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Effects of Synthetic Air Entraining Agents on Compressive Strength of Portland Cement Concrete – Mechanism of Interaction and Remediation Strategy

FINAL REPORT
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16. Abstract

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SUMMARY

This document reports the results of a comprehensive study pertaining to the determination of causes and mechanisms resulting in a reduction of strength in concrete mixtures containing Synthetic air entraining admixtures. The study involved experimentation with concrete mixtures containing both the Synthetic and Vinsol resin admixtures. Tests involved determination of air content for fresh concrete and air bubble characteristics including size and distribution for hardened concrete. Compressive strength of concrete samples were determined at ages 7, 14, 28, and 56 days. Petrographic analysis of the hardened samples revealed that the Vinsol resin admixture produces more of the smaller bubbles desirable for protection against frost. However, Synthetic admixtures induce more of the larger air bubbles within the cement paste. This explains the much lower compressive strengths associated with Synthetic air concretes. It is believed that the larger air bubbles are produced due to the inability of the Synthetic air entraining agents to lower the surface tension in the mixture. This will allow the smaller air bubbles to coalesce into larger ones. Based on the results of this study, recommendations were made that follow up research should involve determination of surface tension in such mixtures.

EFFECTS OF SYNTHETIC AIR ENTRAINING AGENTS ON COMPRESSIVE STRENGTH OF PORTLAND CEMENT CONCRETE – MECHANISM OF INTERACTION AND REMEDIATION STRATEGY

1. STATEMENT OF THE PROBLEM

Air entrained concrete comprises the bulk of the cementitious material employed in the construction of highway infrastructure. Besides improving freeze-thaw durability, air entrainment increases the workability of concrete, and therefore allows for a reduction in water to cement ratio (*w/c*). Lower *w/c* ratios that can be used with air entrained concrete and better compaction characteristics result in more impermeable concrete and a better overall resistance to aggressive agents.

In recent years, Synthetic air entraining admixtures have been more readily available in construction projects. Data accumulated over the past decade indicates that Synthetic air entraining agents cause a rather large reduction in compressive strength. An average bias of 700 psi, 600 psi, and 300 psi has been estimated with Class A, Class B White, and Class B Concrete respectively. According to NJDOT observations, this decrease in strength has been isolated independently of parallel contributions from varying air content, ambient temperature, Portland cement quality control, and alkali contents in Portland cement. Such losses in strength were not observed with Vinsol resin type air entraining agents. Therefore, the problem is why synthetic air entraining admixtures give rise to greater strength loss.

2. RESEARCH OBJECTIVES

The primary objectives of the research described in this report were:

a) To determine the cause and the mechanism for reduction in compressive strength when Synthetic air entraining admixtures are used.
b) To provide a solution for the problem based on the results of the investigation performed in the first stage of the research.

3. BACKGROUND

Full comprehension of the research approach undertaken during this investigation requires a brief description as to the mechanisms of air entrainment, and the testing procedures for determination of air content and air bubble characteristics in concrete. This is accomplished in this section of the report.

Frost Resistance of Concrete by Air Entrainment

Air bubble spacing in the order of 0.1 to 0.2 mm within every point in the hardened cement is the most important factor for protection of concrete against damage by frost. In other words, it is not the total air, but the void spacing which provides protection against frost. By adding small amounts of air entraining agents (e.g., 0.05% by weight of cement) it is possible to incorporate 0.05 to 1 mm bubbles. For a given volume of air the degree of protection against frost action will vary depending on the bubble size, number of bubbles, and bubble spacing. Therefore, volume of entrained air is not a sufficient measure for protection of concrete against frost action, but it is the easiest criterion for the purpose of quality control of concrete mixtures.

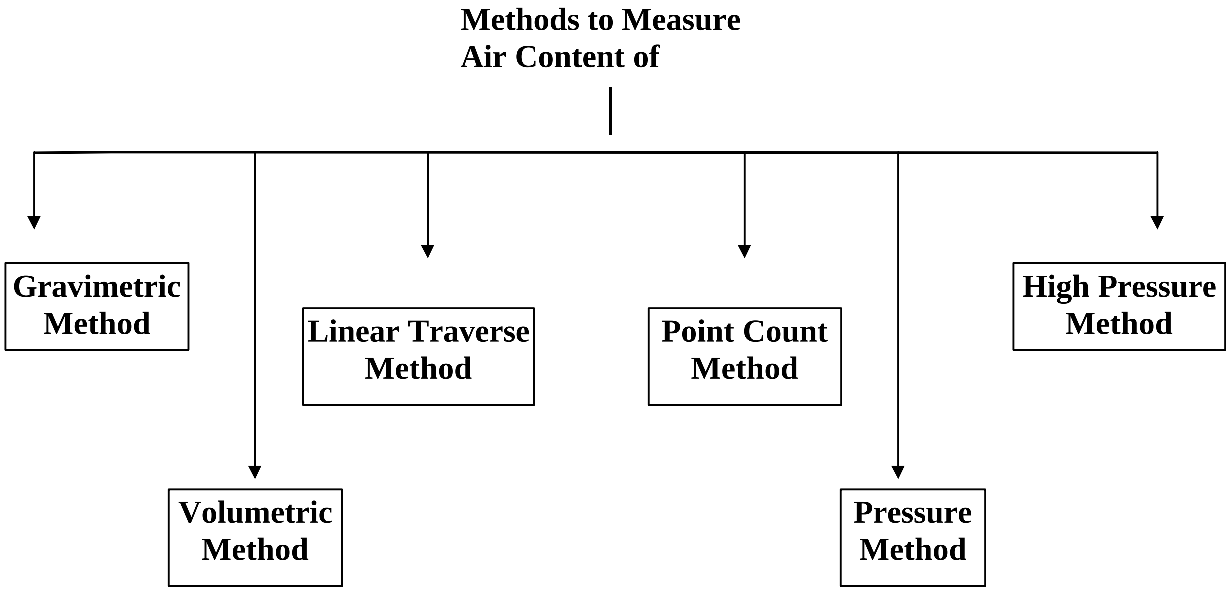
Air bubbles are contained within the cement paste portion of the concrete. The cement paste content of a concrete is generally related to the maximum aggregate size. Concretes composed of larger aggregate sizes contain less cement paste than concrete made of smaller aggregates. Therefore, concretes with more cement paste (i.e., smaller aggregate sizes) require more air bubbles for protection against frost damage. For instance, concretes containing $\frac{3}{8}$ inch maximum size aggregate require 7.5% air under severe exposure conditions whereas for 1 inch aggregate size air content of 6% is sufficient for protection against frost damage. Aggregate grading also afects the volume of entrained air. In general, the volume of entrained air is decreased by an excess of very fine sand particles. Addition of mineral admixtures such as fly ash, or the use of very finely ground cements decrease air content in a similar way as in very fine aggregates. Furthermore, more air is entrained into a cohesive concrete than a very wet or a very stiff one.

Air Entraining Admixtures

Air entraining admixtures pertain to the class of surface-active chemical known as surfactants. Surfactants consist of long-chain molecules one end of which is hydrophilic (water-attracting) and the other hydrophilic (water-repelling). The hydrophilic end contains one and or more polar groups responsible for ionic actions when adsorbed at the air-water, and cement-water interfaces. At the air-water interface the polar groups are oriented towards the water phase lowering the surface tension, promoting bubble formation and countering for the tendency of dispersed bubbles to coalesce. At the cement-water interface where directive forces exist in the cement surface, the polar groups become bound to the cement with the non-polar groups oriented towards water, making the cement surface hydrophobic so that air can displace water and remain attached to the cement as bubbles. Air entraining admixtures generally consist of salts of wood resins, petroleum acids, and some synthetic detergents.

Methods for the Determination of Air Content in Concrete

A number of techniques are available for the determination of air content in fresh or hardened state. These methods include the following:



I-Gravimetric Method

The air content of fresh concrete can be calculated from its measured unit weight and from the weights and densities of its ingredients. This proedure is standardized by ASTM (C 138). This technique is highly accurate provided that the densities of concrete constituents are accurately determined. Time required for measurement of air varies depending on whether the accurate unit weights of constituents are available or they need to be measured. Time required may vary from 45 minutes to about 2 hours.

