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Air content and size distribution of air voids in hardened cement pastes using the section-analysis method

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Abstract

The section-analysis method was used to determine the air-void parameters of hardened cement pastes. The results are compared to those obtained using the point-count method. Five series of air-entrained cement pastes with different water-to-cement ratios were prepared. The point-count method was conducted using a stereoscopic microscope at 50× and the section-analysis method was applied by taking photographs at 80× and reproducing the surface under study. The air content, specific surface, and spacing factor were determined using the point-count method. The air content and the air-void size distributions were obtained using the section-analysis method. For the size of surface area examined and for the air content tested in this study, the results show that the air contents determined using the section-analysis method are lower than the air contents determined using the point-count method by 1.4% to 34.3%. The air-void size distributions obtained using different calculation procedures compare well to each other for all series.

Keywords: Air void system; Petrography; Cement paste; Section analysis; Point count; Spacing factor

Stereology (also known incorrectly as quantitative microscopy) originally was defined as the body of methods used for the investigation of the magnitudes of microstructural features in a three-dimensional material, when only two-dimensional sections through solid bodies or their projections on a surface are available. For several decades, quantitative microstructural analysis has been an important component in the characterization of materials. There are three methods of obtaining data from polished sections or micrographs: the point-count method, the linear-analysis method, and the section-analysis method. These techniques apply to any material and to a variety of shapes of the microstructural features of interest. The principles and the analysis procedures of each technique have been described in the literature [1–3]. The section-analysis technique was mathematically developed much earlier than the linear-analysis and point-count methods and is used extensively in metallurgy for particle size distribution [3]. The section-analysis method has been proven to provide information about size distribution of spheres in a solid more accurately than the linear-analysis method [4]. The section-analysis technique has been used for microstructure analysis to determine total porosity of cement pastes [5]. However, to the authors' knowledge, it has not been used before for air-void analysis in hardened cement-based materials.

Air-void analysis is performed on a smooth, ground concrete section to determine certain air-void parameters and assess whether the concrete mixture contained a proper air-void system. The air-void analysis is carried out using optical microscopes, mainly the stereoscopic microscope. The methods currently used for air-void analysis in hardened concrete are the modified point-count method and the linear-traverse method. These techniques have been standardized and are described in detail in ASTM C 457 for use on ground concrete sections. The air-void parameters determined using the standardized procedures are the air content, the specific surface, and the spacing factor. These parameters provide an indication of the content and size distribution of the air voids in the cement paste. The section-analysis method requires measuring areas of circles on a concrete section, which is very difficult, if not impossible, to measure with a stereoscopic microscope.

Image analyzers were developed during the last two decades and were begun to be used for air-void analysis in concrete. However, the research carried out so far compared the results obtained applying the linear-traverse method using an image analyzer to the results obtained using the optical microscope [6]. The section-analysis method could be used very easily with the image analyzers to determine air content and air-void size distributions.

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Table 1
Summarized results using the point-count method on air-entrained cement pastes

Specimen	Water-to-cement ratio	Total number of points	Length of traverse (mm)	Air content (%)	Specific surface (mm^{-1})	Spacing factor (mm)
A-1	0.40	523	1234.44	2.68	43.27	0.25
A-2	0.40	493	1163.32	2.43	47.48	0.24
B-1	0.45	547	1292.86	1.65	32.72	0.41
B-3	0.45	547	1292.86	1.83	46.85	0.28
C-2	0.50	529	1252.22	1.51	45.16	0.31
C-3	0.50	567	1343.66	1.41	52.76	0.27
D-1	0.55	571	1353.82	1.40	36.30	0.40
D-3	0.55	509	1191.26	2.95	17.56	0.60
F-2	0.60	539	1272.54	1.48	67.09	0.21
F-3	0.60	482	1120.14	2.90	44.09	0.24

1. Purpose and significance

Both standardized techniques currently used for air-void analysis are time consuming and tedious: it takes about 2 to 3 h for an experienced person to complete a measurement. It has been reported that the determination of the specific surface and the spacing factor depends on the air-void system and the presence of entrapped air voids [7,8]. Therefore, it is questionable whether a single value is adequate to provide information about the air-void size distribution. In addition, the spacing factor is not an absolute determination of the frost resistance of concrete: two concretes with the same spacing factor might behave in totally different ways to frost attack.

