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TESTS OF HIGH-RANGE WATER-REDUCING ADMIXTURES.(U)

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TESTS OF HIGH-RANGE WATER-REDUCING ADMIXTURES

by

Bryant Mather

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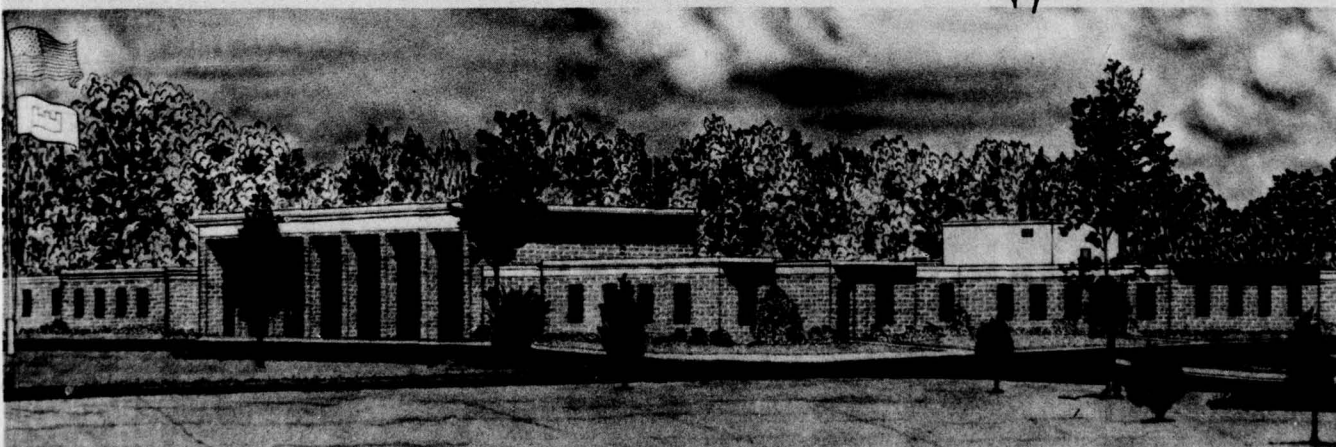
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20. ABSTRACT (Continued).

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PREFACE

This paper was prepared at the request of Mr. V. M. Malhotra of the Canada Centre for Mineral and Energy Technology (CANMET) for inclusion in the Symposium mentioned on page 1 of the paper. It is based on work done under the Civil Works Research and Development Program of the U. S. Army Corps of Engineers, Work Unit 31338. The results were reported in U. S. Army Engineer Waterways Experiment Station (WES) Technical Report C-76-3 by William O. Tynes except for more recent work by Kenneth L. Saucier mentioned in the Appendix. The paper was cleared for public release by the Public Affairs Office, Office, Chief of Engineers, on 28 March 1978.

The work was done in the Concrete Laboratory, WES, under the supervision of Mr. John M. Scanlon, Chief, Engineering Mechanics Division. Directors of WES during these investigations and the preparation of this report were COL G. H. Hilt, CE, and COL J. L. Cannon, CE. Technical Director was Mr. F. R. Brown.

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Tests of High-Range Water-Reducing Admixtures¹

by

Bryant Mather²

Abstract

At the Waterways Experiment Station, four high-range water-reducing admixtures were tested for compliance with the requirements for water-reducing admixtures given in ASTM: C 494-71. None passed the test for frost resistance; all met all other requirements. Water reductions ranged from 18 to 25 percent at recommended dosages. The reference concrete mixtures had durability factors ranging from 57 to 89 (Average 76); the test concretes had durability factors ranging from 5 to 77 (Average 36). A reference specimen from a batch having a DFE = 84 had a bubble-spacing factor ($\bar{L}$) of 0.003. Test concretes showed values of $\bar{L}$ rising from 0.004 to 0.012 as the DFE dropped from 77 to 14; in spite of a proper air content in the freshly mixed concrete.

¹ Prepared for presentation at International Symposium on Superplasticizers in Concrete sponsored by Canada Centre for Mineral and Energy Technology, Ottawa, Canada, 30-31 May 1978.

² Chief, Concrete Laboratory, US Army Engineer Waterways Experiment Station, P. O. Box 631, Vicksburg, MS 39180, USA.



Introduction

In the last few years products described as water-reducing admixtures that permit a concrete mixture to have of the order of 20 percent of the mixing water removed without loss of slump have appeared on the market. Advantage can be taken of this effect in the same ways that advantage has been taken of the effect of the several well-known water-reducing admixtures that have been available for many years, i.e., (1) concrete can be made having greater workability and easier placement with no reduction of cement content and no significant change in strength; or (2) the workability can be kept the same, the water content reduced, the cement content left the same, and higher strengths obtained; or (3) the workability and strength can be kept the same while the cement content is reduced, with resulting cost savings.

The products that have achieved the greatest recent prominence seem to be marketed primarily for increased strength or increased fluidity rather than reduced cost of materials.



Tests Made

The work done thus far at the Waterways Experiment Station has involved tests according to ASTM: C 494-71 (= CRD-C 87-72) to see if the products tested would comply with the requirements for water-reducing admixtures. We made specimens from 34 batches of concrete in connection with tests of admixtures marketed under four brand names from sources in the United States, Japan, Italy, and Germany.