II-Volumetric Method

The volumetric or direct method is based on determining the air content of fresh concrete by removal of the air from a measured volume of concrete and measurement of the volume of air directly. This method is standardized by ASTM (C 173). The instrument for performing this process is called a Roll-A-Meter. Time required for proper measurement of air content is about 45 minutes.

III-Pressure Method

This method is most widely employed due to it's simplicity and the relatively short time period involved in testing (about 20 minutes). It is based on Boyle's law and accordingly since air is the only compressible ingredient of concrete, any reduction in the volume of a sample of fresh concrete due to an increase of external pressure is attributed to air in the specimen. By increasing pressure on a sample in a closed container, and measuring the resulting decrease of volume, the quantity of air in the sample can be calculated. The ASTM designation for this technique is C 231.

IV-Linear Traverse Method

The linear traverse method pertains to the measurement of air content in hardened concrete. It involves cutting the specimen, polishing the cut surface, and measuring the fraction of the total area occupied by sections of air bubbles. By using this technique very important information about the air void characteristics of the sample can be determined. This information include air bubble size, distribution, spacing, and total air content. The ASTM designation for this method is C 457.

V-Point Count Method

This is another method for determination of air content on hardened concrete. It is based on statistical considerations and requires a finely ground plane cross-section of the specimen. In this procedure, a rectangular grid is placed on the plane specimen surface, and each grid intersection that falls within a void section is counted. The air content is equal to the number of such coincidences with voids divided by the total number of grid intersections. This technique is also described in ASTM C 457.

VI-High Pressure Method

The high-pressure method is also applicable to hardened concrete. It involves compressing the air in an oven dried and presoaked (for 48 hrs) specimen by means of hydraulic pressure of about 5000 psi (as opposed to 10 psi for fresh concrete in the pressure method). After applying correction factors, a value for air content is obtained. This technique has not gained wide spread usage.

4. INVESTIGATIVE PROCEDURES

The logical approach in this investigation was to compare the air content and compressive strength characteristics of the Vinsol and Synthetic resin type admixtures through a detailed experimental program. The general overview of research program is briefly described here.

The experimental program is outlined in table 1. Experiments included preparation of two series of concrete samples with Synthetic and Vinsol resin admixtures. The mix proportions and constituent materials in all the concretes were identical in every respect except for the air entraining agent type (table 2). The dosage of air entraining admixture in most of the DOT applications corresponded to 1.5 oz/100 pounds of cement per cubic yard. The experimental program included mixtures containing 1.0, 1.5, and 2.0 oz of admixture per 100 pounds of cement. Air content of fresh concrete samples were measured by using the gravimetric and the pressure test methods. Fifteen 4X8 inch concrete cylinders were fabricated for each of the samples listed in table 1 in order to determine the compressive strengths at 7, 14, 28, and 56 days, and prepare slices for the petrographic analysis of samples. Average of three cylinders was used for computation of strength at each age level. For each sample, three cylinders were sliced and used for petrographic analysis. Compressive strength results were correlated against the air content and air entraining admixture type. It is expected that the samples with synthetic air to exhibit strength losses similar to those previously obtained by others. The experimental program included both Synthetic and Vinsol resin admixtures. Data shown in table 1 corresponds to air content as measured in the fresh state by the gravimetric as well as the pressure methods.

Air content and the air bubble characteristics in the hardened state were determined by the automated linear traverse system shown in Fig.1. The automated technique employs digital image processing principles. Surface of the slices taken from the concrete cylinders were polished through a rigorous procedure and prepared for image analysis (Fig.2). As per ASTM C-457, a minimum traverse length of 95-inch is required for petrographic analysis of concrete with a maximum aggregate size of 1-inch. A typical slice is traversed along eight parallel strips, one-tenth of an inch apart. Each strip can accommodate 13 images of 0.2-inch of width. Each image is traversed along five random lines. This process yields a total traverse length of 104 inches computed as in the following:

$$5\times0.2\times13\times8 = 104 \text{ inch}$$

Spacing factor, specific surface air content and air bubble characteristics were evaluated from the linear traverse results. Typical out put of image processing system in terms of bubble frequency and size is shown in Fig.3. In addition to the measurements, the digital images of the polished surface of the hardened samples were analyzed under the microscope in order to develop a global mapping of air void system in the samples. Results from this analysis would be useful in determining the dispersion characteristics of bubbles. The exact bubble size, spacing, and dispersion characteristics of the Vinsol and Synthetic resin air entrained concretes were determined through petrographic analysis of polished concrete slices. Analysis of these results revealed the nature of the problem associated with the use of synthetic air entraining agents in concrete. These results will be discussed in the later sections of this report.

Table-I Experimental Program

Sample No	Dosage of AEA	Air content (%)			Compressive Strength (ksi)				spacing factor	specific surface
	(oz/100 lb cemet)	Gravimetric	Pressure	Linear traverse	7d	14d	28d	56d	(mm)	(sq. in/cu. in)
R1217	1.0	8.60	7.00	6.7700	Calibration tests				0.1291	790.39
A1218	1.0	6.50	7.50	6.4196	Calibration tests				0.1750	615.11
R0120	1.0	3.60	3.10	6.1521	5.391	6.005	6.095	6.987	0.1610	690.21
A0122	1.0	6.20	5.60	7.1179	3.872	4.293	5.021	5.299	0.1460	660.08
A0407	1.0	6.20	5.80	7.0163	4.345	4.430	5.371	5.641	0.1136	811.10
R0414	1.0	6.40	5.90	6.9691	4.330	4.927	5.384	5.890	0.1222	866.71
C0421	0.0	2.20	1.80	2.9168	5.213	5.586	5.961	6.528	0.2553	1004.11
R0422	1.5	8.04	6.60	8.7412	3.573	3.904	4.095	4.618	0.1066	988.96
A0423	1.5	6.44	6.00	7.0361	3.697	4.407	4.751	5.091	0.0993	741.23
R0427	2.0	7.90	7.50	8.4272	3.776	4.672	4.630	4.954	0.0827	990.79
A0428	2.0	9.60	8.50	10.018	2.846	3.329	3.373	3.779	0.1020	676.08
R0429	1.0	7.29	6.60	8.0347	3.831	4.512	4.609	5.307	0.0921	933.84
A0501	1.0	6.70	6.60	7.4141	3.683	4.018	4.438	4.962	0.1024	909.91
R0707	1.5	5.20	5.40		4.537	4.641	6.011(71days)			
A0708	1.5	5.45	5.62		4.289	4.400	5.857(72days)			

Note: The prefixes A and R in the sample number refer to the synthetic (AEA15) and vinsol resin (AER) type air entraining agents

I

Mix Design Requirements and Criteria

Mix design for all the samples tested here corresponded to standard NJDOT class-A concrete. Mix design for 1-cubic yard of concrete is given in table 2. The basic characteristics, properties and materials used for class-A concrete in this study are given below:

- Required strength: 4200 psi
- Desired sir content: 6%
- Maximum size of aggregate employed was 1.0-inch conforming to the ASTM C 33 gradation requirements.
- Essroc type-II Portland cement conforming to ASTM C 150 was employed in all mixes.
- Water–reducing agent: Plastocrete161: Dosage: 4 oz per 100 lbs. as per ASTM C 494 was employed in all mix categories.
- The effective absorption of the aggregate in dry-air-state was measured at 1.5% per ASTM C 127. This measurement was employed in order to account for the absorbed water in the mix design.
- ASTM No. 2 grade river sand was employed as the fine aggregate in all mixes.