The purposes of this study were a) to use the section-analysis technique to characterize the air-void system of hardened cement pastes, b) to compare the results obtained using the section-analysis technique to the results obtained using the point-count technique, and c) to compare the air-void size distributions obtained from the section-analysis data using three different calculation procedures. As a first approach for the section analysis, instead of an image analyzer, an area of about 100 mm^2 from each specimen tested

Table 2
Distibution of circular section sizes for the air-entrained cement pastes obtained using the section-analysis method

Section diameter at 80× (mm)	Real section diameter (mm)	Section area (μm^2)	Number of circular sections A-2	B-3	C-3	D-1	F-2
2	0.0250	490.87	35	9	8	40.5	33
3	0.0375	1104.47	48	23.5	25	19	41.5
4	0.0500	1963.50	38.5	24.5	21.5	22	25
5	0.0625	3067.96	52.5	35.5	29.5	8	24.5
6	0.0750	4417.86	31	20	22	5	16.5
7	0.0875	6013.20	14	12	10	3	6.5
8	0.1000	7853.98	23	18	3	4	8.5
9	0.1125	9940.20	16	7.5	5.5	5	2
10	0.1250	12271.85	1	4.5	2	6	7
11	0.1375	14848.93	11.5	4	3	3	3
12	0.1500	17671.46	5	1.5	2	1	4
13	0.1625	20739.42	4	5	1	0.5	0
14	0.1750	24052.82	1	1	0	1	1
15	0.1875	27611.65	1	1	2	0	2
16	0.2000	31415.93	1	4	0	0	0
17	0.2125	35465.64	0	2	2.5	0	0
18	0.2250	39760.78	0	1.5	1.5	0	1
19	0.2375	44301.37	0	1	0	0	1
20	0.2500	49087.39	1	0.25	0	0	1
21	0.2625	54118.84	1	2	4	0	2
23	0.2875	64918.07	0	1	0	0	0
25	0.3125	76699.04	0	0	0	0	1
30	0.3750	110446.62	0	0	0	0	2
31	0.3875	117932.44	0	1	0	0	0
33	0.4125	133640.41	1	0	0	0	0
34	0.4250	141862.54	0	0	1	0	1
36	0.4500	159043.13	0	0	0	0	1
60	0.7500	441786.47	0	0	0	1	0

was photographed and reproduced by pasting the photographs next to each other.

2. Preparation of the sections

2.1. Materials used and preparation of specimens

Five series of air-entrained cement pastes were produced having the following water-to-cement (w/c) ratios: 0.40, 0.45, 0.50, 0.55, and 0.60. The cement used was type I Portland cement from the Allentown Cement Company (Allentown, PA). The water used was deionized water. The air-entraining admixture (AEA) used was a modified salt of a sulfonated hydrocarbon (DAREX) supplied from W.R. Grace Co., Cambridge, MA. The AEA was used at 0.2% by mass of the cement and was added to the materials without adjustment of the total volume. The AEA was first diluted in the water. All subsequent mixing and casting operations were done according to ASTM Standard C 109 except that the product was paste rather than mortar. From each series, three 50.8-mm (2-inch) cubes were cast in brass molds. All mixing operations were carried out at room temperature, approximately 24°C in an air-conditioned laboratory. Immediately upon completion of molding, the specimens were covered with a plastic wrap and kept in a moist, sealed plastic container for 24 h. After 24 h, the molds were stripped and the specimens kept in a moist, sealed plastic container until testing.

