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Image Analyses for Porosity Characterization of Porcelain Tiles with Different Thickness

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Abstract

The physical properties of materials provide some of the most fundamental information regarding the material characterization. Porosity is one of the physical properties which affects the unit density, water absorption, strength, heat and sound insulation of materials. In this study, porcelain tiles with different thickness were sintered at same temperature, after that the cross-section of tiles were inspected with Scanning Electron Microscope (SEM). The porosity percentage and pore size of tiles from SEM images was calculated by using Scandium Image Analysis software. The aspect ratio of pores analyzed using Fiji Image Analyses Program. The minimum porosity percentage was achieved at average thickness. These porosity results were compared with total porosity percentage measured with He pycnometer. The highest percent of spherical pore (aspect ratio equals to 1) was found at the highest thickness but the smallest pore percentage found at the lowest thickness. It was found that the image analysis programs were effective tools for material characterization.

Key words
Porosity; Image analyses; Scanning electron microscope; Microstructure

Farklı Kalınlıklardaki Porselen Karoların Gözenek Karakterizasyonu için Görüntü Analizi Programlarının Kullanılması

Özet

Malzemelerin fiziksel özellikleri, malzeme karakterizasyonu için gereken temel bilginin büyük bir kısmını sağlamaktadır. Gözeneklilik, malzemenin birim yoğunluğunu, su emmesini, mukavemetini, ısı ve ses yalıtımını etkileyen fiziksel özelliklerden biridir. Bu çalışmada, farklı kalınlıklardaki porselen karolar aynı sıcaklıkta sinterlendikten sonra karoların kesit alanları Taramalı Elektron Mikroskobu ile incelenmiştir. Taramalı Elektron Mikroskobu görüntülerinden karoların por yüzdesi ve por boyutu Scandium görüntü analizi programı kullanılarak hesaplanmıştır. Por boyutlarının a/c oranı ise Fiji görüntü analizi programı kullanılarak analiz edilmiştir. En düşük por miktarına orta kalınlıktaki karolarda ulaşılmıştır. Görüntü analizi ile belirlenen por yüzdesi, Helyum piknometresi ile ölçülen toplam por yüzdesi ile kıyaslanmıştır. a/c oranı 1'e eşit olan küresel porların yüzdesi en kalın karolarda elde edilirken, en küçük gözenek yüzdesi en düşük kalınlıktaki karolarda elde edilmiştir. Görüntü analiz programlarının malzeme karakterizasyonu için etkili araçlar olduğu belirlenmiştir.

Anahtar kelimeler
Por; Görüntü analizi; Taramalı elektron mikroskobu; Mikroyapı

1. Introductıon

The porosity in microstructure is one of the physical properties of ceramic materials and one of the significant factors that affects the mechanical properties. The desired strength value cannot be achieved due to the defects such as porosity, crack occurred during production of ceramic materials and development of microstructure after firing. Because these defects form regions of stress concentration in the microstructure (Callister, 1994). Accordingly, as the amount of porosity increases in the material the area which carries the applied load reduces and the formed strain to the applied load increases in the body (Richerson, 1992). As the porosity amount, pore's shape and distribution of pores in the ceramic material effects properties such as strength, water absorption. (Ivanchenko et al. 1999; Ece and Nakagawa 2002; Karamanov and Pelino 2008; Arias et al. 2003; Zanelli et al. 2004). For example, pores which reaches to the surface after firing in low green density tiles and pores which opens after polishing process of porcelain tiles affects negatively strength (Griggs et al. 1996). So the material should have homogeneous pore size distribution and high density initially (Orts et al. 1993).

Bulk density of bodies is determined by the Archimedes method by Equation 1. According to this method, a sensitive scale is used for measure dry weight (W1) of fired samples, in the water weight (W2) after boiling in water 4 hours and wet weights (W3) of samples after taken from the water and their surfaces are dried.

$$\text{Bulk density}= \frac{W_1}{W_3 - W_2} X\rho_{H_2O} \qquad (1)$$

Different methods are used to determine the amount and size of pores. For determination of total porosity by Helium py observed pressure change between two chambers. With no sample present in the measuring chambers, the pressure in each chamber is the same. When a sample is introduced, a difference in pressure is observed. The difference is in relationship to the volume of the samples by the gas law. If the sample is fine power, generally < 45 µm, open and closed pores are destroyed, and the result is called as the true density.

$$\text{Total porosity (\%)}= \frac{(\rho_t - \rho_b)}{\rho_b} x100 \qquad (2)$$

In the equation; *P* (%): total porosity (%), ρ_t: theoretical density (g/cm^3) and ρ_b: bulk density (g/cm^3) (Andreola et al. 2000).

