

DISTRIBUTION OF VOIDS IN FIELD CONCRETE

by

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(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

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SUMMARY

This study was intended to evaluate the air void characteristics of concrete in an attempt to identify, quantitatively or semiquantitatively, different types of voids and to predict their influence on strength and durability. At the outset, it was anticipated that total air contents and water-cement ratios of concrete mixtures would reflect the presence of excess water or air voids. However, because it is difficult to determine the water-cement ratio of hardened concrete, an attempt was made to relate the water-cement ratio and the air content, taken as independent variables, to the parameters of the void system that are readily determined from the hardened concrete. The parameters of the void system considered were:

1. The specific surface and the spacing factor calculated from linear traverse data.
2. The constants of a continuous chord frequency distribution curve derived from a probability density function.
3. The mean diameters and the number of bubbles per unit volume determined by a graphical method.

The concrete samples were prepared in the laboratory, and the variables included the amount of air entrainment, the water-cement ratio, the mixing time, and the mixture temperature. The data on the void system were obtained by utilizing the chord lengths of voids determined by the linear traverse analysis of concrete specimens. Within the framework of the study it was not possible to correlate the combined effect of water and air content with the above mentioned void parameters. A good correlation was obtained between the air contents of fresh and hardened concretes that were properly measured. Satisfactory relationships were established between the measures of resistance to freezing and thawing (weight loss, relative dynamic modulus of elasticity, and the surface rating), and certain parameters of the void system (the spacing factor, the specific surface, and the diameter of the bubbles).

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INTRODUCTION

Over the years some concrete samples from projects administered by the Virginia Department of Highways and Transportation and utilizing approved materials have exhibited lower than anticipated strengths. In most cases petrographic examination of these samples has revealed that the void contents measured in hardened concrete were considerably higher than the air contents recorded in the field for the corresponding fresh concretes. These findings are not in keeping with the results of a study performed at the Research Council nor data from other sources, both of which have shown that the void contents of fresh and hardened concrete samples representing the same conditions usually agree closely.^(1,2) At the Research Council, this agreement was found to be within ± 1 percentage point in air content. The higher void contents observed in some of the hardened field concretes could be attributed to extra air entrainment, improper measurements of the air content, inadequate consolidation of the fresh concrete, or the addition of excess water to the mixture, especially the addition of water subsequent to the field measurements of air content. The distinction between entrained air voids and the voids from water is based primarily on the shape and size of the voids as determined in petrographic examinations.

It would be desirable to have a quantitative or semi-quantitative method for differentiating between small voids measured in freshly mixed concrete and those generated by any subsequent addition of water. Therefore, this investigation was intended to evaluate the air void characteristics of concrete in an attempt to identify different types of voids generated by air entrainment or extra water and to predict their influence on strength and durability.

OBJECTIVES

The initial objectives of this study were listed in the

working plan⁽³⁾ as follows:

1. To investigate the increased air content observed in hardened concrete from field samples as compared to measurements for freshly mixed, air entrained concrete. Chord and bubble size distributions will be calculated by computer from existing linear traverse data for lab and field concrete specimens.
2. To transfer the visual linear traverse data on the void sizes classified as greater or smaller than 1 mm from a plane to a volume. These results will be compared with the quantity of voids in three dimensions calculated from the chord distributions of the linear traverse data by means of computers.
3. To examine the possibility of altering the recording device of the linear traverse machine so as to record onto a computer tape.

However, as the study progressed it was decided that the objectives given below would be more appropriate.

1. To relate the void contents of the same batches of concrete at the fresh and hardened stages.
2. To differentiate between the water voids and air voids.
3. To relate measures of durability — weight loss, relative dynamic modulus of elasticity and the surface rating — to the following parameters of the void system: specific surface, spacing factor, mean diameter of bubbles and the number of bubbles per unit volume, and the air content of voids having a diameter < 1 mm.

The initial third objective was considered to be an evaluation and was achieved.

BACKGROUND

Pore Structure

The amount and the size of pores and voids in concrete influence its properties. In freshly mixed concrete, there are both water- and air-filled voids. As hydration proceeds, gel pores and capillary pores are produced in the space initially

occupied by water. Depending on the water-cement ratio and the degree of hydration of the cement, capillary pores may be considered to range from $8 \times 10^{-3} \mu\text{m}$ to $13 \mu\text{m}$ in hardened concrete.⁽⁴⁾ Gel pores are generally much smaller than the capillary pores, with diameters about $1.5 \times 10^{-3} \mu\text{m}$ to $2 \times 10^{-3} \mu\text{m}$.⁽⁵⁾ The concrete also contains other types of voids. Of these, the ones in the range from 10 to 1,000 μm with the majority under 100 μm are from purposefully entrained air and the larger ones are from unintentionally entrapped air or water voids. Aggregates also contain pores of different sizes. These pores usually are at least the size of the largest capillary pores in the concrete. Voids of any size in concrete generally reduce its strength; however, the far more important protection the small and spherical entrained air voids provide against damage from freezing and thawing necessitates the use of air entrainment at an optimal level in highway structures. On the other hand, the entrapped air or water voids are undesirable and should be minimized or removed by proper consolidation and/or mixture proportioning.

Air Content Determination

Generally, in the laboratory the air content measurements are performed on freshly mixed, normal weight concrete in accordance with ASTM Method C231. This is a pressure method based on the principle that entrained or entrapped air is the only compressible portion of the fresh mixture.⁽⁶⁾ In the field the air contents are determined in a simple way using a pocket size indicator called the Chace air meter. In both methods the water filled voids are undetected. The void system in hardened concrete is determined using ASTM Method C457-71, which includes the linear traverse and the modified point count methods. In the linear traverse method the void content of concrete is determined by integrating bubble chords traversed along regularly spaced lines. Utilizing ASTM C457-71, other useful information such as the specific surface and the spacing factor of the voids and the air-paste ratio can be obtained.

Even though the determinations of air content on fresh and hardened concretes were performed by two entirely different test procedures, it was found that the amounts of air measured on the same concrete at different stages agree closely with each other as mentioned in the introduction.

