vermiculite and sintering temperatures. While it was 39.17% for pure bricks fired at 800 degrees, it was 36.15% for bricks fired at 1000 degrees. While this rate was 35.52% in samples with 20% vermiculite added and fired at 800 degrees, it was 30.52% in samples produced at 1000 degrees. Similarly, the densities increased in proportion to the sintering temperature and the amount of vermiculite. While the density obtained for pure bricks at 800 degrees is 1.55 g/cm<sup>3</sup>, the density for 1000 degrees is calculated as 1.61 g/cm<sup>3</sup>. In 20% vermiculite added samples, these values were measured as

density 1.61 g/cm<sup>3</sup> for 800 degrees and 1.63 g/cm<sup>3</sup> for 1000 degrees. Water absorption values also decreased with increasing temperature and additive amount. While it was 25.06% at 800 degrees without additives, it was found to be 23.48% at 1000 degrees. For baked bricks with vermiculite addition, these values are 20.44% and 19.63%. The strength test results showed that there was an increase with increasing sintering temperature and the addition of vermiculite. The measured value of 9.05 MPa in bricks fired without additives at 800 degrees became 13.82 at 1000 degrees. The 3-point bending strength results of 20% vermiculite added samples are 9.51 MPa and 15.96 MPa (Table 2, Figure 2).



#### Figure 2



Figure 2 The Bending Strength and Porosity Graphs of Brick Samples are Presented Here

Figure 3 shows that the brownish colour of the samples increased from light to dark with increasing vermiculite when examined. The sintering temperature increase resulted in the colour becoming darker.





Figure 3 Macro Images of Bricks Samples

Phlogopite, tridymite, anorthite and quartz phases were detected in the XRD graphs obtained after firing of bricks with and without vermiculite added. Anorthite (96-900-0363), tridymite (96-901-3394) and quartz (96-900-9667) phases were detected in pure bricks fired at 1000 degrees. Firing temperature caused an increase in the apparent peak and intensity of the tridymite and anorthite phases, as observed. In bricks fired at 1000 degrees with 20% vermiculite, phlogopite, tridymite, anorthite and quartz phases were detected as the firing temperature increased and the peak densities of crystallized anorthite and tridymite phases in the material increased (Figure 4).



Figure 4 The XRD Patterns of Brick Samples Sintered at 1000  $^\circ C$  with No Doping and 20% Vermiculite Added

It was calculated that with increasing firing temperature and increasing vermiculite, the porosity of the samples decreased and their density increased. In SEM images, it was observed that the micropores in the structure became smaller as the temperature and vermiculite content increased. General A and B, C regional results were used to evaluate the EDX analysis results of brick samples baked at 1000 degrees with 20% vermiculite. XRF data confirms that the overall results from region a are correct. The X-ray pattern findings of the sample are in accordance with regional points B and C. There is quartz phase at point B and phlogopite phase at point C (Figure 5 and Figure 6).



Figure 5 SEM Images of B20V800 and B20V1000 Brick Samples

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#### Figure 6



rigure o LDA Analyses of D20V 1000 Drick Samples.

## 4. PEARSON CORRELATION COEFFICIENT, T-TEST AND P VALUE

This study is aimed at calculating the relationships between bending strength and porosity properties in ceramic bricks doped with vermiculite. The strength of the relationship between two quantitative variables, regardless of whether it is positive or negative, is represented by the correlation coefficient. The Pearson correlation coefficient will be used since our data is based on continuous variables. The statistical measure of the strength of the association between two variables and their relationship with one another is known as the Pearson correlation coefficient, sometimes known as Pearson's r. To put it simply, the Pearson correlation coefficient determines how much a change in one variable affects another. The significance of the correlations will also be tested. The final step is to create the graphics.

A linear relationship between two variables can be determined by correlation. To put it differently, it illustrates whether changes in variables have an impact on each other. Correlation coefficient is a value used to show the relationship between variables. The variable takes a value between -1 and 1 when calculated with the formula below. A negative relationship is indicated by negative values, and a positive relationship is indicated by positive values. A perfect relationship exists when the values are 1 or -1. When the values approach 0, the relationship between the variables decreases (Figure 7).