Table II. Mix Design (1 Cubic Yard)

Type & Serial No.	***Cement***	**Coarse Agg.**	**Fine Agg.**	***Max. Water***	***Design Water***
A AE563510M1	625 lbs.	1942 lbs.	1167 lbs.	33.24 Gals	30.97 Gals

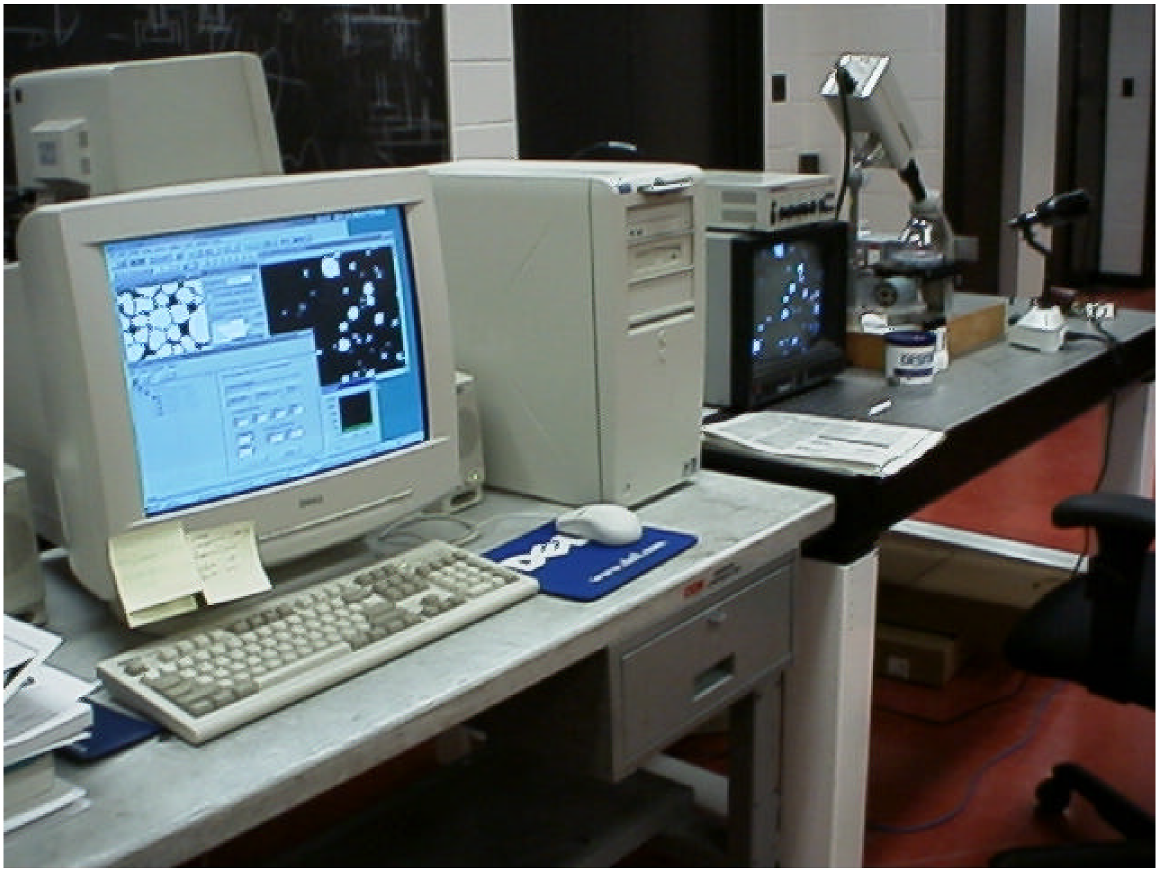


Fig.1 The automated linear traverse system.

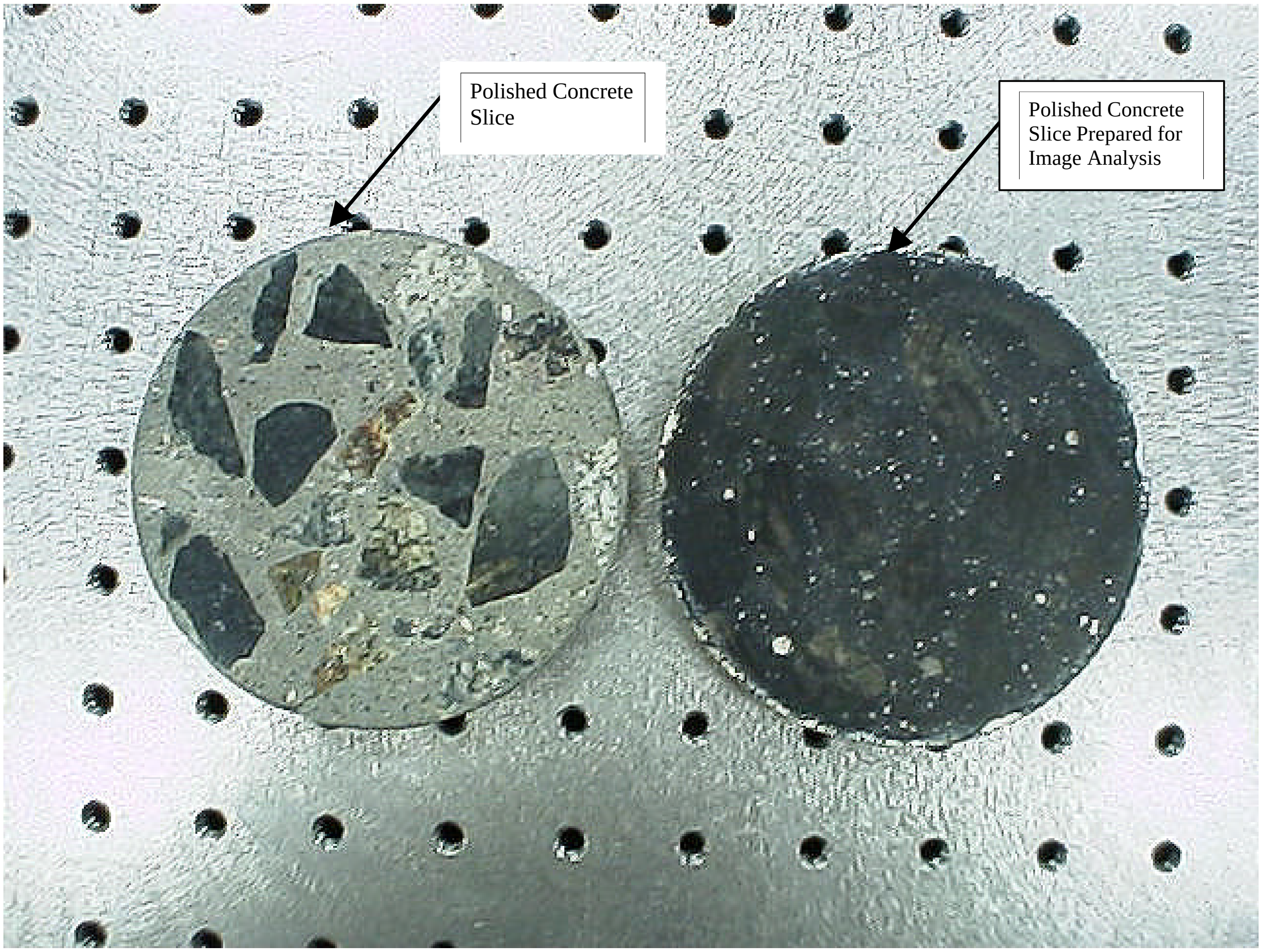
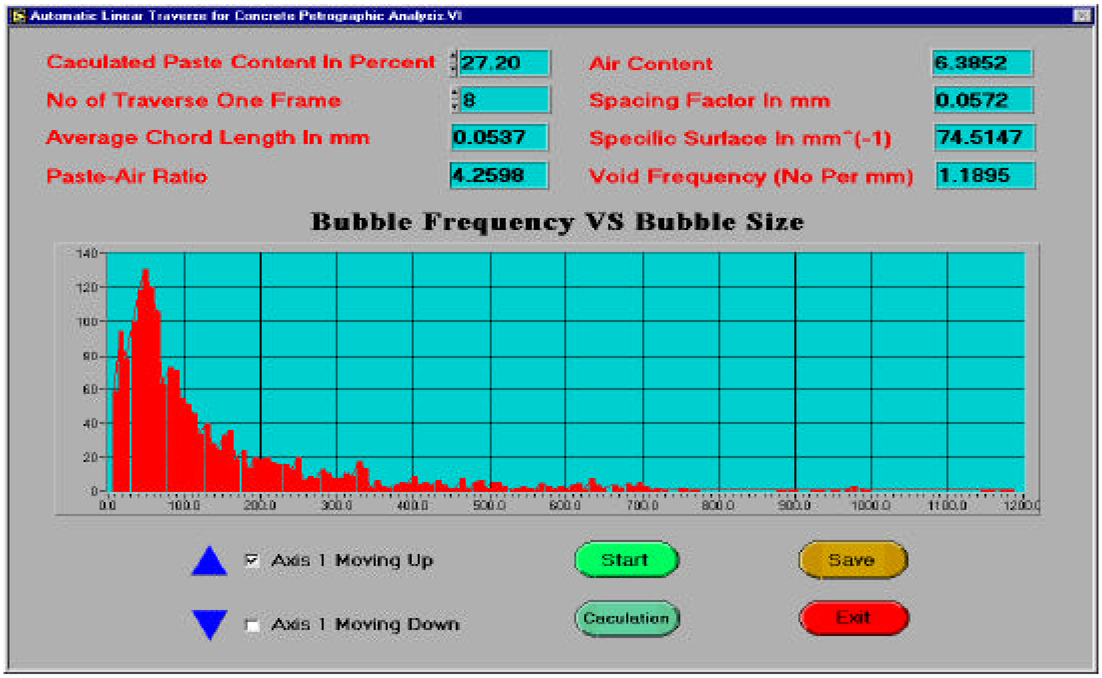