Freezing Mechanism

The freezing and thawing deterioration is caused by the expansion of freezing water either in the aggregates, the paste, or both. Water occupies 9% more space after freezing. Therefore, if concrete is completely or critically saturated some of the water will tend to be forced out of the pores during

freezing. Internal hydraulic pressures can develop to an extent that will rupture the concrete as the flow of water is resisted by the surrounding concrete.(7)

Many capillary pores are large enough that water can freeze in them, but water cannot freeze in small gel pores at normal freezing temperatures. However, gel water can diffuse into capillary pores and thus contribute to the growth of ice crystals that can cause expansion sufficient to bring about disintegration.(8)

Deicing salts also contribute to the deterioration of concrete caused by freezing and thawing. Their harmful effects result from the possible generation of osmotic pressure, the production of additional moisture by melting the surface ice or snow, and, at high concentrations, the formation of salt crystals in subsurface voids.(9)

Air Entrainment

Air voids purposefully created in concrete improve certain characteristics of the fresh as well as the hardened concrete. These small, nearly spherical voids are isolated and are almost uniformly distributed throughout the paste. Entrained air voids mainly protect the hardened concrete from frost damage, but in addition they improve the workability and reduce segregation and bleeding in plastic concrete.(10,11,12)

Initially the air voids are filled with air and are difficult to fill with water because of their relatively large size.(13) During freezing some of the water forced out of the critically saturated capillary or aggregate pores generates hydraulic pressures in the surrounding paste. If the excess water moves into an air void, the pressure buildup in the paste does not reach a level that causes failure. The generation of internal pressure is affected by parameters such as the permeability of the paste, the degree of saturation, the distance from the capillary pore to an air void, and the rate of freezing.(9) The air voids should be spaced closely and uniformly in order to relieve the internal pressure that could cause disruption in concrete.

Powers Spacing Factor

Powers introduced the concept of the spacing factor, \bar{L} , to estimate the average spacing of bubbles in paste.(14,15) As given in ASTM C457,

$$\bar{L} = 1/(4 \times n_p) \quad \text{when } P/A < 4.33, \text{ and}$$

$$\bar{L} = 3/\alpha [1.4 (P/A) + 1]^{1/3} - 1 \quad \text{when } P.A \geq 4.33,$$

where,

\bar{L} = the spacing factor,

n_p = the average number of air void sections intersected per unit length of traverse in the paste,

P = paste content, in percent,

A = the total volume of air voids, in percent, and

α = the specific surface of the air voids.

\bar{L} indicates the average distance water must travel to reach a protective void and thus reduce the internal hydraulic pressure. Powers estimated the maximum allowable spacing factor to be 0.01 in. (250 mm) for satisfactory freeze-thaw performance. Subsequently, Mielenz et al. suggested a maximum effective spacing factor of 0.008 in. (0.2 mm).⁽¹⁶⁾

Philleo Spacing Factor

Philleo points out that Power's spacing factor, \bar{L} , can be applied only to concrete with similar void size distributions since it is based on the total air content and surface area.⁽¹⁷⁾ Also, \bar{L} is an average spacing factor and the performance of concrete might be determined by that half of the bubbles that are larger than the average spacings.⁽¹⁷⁾

To eliminate the above limitations, Philleo introduced a spacing factor concept based on a knowledge of the total volume of air in the paste and the number of voids per unit volume of the paste. Under his concept, the fraction of paste within a specified distance of the nearest void is calculated as

$$F' = 1 - \exp. \left\{ \begin{array}{l} -4.19(SN^{1/3})^3 - 7.80(SN^{1/3})^2 \left[-\log(1-A) \right]^{1/3} \\ -4.84(SN^{1/3}) \left[-\log(1-A) \right]^{2/3} \end{array} \right\},$$

where

S = the Philleo spacing factor,

A = the fraction of air in paste,

F' = the fraction of air-free paste within distance S from edge of nearest void, and

N = the number of bubbles per cubic inch.

The void distribution needed for the calculation of the Philleo spacing factor can be obtained by a graphical method presented by Lord and Willis.⁽¹⁸⁾ In their method, chord lengths obtained by the linear traverse are classified into discrete small groups, a chord frequency distribution is generated, and then the number of voids in each size group is computed.

Research by Larson et al. has shown that Philleo's spacing factor is a reliable indicator of the freeze-thaw resistance of concrete.⁽¹⁹⁾

PROCEDURE

In the current practice at the Research Council, to differentiate between the small entrained voids and the large undesirable voids, the voids are separated into two groups on the basis of size, with the dividing line being a void diameter of 1mm (0.04 in.) on a plane surface. The diameters of the bubbles are determined visually and the total amount of voids is calculated from the total chord lengths intercepted along a length of traverse. In this study, a means of differentiation of bubbles other than the above mentioned visual way was sought. It was anticipated that the water-cement ratio (w/c) and the air content (A) of the concrete mixtures would reflect the presence of extra water voids or air voids in concrete. However, because it is difficult to determine the w/c in hardened concrete, an attempt was made to correlate the w/c and A, taken as independent variables, to the specific surface and the spacing factor that can readily be determined from the linear traverse data from hardened concrete. The specific surface (α) and the spacing factor (\bar{L}) give an indication of the performance of concrete exposed to freezing and thawing. This assumption was investigated by relating the measures of freezing and thawing durability, which are the weight loss (WL), relative dynamic modulus of elasticity (RDM), and the surface rating (SR), to α and \bar{L} . Concrete samples were fabricated in the laboratory to obtain values for w/c, A, α , and \bar{L} as explained later under "Concrete Specimens and Test Results". Some of the specimens were exposed to rapid freezing and thawing and the WL, RDM, and SR were calculated and correlated to α and \bar{L} by a linear regression analysis. Also, a relationship between the air contents of the same concrete measured at the fresh and hardened stages was established. Other specimens were obtained from a past laboratory study and the field and were evaluated qualitatively to determine the relations between w/c, A, and α and \bar{L} .

At the Research Council, the individual chord lengths that are summed to obtain the total chord length are recorded on paper tape. These individual chord lengths were utilized to obtain void system parameters through a probabilistic and a graphical approach. The data needed were obtained from the laboratory and field specimens mentioned above. In the probabilistic method a continuous curve was obtained and the constants of this curve were found under different mixture characteristics. In the graphical method, the parameters derived were the mean diameters and the number of bubbles per unit volume. As in the case with α and \bar{L} , the parameters from the probabilistic and the graphical methods were correlated with w/c and A . The relationship sought was

$$y = C_0 + C_1 (w/c) + C_2 (A),$$

where

y = the void parameter that gives the best correlation with w/c and A ,

C_0, C_1, C_2 = constants

w/c = the water-cement ratio, and

A = air content.

