EFFECTS OF FLAKE AND PELLET FORM OF CALCIUM CHLORIDE ON THE COMPRESSIVE BREAKING STRENGTH OF STANDARD PORTLAND CEMENT CONCRETE

Thesis for the Degree of M. S.

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Roger John Claus

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This is to certify that the

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EFFECTS OF FLAKE AND PELLET FORM OF CALCIUM CHLORIDE ON THE COMPRESSIVE BREAKING STRENGTH OF STANDARD PORTLAND CEMENT CONCRETE

presented by

ROGER JOHN CLAUS

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C. M. Cade

Major professor

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A THESIS

Submitted to the School of Graduate Studies of Michigan
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AN ABSTRACT

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Approved Charles O. Harris

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The objectives of this study were: (1) to determine the changes in the compressive breaking strength of standard portland cement concrete due to the addition of various amounts of flake and pellet form of commercial calcium chloride, and (2) to determine whether or not one form is superior to the other form.

Concrete mixes of constant design containing calcium chloride in the amounts of 0, 1, 2, 3, and 4 percent by weight of cement were placed in 6x12 inch steel molds. These five batches were poured consecutively, and constituted a series. Specimens of each series were tested at the ages of one day, three, seven, and twenty-eight days. For each form of calcium chloride, six series were poured; in other words, there were six specimens for each variable.

Results of the strength tests indicated the following conclusions:

(1) Additions of either form of calcium chloride in any percentage (1 to 4) increased the strength of the concrete at all ages studied.

(2) The optimum amounts of the commercial calcium chlorides were from 2 to 3 percent.

(3) Peladow was equal to or slightly superior to Dowflake.

(4) On the basis of content of pure anhydrous calcium chloride, one form was about as effective as the other form.

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I. INTRODUCTION

A. Objectives

The objectives of this study were: (1) to determine the changes in the compressive breaking strength of standard portland cement concrete due to the addition of various amounts of flake and pellet form of commercial calcium chloride, and (2) to determine whether or not one form is superior to the other form.

This study was limited to the total effects of the two forms of calcium chloride upon the strength of the concrete without regard for the individual strength contributions of the several chemical compounds of cement.

B. History

Calcium chloride is a white, very deliquescent salt; that is, a solid which gradually passes into solution by absorbing moisture from the air. It is obtained from natural brines, and also is formed as a by-product in the manufacture of a number of compounds.

For many years, calcium chloride has been used as a drying agent. It is used to lay the dust on roads, and occasionally is applied to the surface of a newly laid concrete road to prevent evaporation of the mixing water.

Certain amounts of this salt mixed integrally with concrete are known to increase considerably the early strength of concrete. Calcium chloride has been used in this manner for the past twenty-five years.

As an admixture to portland cement concrete, calcium chloride has been the subject of considerable research. Perhaps the most notable research was that done by Paul Rapp at the National Bureau of Standards in the early nineteen thirties. Among his conclusions are that: (1) the workability of the concrete was increased by the addition of calcium chloride up to and including 3 percent of commercial calcium chloride (up to 2 1/4 percent pure anhydrous calcium chloride), (2) in regard to strength, 2 percent is the optimum amount of commercial calcium chloride (1 1/2 percent pure) for standard portland cement concrete cured at 70 degrees Fahrenheit.

The American Society for Testing Materials has a standard specification (D98-48) for calcium chloride used in concrete. The first tentative specification was published in 1921. The present one was adopted as standard in 1948. It requires that the calcium chloride be in the form of flakes with 100 percent passing a 3/8-inch sieve, 20 percent maximum being retained on a 1/4-

inch sieve, and 10 percent maximum passing a No. 20 (840-micron) sieve. A micron is one-millionth of a meter.

Since World War II, the Dow Chemical Company has found it expedient to manufacture calcium chloride in the form of pellets which they call Peladow. At the same time this company manufactures the flake form under the trade name of Dowflake. This latter form is in substantial agreement with the specification of the American Society for Testing Materials. A physical comparison of the two forms may be had from the photograph in Figure 1 and the sieve analyses in Table I. In addition to Peladow's not conforming physically to the aforementioned specification, it also contains more alkali chlorides than allowed by that specification.

C. Reasons for this Study

Since the work of Rapp, almost two decades have passed. Therefore it was thought desirable to substantiate earlier results using present day cements. Also, the production of the pellet form of calcium chloride logically presents this question: What effect does the form of this salt have as an admixture for portland cement concrete?