2.2. Preparation for testing

When the cubes were 28 days old, they were cut to provide samples for examination. Each cube was cut with a diamond saw 15 mm from the top surface, providing a section parallel to the finished surface. The cut section at the bottom part of the cube was polished to provide a smooth surface for microscopic observations. The preparation included grinding and polishing, which were accomplished by hand on a rotating wheel. Grinding was done by using silicon-carbide grit no. 150 from the Norton Company (Worcester, MA) as abrasive. Successively, two finer abrasives were used for polishing. The medium material was silicon-carbide grit no. 2500, and the fine material was grit no. 1900 aluminum oxide-titanium oxide, both from Lapmaster International (Morton Grove, IL). The purpose of polishing the surface was to remove any deformations induced during the grinding process. At the end of each preparation stage, the specimen was cleaned thoroughly to remove any polishing material from the previous stage and prevent the formation of pits and scratches. No materials were used to enhance contrast between the air voids and the cement paste matrix.

Table 3
Results of air content measured using the section-analysis method

Specimen	Number of sections	Total area scanned A_0 (mm^2)	Total area of circles A_S (mm^2)	Air content (%)
A-2	285.5	92.17	1.54	1.67
B-3	180.5	87.63	1.46	1.67
C-3	143.5	97.61	1.09	1.11
D-1	119.0	91.83	0.84	0.92
F-2	186.5	101.45	1.51	1.48

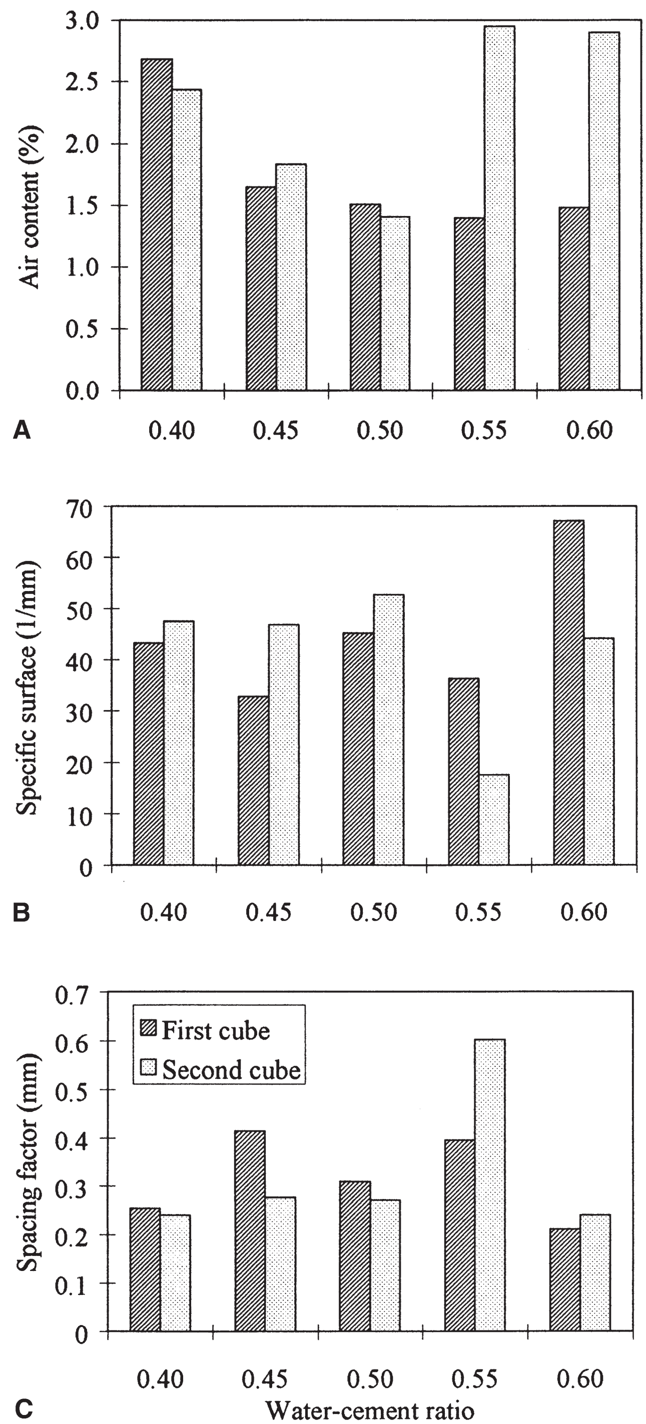

Fig. 1. Air-void parameters determined using the point-count method on cement pastes having different water-to-cement ratios. (*A*) Air content; (*B*) specific surface; (*C*) spacing factor.