The statistical significance of the correlation between Pearson's coefficient and other factors is high. The relationship between two variables is the subject of examination. It endeavors to depict the relationship between two variables by drawing a line through their data. The Pearson correlation coefficient equation 1 was used to calculate the relationship between the variables. A positive or negative outcome can be expected from the relationship that is calculated. If the result is negative, there is a negative correlation between the two variables, and when the result is positive, there is a positive correlation relationship between the variables.

Pearson correlation coefficient (r):

$$r = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n \sum x^2 - (\sum x)^2][n \sum y^2 - (\sum y)^2]}}$$
(1)

N = The total number of point pairs.  $\Sigma xy$  = Pair scores' sum of products is xy  $\Sigma x$  = The total sum of x values  $\Sigma y$  = The total sum of y values  $\Sigma x^2$  = Sum of squared x values  $\Sigma y^2$  = Sum of squares of y values

Based on the survey sample, a t-test can be used to assess whether the Pearson correlation coefficient is substantially different from zero. The sample size is denoted by n, and the correlation coefficient by r.

$$t = r \sqrt{\frac{n-2}{1-(r)^2}}$$
(2)

To ascertain the significance and validity of the Pearson correlation coefficient, a t-test is conducted based on the examined sample. This test determines whether the coefficient significantly differs from zero. Subsequently, a p-value is derived from the computed test statistic t. If the obtained p-value falls below the designated significance level, typically set at 5%, the null hypothesis is refuted; otherwise, it is retained.

In the context of a t-test, the conventional threshold for significance is typically set at p = 0.05. Essentially, the p-value represents the likelihood of observing a mean difference purely by chance, assuming there is no true disparity within the population. When the p-value obtained from a t-test is below 0.05, the result is deemed statistically significant. Conversely, if the p-value exceeds 0.05, the finding is considered insignificant. The p-value is derived from the t distribution table and is calculated as 2 times the probability of obtaining a value greater than the observed t statistic, where T follows a t distribution with degrees of freedom equal to n - 2.



Figure 8 Bending Strength - Porosity Correlation Graphs of Ceramic Bricks.

A correlation coefficient of -0.769 was determined for ceramic brick (Figure 8). The negative slope indicates that as one variable increases, the other variable decreases, illustrating a negative linear relationship. This suggests that changes in one variable are inversely related to changes in the other variable.

When the correlation coefficient (r) exceeds 0.5 or falls below -0.5, it indicates that the data points closely align with the best-fit line, indicating a strong negative correlation. In this study, a strong negative correlation of -0.769 was observed between the bending strength and porosity properties of vermiculite-enhanced ceramic brick. The calculated t-test values were -6,365. Based on the t distribution table, the p-value was determined to be less than 0.001 for ceramic brick (refer to Table 3). Consequently, it has been established that there exists a robust negative association between 3-point bending strength and porosity in vermiculite-augmented ceramics.

l'able 3					
Table 3 Correlation Coefficient (r), t-test and p Values of Ceramic Brick					
Materials	Correlation coefficient, r	t	р		
Ceramic Brick	-0,769	-6,365	<0,001		

## 5. CONCLUSIONS AND RECOMMENDATIONS

Benefitful outcomes were achieved through the investigation of vermiculite's use in brick manufacturing in this study.

- As the sintering temperature increases, the results show an increase in bulk density, shrinkage, and bending strength values. The sintering temperature increased, leading to a decrease in porosity and water absorption values. Vermiculite added samples also experienced increases and decreases.
- 2) The red-brownish color of the sintered samples, due to the iron oxide content, increased from light to dark with the addition of vermiculite and increasing sintering temperatures.
- 3) Phlogopite, Anorthite, Quartz, and Tridymite were revealed in the XRD pattern of bricks sintered at 1000 °C, which were not doped. The firing temperature increased, resulting in an increase in the apparent peak and intensities of the tridymite and anorthite phases.

- 4) There is a strong negative correlation of -0.769 between the bending strength and porosity properties of vermiculite-added ceramic bricks.
- 5) The calculated t-test value is -6,365. From the t distribution table, the p value was found to be p<0.001 for all three ceramic materials.

As a result, it has been revealed that there is a strong negative relationship between 3-point bending strength and porosity in vermiculite added ceramics.

## **CONFLICT OF INTERESTS**

None.

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