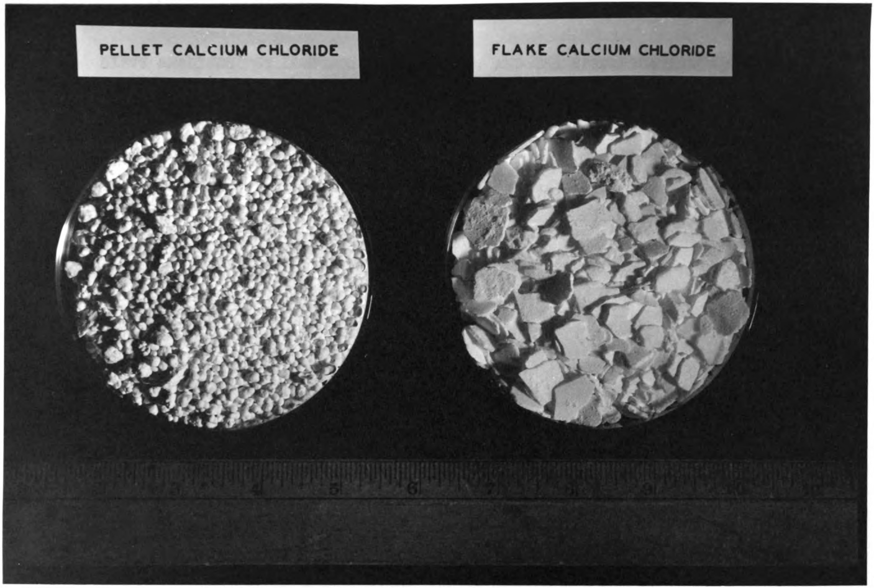

Fig. 1. Comparison of Dowflake and Peladow

TABLE I

SIEVE ANALYSES[1] OF DOWFLAKE AND PELADOW, AND THE SPECIFICATION[2] FOR FLAKE CALCIUM CHLORIDE

Sieve Size	Peladow		Dowflake		Specification for Flake Calcium Chloride	
	Percent Retained	Percent Passing	Percent Retained	Percent Passing	Percent Retained	Percent Passing
3/8 inch	0	100	2	98		100
1/4 inch	1	99	37	61	20 max.	
No. 20	98	1	54	7		10 max.
Pan	1		7			

[1] Samples were taken from very small lots of calcium chloride.

[2] This is the American Society for Testing Materials Specification D98-48.

D. Scope of this Study

This study is a part of a broad calcium chloride research program initiated and financed by the Dow Chemical Company. It is expected that the investigation will include the effect of the flake and pellet form of the salt upon most of the physical and chemical properties of portland cement mortar and concrete. Both standard and high early strength cements will be used, with and without an air-entraining agent. Also, the calcium chloride will be added in both the dry and liquid form.

In this study, only standard (normal) portland cement was used, and Dowflake and Peladow were added to the mixer in dry form only. The mix design was the same for every batch. The intention was that there be only two variables. They were the amount and form of calcium chloride used as an admixture.

Both forms of the salt were added to the mixer in the amounts of 1, 2, 3, and 4 percent by weight of the cement.

In addition to the study of the effects of the chlorides upon strength, measurements of consistency (slump test) and of entrained air were made.

For each form of calcium chloride, 120 specimens were tested for strength at the ages of 1, 3, 7, and 28 days.

II. SCHEDULE OF SPECIMENS

Concrete test cylinders were identified according to series, amount and type of calcium chloride, and age. Each series consisted of five batches of concrete, a control batch of plain concrete and batches containing 1, 2, 3, and 4 percent, respectively, of commercial calcium chloride by weight of cement. From each batch there were made five 6x12 inch concrete cylinders, four of which were tested at the ages of 1, 3, 7, and 28 days, respectively. This report does not include the results of the 90 day tests. Each specimen was identified by symbols. For instance, I-PD2-4-7 indicated type one (normal or standard) cement, Peladow added dry in the amount of 2 percent, series number four, and to be tested at seven days.

The results in this report are based upon the average of six series using the flake form and six series using the pellet form of calcium chloride. In other words, of the 300 specimens which were formed, 240 were tested.

III. CONDUCT OF THE RESEARCH

A. Concrete Mix

1. Aggregates. The aggregates were donated by the American Aggregate Corporation of Brighton, Michigan. These were natural aggregates, and were to conform with the Michigan State Highway Department specifications 2NS for sand and 6A for gravel. As shown in Table II, the coarse aggregate was deficient in material passing the 1/2-inch sieve. This lack of some fine material in no way affected the validity of the test results. The mix was a little harsh, but still workable and plastic.

Upon receiving the aggregates in one shipment, they were stored outside. Loss and contamination were prevented by a baseboard around the piles and a waterproof canvas tarpaulin over each pile. The aggregates were brought to approximately room temperature by transferring them to inside bins two or three days before using.

2. Cement. Specimens of the twelve series reported in this paper were made using type one portland cement. This is the standard cement used for general concrete construction. Arrangements were made with the

TABLE II

SIEVE ANALYSES OF AGGREGATES
AND THEIR SPECIFICATIONS

Aggregate	Sieve Size	Percent Passing	Specifications* Percent Passing
Coarse	2 in.	100	100
	1 1/2 in.	100	95-100
	1 in.	71	60-90
	1/2 in.	14	25-55
	No. 4	0	0-8
Fine	3/8 in.	100	100
	No. 4	97	95-100
	No. 8	86	65-95
	No. 16	66	35-75
	No. 30	46	20-55
	No. 50	21	10-30
	No. 100	6	0-10

* Specifications are those of the Michigan State Highway Department for 2 NS natural sand and 6A gravel.