Air-entrained concrete mixtures, containing 1-in. (25.4-mm) nominal maximum size aggregate, were proportioned with and without the water-reducing admixtures under test. The cement content was 517 lb/yd³ (307 kg/m³); slump was 2-1/2 ± 1/2 in. (63.5 ± 12.7 mm); and the air content when determined by ASTM Method C 231-75 was 6.0 ± 1 percent. The difference between the air content of the reference concrete and that of the concrete containing the admixture under test did not exceed 0.5 percent. A reference laboratory Type II cement was used rather than a

blend as specified in ASTM: C 494.* The water-reducing admixture was introduced as the last ingredient to the batch. Following such introduction, the specified 2-min mixing, 3-min rest, and 1-min remixing were done.

The liquid water-reducing admixtures were batched by weight. However, in order to compute dosage in the same units as were used to batch the air-entraining admixture, it was necessary to compute the unit weights. The values obtained were:

<u>Admixture</u>	<u>ml/lb</u>	<u>Solids, %</u>
SM-21-74	405	20
SM-28-74	370	42
SM-4-75	380	34

The relation of unit weight to reported solids content is shown in Figure 1.

The quantity of air-entraining admixture varied with the different cements and water-reducing admixtures and also as the amounts of the admixtures used changed. For some of the admixtures a greater amount of air-entraining admixture was required than used in the control mixture to obtain the desired air content, and for others a smaller amount was required. The relationships are shown in Figure 2.

Three batches of concrete were made for each major test condition.

As a result of reported differences between test results in this study and those of others with regard to resistance to freezing and thawing of concrete made using two of the admixtures, additional mixtures were made to investigate this discrepancy. It was suggested that the quantity of admixture being used may have caused the low durability factor or the slump was not high enough. The slump and air contents of a later mixture were varied to provide information as to the effect of higher slump and using a higher initial air content and slump. The air content was reduced from 10 to 5.2 percent; the slump was lowered from

* A proposal to revise C 494 to permit a laboratory to use a reference cement is under study in the appropriate subcommittee of ASTM Committee C-9.

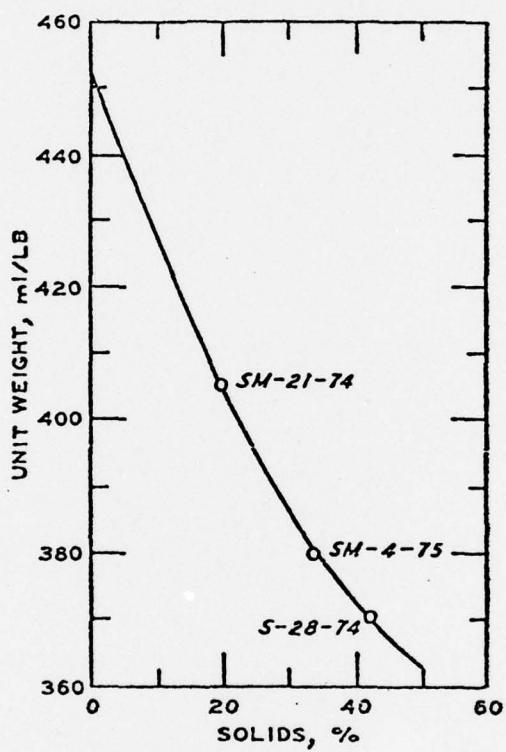


Figure 1. Relation of solids content (as reported by producer) to unit weight of liquid admixtures