One of the methods which shape of the pore is determined by is the optical microscope. But optical microscopy has become inadequate for in-depth control of ceramic microstructures, including the most traditional ones.

In recent years, with the development of computer software programs, image processing and image analysis methods have become more readily available. Size and shape distribution of porosity can be obtained using the images collected by optical microscopy, SEM and TEM. Porosity is determined as the ratio between the area of pores and the total area of the sample analyzed. The image analysis software transforms the information collected in a binary signal as a grayscale. Images must have significant contrast to distinguish objects to be analyzed from the rest of matrix. It is possible to calculate the porosity percentage by using Eq. 3 (Andreola et al. 2000; Jordan et al. 1996):

$$P\% = \frac{PoresArea}{TotalAreaCenter} \times 100 \qquad (3)$$

In this study, changing of porosity amount, size and morphology is analyzed comparatively by Archimedes method, Helium pycnometry and the image analysis method in porcelain tile bodies of different thickness have the same composition and subjected to the same firing conditions.

2. Experimental Procedure

Standard porcelain tile granules were used for preparing samples in this study. Samples were prepared by uniaxial pressing technique in a 50 mm x 100 mm rectangular die at 216 kg/cm^2 (with different thicknesses: 2.5 mm, 5 mm and 7.5 mm), and dried at 110˚C. For each thickness (Fig.1), four tiles were prepared. The firing step was carried out in a industrial roller kiln at 1182˚C temperatures with industrial fast-firing cycle (total 50 minutes including cooling).

After sintering, the bulk density and the total porosity (open and closed pores) of a material was measured by using Archmedes Method and Helium pycnometry. The bulk density can be determined using the techniques, e.g., liquid or powder immersion of the bulk sample. In this work, it was determined by water immersion (Andreola et al. 2000). The measure of pycnometry used the Quantachrome Model MVP-1 Multipycnometer. The sample microstructures were examined by scanning electron microscopy (EVO-50, Carl-Zeiss). The SEM samples were polished using a polishing machine (Metkon- Forcipol-300-1N) and their surface etched for 35-40 s with 5 weight

(a) (b) (c)

Figure 1. Prepared tiles with different thickness (a) 2.5 mm, (b) 5 mm and (c) 7.5 mm

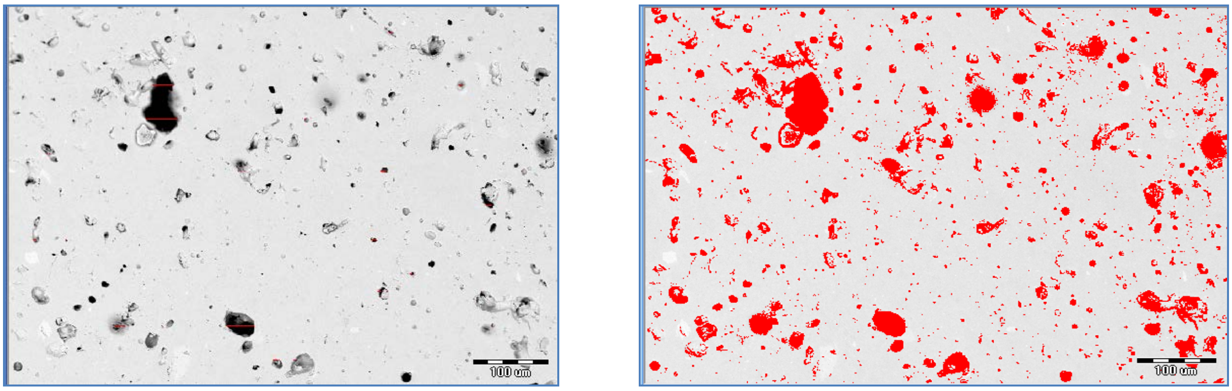
percentage of HF solution. Additionally, the total porosity and pore size ranges of samples were calculated by using Scandium Image Analysis software. Aspect ratios of the tiles were determined with Fiji program. 6 images were inspected for each thickness.