Peerless Cement Company of Detroit to obtain from one grind all of the cement for the project.

3. Calcium chlorides. The calcium chlorides used as an admixture were supplied by the Dow Chemical Company. In addition to the difference in the form of the two products, there is a considerable difference in purity. Dowflake contained 77.2 percent pure anhydrous calcium chloride and Peladow 96.1 percent.

4. Proportioning. The concrete mix was proportioned according to the mortar-voids method as practiced by the Michigan State Highway Department. All mixes were designed for a cement content of 5.5 bags per cubic yard of concrete, and a relative water content of 1.30. This resulted in a water-cement ratio of 6.06 gallons per sack of cement and dry weight proportions of 1 part cement to 2.46 parts of sand to 3.77 parts of gravel.

Trial batches indicated that the relative water content of 1.30 would give the control batches a desired four inch slump.

B. Mix Procedure

1. Weighing materials. The aggregates, cement, and water were weighed on a Toledo platform scale reading directly to two ounces. Another Toledo scale was used to weigh the calcium chloride to the nearest gram.

A capacity batch from a one cubic foot mixer provided concrete for five cylinders and the entrained-air indicator bowl.

2. Mixing. The most satisfactory mixing was obtained when a certain procedure was followed. After the coarse and fine aggregates were added, the mixer was turned on and the cement added. Then the water was deposited in a manner that minimized the clogging of the blades with cement paste. Finally, the calcium chloride was added.

A stop watch was used to time the mixing which was two minutes for every batch. The interval was reckoned from the instant all the water was in the drum.

After the batch was discharged into a large mixing pan, the drum was brushed out to insure the correct amount of cement paste for each batch. The concrete was then hand-mixed just enough to correct segregation.

3. Measurements of slump and entrained air. Immediately following the remixing by hand, two slump tests were made for each batch. At the same time, the air content of the concrete was determined by means of a Klein-type air meter.[1]

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[1] See pages 279 and 362 of reference number 2.

4. Molding specimens. The concrete was scooped from the large mixing pan into the five steel molds. Each of three approximately equal volume layers was rodded with twenty-five strokes of a standard tamping rod. The top layer was struck off with a steel trowel and finished with a wooden smoothing tool.

The molds were nominally six inches in diameter and twelve inches long. They were made of one-eighth inch thick seamless steel tubing cut to provide one longitudinal joint. This joint was closed by means of two bolt-clamps. Each cylinder was bolted to a ten inch by ten inch by one-half inch steel base plate (see Figures 2 and 3). To prevent loss of mixing water, the longitudinal joint was sealed with masking tape and hot paraffin was applied around the circumference of the mold at the base plate. Before a series was poured, the assembled mold and base plate was oiled with mineral oil. After each use the parts were cleaned bright by means of a wire wheel brush driven by a flexible shaft machine. The above procedure resulted in test specimens of excellent appearance.

C. Curing

Soon after the last batch of concrete was molded, wet burlap was placed over the twenty-five cylinders.

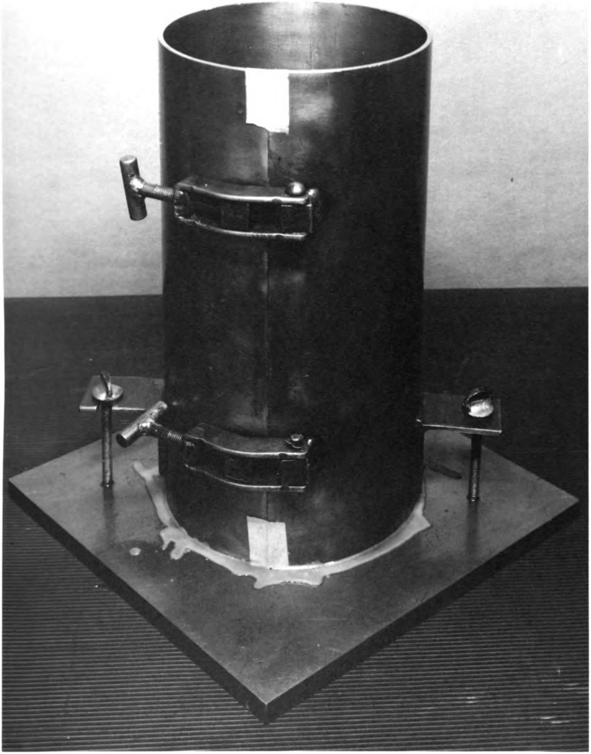

Fig. 2. Detail of mold assembly

Fig. 3. Molds as used for each series

Approximately twenty-four hours later the specimens were removed from the molds. Then each cylinder was marked, and all but the five for the one day test were placed in the moist room. The temperature and the relative humidity of this room are controlled at 70 degrees Fahrenheit and 98 percent, respectively. Cylinders were stored under these conditions until they were capped before testing.