Fig.2 Sliced Concrete Samples for the Automated Image Analysis System



Automatic Linear Traverse for Concrete Petrographic Analysis.VI

Caculated Paste Content In Percent: 27.20

No of Traverse One Frame: 8

Average Chord Length In mm: 0.0537

Paste-Air Ratio: 4.2598

Air Content: 6.3852

Spacing Factor In mm: 0.0572

Specific Surface In mm^(-1): 74.5147

Void Frequency (No Per mm): 1.1895

☑ Axis 1 Moving Up

☐ Axis 1 Moving Down

Fig.3 Typical computer screen output of the image processing system.

5. EXPERIMENTAL RESULTS

Comparison of strength data of Vinsol and Synthetic mixes in table 1 supports the previous observations of NJDOT in terms of strength loss associated with concretes entrained with Synthetic type admixtures. The compressive strength of the air entrained concretes for the Synthetic and Vinsol resin mixtures containing 1.0 oz/100 pounds of admixture are compared in Fig.4. As shown in Fig.4, the Synthetic air concrete exhibited lower compressive strengths than that of the Vinsol resin mixture at all ages. As shown in Figs. 5 and 6, similar comparisons could be made for the 1.5 and 2.0 oz dosages of admixtures. The 1.5-oz dosage did not produce consistent results in all cases, and more experiments will be performed in future research to develop consistent data for this dosage.

Results from the image analysis of the hardened concrete samples are given next. As discussed earlier, the automated image analysis system facilitated a more thorough examination of the network of air bubbles within the hardened paste. Typical data corresponding to the air bubble size distribution and count (frequency) for the control sample (non-air-entrained concrete) is shown in Fig.7. For air entrained concrete, data were compared in a number of different ways in order to understand the effects of sir entraining admixture type on the bubble characteristics. Figs. 8 and 9 correspond to the comparison of typical Synthetic and Vinsol resin mixtures at the same dosage of air entraining admixture.

In a similar manner, tables 3 and 4 correspond to comparison of air bubble size distribution for mixtures containing similar dosages of air entraining admixtures. Finally, the air bubble distribution of the Synthetic and Vinsol resin mixtures at the same air content are compared in Fig.10 and table 5. As shown by all these results, Vinsol resin admixture produces more of the smaller bubble sizes desirable for protection against frost. In fact, comparison of air bubble size count reveals that the main reason for higher air contents in Synthetic mixtures is due to the increased number of larger air bubbles within the cement paste. This is illustrated in the magnified images of the Synthetic and Vinsol mixtures in Fig.11. As shown in Fig.11, in comparison with Vinsol resin concretes, the air bubble system in Synthetic air concrete exhibits larger cluster of air bubbles within the same area of paste. This in turn explains the much lower compressive strengths associated with Synthetic air concretes. In other words, Synthetic air entraining agents produce larger air bubbles that are not desirable for resistance against frost protection and result in lower compressive strengths.

Rationale

Preliminary experimental results indicate that the compressive strength loss in Synthetic concrete mixtures is due to the larger air bubbles in these mixtures. Most of the Synthetic air entraining admixtures are derived from petroleum acids, and Synthetic detergents. The hydrophilic component of these admixtures is responsible for ionic actions when adsorbed at the air-water, and cement-water interfaces. This action, results in lowering the **surface tension** promoting bubble formation and countering for the tendency of dispersed bubbles to coalesce. The surface tension has to be sufficiently lowered in order to prevent formation of larger size bubbles through coalescence. As per foregoing discussions, it is believed that the Synthetic admixtures do not sufficiently lower the surface tension to the appropriate levels for formation of smaller and more stable air bubbles.

One possible reason for inability of the Synthetic admixtures in lowering the surface tension is the chemical composition. In particular, the concentration of the components in the admixture. It is possible that a modification to the concentration of the admixture is all that is required to improve the admixture. Factors other than concentration levels may have been the cause for larger air bubbles. For all these reasons, it is necessary to compare the surface tension of Synthetic and Vinsol resin air entraining admixtures through careful measurements. This needs to be done for a range of admixture dosages in the concrete mixtures. Petrographic analysis shall be performed in order to correlate surface tension to air bubble distribution and surface tension of the admixture. Analysis of these results will enable the manufacturers to make the necessary adjustments to the chemical composition of their admixtures.

6. CONCLUSIONS

An experimental program of research was undertaken through which it was possible to determine the cause and mechanisms leading to the lower compressive strengths in concrete mixtures containing Synthetic air entraining admixtures. Research involved testing of concrete samples in fresh as well as hardened state. Petrographic analysis of the hardened samples revealed that the Vinsol resin admixture produces more of the smaller bubbles desirable for protection against frost. Whereas, Synthetic admixtures induce more of the larger air bubbles within the cement paste. This explains the much lower compressive strengths associated with Synthetic air concretes. It is believed that the larger air bubbles are produced due to the inability of the Synthetic air entraining agents to lower the surface tension in the mixture. This will allow the smaller air bubbles to coalesce into larger ones. Based on the results of this study, recommendations were made that follow up research should involve determination of surface tension in such mixtures. This will be done in the follow up stud