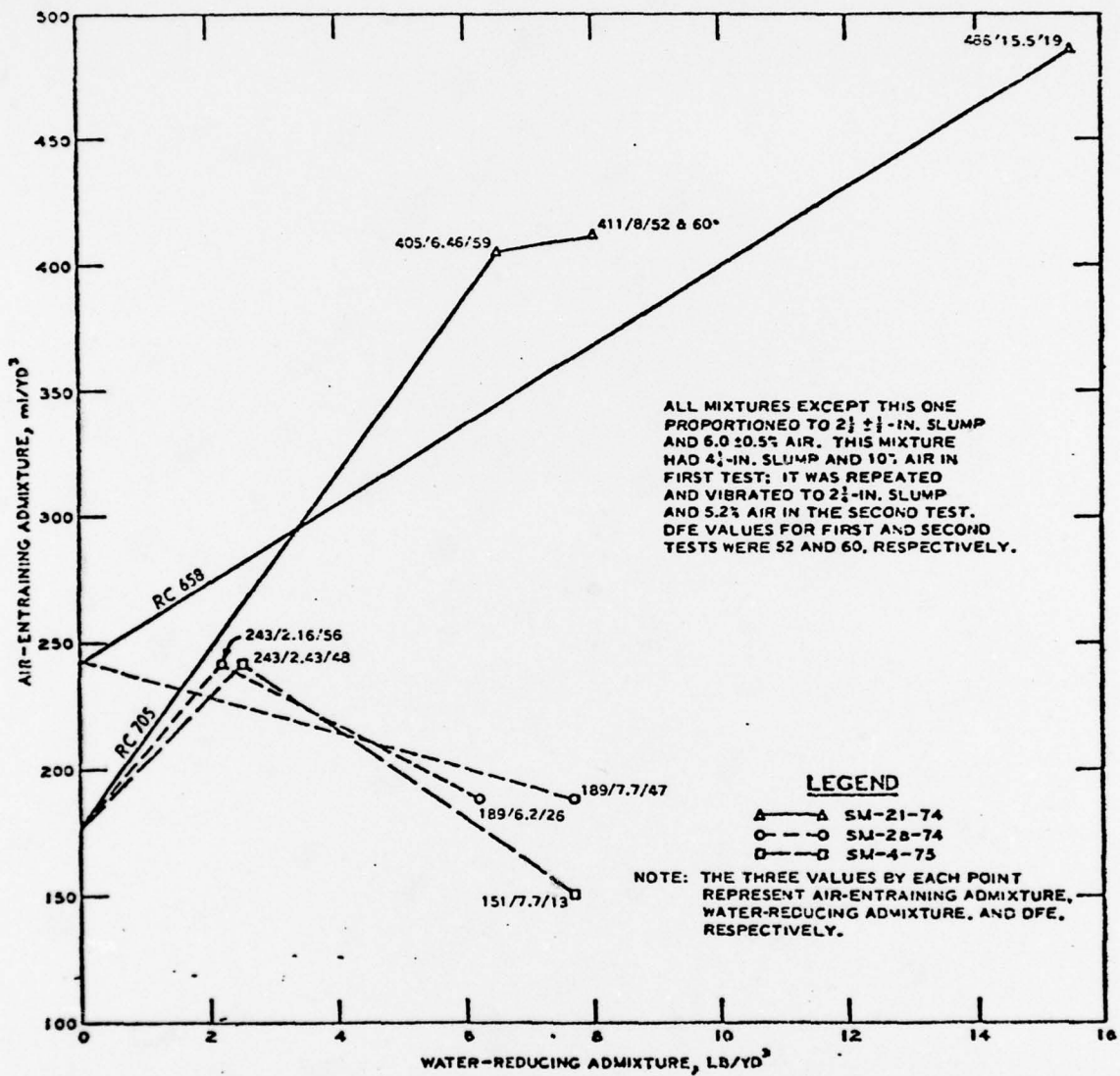


Figure 2. Effect of water-reducing admixtures on air-entraining admixture demand

4-1/4 in. (108.0 mm) to 2-1/4 in. (57.2 mm) on one batch, and the other batch was used as produced. This reduction in air content was accomplished by vibrating the complete batch of concrete 2 min with an internal vibrator.

When used in recommended amounts, water-reductions ranged from 18 to 25 percent. When used at lesser amounts water reductions were as low as 12.6 percent. At recommended amounts the 0.45 w/c for the control was reduced to 0.34 to 0.37 and the strengths correspondingly increased (see Figure 3). Values of compressive strength as percent of reference were:

	Age, Days				
	<u>3</u>	<u>7</u>	<u>28</u>	<u>180</u>	<u>365</u>
B	198	174	137	124	141
C	193	170	155	155	153
E	174	142	132	125	-

Values for flexural strength were (see Figure 4).

B	135	151	126	-	-
C	119	138	127	-	-
E	121	120	118	-	-

In the tests for drying shrinkage the reference specimens always showed less than 0.030 percent length change, hence the requirement is that the test specimens values should not exceed the reference values by more than 0.010. The actual differences were -0.002 and -0.001.

Frost Resistance

The only problem of consequence we encountered--and it was encountered with all four of the products tested--was in the development of an appropriate air-void system to produce adequately frost-resistant concrete when tested in the standard manner in the laboratory as prescribed in ASTM: C 494.

Before pursuing in greater detail the actual results of the tests and the efforts to understand the causes and significance of the results, let me digress for a moment and make some remarks about my interest in the matter and the relationship of that interest to commercial products.