D. Capping and Testing

Only the top ends of the specimens were capped, as the bottom ends were plane within the limits specified by the American Society for Testing Materials specification C192-49.

To facilitate capping, a bench was designed to provide for the accurate capping of ten specimens (see Figures 4 and 5). After the gypsum plaster caps were allowed to harden about one hour, the specimens were tested.

During the hardening time, the cylinders were measured to determine the cross-sectional area. As all of the steel molds were slightly out-of-round in varying amounts, an average diameter was sought by measuring each specimen at the top, middle, and bottom in such a manner that each diameter made an angle of 120 degrees

Fig. 4. Capping bench for ten specimens

Fig. 5. Detail of specimen capping

with the preceding one. By means of a dial-equipped caliper, diameters were measured directly to one-hundredth of an inch (see Figure 6).

Then the cylinders were placed in a three hundred thousand pound capacity Rhiele hydraulic compression testing machine. Load was applied at the rate of thirty-five pounds per square inch per second. The maximum load carried by the specimen was generally recorded to the nearest five hundred pounds. (A second dial reads to one hundred pounds for loads under sixty thousand pounds.)

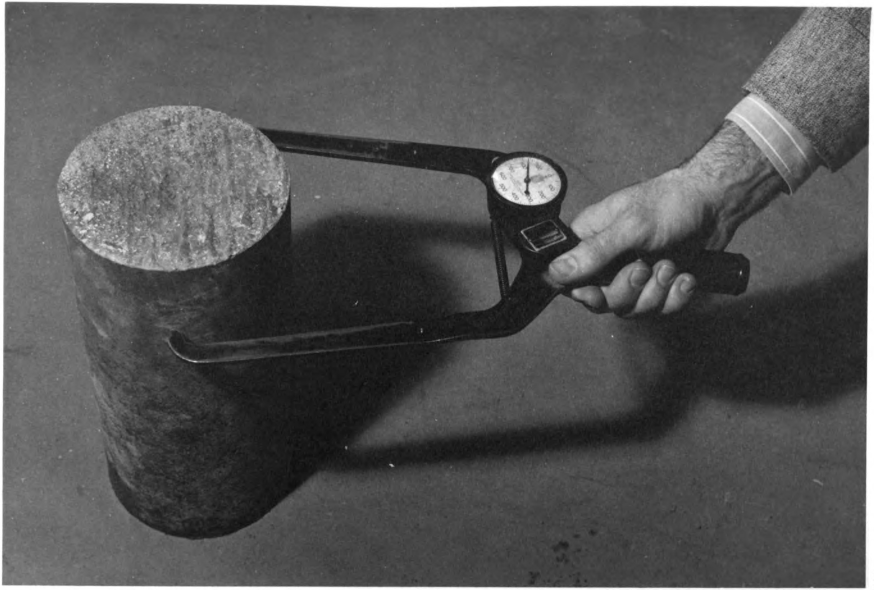

Fig. 6. Measuring a diameter of a specimen

IV. RESULTS

A. Slump--A Measure of Consistency

As may be seen from Figure 7, the average slump gradually increased from four to five and one-half inches as the Dowflake was increased from zero to 4 percent. On the other hand, Figure 8 shows that the average slump increased with concretes containing 1 and 2 percent of Peladow, and then decreased with those containing 3 and 4 percent. Each point on these graphs represents the average slump of six batches of concrete.

The control batches (the zero percent calcium chloride batch of each series) were designed to have about a four inch slump. This was realized in the flake series, while in the pellet series the average slump was about five and one-half inches. However, in both series the individual control batch slumps varied widely from the aforementioned averages. Although precautions were taken, these variations were probably due to some segregation of coarse aggregate and to non-representative sampling for moisture determinations. The writer does not believe that this variation of slump appreciably affected the comparison of the two forms of calcium chloride. However, in the interest of consistency, series having either very high or very low slumps will be re-