3. Results and Discussion

The density and porosity (%) values of tiles with different thickness ratio after sintering are determined by Archimedes and Helium pycnometry methods are shown in Table 1. Archimedes method depending on the amount of water entering the open pores of samples when calculating bulk density, Helium gas pycnometer, depending on the method of calculation which is performed by gas entering to the pores with the help of pressure. For this reason, the bulk density values determined by the Archimedes method differs from the theoretical density and total pore volumes obtained by Helium pycnometer. Bulk densities of tiles with different thickness ratio were not significantly different. Theoretical density changed depending on the thickness ratio of the tiles with the effect of varying porosity. In general, when the thickness decreases, the amount of porosity decreases. Selecting a representative sample within the each thickness range, back scattering electron images from the samples were analyzed by Scandium image analyzing program. The analyzed images obtained by SEM images were shown in Figures 1-3 and porosity %, pore size distribution were given in Table 2. When the pore size distribution was analyzed, the most pore size % was achieved at 0-10 µm range for all samples. It was found that for the tile sample of 7.5 mm in thickness, the amount of > 20 µm pore size is higher and the amount of 0-10 µm porosity is lower than the other samples. There was no significant difference is observed samples 5.0 and 2.5 mm in thickness about the percentage of porosity and the pore size distribution. When the average amount of the total pore obtained with Helium pycnometer compared to the amount of porosity obtained with image analysis program (Scandium) by using electron images of the color reflected back portions (contrast difference: porosity), the porosity values of two techniques' close to each other. As the amount of thickness increases at microstructure images, the presence of larger pores is noteworthy. In this respect, the method of image analysis give information about the dimensions of porosity in addition to the amount of porosity is superior to the other methods.

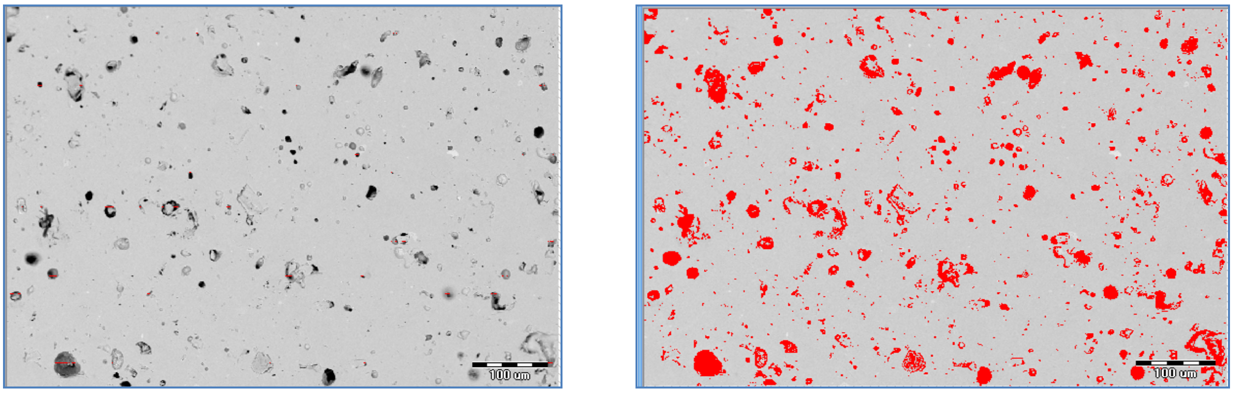
Table 1. Amount of total porosity and densities of tiles with different thickness amount

Thickness amount of tiles (mm)	Theoretical density (g/cm^3)	Bulk density (g/cm^3)	Total porosity (%)	Mean of Total Porosity (%)
7.5	2.49	2.36	5.82	5.35± 0.32
	2.48	2.36	5.17	
	2.48	2.36	5.25	
	2.48	2.36	5.13	
5.0	2.45	2.37	3.53	3.46± 0.37
	2.46	2.37	3.80	
	2.44	2.37	2.93	
	2.45	2.37	3.58	
2.5	2.46	2.36	3.97	3.56± 0.46
	2.46	2.37	3.87	
	2.45	2.36	3.43	
	2.43	2.36	2.95	