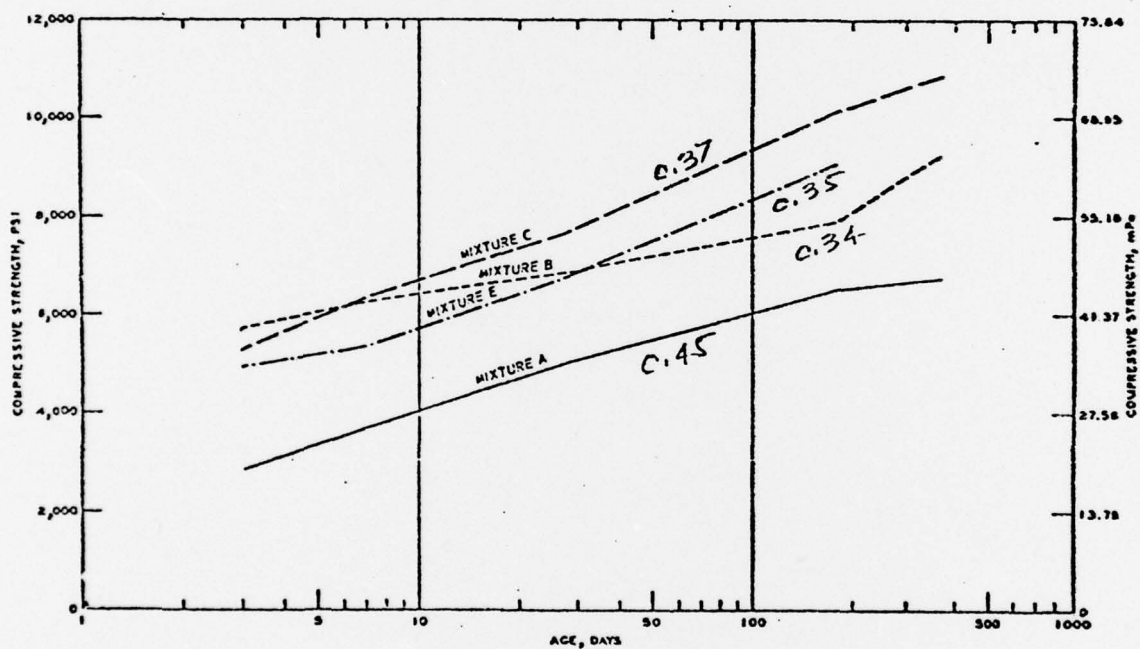


Figure 3. Average compressive strength of control mixture (A) and mixtures containing high-range water-reducing admixtures.

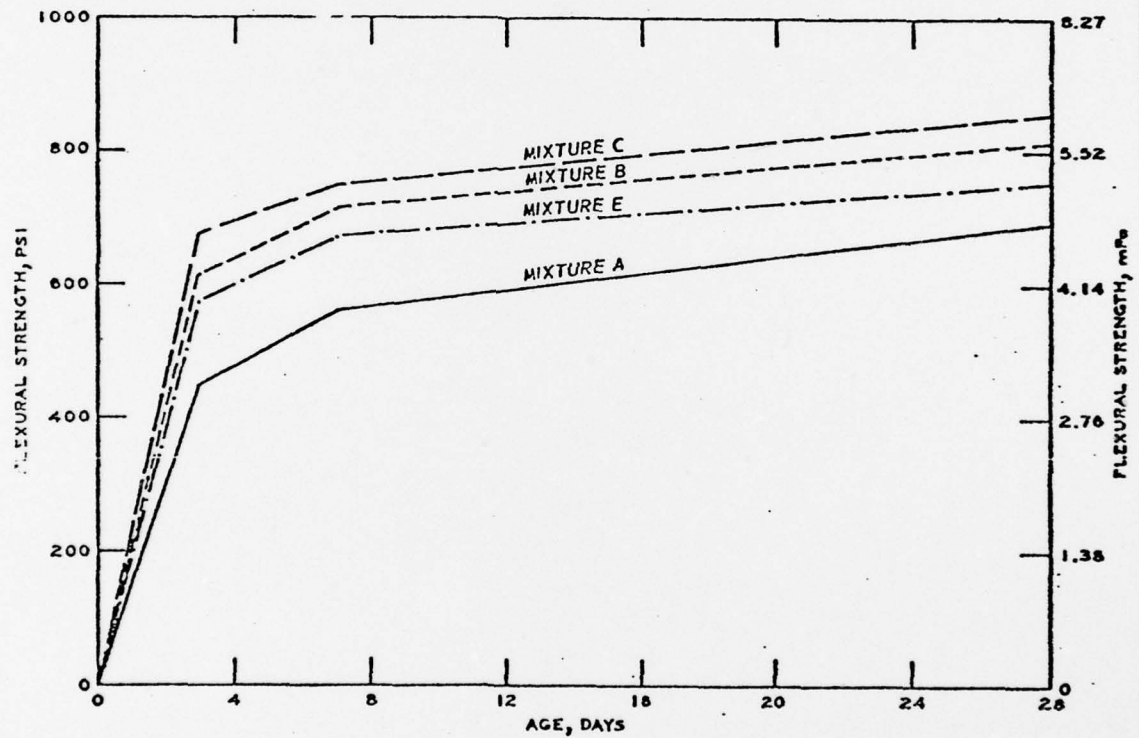


Figure 4. Average flexural strength of control mixture (A) and mixtures containing high-range water-reducing admixtures.

In 1973 representatives of the firm (SKW) in Germany that produces one well-known product and representatives of the firm (Kao) in Japan that produces another well-known product visited the Concrete Laboratory in Vicksburg and described their products and some of the results that had been obtained with them. The impression that I derived from the discussions with the people from Germany was that they were primarily interested in exploiting the potentialities of their product to produce a concrete that would be very fluid without being weak and subject to segregation. Essentially they were going to take advantage of water reduction by saving labor. The representatives from Japan on the other hand, mostly talked about the use of their product to obtain concrete of higher strength than would otherwise be obtained. Neither group appeared particularly interested in exploiting the potentialities of its water-reducing admixture for the purpose of reducing the cost of the materials in concrete by achieving concrete of the desired slump and the desired strength with less cement or, preferably, with an increased ratio of pozzolan or slag to portland cement.