In this study the above relationship was not established satisfactorily. However, if it could be, a table of y values would be generated for various combinations of w/c and A . Further, an estimate of the w/c for a field sample for which A and y have been measured could be attempted.

The parameters from the graphical method were also related to the measures of durability — WL, RDM, and SR — to find the parameter giving the best indication of the freezing and thawing resistance.

Specimens and Test Results

Present Laboratory Study

Materials

In the laboratory 2-ft.³ (572-dm³) batches of concrete were prepared utilizing Type II cement having the chemical and physical characteristics given in Table 1. The coarse aggregate used was granite gneiss with a specific gravity of 2.78, an absorption of 0.5, and dry rodded unit weight of 100 lb/ft.³ (1.6 Mg/m³).

Table 1

Chemical and Physical Characteristics
of Type II Cement Used

<u>Chemical</u>		<u>Physical</u>	
SiO ₂	22.13	Fineness (Blaine)	3,660
Al ₂ O ₃	4.47	Soundness (%)	0.03
Fe ₂ O ₃	2.82	Time of Set (min.)	155
CaO	63.67	Compressive Strength (psi)	
MgO	2.54	1-day	1,420
		3-day	2,700
SO ₃	2.54	7-day	3,500
Total Alkalies	0.68	Air Content (%)	10.2

Note: 1 psi = 6.89 kPa

The fine aggregate used in the first six batches shown in Table 2 was quartz sand with a specific gravity of 2.59, a fineness modulus of 2.99, an absorption of 0.9, and void content of 50.0%. In the seventh batch, sand with a higher void content, 52.1%, was used. It had a specific gravity of 2.60, fineness modulus of 2.70, and absorption of 1.1%. The eighth batch incorporated a sand with a relatively low void content of 47.5%, a specific gravity of 2.58, and an absorption of 0.7. In all of the mixtures a commercially available air entraining solution, a neutralized vinsol resin, was used. Also, a water reducing and a set controlling admixture was utilized in all the batches except the first one. This admixture is commercially available and is a blend of lignosulfonic and hydroxylated carboxylic acid. Both of the admixtures are approved by the Virginia Department of Highways & Transportation for use in concrete.

TABLE 2

Summary of Batches Prepared in the Laboratory

Batch Number	Variable	W/C	Mix Temp. °F	Slump, in.	Air, % (Fresh)	Air, % (Hardened)	Air, % < 1 mm Diameter	Specific Surface, in. -1	Spacing Factor, in.	28-day Comp. Strength, psi
1 A	Reg. A 4	0.42	70	2.6	5.0	5.3 *	3.4 *	607 *	0.0080	6,370
1 B	Add water	0.54	70	6.6	5.7	5.9	4.0	615	0.0074	4,930
2	High air	0.42	70	7.0	11.1	10.2	7.4	592	0.0047	5,820
3	High air	0.39	72	3.6	11.0	7.8	6.1	831	0.0042	6,380
4 A **	High air	0.42	72	4.1	8.2	6.1	4.3	745	0.0060	6,415
4 B **	Agitate 90 min.; add water	0.49	72	4.4	6.2	5.7	3.5	538	0.0088	5,575
5 A	Reg. A 4	0.42	69	3.8	6.2	5.0	3.4	692	0.0070	6,960
5 B	Add water	0.46	70	5.5	5.3	4.6	2.9	690	0.0075	6,385
5 C	Mix 25 min.	0.46	76	1.2	3.4	2.9	1.5	566	0.0112	7,240
5 D	Add water	0.52	74	5.0	4.0	3.8	2.2	562	0.0106	5,870
6 A	Temperature	0.42	102	2.1	4.6	3.6	2.3	650	0.0087	7,240
6 B **	Add water	0.51	100	5.0	6.8	5.4	3.3	598	0.0082	6,145
6 C **	Agitate 75 min.; add water	0.58	90	4.0	5.4	5.5	3.5	568	0.0091	6,065
7 A	Temp.; F. A. high void content	0.42	101	0.4	3.9	3.1	1.6	503	0.0120	--
7 B **	Add water	0.50	101	2.9	5.0	4.6	2.8	526	0.0099	6,610
7 C	Agitate 35 min.	0.50	101	0.9	4.3	3.4	1.6	391	0.0156	--
7 D **	Add water	0.58	101	3.0	4.7	4.1	2.3	514	0.0113	6,004
8 A **	Temp.; F. A. low void content	0.42	100	2.7	7.2	4.3	2.9	632	0.0083	7,130
8 B **	Agitate 30 min.	0.42	100	1.0	4.6	3.8	2.0	516	0.0107	7,920
8 C **	Add water	0.51	98	4.2	6.2	5.7	3.3	569	0.0085	6,064

NOTE: $t_c^0 = (t_F - 32)/1.8$;

1 in. = 25.4 mm; 1 psi = 6.89 kPa.

* = Average of 3 specimens

** = Two prisms were prepared for freezing and thawing test.

Batches of Samples

Class A4 (superstructure) concrete of the Virginia Department of Highways & Transportation was used in the control batches. This concrete has a minimum 28-day compressive strength of 4,000 psi (27.6 MPa). The specified minimum cement content is 634 lb./yd.³ (376 kg/m³) and the maximum w/c is 0.47. A slump of 2-4 in. (50-100 mm) and an air content of 6 1/2 % \pm 1/2% are required. Initially, eight batches of concrete were prepared in the laboratory. The mixture variables were the amount of air entrainment, the w/c, the mixing time, and the mixture temperature as shown in Table 2. In most cases the batches were retempered to obtain the different variables such as the w/c and the mixing time. In the first column of Table 2 the numbers indicate the batch numbers and the letters designate the different stages of mixing at which the variables were introduced. The specimens were prepared in accordance with ASTM C192-69. When a mixture variable was introduced into the batch a 6 x 12 in. (150 x 300 mm) cylinder was prepared for petrographic examination. However, in the first batch for a regular A4 concrete three cylinders were fabricated to determine any possible variability in measurements. Each cylinder was cured in the moist room for at least 28 days and then slabs with finely lapped surfaces, one taken 2 in. from the top and the other 2 in. from the bottom, were prepared for petrographic examination in accordance with ASTM C457-71. For each mixture variable three 3 x 3 in. (150 x 300 mm) cylinders were fabricated, moist cured, and tested to determine the compressive strengths at 28 days in compliance with ASTM C39-72. Also two prisms of 3 x 4 x 16 in. (75 x 100 x 400 mm) were prepared from some batches as indicated in Table 2. They were moist cured for 14 days, air dried for 7 days, and then subjected to rapid freezing and thawing in the presence of 2% NaCl₂ following ASTM C666-73.