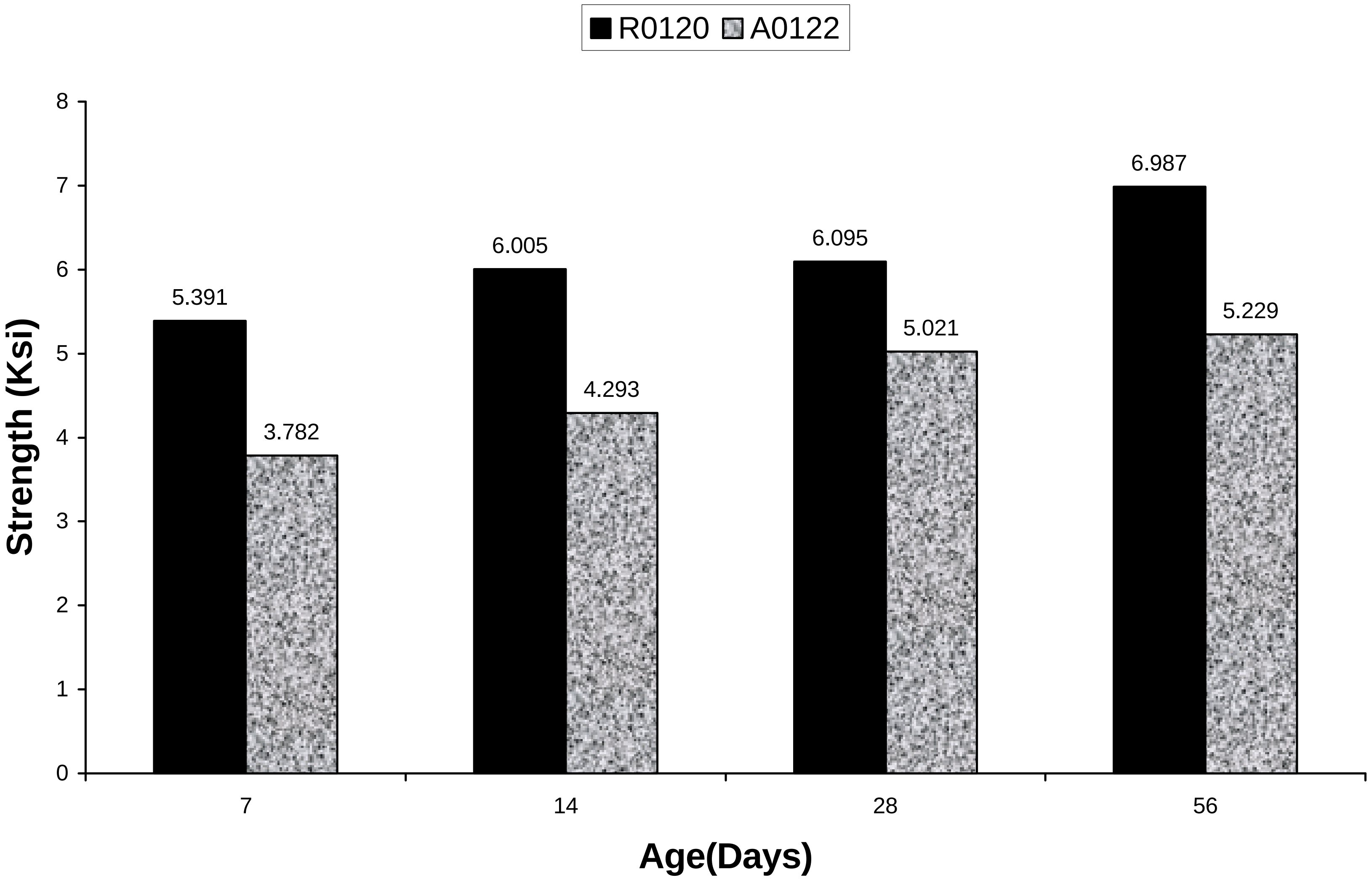


Fig.4 Comparison of compressive strengths for Synthetic (A0122) and Vinsol (R0120) resin concretes (at 1.0 oz).

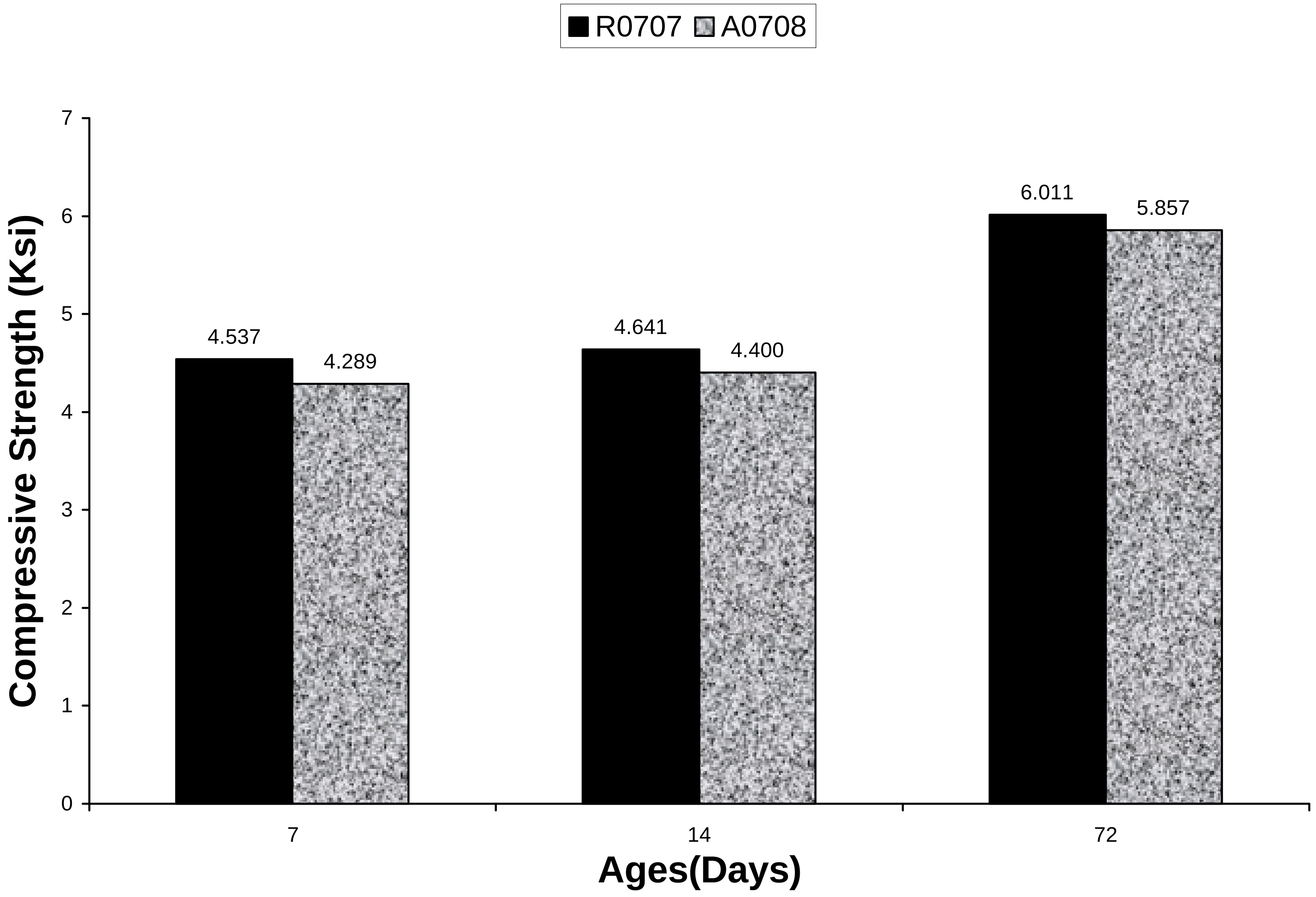


Fig.5 Comparison of compressive strengths for Synthetic (A0708) and Vinsol (R0707) resin concretes (at 1.5 oz).