In the meantime, our attention was called to a product produced by NOPCO Chemical Division of Diamond Shamrock, and to products of certain other manufacturers. Later a product marketed in Italy was sent to us for test for use in construction being carried out under the supervision of the Corps of Engineers in Saudi Arabia. The tests on this product were conducted as part of the normal materials evaluation for field use which is simply to find out whether or not it met the Corps of Engineers specifications which are the same as ASTM: C 494. The report that was produced stated that the product did meet all of the requirements except the one relating to resistance of freezing and thawing which is not of great importance in connection with acceptability for use in Saudi Arabia.

Samples of the other materials were tested in the laboratory as a part of work authorized by the Office, Chief of Engineers under Civil Works Research work unit relating to investigations of new materials.

The report on the work done in connection with the Civil Works research unit was published in April 1977.*

There are certain differences in the performance of these products in the tests that were done in our laboratory but it is our opinion that these differences are not especially important, at least from the standpoint of the interest that we have in the subject. The similarities are greater than the differences. All of these products including the product from Italy mentioned previously, permit water reduction typically of the order of twice as much as conventional water-reducer admixtures. However, we have not yet been able with any degree of success to produce concrete using any of these materials that will give satisfactory resistance to freezing and thawing using the apparatus and procedures that are used at our laboratory and which we believe to be in compliance with the requirements of ASTM: C 494.

The freezing and thawing resistance evaluation set forth in ASTM: C 494 involves fabricating standard concrete beams from two concrete mixtures, one containing an air-entraining admixture used at an appropriate dosage to give satisfactory resistance to freezing and thawing when the air content is in the range 6.0 ± 1.0 percent. The other mixture is made with the same aggregates, cements, air-entraining admixture, and with the chemical admixture under test and the air-entraining admixture dosage adjusted to give essentially the same air content as the control mixture, so that the difference between the air content of the reference concrete and that of the concrete under test will not exceed 0.5. Otherwise, the tests are made in accordance with C 666, procedure A, beginning the freezing at an age of 14 days and calculating the relative durability factor as described in C 260, with the requirement being that the relative durability factor be not less than 80.

* Tynes, William O., "Investigation of Proprietary Admixtures," US Army Engineer Waterways Experiment Station, Technical Report C-77-1, 1977 (NTIS AD A 039 612).

There are a few comments that might be made about these requirements. In the first place, the requirement pertaining to the selection of the air-entraining admixture states that it shall be a material such that when used in the mixture that is proposed as the reference concrete to entrain the specified amount of air, the concrete will be of "satisfactory resistance to freezing and thawing." It then goes on to say that if no material has been designated by the person or agency for whom the testing is to be done, the laboratory will use neutralized Vinsol resin. There would appear to be an implicit assumption that if the laboratory uses Vinsol resin and fails to get "satisfactory resistance to freezing and thawing" in the reference concrete, when the air content of that concrete as measured in the specified manner is between 5.0 and 7.0, it will be necessary to approach the solution of this problem through other means such as choice of cements or choice of aggregates. However, this continues to beg the question of what constitutes "satisfactory." Since the aggregates and cements and air-entraining admixture are constant, it perhaps does not unfairly affect either the consumer's risk or the producer's risk if the reference concrete is, in one laboratory, selected to be one which, in that laboratory, gives a durability factor of 95, whereas in another laboratory the reference concrete it uses gives a durability factor of only say 65.

The intent of the requirement is that the presence of the water-reducing admixture should not adversely affect the frost resistance of properly air-entrained concrete made with cements, aggregates, air-entraining admixtures, and mixture proportions appropriately to making frost-resistant concrete in the absence of a water-reducing admixture. This is why the requirement is expressed as "relative durability factor" not less than 80. The assumption presumably is that with the required number of specimens and the inherent variability in the test, if the results come out as $RDF = 80$ or higher, it may be concluded that no proof has been found of a tendency for the water-reducing admixture to adversely affect frost resistance.

With conventional water-reducing admixtures it has often been observed that the quantity of air-entraining admixture required to

produce the stipulated level of air content in the concrete in the test mixture in which the water-reducing admixture is used is substantially less than in the control mixture. I do not believe that conclusive studies have been reported as to whether this reduction represents air entrainment by the water-reducing admixture or simply alteration of the physical-chemical environment so as to permit the air-entraining admixture to operate more efficiently and, hence, produce the desired air-void system at a lower dosage. I have heard speculation more often in support of the latter point of view.

In any event, in the present studies, it was observed that in some cases the quantity of neutralized Vinsol resin that was needed to give concrete of the stipulated air content increased as the dosage of the exotic water reducer increased and in other cases it decreased. Some of the differences were quite substantial. In one case, the quantity of neutralized Vinsol resin required to produce the air content increased from approximately 250 ml/yd³ in the reference concrete to nearly 500 ml/yd³. On the other hand in the tests of the Italian product the quantity of NVR used in the test concrete dropped to 73 ml/yd³ from 178 in the reference concrete.

The 34 batches of concrete previously mentioned involved 18 batches containing the exotic water-reducing admixtures and 16 reference mixtures with only the air-entraining admixture. The reference concretes and the comparable test concretes were always made concurrently using cements, aggregates, and air-entraining admixture from the same reference lots. The work was done over a period of time and involved two different cements: one made in Alabama the other made in Texas. Two freezing and thawing test specimens were made from each batch of concrete. The 32 reference specimens had durability factors ranging from 57-89 (average 76); the 36 test concrete specimens had durability factors ranging from 5-77 (average 36); or considerably less than required ($0.8 \times 76 = 61$).