3. Testing procedure

3.1. Point counting

Two polished surfaces from each series were analyzed using the point-count method, and one of the two surfaces was examined using the section-analysis method. The microscope used for the point-count method was a Wild M5 stereoscopic microscope (Heerbrugg, Switzerland), which was operated at a magnification of 50×. The system included a hand-driven linear-traverse device. Two spotlight microscope lamps were used to provide illumination at a low and variable incident angle to the surface, so that the air voids were demarked by a shadow. The total number of points was measured for each surface, as well as the number of points crossing air voids. The air content, spacing factor, and specific surface were calculated for each cube separately using the equations given in ASTM C 457. The results of the measurements are presented in Table 1, summarized for all the specimens tested. The area scanned on each polished section was approximately 40 mm × 40 mm.

3.2. Section analysis

Section analysis was carried out on the surface of the same samples that were used for the point-count method. A picture was taken from one area on the polished surface of the specimen at a magnification of 80×. The cube was moved parallel to its initial position in such a way that a neighboring area was photographed. The procedure was followed until approximately 70 pictures were taken from each surface photographing an area of about 100 mm^2 from each cube. It proved very helpful for later reconstruction of the surface by the photographs, to ensure some overlap in the small areas photographed each time. To control the accuracy of detection of air voids on the pictures, the position of each void was observed through the optical microscope and noted during photographing with the aid of a coordinate system integrated in the microscope. Because this procedure proved to be time consuming, only one surface from each series of cement pastes was examined. After processing and developing the films, the photographs were mounted next to each other. This was done to measure the total area correctly without measuring twice the parts of the photographs that were overlapping. With the aid of a circle template, the circular sections were located on the surface and circled with a preassigned diameter circle that fit best to the circular section of the picture. After this was done for all the circles on the photographs, the number of circles for each size was counted. Table 2 presents the results of the size distributions of the circular cross-sections measured on the surface of each cube. The total area of the surface scanned was measured, as well as the total area occupied by circular sections, to calculate the air content. The results of air content for all the samples examined are presented in Table 3.

The air content was determined using Eq. (1). The air content determined using the section-analysis method ranges between 0.92% and 1.67%.

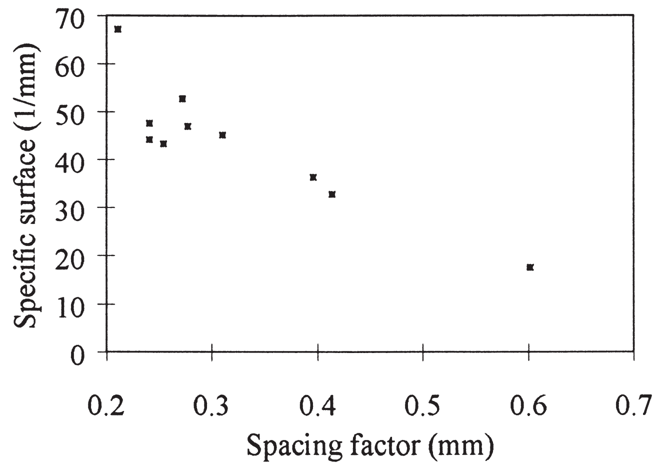

Fig. 2. Specific surface vs. spacing factor determined using the point-count method.

$$A = \frac{A_S}{A_0} \cdot 100 \tag{1}$$

Where A = air content (%), A_S = total area of circular sections (mm^2), and A_0 = total area scanned on the surface (mm^2).