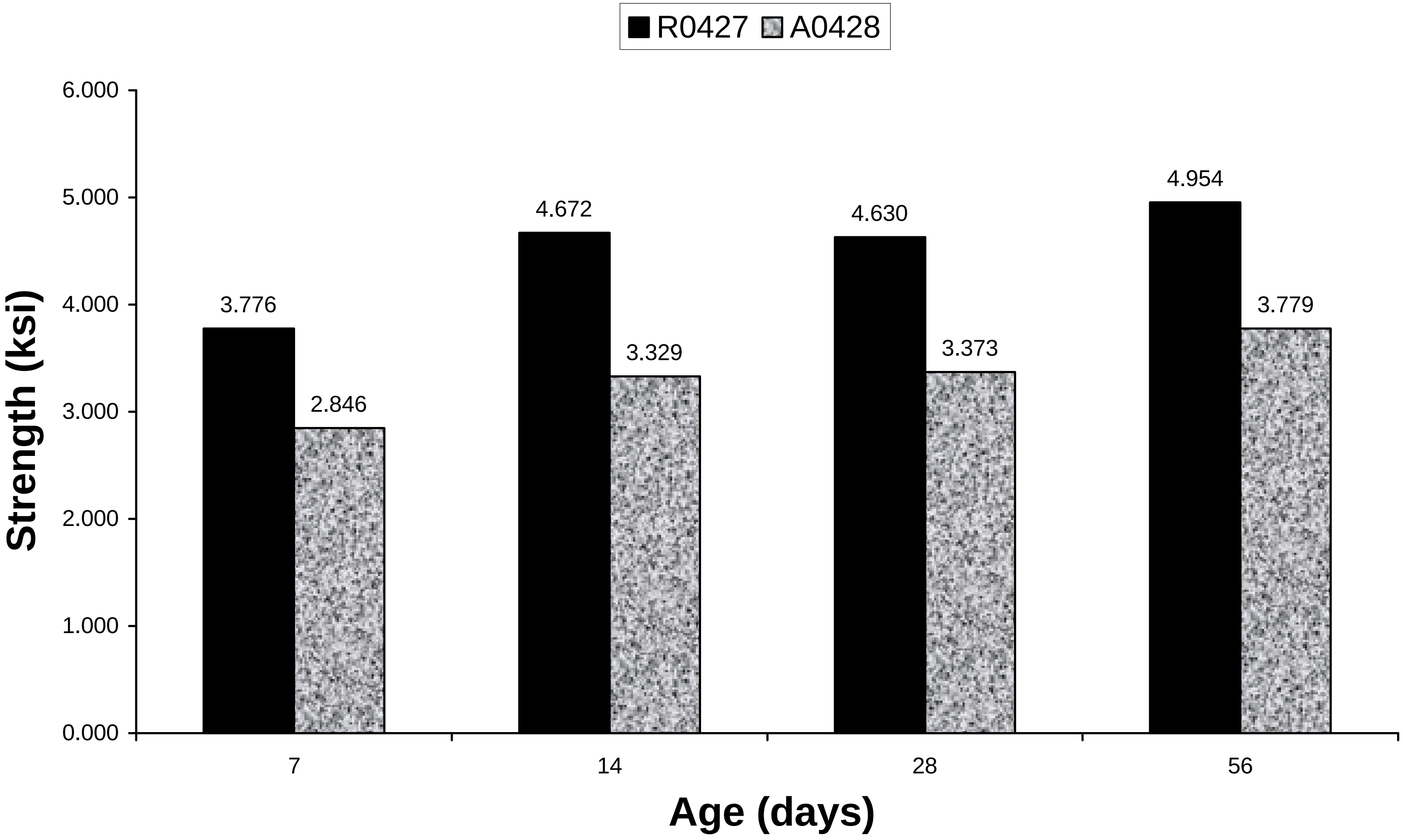


Fig.6 Comparison of compressive strengths for Synthetic (A0428) and Vinsol (R0427) resin concretes (at 2.0 oz)

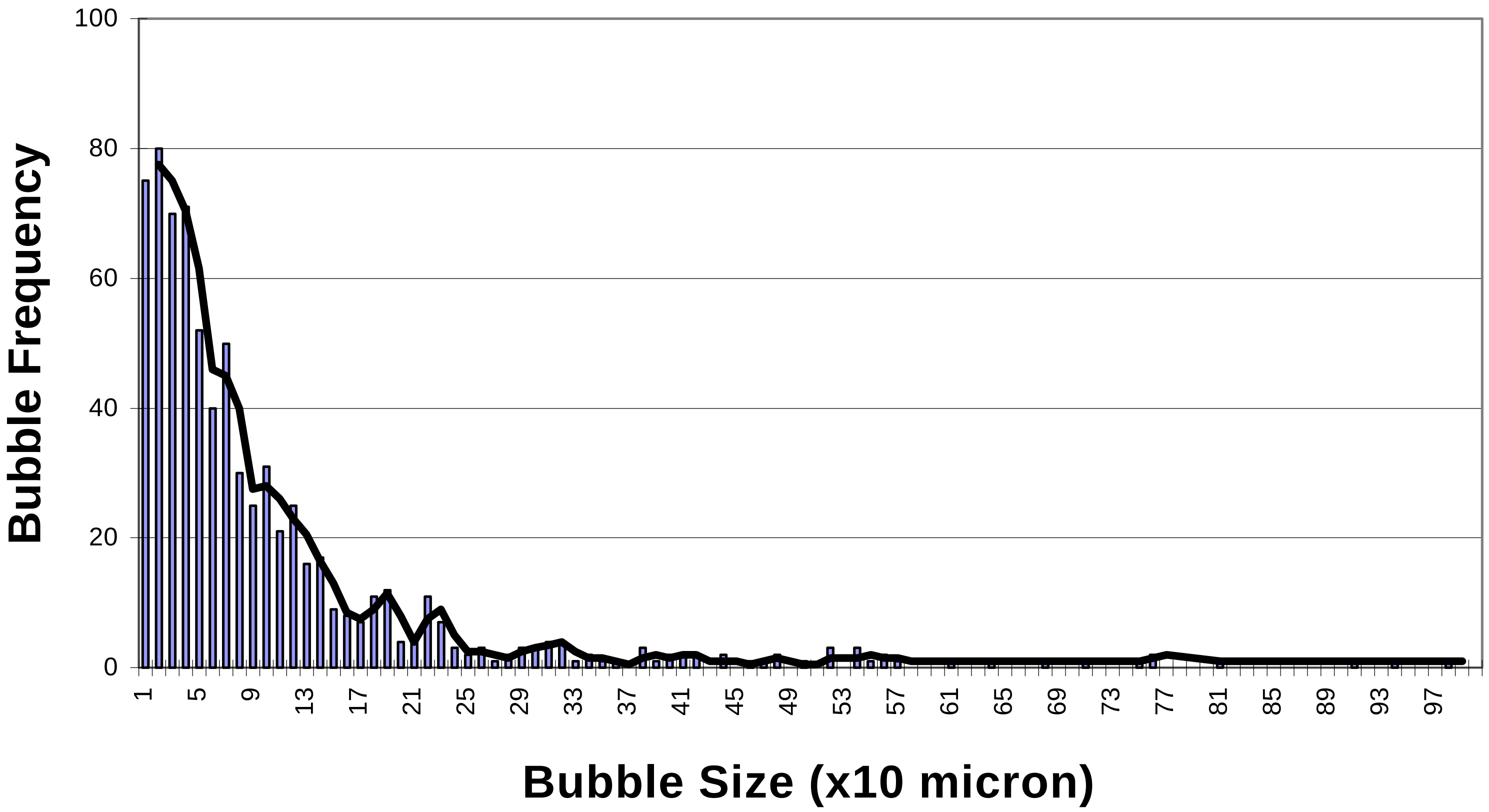


Fig.7 Bubble size distribution in the control sample (non-air entrained concrete).