There is no question in my mind that the almost universal failure of the test mixtures to show good frost resistance was due solely to our failure to produce in these mixtures a suitable air-void system.

Six specimens were selected and examined by ASTM: C 457 for bubble-spacing factor ($\bar{L}$): one from a reference batch with a DFE of 84 had $\bar{L} = 0.003$; one with each of the three products tested in the research series; and one with two of these tested at reduced dosage on the recommendation of the representatives of the producers. Data on the specimens on which bubble-spacing factor information was obtained are shown below:

Admixture	DFE 300	Air				$\bar{L}$	
		Pressure	Microscope			in.	mm
			Entrained	Entrapped	Total		
None	84	6.3	5.5	0.6	6.1	0.003	0.08
B	32	6.2	2.4	1.2	3.6	0.011	0.28
B1	77	6.6	5.8	0.8	6.6	0.004	0.10
C	55	6.0	3.8	1.1	4.9	0.008	0.20
C1	26	5.8	2.8	0.3	3.1	0.009	0.23
E	14	5.7	2.7	1.0	3.7	0.012	0.30

The values follow, in general, a regular relation of decreasing DFE with increasing $\bar{L}$, in which DFE = 50 corresponds with $\bar{L}$ = about 0.008 in. One batch of concrete with one high-range water reducer gave DFE = 77 and $\bar{L}$ = 0.004 in. A second batch, intended to be identical, gave DFE's of 38 and 43 (see Figure 5).

At that point we set out to see how our findings compared with those of others. The pattern that continues to emerge is very clouded--and very interesting. I report it to you in the pieces in which it has come to my attention--in no particular order.

1. A visitor, knowledgeable about concrete materials properties, from Japan, in our laboratory in the spring of 1976 asked if we had studied these materials. I described our experience and sought his. He said he'd let me know. About 10 months later the representative of the marketer of an admixture asked me to be sure in the future if I described our results, also to state that the producer's representative had told me that others had found satisfactory freeze-and-thaw results using the product of interest to him.