4. Analysis of results and discussion

The results of air content, specific surface, and spacing factor calculated using the point-count method are presented in Fig. 1 separately for each specimen examined. The air content calculated by the point-count method ranges between 1.4% and 2.95% (Fig. 1A). Even some samples from the same mixture showed variation in the results (e.g., D-1

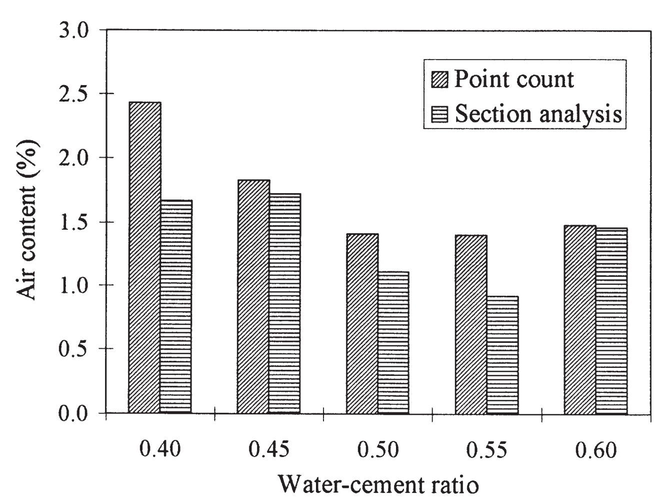

Fig. 3. Comparison of the air content determined using the point-count and section-analysis methods for cement pastes having different water-to-cement ratios.

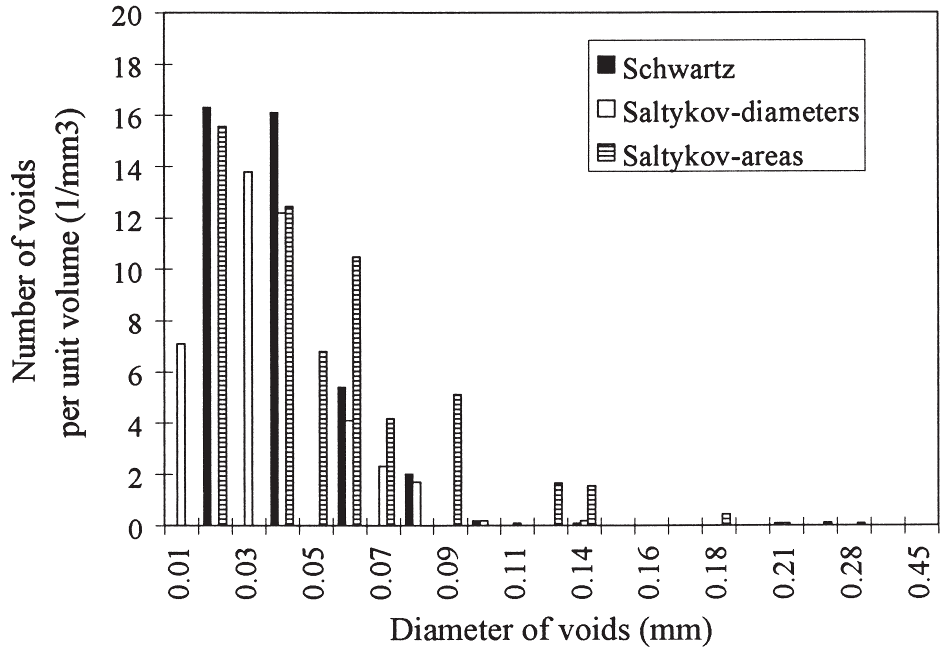

Fig. 4. Air-void size distributions obtained using three different section-analysis procedures on a cement paste with water-to-cement ratio = 0.40.

and D-3, F-2 and F-3). This disagreement could be due to the small surface area examined, which might not be representative of the solid. The values of specific surface calculated for each specimen by the point-count method are presented in Fig. 1B. The values of specific surface range between 17.56 and 67.09 mm^{-1}. The spacing factors calculated for each cube tested are presented in Fig. 1C.

By comparing the results from specimens A-1, A-2 and F-2, F-3, we observe that for a similar spacing factor (0.24 mm), the specific surface might be similar (43.27 and 47.48 mm^{-1}) or quite different (67.09 and 44.09 mm^{-1}). Specimens A-1, A-2, and B-3 have similar specific surfaces (43.27, 47.48, and 46.85 mm^{-1}) and spacing factors (0.24, 0.25, and 0.28 mm), but the air contents are different (2.68%, 2.43%, and 1.63%).