Results

The measurements of mixture temperature, slump as determined by ASTM C143, and air content of the fresh concrete as determined by ASTM C231 are shown in Table 2. The void content of hardened concrete, the specific surface, and the spacing factor calculated in accordance with ASTM C457-71, and the compressive strength values are also depicted in the table.

The three specimens obtained from the first batch to determine testing variability yielded a standard deviation of 87 in.⁻¹ (3.4⁻¹ mm) for the specific surface values and 0.0004 in. (0.010 mm) for the spacing factor. The coefficients of variation are 14% and 5%, respectively.

The void contents of the fresh and hardened concretes are shown in Figure 1. A linear regression analysis was employed based on the least square method and the line of best fit was determined and is represented in Figure 1 by the broken line. The correlation coefficient calculated was 0.88. The data indicate that void contents measured on hardened concrete are in good agreement with air contents determined on related samples of fresh concrete. This finding is consistent with those from earlier studies.(1,2)

The void contents of fresh and hardened concretes obtained from freshly mixed samples not subjected to retempering showed a good correlation coefficient of 0.85. The line of best fit was found to be $y = 1.1 + 0.7 \alpha$, where α and y are the void contents of the fresh and hardened concretes, respectively.

The specific surface values range from 391 in.⁻¹ (15.4 mm⁻¹) to 831 in.⁻¹ (32.7 mm⁻¹) and the spacing factor from 0.0042 in. (0.1067 mm) to 0.0156 in. (0.3962 mm), with the maximum values of the former corresponding to the minimum values of the latter. The compressive strength values of 3 x 6 in. (150 x 300 mm) cylinders ranged from 4,930 psi (34.0 MPa) to 7,920 psi (54.6 MPa). Those with a low w/c and air content attained the higher compressive strengths, while all exceeded the minimum A4 requirements. Prolonged mixing or agitation tended to reduce the specific surface of bubbles. Adding water into the mixture increased the void content of the hardened concrete, and prolonged mixing or agitation tended to decrease it, as would be expected. The slump of the mixtures was also increased by the addition of water, but additional mixing and agitation reduced it.

The rapid freezing and thawing data are summarized in Table 3. Only the sixth batch showed a weight loss and surface rating in excess of the criteria established by the Research Council for satisfactory performance; namely, a maximum weight loss of 7% and a surface rating of 3 or less. Mixtures with a low w/c and adequate air entrainment exhibited satisfactory resistance to freezing and thawing. In general, the addition of water had adverse effects on the resistance to freezing and thawing.

It was observed that the freezing and thawing results reflected the fact that the specimens did not show a wide range of air contents and most were adequately air entrained so that the specimens generally performed well. Therefore, two other available concrete batches were included in the study to achieve a wider range of performance. The first batch did not contain air entrainment and was prepared with Type II cement. It had an air content of 3.0% as measured by the pressure method. The petrographic examination revealed a void content of 3.2%, a specific surface of 200 in.⁻¹ (7.9 mm⁻¹), and a spacing factor of 0.0308 in. (0.7823 mm). The rapid freezing and thawing test