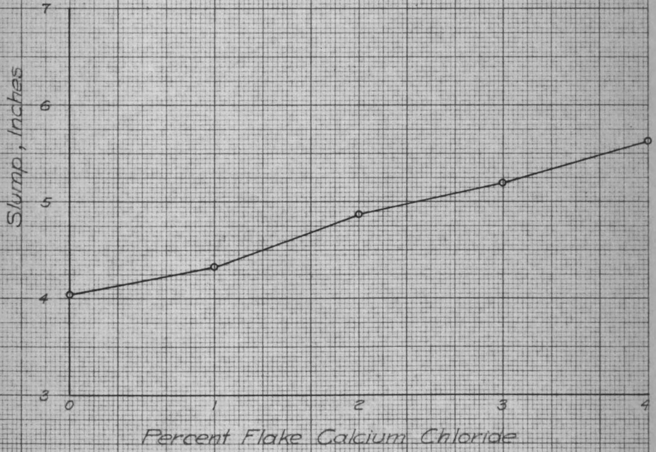

Fig. 7. Effect of flake calcium chloride on slump

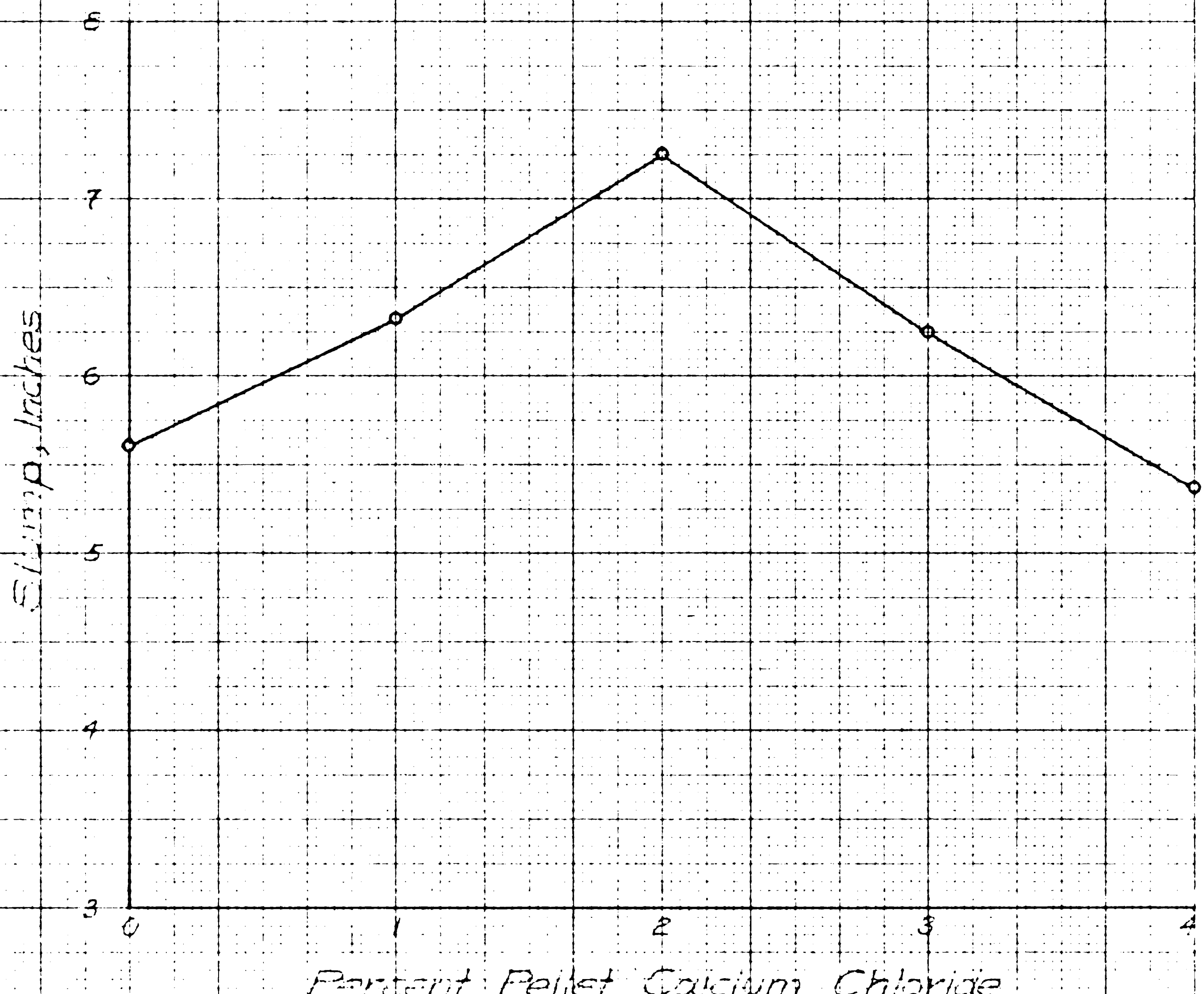

Fig. 8. Effect of pellet calcium chloride on slump

poured. These changes will be made when this study is incorporated as part of the complete calcium chloride project.

B. Air Content

Apparent air content averages for the freshly mixed concrete batches are shown in Figures 9 and 10. The results indicate that the addition of either form of calcium chloride in the amounts studied slightly increases the entrained air. Because of certain probable laboratory errors, and because the change is not great, no more specific statement appears to be justified.

C. Compressive Breaking Strength

Results of the tests graphed as shown in Figures 11 and 12 indicate that the addition of either form of the chloride in any amounts tested increases the strength at all ages.

An interesting variation of this same data is presented in Figures 13 and 14. These graphs readily show that there is an increase in strength up to the addition of 2 or 3 percent calcium chloride, and then (with the exception of the one day test on Peladow) a decrease in strength as 4 percent is added.