A plot of specific surface vs. spacing factor is presented in Fig. 2. It is expected that for similar air contents, the specific surface tends to decrease as the spacing factor in-

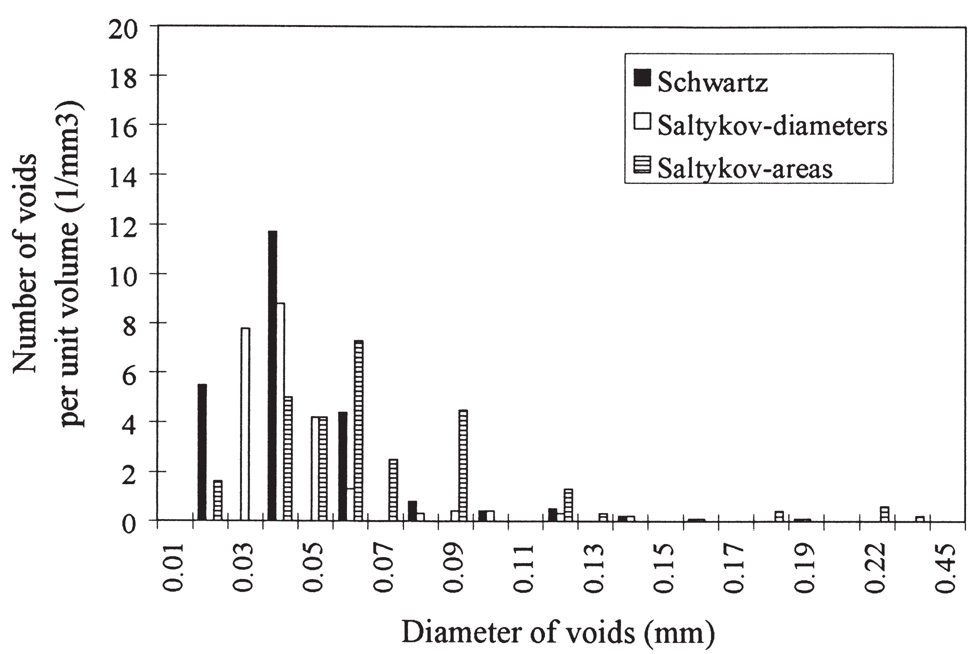

Fig. 5. Air-void size distributions obtained using three different section-analysis procedures on a cement paste with water-to-cement ratio = 0.45.

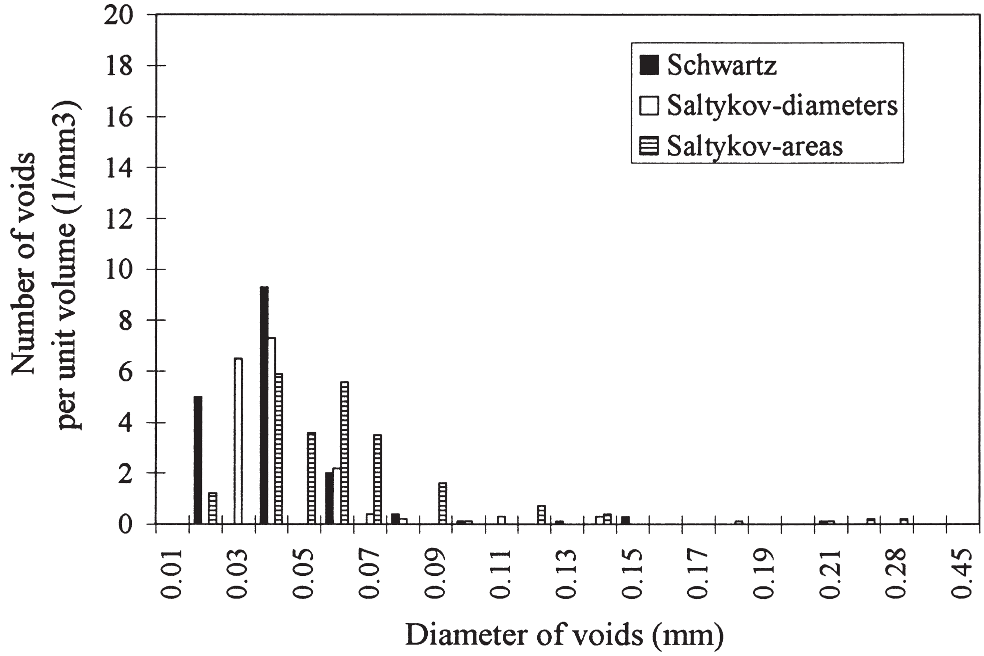

Fig. 6. Air-void size distributions obtained using three different section-analysis procedures on a cement paste with water-to-cement ratio = 0.50.