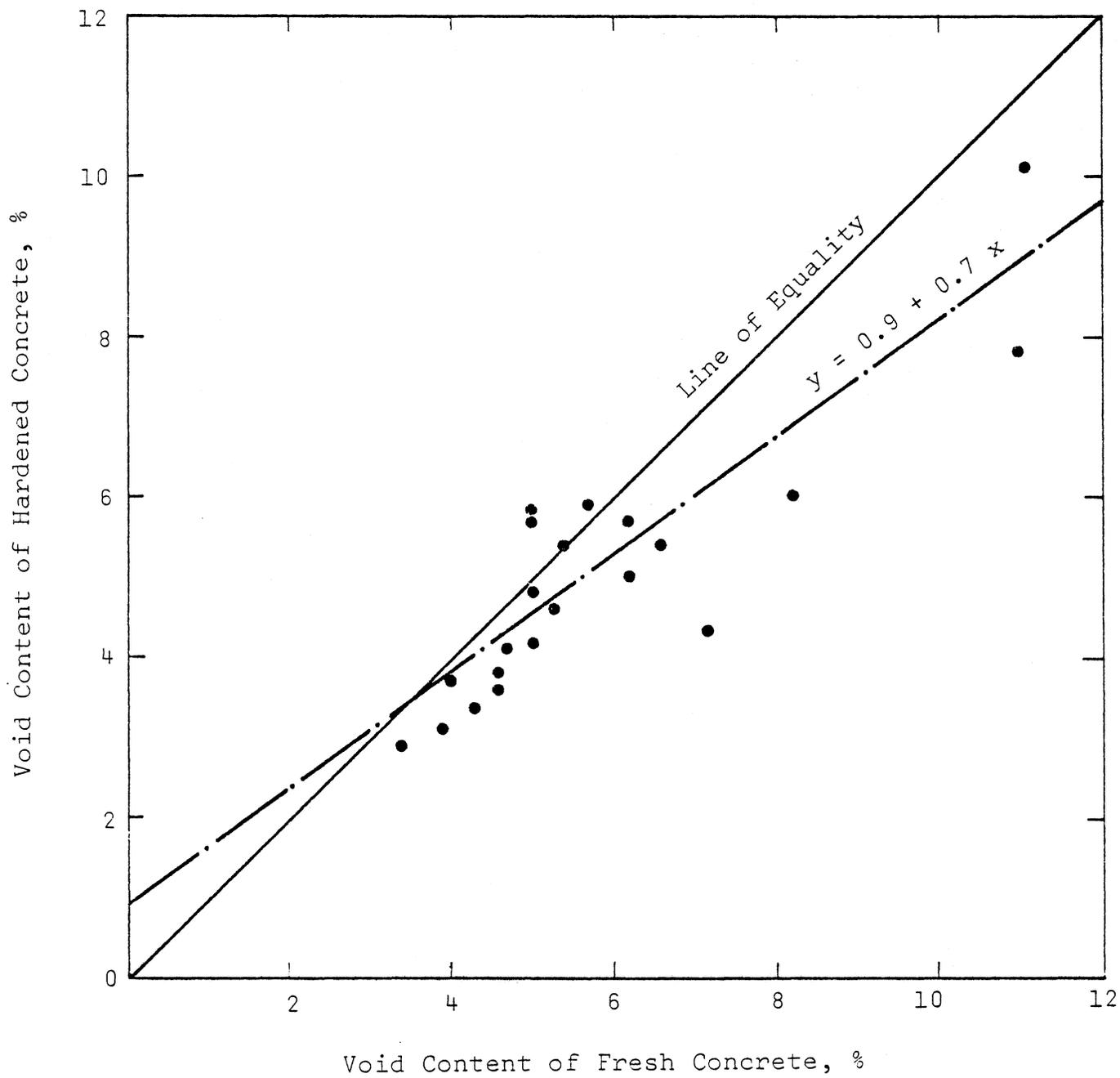


Figure 1. Void contents in fresh and hardened concretes.

Table 3

Rapid Freezing and Thawing Data

Batch Numbers	W/C	Air Content, %	Weight Loss, %		Durability Factor		Surface Rating	
			100 cycles	300 cycles*	100 cycles	300 cycles*	100 cycles	300 cycles*
4A	0.42	6.1	0.4	1.1	100	100	1.0	1.5
4B	0.49	5.7	1.2	5.4	98	97	1.6	3.0
6B	0.51	5.4	3.6	11.0**	96	70**	2.4	4.0**
6C	0.58	5.5	4.1	11.8**	98	72**	2.5	4.0**
7B	0.50	4.6	0.7	2.4	99	100	1.3	2.3
7D	0.58	4.1	1.5	5.2	99	92	1.7	2.7
8A	0.42	4.3	0.4	0.9	99	100	0.8	1.6
8B	0.42	3.8	0.5	1.6	98	100	1.2	2.0
8C	0.51	5.7	0.8	2.3	100	100	1.2	2.2

*Average of two specimens.

**Projected from 225 cycles.

for this concrete mixture had to be terminated at 55 cycles due to heavy deterioration. The weight loss, dynamic modulus of elasticity, and the surface rating values were projected to 100 cycles and found to be 14%, 27, and 4, respectively. The second batch was prepared with Type IP cement. The petrographic examination yielded a void content of 2.6%. The weight loss, dynamic modulus of elasticity, and the surface rating were 5.8%, 100, and 3.5 at 100 cycles.

The w/c and the air content, taken as independent variables, were regressed with the specific surface, α , and then with the spacing factor, \bar{L} , taken as dependent variables for the specimens incorporating Type II cement. When the specific surface and the spacing factor were taken as dependent variables, correlation coefficients of 0.517 and 0.559 were obtained. Both of these values were assumed to yield poor correlations; i.e., only about a fourth of the variation in the dependent variable was explained by the relationship. Thus, it was not possible to regress the w/c and the air content satisfactorily with either α or \bar{L} on the basis of the limited number of samples prepared.

The values for freezing and thawing resistance, weight loss, relative dynamic modulus of elasticity, and surface rating at 100 cycles, each taken separately as a dependent variable, were correlated with the w/c and A taken as independent variables utilizing a multiple regression analysis. Low correlation coefficients of 0.546, 0.411, and 0.659, respectively, were obtained. The same values of durability were correlated linearly to α , \bar{L} , and the air content of voids < 1 mm. The correlation coefficients, summarized in Table 4, indicate a satisfactory correlation, except in the case of surface rating versus the air content of voids < 1 mm. The spacing factor correlates very well with the weight loss and dynamic modulus of elasticity. The lower correlation factors with surface rating would be expected because of the subjective nature of the ratings.

Table 4

Correlation Coefficients for Measures of Durability
Related to the Specific Surface, Spacing Factor, and Air
Content of Voids Having a Diameter < 1 mm

<u>Variables</u>	<u>Specific Surface</u>	<u>Spacing Factor</u>	<u>A < 1mm</u>
Weight loss	-0.845	0.930	-0.771
Dynamic modulus	0.829	-0.941	0.735
Surface rating	-0.760	0.778	-0.699

Previous Laboratory Results

In a previous, unpublished study at the Research Council, water was added at intervals during the mixing of a batch of concrete utilizing 611 lb./yd.³ (9.8 Mg/m³) of cement. The slump, air content, and temperature of the mixture were then determined (see Table 5), and specimens for compressive strength testing and petrographic examination were molded. This batch of concrete contained an air entraining agent and a set retarding admixture.

There was a decrease in strength as the w/c increased, as would be expected. The additional water and mixing did not change the specific surface or the spacing factor significantly. Even though more voids are introduced into the mixture as a result of extra water, the additional mixing tends to dissipate some of the air voids and possibly decrease their size or divide the large air voids.

Field Specimens

Included in the investigation were a few specimens prepared for a project on two-course bonded bridge deck construction conducted by S. S. Tyson of the Research Council.⁽²⁰⁾ Class A4 concrete was used in the specimens and the w/c was kept at its maximum permissible limit of 0.47. The slump, temperature, and air content of the mixture were obtained in the field and are shown in Table 6. Also, specimens were prepared for compressive strength testing and petrographic examination, and the results are summarized in Table 6.

The specific surface values ranged from 614 in.⁻¹ (24.2 mm⁻¹) to 772 in.⁻¹ (3.04 mm⁻¹) and the spacing factors from 0.0053 in. (0.1346 mm) to 0.0078 in. (0.1981 mm), with the highest specific surface values corresponding to the lowest spacing factor values. The compressive strengths from 6 x 12 in. (150 x 300 mm) cylinders varied from 3,710 to 4,620 psi (25.6 to 31.8 MPa).

Specimen number 14 in Table 6 was obtained from a bridge deck concrete that showed poor consolidation. The high spacing factor of this specimen made it susceptible to deterioration from freezing and thawing.

The laboratory specimens from the previous study and the field specimens both were evaluated qualitatively; no direct quantitative comparisons were made with the present laboratory samples since the admixtures used were unknown or different in all cases.