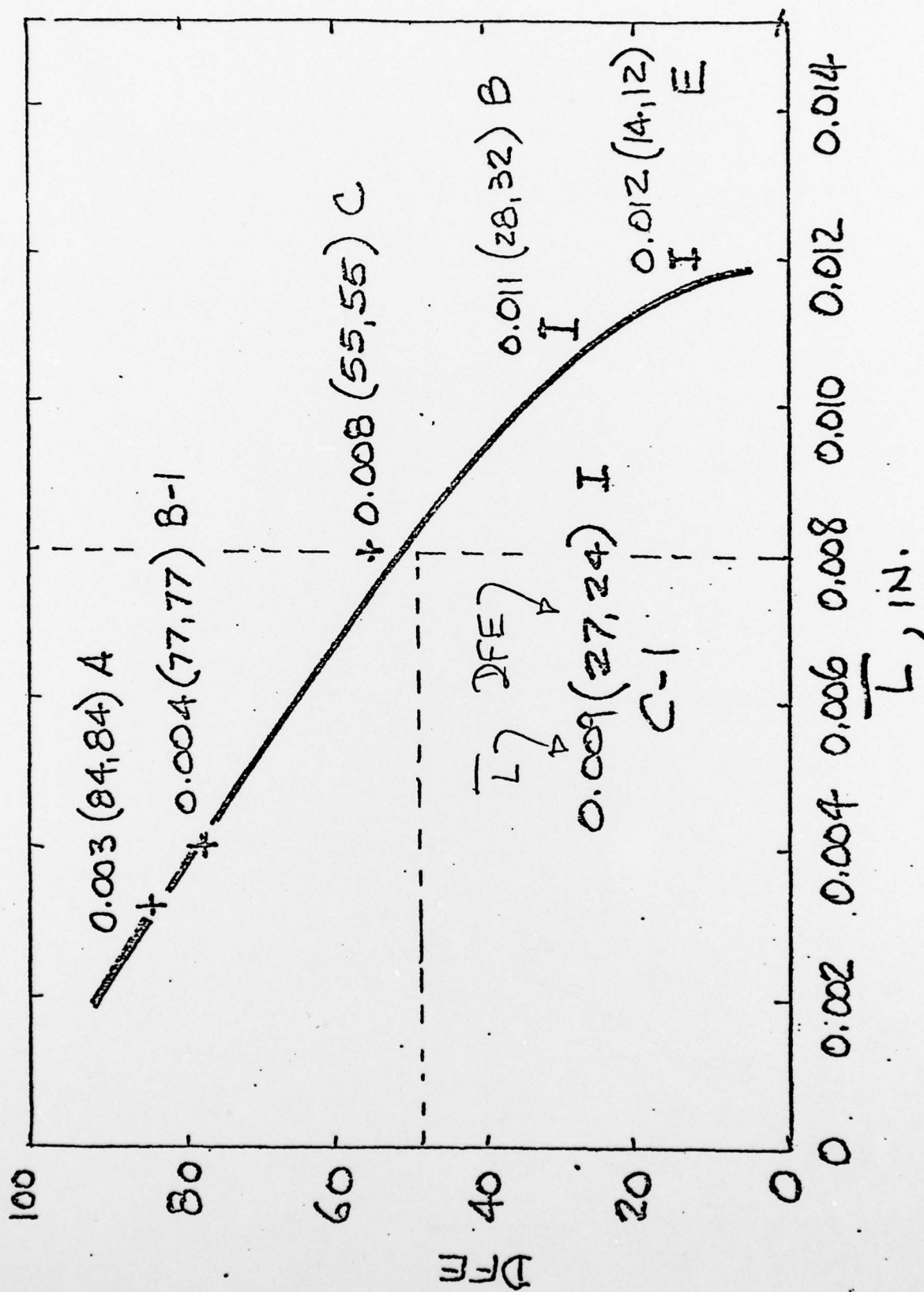


Figure 5

2. In seeking to find ways of overcoming the difficulty or developing an explanation of what caused it, I sought details of tests made elsewhere in which satisfactory behavior was found. When I sent copies of my correspondence on these to representatives of both of the major products, I got a reply from one: "I must ask that in the future any reports or technical information which we send you be kept in strict confidence and not shown to a competitor." This suggests that some of the marketers don't really want the problem solved, they want it overlooked.

3. Data from one reputable private laboratory showed DFE = 88 for the reference and 87 for the test mixture. However, in the detailed reports the sources of the aggregates were given. This aggregate combination in air-entrained concrete would be expected to have a DFE in the range 50 to 60 rather than 90 based on Corps of Engineers test data from two different Corps laboratories.

4. I was advised by a representative of one marketer that "if you plan to cite your results I would ask that you mention the enclosed data as well to give a balanced picture." I am more than happy to do so since these data reveal the present sad state of freezing and thawing testing in the USA. Before presenting these data, I would like to call to your attention what I believe to be the essential characteristics of a performance test for resistance of concrete to freezing and thawing.

a. There is no point in making a freezing-and-thawing test where choice of concrete materials is the variable under study unless the specimens are frozen in a critically saturated state and kept so saturated throughout the testing. If the concrete isn't critically saturated, it doesn't matter whether or not the paste is provided with a proper air-void system or whether the aggregate is sound. This is why freezing in air (ASTM: C 666, Procedure B) is explicitly not allowed by C 494; when freezing in air is done it is easy to lose just enough water (especially from low water-cement ratio concrete) to drop below critical saturation (91 percent).

b. A freezing and thawing test is worthless unless it rapidly destroys critically saturated non-air-entrained concrete and properly air-entrained concrete made with unsound saturated coarse aggregate.

5. Now for the data:

a. In the laboratory of a state department of transportation it was found that using the high-range water reducer at a given dosage in air-entrained concrete the DFE was 70-80 and at the same dosage in non-air-entrained concrete the DFE was 75-83.

b. In the laboratory of a second state department of transportation it was found at two cement factors that the reference and test DFE's were 103, 103 and 103.7, 102.7, respectively; at one cement factor the salt scale ratings were 1.0/1.5 at the other 1.5 and 1.0.

c. In the laboratory of a third state department of transportation F&T losses were reported in percent; the values were 0.2, 0.3, 0.3, and 0.4 for the controls and 0.2 to 0.5 for the test--which it regarded as "negligible." The tests were made at 1 cycle/day for 25 cycles in a 10 percent NaCl solution.

d. The laboratory of a fourth state department of transportation reported durability factor of 99.6 for the test concrete.

e. So much for the data I was asked to report.

6. Another US Government agency has tested a high-range water reducer and told us it failed the F&T requirement.

7. A producer of conventional admixtures has told us that their tests of a high-range water reducer showed failure in F&T and an excessive air-void spacing factor.

8. In the 1976/77 Zement Taschenbuch of the Verein Deutscher Zementwerke there is a chapter by Dr. Bonzel from Dusseldorf on Fließbeton or Flowing Concrete which contains a section (2.4.3.4) on frost resistance. This section says that the rules affecting frost resistance of normal concrete also apply to fluidified concrete and it must, to be resistant, have a bubble spacing factor not larger than 0.20 mm. They then add: "Since a detrimental change in size and distribution of air voids by the fluidifying medium or the fluid concrete

consistency cannot be excluded in each case, the total air content of the fresh concrete can be considered sufficient only when the combination has been tested for spacing factor."

This was translated for me by one of the most experienced workers on frost resistance in Germany, she remarked: "The writer was not too sure about the frost resistance of Fließbeton...One thing is sure: he did not mean to say it was enough to get the right air content, the spacing factor is the decisive thing, it must be less than 0.20 mm."

So I conclude that somewhere somebody has the problem of finding out how to produce concrete with high-range water reducers having a bubble spacing factor of 0.20 mm (= 0.008 in.) or less in a controllable, predictable way. Maybe somebody has--but Dr. Bonzel in Dusseldorf seems worried; the Japanese National Railways are reported to be worried; only the Corps of Engineers in Saudi Arabia is not worried--for obvious reasons.