creases. The results presented in Fig. 2 seem to follow the expected relationship.

The results of air content determined using the point-count method and the section-analysis method are compared in Fig. 3. The results show that the air content calculated by the point-count method is in general agreement with the air content determined using the section-analysis method. The air contents obtained using the section-analysis method are lower than those obtained by the point-count method by 1.4% to 34.3%.

4.1. Air-void size distributions

The section-analysis technique has the major advantage over the point-count technique in that it can give air-void size distributions, whereas the point-count technique can-

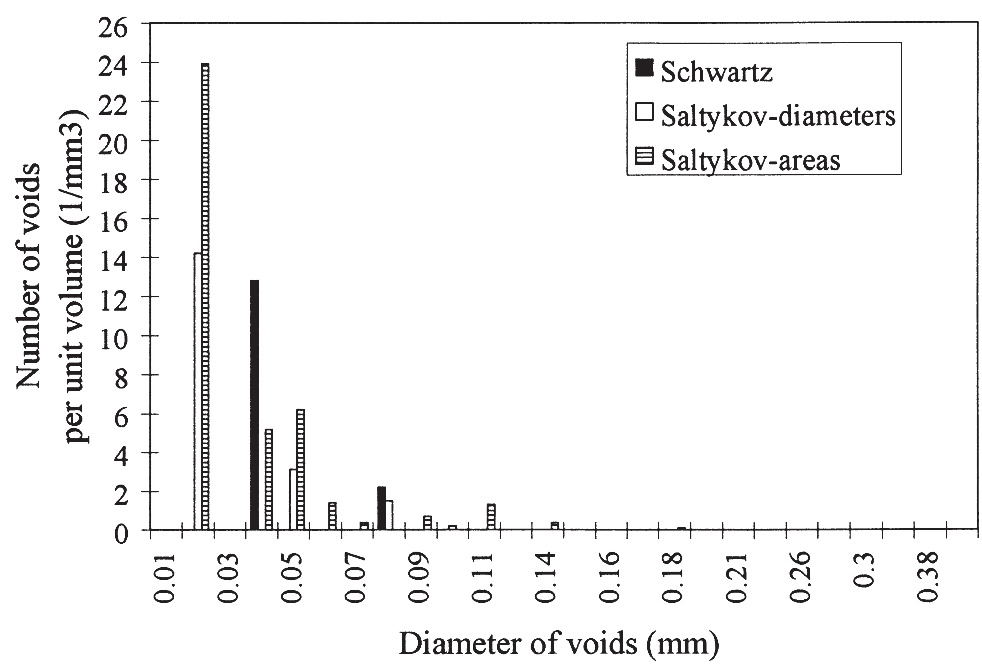

Fig. 7. Air-void size distributions obtained using three different section-analysis procedures on a cement paste with water-to-cement ratio = 0.55.