Table 5
Summary Test Results from Previous Study

Batch Number	w/c	Mix Temperature, °F	Slump, in.	Percent Air, Fresh Concrete	Percent Air, Hardened Concrete	Specific Surface, in. ⁻¹	Spacing Factor, in.	28-day Compressive Strength, psi
9A	0.49	69	3.8	7.0	6.0	767	0.0060	5,100
9B	0.56	72	3.5	6.2	4.8	718	0.0069	5,050
9C	0.64	73	3.4	5.2	5.1	764	0.0069	4,510

Note: 1 in. = 25.4 mm; 1 psi = 6.89 Mpa
 $t_{C}^{0} = (t_{F}^{0} - 32)/1.8$

Table 6
Samples Obtained from Tyson Study and the Field

Batch Number	w/c	Mix Temperature, °F	Slump, in.	Percent Air, Fresh Concrete	Percent Air, Hardened Concrete	Specific Surface, in. ⁻¹	Spacing Factor, in.	28-day Compressive Strength, psi
10	0.47	79	3.2	7.4	7.8	772	0.0053	3,710
11	0.47	82	4.9	4.6	5.8	715	0.0066	4,620
12	0.47	85	5.0	6.0	6.3	614	0.0078	4,390
13	0.47	86	5.5	7.0	7.5	674	0.0059	4,330
14	0.47	---	---	---	5.1	563	0.0089	---

Note: 1 in. = 25.4 mm; 1 psi = 6.89 kPa; $t_{C}^{0} = (t_{F}^{0} - 32)/1.8$

Probabilistic Method

It is suggested in the literature⁽¹⁹⁾ that the chord frequency distribution of the voids in concrete can be approximated by a continuous function as

$$\phi(x) = \frac{a x}{b^x},$$

where

$\phi(x)$ = the frequency,

x = the chord size, and

a and b = constants for a given distribution.

This equation, as noted by DeGroot,⁽²¹⁾ can be derived from the general gamma distribution with parameters α and β that has a continuous distribution whose probability density function f is

$$f(x) = \begin{cases} \frac{\beta^\alpha}{\Gamma(\alpha)} x^{\alpha-1} e^{-\beta x} & \text{for } x > 0 \\ 0 & \text{for } x \leq 0 \end{cases}$$

for $\alpha = 2$,

$$f(x) = \frac{\beta^2 x}{e^{\beta x}} = \frac{a x}{b^x} = \phi(x)$$

Based on the available literature and the shape of the chord histogram the special case of the gamma distribution given above was chosen as the most preferred one.

If the frequency distribution of chords could be represented by the above probability density function, there would be a possibility of studying the characteristics of concrete by a few chord lengths that would be sufficient to summarize the complete data set.

To evaluate the validity of the probabilistic model, the probability density function $\phi(x)$ was regressed linearly to the observed chord lengths on some laboratory specimens. The frequency distribution of the chord lengths was plotted in chord intervals of 20 μm for an air entrained and a non-air entrained concrete as shown in Figure 2. In the air entrained concrete the

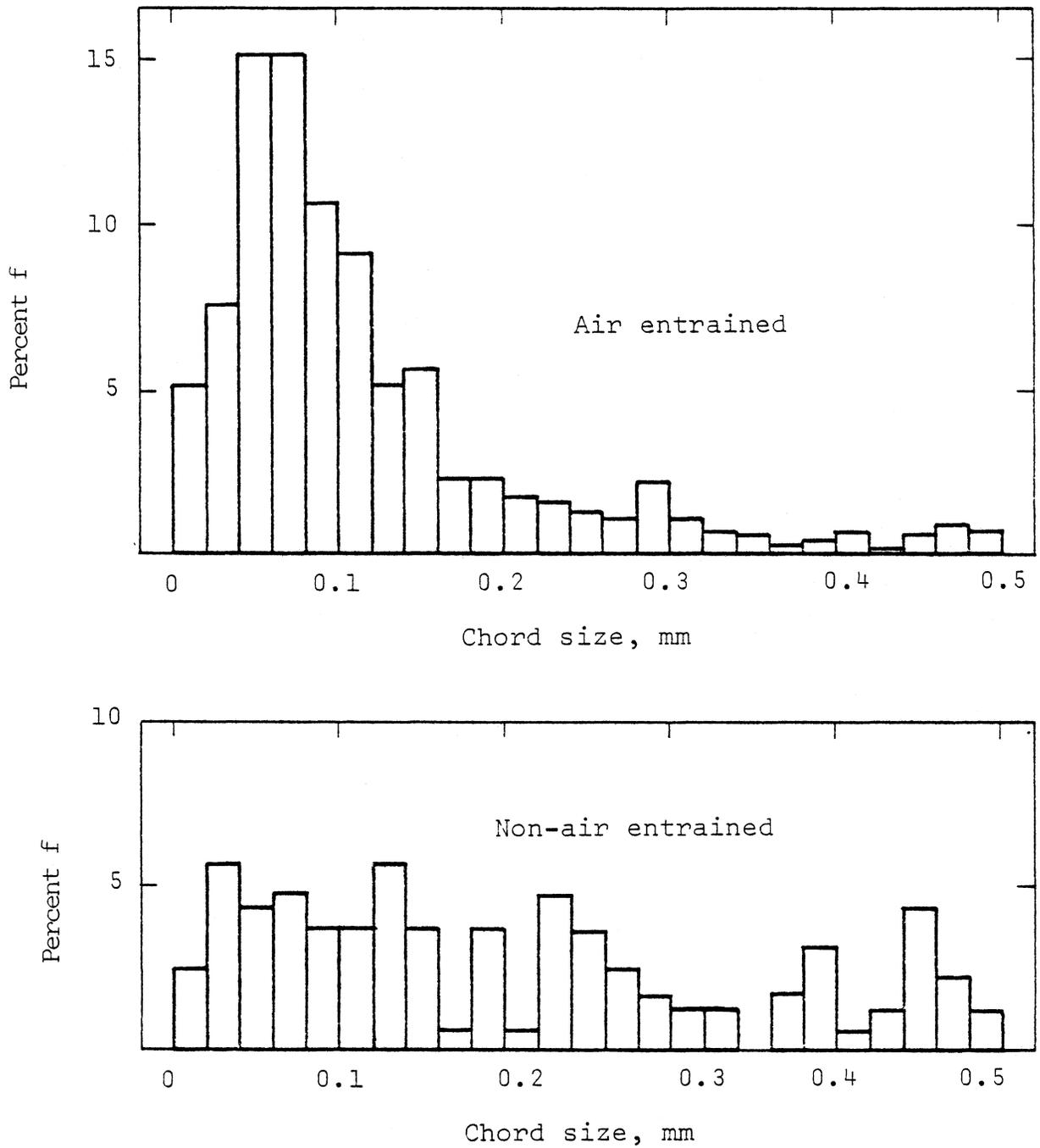


Figure 2. Frequency distribution of chord lengths for air entrained and non-air entrained samples based on 20 μm (7.9×10^{-4} in.) chord intervals up to 0.5 mm (2×10^{-2} in.).