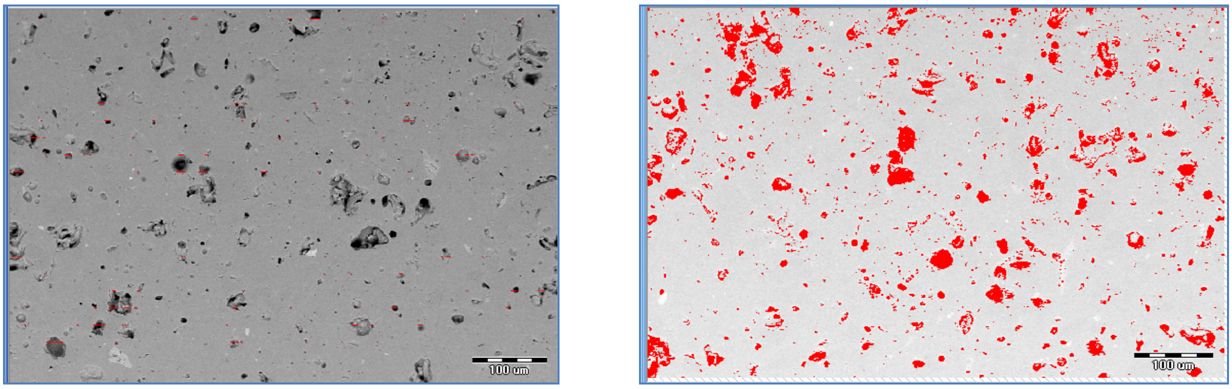
(a) (b)

Figure 1. a)Backscattered electron image, b)the image analysed by Scandium program of tile with 7.5 mm



(a) (b)

Figure 2. a)Backscattered electron image, b)the image analysed by Scandium program of tile with 5.0 mm



(a) (b)

Figure 3. a)Backscattered electron image, b)the image analysed by Scandium program of tile with 2.5 mm

Table 2. Mean pore size distributions of tiles with different thickness amount

Thickness amount of tiles (mm)	Image analysis results for total porosity (%)	Size distribution of porosity (%)		
		0-10 µm	10-20 µm	>20µm
7.5	4.86	85.82	6.71	7.47
5.0	2.36	91.66	7.23	1.11
2.5	2.33	95.80	3.27	0.93

The aspect ratios of images taken by electron microscope is inspected with Fiji image analysis program. The percentage of spherical pore (aspect ratio=1) for the 7.5 mm thick tile is 44.30%, for the 5.0 mm medium thick tile is 37.21% and the thin tile is 37.35%, respectively. The spherical pore percentages are similar for the thin and medium thick samples. The spherical pores contain less tensile stress regions than the ellipse-shaped pores. Stathis et al. (2004) inspected if total porosity significant on the strength in isolated spherical pores containing bodies and they indicated that total porosity value is a variable on the strength only the systems containing interconnected pores. Furthermore, the general trend for laminar pore character at higher pressures (and as a function of higher aspect ratio) of die-pressed bodies (Rice, 1998). When all the samples pressed at same pressure, the masses for the shaping is less for 5.0 and 2.5 mm thick tiles than 7.5 mm thick tile. This can be a reason of lower the spherical pore percentages for 5.0 and 2.5 mm thick tiles than 7.5 mm thick tile.

4. Conclusion

Images obtained by electron microscopy techniques, which now can be considered not only as research tools, but also as very valuable instruments for quality control of microstructures. In this study affect of thickness on microstructure change was investigated. It was determined that when the thickness amount of tiles decrease, percentage of porosity and size of porosity of tiles decrease. The percentage of spherical pore of tiles was obtained by the Fiji program. According to this measurement, Fiji program can be give information about strength of tiles.

According to total porosity measurements, image analysis technique has a good correlation with helium pycnometry. There is only maximum ~ 1.2 % changes at the porosity levels.

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