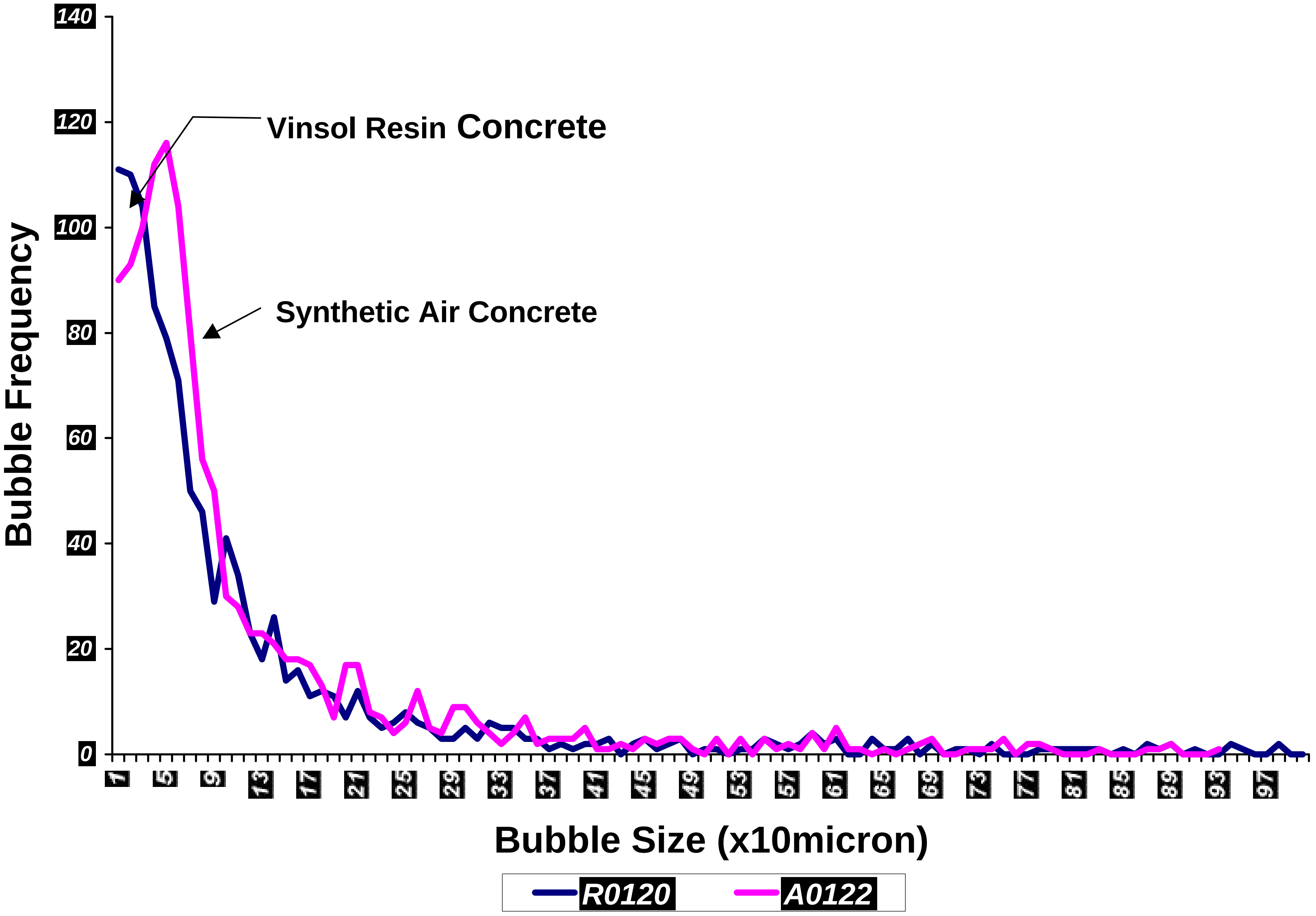


Fig.8 Comparison of bubble size distribution for samples containing 1.0 oz of air entraining admixture.

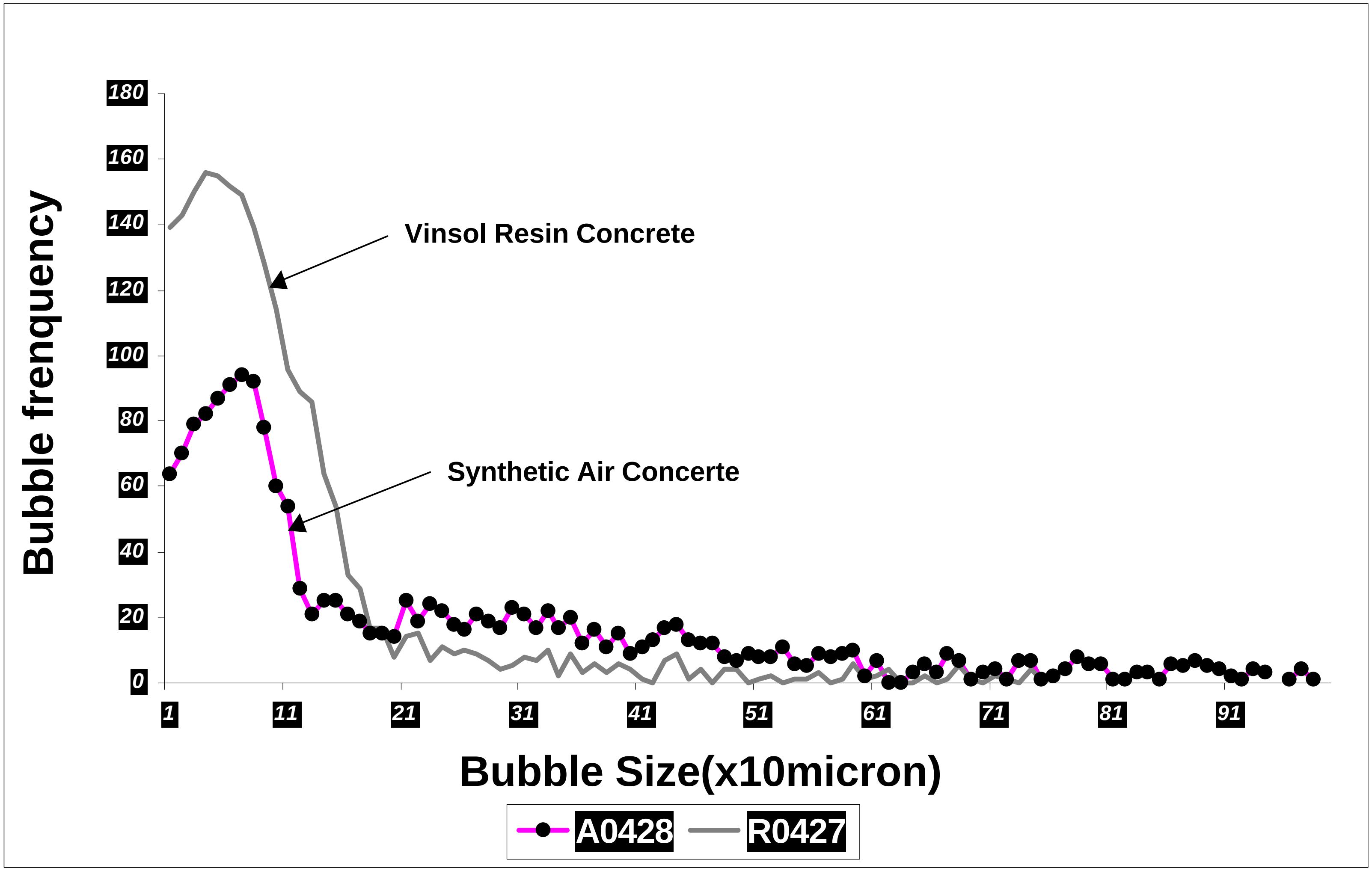


Fig.9 Comparison of bubble size distribution for samples containing 2.0 oz of air entraining admixture.

Table III Comparison of air bubble distribution in Synthetic and Vinsol resin mixtures at 1.0 oz

Diameter of air bubble *(μ)	Number of bubbles per total 104 inch traverse length	
	Vinsol Resin	Synthetic Resin
20	221	183
40	189	212
60	150	220
80	96	136
100	70	80
120	57	51
140	44	44
160	30	36
180	23	30
200	18	24
250	38	32
300	22	39
350	22	23
400	9	16
500	17	17
600	17	18
700	13	14
800	7	11
900	9	5
1000	6	3
1500	12	10
>1500	29	2
Total	1099	1216
Air content (%) by the linear traverse method	6.1521	7.1179
Specific surface α (sq mm/cu mm)	27.1735	25.9873
Mean chord length (mm)	0.1472	0.1539
Paste content (%)	27.2	27.2
Spacing factor $\bar{L}$ (mm)	0.1610	0.1460

Data from R0120, and A0122

*The figure give upper limits of diameter intervals; for example, 80 corresponds to 60 to 80 μ.

Table IV Comparison of air bubble distribution in Synthetic and Vinsol resin mixtures at 2.0 oz[*1]

Diameter of air bubble *[2] (μ)	Number of air bubbles per total 104 inch traverse length	
	Vinsol Resin	**Synthetic Resin**
20	282	134
40	306	161
60	307	178
80	288	186
100	242	138
120	185	83
140	150	46
160	87	46
180	46	34
200	25	29
250	56	108
300	44	96
350	36	97
400	22	63
500	30	129
600	16	76
700	16	39
800	10	46
900	4	36
1000	3	18
1500	9	13
>1500	7	6
Total	2161	1753
Air content (%) by the linear traverse method	8.4272	10.0182
Specific surface α (sq in/cu in)	990.79	676.08
Paste content (%)	27.2	27.2
Spacing factor $\overline{L}$ (mm)	0.0827	0.1020

[*1] Data from R0427 and A0428

[*2] The figure give upper limits of diameter intervals; for example, 80 corresponds to 60 to 80 μ.