Note: 1 in. = 2.54 mm

chord lengths were gathered at the small size range to form an apparent peak, whereas in the non-air entrained concrete an irregular distribution of chord lengths was observed. Thirteen air entrained samples were chosen from the lab and the field specimens at random. The chord distribution function $\phi(x) = a \times b^{-x}$ was put in the form of a straight line $y = a + b^x$ as

$$\log \frac{\phi(x)}{x} = \log a - (\log b) x$$

then a linear regression analysis was performed on the chord distribution data.

Initially the chord lengths up to 2 mm (0.08 in.) were included in the regression and constants a and b were obtained in addition to the correlation coefficients. The regression analysis indicates how well the function represents the data points. The correlation coefficients ranged from 0.797 to 0.881. However, it was found that this continuous curve ignores the peak values in the chord frequency distribution. The constants a and b were correlated to A (the air content of the hardened concrete) and α (the specific surface) linearly. When a was related to A and α , linear correlation coefficients of -0.45 and -0.09 were obtained; and when b was related to A and α , values of 0.11 and 0.40 were calculated. These correlation coefficients were not considered to be satisfactory. For better representation of the peak values, the chord lengths less than 0.2 mm were considered and the continuous function was fitted. The correlation coefficients ranged from 0.864 to 0.996. However, the constants of the function still did not correlate satisfactorily with the air content and specific surface with the correlation coefficients ranging from 0.22 to -0.52. The constants of the distribution function were also calculated by substituting two points from the chord histograms of all the lab specimens as suggested by Larson et al. One of the points was taken at the maximum frequency and the second from a decreasing portion of the histogram. When a was correlated to A and α linearly, correlation coefficients of -0.13 and 0.57 were calculated, and when b was correlated, coefficients of -0.21 and 0.24 were obtained.

Based on the correlation coefficients it is concluded that a satisfactory linear relationship was not established between the constants of the continuous curve and the air content or the specific surface of the mixture. The first two initial objectives of this study were based on the derivation of the bubble size distribution from a continuous chord distribution curve. However, the continuous function chosen, $\phi(x) = ax/b^x$, did not represent the chord lengths satisfactorily as originally anticipated; therefore, a graphical method was undertaken that could yield some insight into the void system and help in differentiating between the water voids and the air voids.

Graphical Method

Lord and Willis have suggested a graphical method that enables one to calculate the bubble diameters and the number of bubbles per unit volume based on chord frequency distribution histograms.⁽¹⁸⁾ The histograms based on 20 μm intervals were tabulated and processed by computer to determine the number of bubbles per cm^3 of concrete and the corresponding bubble diameters for each interval. In the calculations, the number of chords per unit traverse length per interval is divided by the median value of the chord length group. The ratio for the smallest median group is then subtracted from the ratio of the next largest median group. When the difference is multiplied by the constant, $2/\pi$, the number of spheres with diameters equaling the upper limit of the first chord interval is found. The graphical method also indicates that the total number of spheres could be obtained if the ordinate of the shortest chord length group is divided by the median and then multiplied by $2/\pi$. However, due to the limit of resolution of the microscope and the difficulty of the operator in observing the small bubbles or small chords, it is possible to miss these small chords and come up with an increasing slope at the initial portion of the curve. When this occurs, the steepest slope of the curve is used to define the initial portion. Also, the part of the curve beyond the maximum point could have ordinates that do not maintain a continuously decreasing slope. In such a case, the successive intervals are included in the calculations till the averaging effect does yield a decreasing slope. Utilizing the Lord and Willis method, in addition to the number of bubbles per unit volume, void parameters such as the arithmetic mean bubble diameter, the diameters of the bubble determined from the mean bubble surface area and the mean bubble volume, and the specific surface were obtained and are summarized in Table 5.

The w/c and the A, taken as independent variables, were correlated with each of the void parameters calculated in the graphical method, which were the diameters and the number of bubbles per unit volume for all the laboratory specimens using Type II cement. The low correlation coefficients ranging from 0.213 to 0.681 obtained were found to be unsatisfactory, since only less than half the variability was explained in the dependent variable by the relationship. The freezing and thawing durability, weight loss, dynamic modulus, and surface rating at 100 cycles were related to the diameters and the number of bubbles per unit volume. Only in the case of diameters determined from the mean volume were satisfactory relationships observed. For this case the correlation coefficient with weight loss was 0.841; with the dynamic modulus it was -0.787; and with the surface rating it was 0.748. The measures of durability

were also correlated with the cube root of the number of voids per unit volume. The correlation coefficients were -0.77, 0.67, and -0.74 for weight loss, dynamic modulus, and the surface rating. These values are higher than those obtained from correlating the total number of bubbles, which yielded correlation coefficients of -0.64, 0.54, and -0.63, respectively.

Also, it was noticed that the specific surface obtained by the linear traverse method and given in Table 2 correlated satisfactorily with the specific surface obtained by considering bubbles at each interval in the Lord and Willis method depicted in Table 7, showing a correlation coefficient of 0.854.

DISCUSSION OF RESULTS

In the laboratory, the w/c of the air entrained specimens ranged from 0.39 to 0.58, the corresponding specific surfaces ranged from 503 in.⁻¹ (19.8 mm⁻¹) to 831 in.⁻¹ (32.7 mm⁻¹), and the number of voids varied from 16,351 to 82,639 bubbles per cm³ (267,880 to 1,353,883 bubbles per in.³). However, Mielenz et al. have found a value of about 150,000 voids per cm.³ (2,457,465 bubbles per in.³) for concrete with a w/c of 0.55.⁽¹²⁾ For their study the observed specific surface for the air entrained concrete ranged from 615 to 1,600 in.⁻¹ (24.2 to 63.0 mm⁻¹), while in the present study values near their lower limit were noted. Also, Larson et al. note that the peak occurring in the frequency distribution of chord lengths is generally below the 50 μm range. In this study the peaks occurred mainly at about 60 to 80 μm.