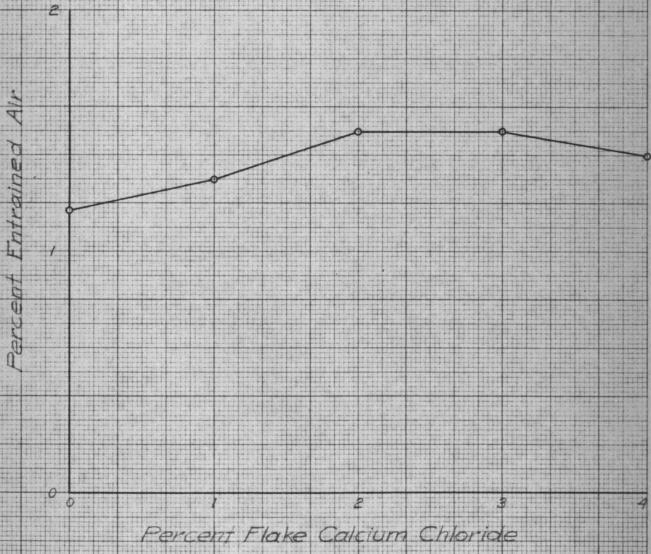

Fig. 9. Effect of flake calcium chloride on air content of freshly mixed concrete

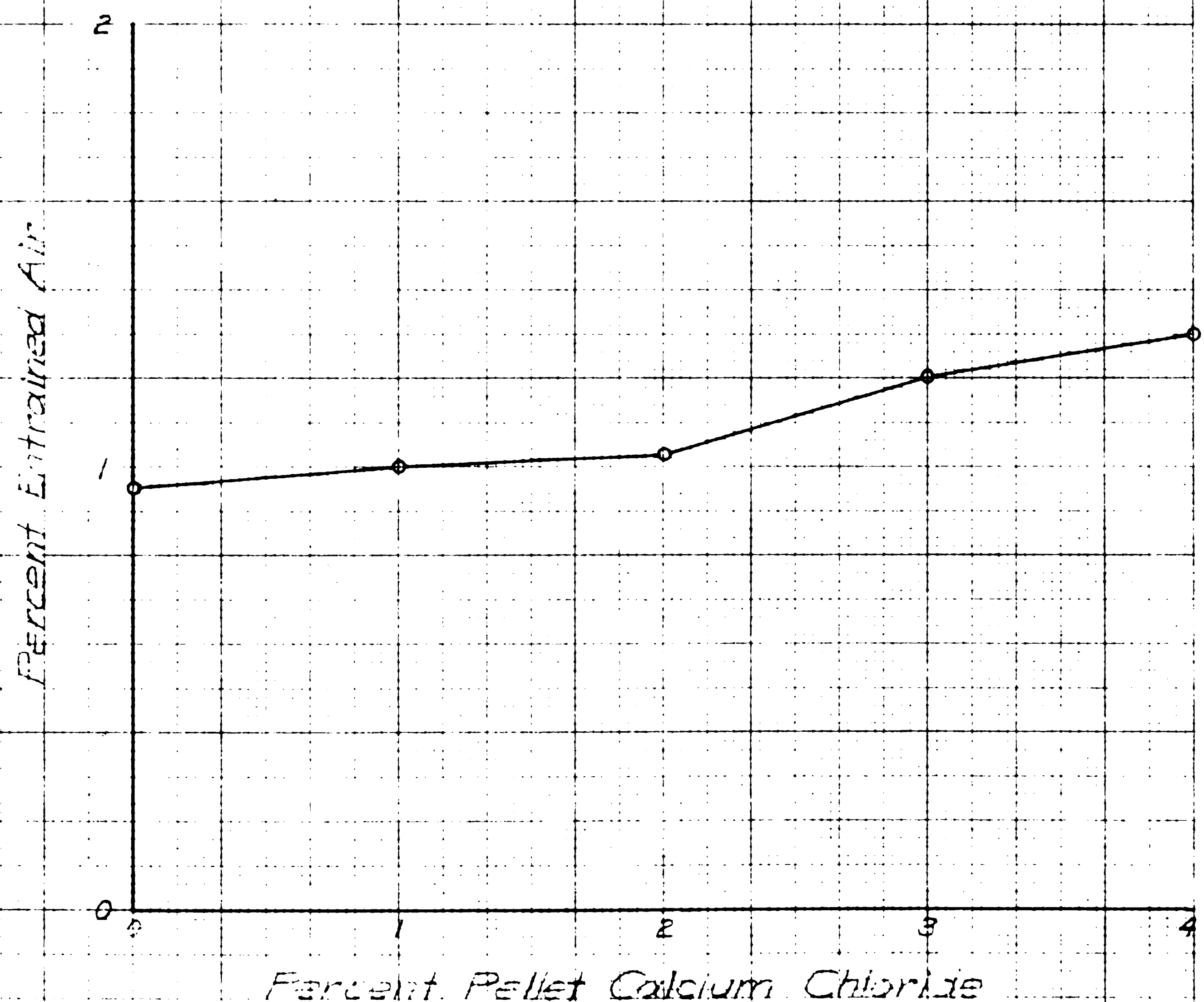

Fig. 10. Effect of pellet calcium chloride on air content of freshly mixed concrete