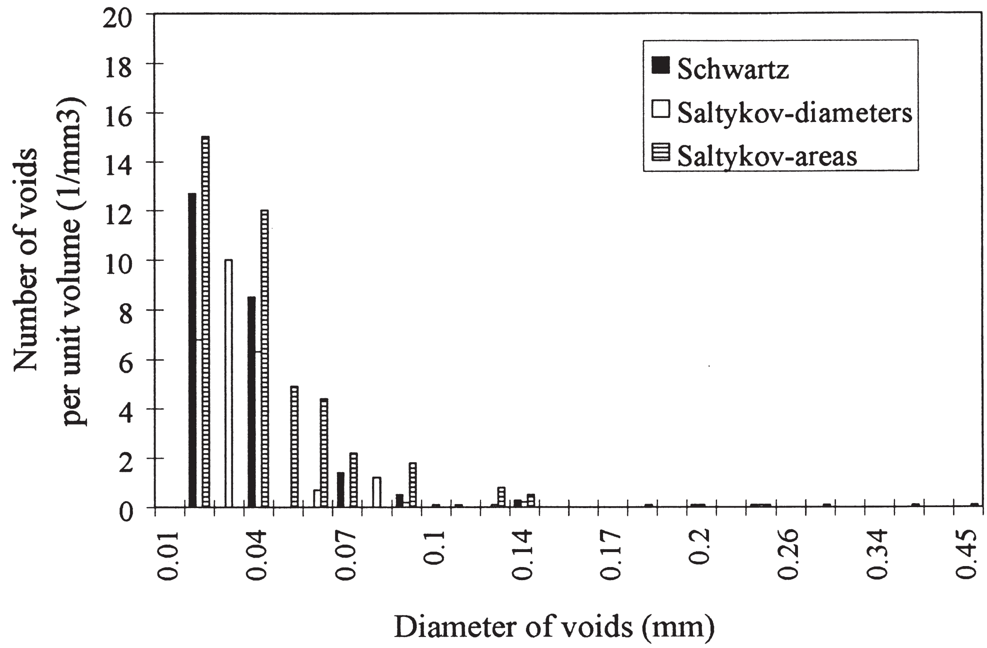

Fig. 8. Air-void size distributions obtained using three different section-analysis procedures on a cement paste with water-to-cement ratio = 0.60.

not. A number of analysis procedures had been developed to provide information about distributions of spheres in a solid. In this study, the following calculation procedures were used to obtain air-void size distributions:

- The procedure developed by Schwartz using circular diameter distributions,
- The procedure developed by Saltykov using circular diameter distributions, and
- The procedure developed by Saltykov using circular areas distributions.

The equations used and the coefficients for obtaining air-void size distributions using circular sections have been presented in the literature for each of the three techniques [9].

The Schwartz analysis and the Saltykov analysis based on diameters of circular sections calculate a fixed diameter for the air voids in each group. The Saltykov analysis based on areas of circular sections gives a range of diameters for each group of air voids. The air-void size distributions calculated by the three different analysis procedures are expressed per unit volume and are shown graphically for each specimen in Figs. 4–8.

By comparing the results of air-void size distributions obtained by the three different analysis procedures, the following observations can be made:

- Each of the calculation procedures results in different values of the radius of the air voids in each group. This results from the way in which the methods were developed. Furthermore, in the Saltykov analysis using areas of the circular sections, the air voids in each group do not have a fixed radius size, but range within radius intervals (however, in the graphs the average radius of each group was used for simplification in comparing results).
- The number of groups obtained by the Saltykov analysis using areas of the circular sections is not fixed (as with the other two methods), but varies depending on the maximum diameter of the circular sections measured.
- The Saltykov analysis using areas of the circular sections tends to spread the air voids to larger radii.
- The Saltykov analysis using areas of the circular sections has not provided any air voids for the group with radii ranging from 0.0125 to 0.016 mm, for all the specimens. This might be the result of the logarithmic scale used for the analysis, which tends to divide voids with smaller diameters into more groups than voids with larger diameters.
- The values of diameters of the air voids determined using the three analysis procedures range between 0.01 and 0.45 mm, which is in agreement with the values given in the literature.
- The air voids seem to follow a log-normal distribution, which already has been noted in previous research [10].

5. Conclusions

For the cement pastes used, the air contents tested, and the surface area scanned in this study, the following conclusions can be drawn:

- The specific surface and the spacing factor do not always agree well with the trends expected from the air content.
- The section-analysis method can be done relatively easily once the air voids are detected.

- The section-analysis method tends to underestimate the air content compared to the point-count method.
- The air voids obtained using the section-analysis method seem to have a log-normal distribution.
- The section-analysis technique can facilitate air-void determination because a smaller area can be tested.
- The Saltykov analysis using areas provides more information about voids with small sizes than the other analysis procedures.

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