All of the aforementioned values indicated that the bubbles obtained in this study were coarser than should be expected. Therefore, a check on the accuracy of the data was desired mainly to find out whether a considerable amount of the small bubbles were being missed during measurements. To verify the validity of the study data, it was proposed that the results of Research Council tests on four standard specimens circulated by ASTM Subcommittee C09.02.07 for comparative measurements would be compared to those of other laboratories participating in the ASTM evaluation. (The interlaboratory studies on the standard specimens were undertaken to determine the precision of ASTM C457.) Data on the air content and specific surface of the four ASTM specimens were gathered from four other participating laboratories. Data on these samples were generated using a magnification of about 100. The Council's data were linearly correlated to those from the other labs and coefficients in excess of 0.994 for the air content and 0.961 for the specific surface were obtained. These values indicated a good correlation. The specific surfaces obtained on the air entrained specimens were close to the lower range suggested by Mielenz et al. The low specific surface values and the visual examination of the samples showed that the bubbles were coarse and fewer in number than anticipated. Therefore,

Table 7
Void System Parameters as Obtained by the Lord and Willis Method

Classification	w/c	D ₁ , μm	D ₂ , μm	D ₃ , μm	Specific Surface, per inch	N
1A ^a	0.42	57	80	117	630	61,730
1B	0.54	86	108	143	602	39,247
2	0.42	107	127	157	637	46,058
3	0.39	82	99	125	766	82,639
4A	0.42	69	90	120	725	69,086
4B	0.49	67	98	139	546	39,981
5A	0.42	54	79	112	675	69,293
5B	0.46	60	80	113	687	61,406
5C	0.46	47	70	120	435	41,242
5D	0.52	58	83	120	597	38,144
6A	0.42	62	86	120	643	39,444
6B	0.51	62	94	131	601	45,876
6C	0.58	70	96	134	588	41,552
7A	0.42	57	83	128	500	29,449
7B	0.50	75	118	165	471	22,408
7C	0.50	71	99	145	494	16,351
7D	0.58	52	79	119	566	41,262
8A	0.42	89	102	132	698	34,007
8B	0.42	53	80	122	540	37,721
8C	0.51	93	116	151	596	30,175
9A	0.49	688	84	111	780	61,889
9B	0.56	736	92	119	756	53,244
9C	0.64	702	86	115	728	68,341
10	0.47	101	121	142	765	52,466
11	0.47	68	81	106	836	79,680
12	0.47	111	131	163	608	28,288
13	0.47	116	132	154	718	36,104
14	0.47	51	73	108	641	67,638
Non-A.E. Spec.	0.47	70	124	224	209	5,020

Note:

- a Three specimens were averaged.
One inch = 25.4 x 103 μm;
One cubic inch = 16.39 cm³.

- D₁ = Arithmetic mean bubble diameter.
D₂ = Diameter of the bubble from the mean bubble surface area.
D₃ = Diameter of the bubble from the mean bubble volume.
N = Number of bubbles per cu cm of concrete.

tests on the ASTM specimens did not exactly indicate whether the small voids were being missed during the linear traverse measurements.

Recently the Research Council investigated a void system of concrete by making a point count analysis of thin sections about 15 μm thick under a 400 magnification with both transmitted polarized light and reflected ultraviolet light. A specific surface value of 1,450 in.^{-1} (57.1 mm^{-1}) was obtained when transmitted polarized light was used, and under the ultraviolet light a specific surface value of 1,257 in.^{-1} (49.5 mm^{-1}) was found. On finely lapped surfaces prepared from the same concrete mixture, traverse measurements under 100 magnification showed a specific surface value of 617 in.^{-1} (24.3 mm^{-1}). Thus, under a 400 magnification the specific surface value found was about twice that obtained under 100 magnification. This finding implies that some of the small bubbles present in concrete can be missed during the linear traverse method of analysis under 100 magnification. The differences in values between the two systems may not be as much as recorded here, and a direct quantitative comparison may not be suitable because different samples and different sample sizes were used, even though the same mixture was tested.

CONCLUSIONS

1. A satisfactory correlation between the w/c and air content combined (such that the effects of excess water and air entrainment voids could be established at the same time) against the parameters of the void system could not be achieved within the framework of this project.
2. Prolonged mixing or agitation reduced the specific surface of voids in concrete.
3. In general, adding water to the mixture increases the void content of hardened concrete and prolonged mixing or agitation tends to decrease it.
4. A reduction in specific surface generally results in an increase in the spacing factor.
5. For a given air content the weight loss, dynamic modulus of elasticity, and surface rating were correlated satisfactorily with the spacing factor, the specific surface, and the diameter of bubbles determined from the mean bubble volume. However, a positive method of differentiating between water voids and air entrained voids was not found from the limited testing conducted.

6. A satisfactory relationship was found between the freezing and thawing durability, expressed in terms of weight loss, and the dynamic modulus of elasticity versus the amount of voids < 1 mm in diameter in a plane surface.
7. There is a possibility that some of the small air entrained voids in concrete can go unnoticed in the linear traverse method based on 100 magnification. Thus, judgement on the durability of concrete may be made on a part rather than complete void system in concrete when only the linear traverse method is considered.
8. A good correlation was found between the air contents of fresh and hardened concretes that were properly measured.
9. Bubbles obtained in this investigation were generally coarse as reflected by the low specific surface and high spacing factor values. However, a study in the 1950's⁽¹⁶⁾ showed higher specific surfaces and lower spacing factors, which indicated the presence of abundant small voids in concrete. It is possible that the recent cement additions as grinding aids, or the water reducers and set retarders in the mixtures, affect the air entraining admixtures, and thus the generation of bubbles.

RECOMMENDATIONS

The air content of hardened concrete and the freezing and thawing durability are closely related to the specific surface and the spacing factor. In a properly designed, consolidated, and measured concrete there is good agreement in air contents at the fresh and hardened stages. Therefore, if the air contents in specifications are achieved in controlled and properly cured concrete mixtures, the durability and strength should be readily attained. In case of disputes it is recommended that the petrographic examination be performed and that the void parameters obtained in accordance with C457 be taken as evidence.

It has been observed that the distribution and the number of bubbles in concrete are very much affected by the type and kind of air entraining agent used, and possibly by cement additions or admixtures which are surface-active in nature. Therefore, it is desirable to investigate the effects of surface-active agents on the air entraining admixtures.

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