We have tried hard to tell what it is we did that gave us the batch that contained a high-range water reducer and a DFE of 77 and $\bar{L} = 0.004$ in. We were not able to reproduce it. We tried a batch of the product from Italy--where the test concrete only took 73 ml of NVR as compared with 178 in the control, where we put in 178 ml of NVR and the slump went to 7-1/2 in. and the air to 13 percent and the DFE went up to 35 from the previous level of 13; we must have improved $\bar{L}$ some.

In our report we raise the question of whether the use of an air-detraining agent is indicated; we do not have funding to study this.

Concluding Statement

None of the foregoing should be taken to indicate that we are other than enthusiastic about the potential of these materials, we agree with Pomeroy who said in the September 1976 issue of the Magazine of Concrete Research: "The most promising field for the wider use of polymers in concrete is as a workability aid and water-reducing agent." We wonder a little about the significance of the remarks of Hewlett and Rixom in Concrete for September 1976 who note: "Superplasticized

concrete releases large quantities of air since the fluid state is conducive to rapid air release. However, residual air content figures are normally in the range 1-3%." They remark later that such concretes have "excellent durability." It should be remembered that in the UK it is not regarded as proper for concrete to be damaged by frost.

Acknowledgement

The tests described and the resulting data presented herein, unless otherwise noted, were obtained from research conducted under the Civil Works Research and Development Program of the United States Army Corps of Engineers by the US Army Engineer Waterways Experiment Station. Permission was granted by the Chief of Engineers to publish this information.

APPENDIX

The foregoing manuscript was completed and submitted to Mr. Malhotra on 1 September 1977. It was accepted on 16 September subject to submittal of originals of the drawings. They were submitted on 26 September 1977. At that time I was fairly pessimistic about the prospects that the agencies marketing high-range water-reducing admixtures in the United States would realize that they had a problem that needed some solution other than merely asserting that the Corps of Engineers Concrete Laboratory didn't know how to run a test of concrete for resistance to freezing and thawing that would give the right answer.

Then there began to be gleams of light at the end of the tunnel. First one, then another, then a third marketing agency let it be known that it believed it had a "slightly" modified version of its product that might behave better in the Corps of Engineers laboratory--emphasizing all the time that their normal previous product was, of course, satisfactory for the rest of the world.

Late in the fall of 1977 we received a sample of one of such modified product and obtained authority and funding to make tests using it. As we had done previously we made six batches of concrete, three with and three without the admixture. In addition, in this case, we vibrated one of the three freezing-and-thawing test specimens from each batch and hand rodded the other two. Either procedure for consolidation is permitted by the applicable ASTM standard. The test results were not significantly different from previous work with material of the same brand except with regard to resistance to freezing and thawing. In these more recent tests there was no significant difference between the frost resistance of the concrete of comparable air content made with and that made without the high-range water reducer. Actually the average DFE values were 80 and 74 so that the admixture concrete had a relative durability factor of 108. There was also no significant difference in air content or frost resistance as a function of whether the concrete in the test specimens was consolidated by vibration or hand rodding. One specimen for example made using 517 lb of cement, 191 lb of water per cubic yard, and 41.1 ounces of high-range water reducer per hundredweight of

cement (3.0 percent liquid/cwt), consolidated by rodding, had an air content freshly mixed of 6.5 percent, air content hardened 5.7 percent, an air-void spacing factor of 0.006 in. (0.15 mm), and a durability factor of 79. The marketer of the admixture is identifying the modified formulation with a letter designation to distinguish it from the product of the same brand name we tested previously. Data relating to these tests are summarized on Table A-1.

TABLE A-1

Tests of Modified High-Range Water-Reducing Admixture

	Control			Test		
Cement, lb/yd ³	517			517		
kg/m ³	307			307		
Water, lb/yd ³	233			191		
kg/m ³	138			113		
NVR, ml/yd ³	178			620		
WRA, lb/yd ³	-			15.5		
Batch	1	2	3	1	2	3
Air, %, rodded	5.8	6.5	5.8	5.5	6.5	5.8
vibrated	5.8	6.5	5.5	5.8	6.5	6.0
DFE ₃₀₀ , rodded ^(a)	84	58	70	86	78	80
vibrated ^(b)	87	73	77	80	71	79
average		74			80	
Comp Str, psi, 3d	1890	1730	1960	4040 (214) ^(c)	4160 (240)	4520 (230)
7d	2710	2540	2750	5410 (199)	5410 (213)	5360 (195)
28d	4680	4340	4360	7320 (156)	6870 (158)	6890 (158)

(a) Average of 2

(b) Single specimen

(c) Percent of control

In accordance with letter from DAEN-RDC, DAEN-ASI dated 22 July 1977, Subject: Facsimile Catalog Cards for Laboratory Technical Publications, a facsimile catalog card in Library of Congress MARC format is reproduced below.

Mather, Bryant

Tests of high-range water-reducing admixtures / by Bryant Mather. Vicksburg, Miss. : U. S. Waterways Experiment Station ; Springfield, Va. : available from National Technical Information Service, 1978.

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1. Concrete admixtures. 2. Concrete durability. 3. Concrete mixtures. 4. Concrete tests. 5. Frost resistance. 6. Water reducing agents. I. United States. Army. Corps of Engineers. II. Series: United States. Waterways Experiment Station, Vicksburg, Miss. Miscellaneous paper ; C-78-3.

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