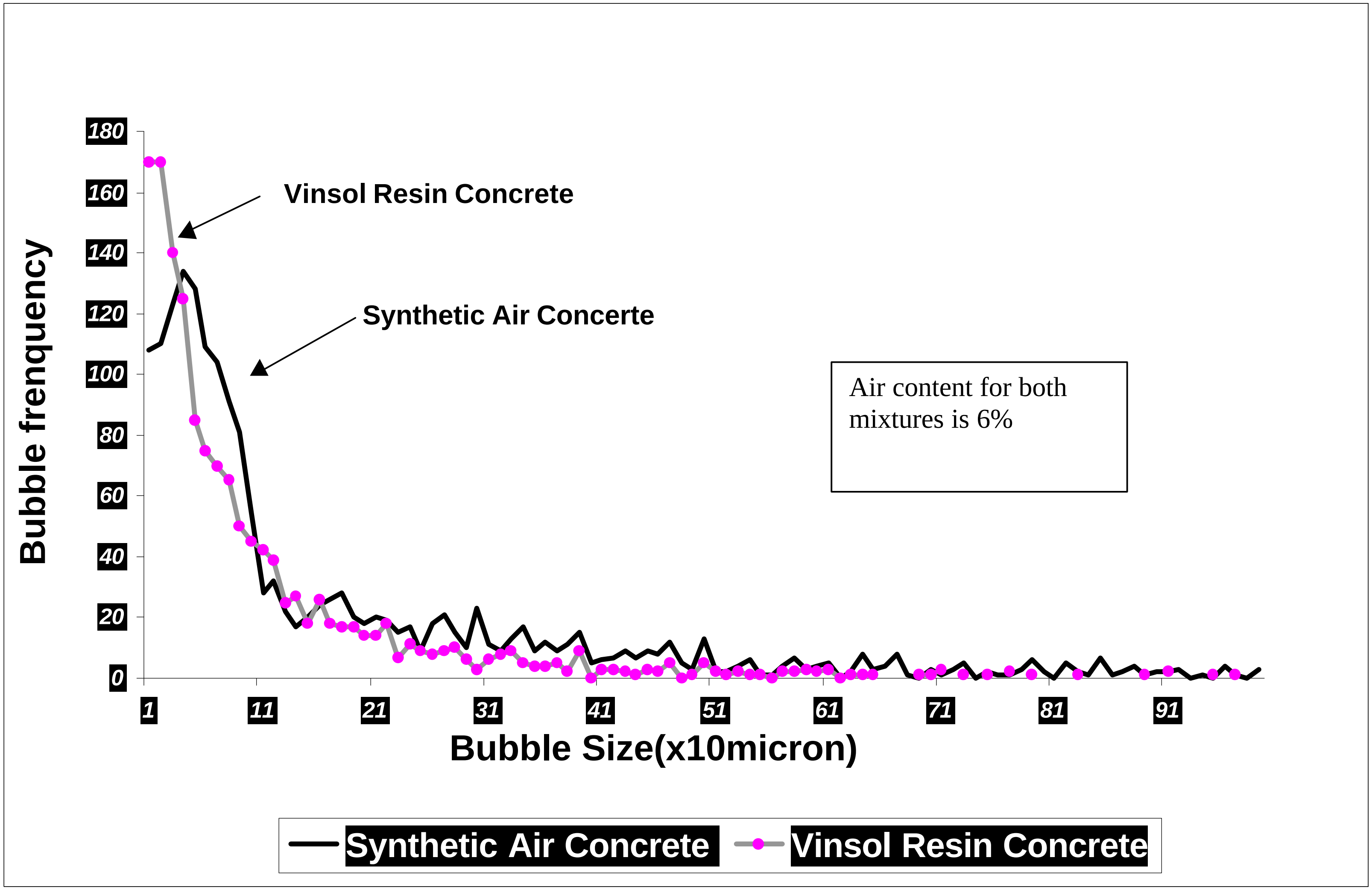


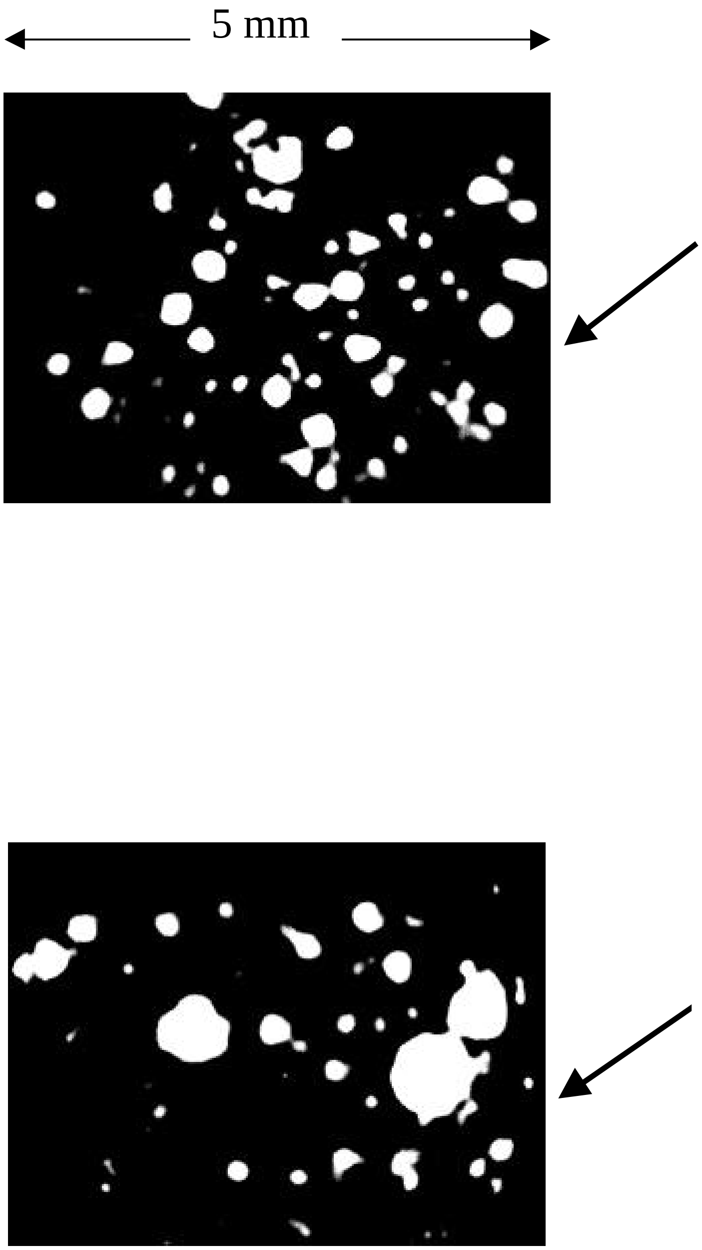
Fig.10 Air bubble size distribution at the same air content.

Table V Comparison of bubble size distribution in Synthetic and Vinsol resin mixtures at the same air content.

Diameter of air bubble *(μ)	Number of the air bubble per total 104 inch traverse length	
	Vinsol Resin	**Synthetic Resin**
20	340	218
40	265	257
60	160	237
80	135	195
100	95	135
120	81	60
140	52	39
160	44	44
180	35	52
200	31	38
250	59	80
300	36	87
350	32	59
400	20	52
500	23	84
600	16	34
700	8	38
800	8	24
900	2	25
1000	4	15
1500	6	17
>1500	11	18
Total	1463	1801
Air content (%) by the linear traverse method	6.9691	7.0361
Specific surface α (sq in/cu in)	866.71	741.23
Paste content (%)	27.2	27.2
Spacing factor $\overline{L}$ (mm)	0.1222	0.0993

*The figure give upper limits of diameter intervals; for example, 80 corresponds to 60 to 80 μ.

Data from R0414 and A0423.



Air bubble system in concrete with Vinsol Resin type admixture

Air bubble system in concrete with Synthetic resin type admixture

Fig.11 Comparison of air bubble size and distribution within the same area of paste.