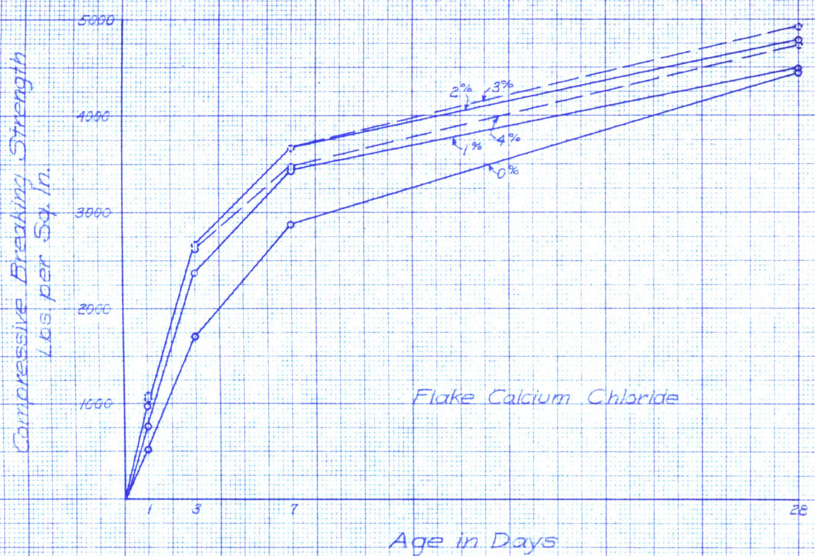

Fig. 11. Strength with particular percent flake calcium chloride versus age

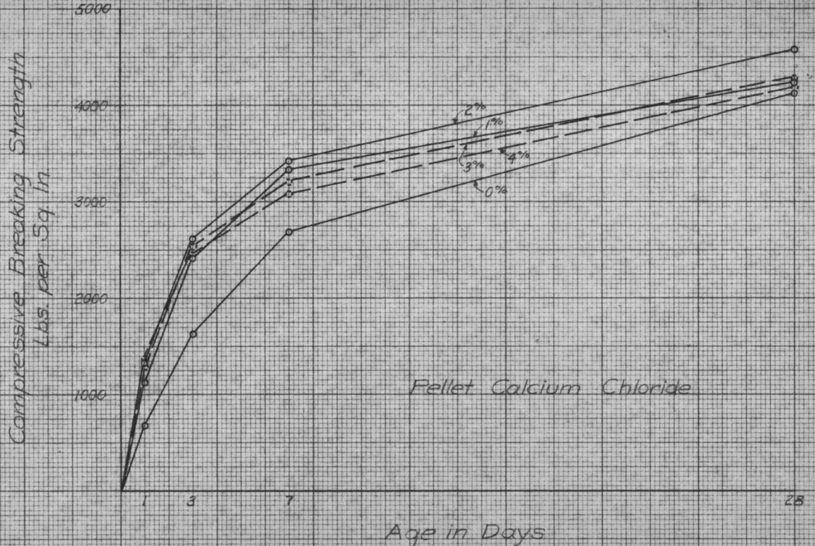

Fig. 12. Strength with particular percent pellet calcium chloride versus age

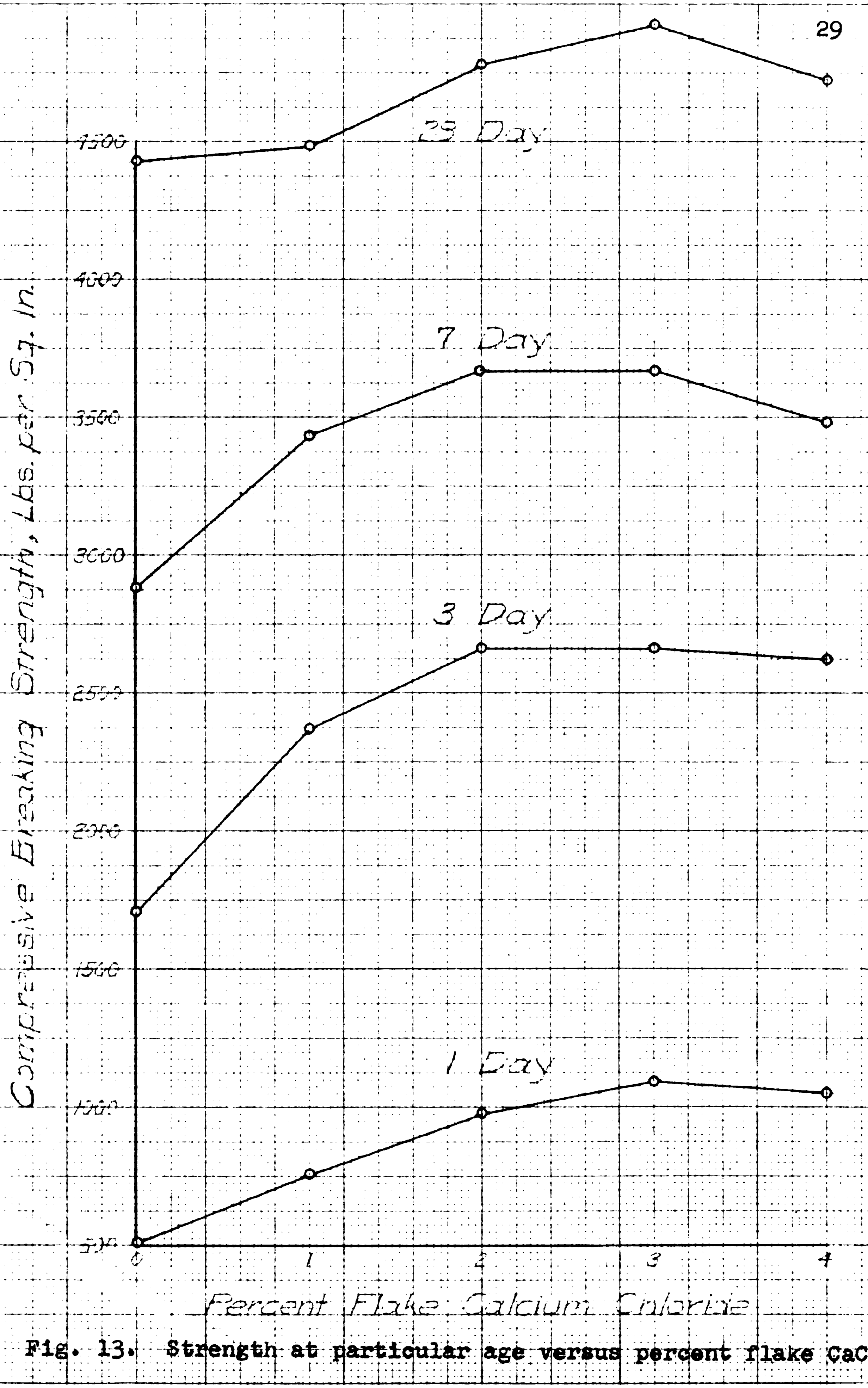

Fig. 13. Strength at particular age versus percent flake $CaCl_2$

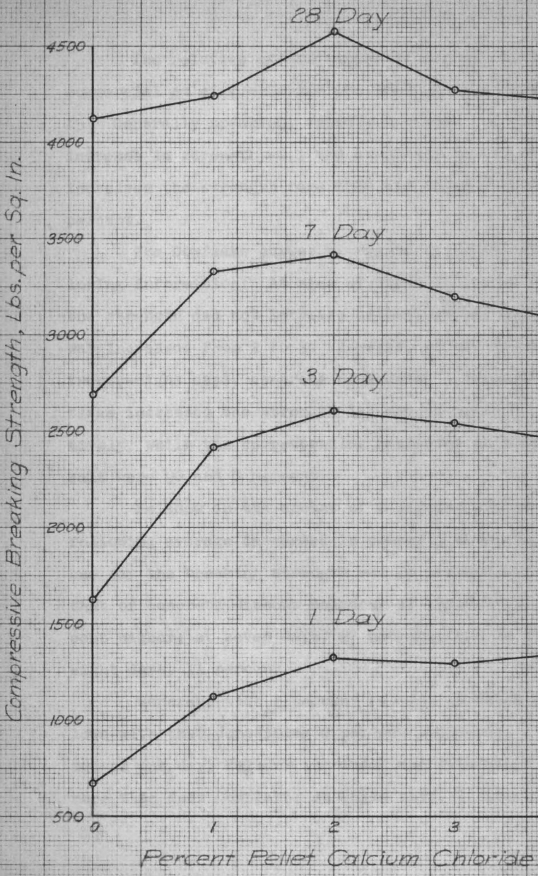

Fig. 14. Strength at particular age versus percent pellet $CaCl_2$

The relative effectiveness of the two forms of commercial calcium chloride can best be seen from Figure 15. Generally speaking, Peladow provided the greater increase in strength at 1 and 2 percent, while Dowflake increased the strength more than did Peladow at 3 and 4 percent.

The Dowflake curves of Figure 15 reveal the following information: At ages of three and seven days, the strength did not increase as the percent chloride was increased from 2 to 3. However, at one day and twenty-eight days, concrete containing 3 percent Dowflake increased the strength 113 and 11 percent, respectively, while the corresponding increases at 2 percent salt were 90 and 8 percent.

Peladow in the amount of 2 percent provided the following average increases in concrete strength: At one day the breaking strength was 96 percent higher than that of concrete without Peladow; at three days, 60 percent higher; at seven days, 27 percent; and at twenty-eight days, 11 percent.

Concrete with 2 percent Peladow compares as follows with that containing 2 percent Dowflake: At the age of one day, Peladow increases the strength 6 percent more than does Dowflake; at three days, 4 percent more;

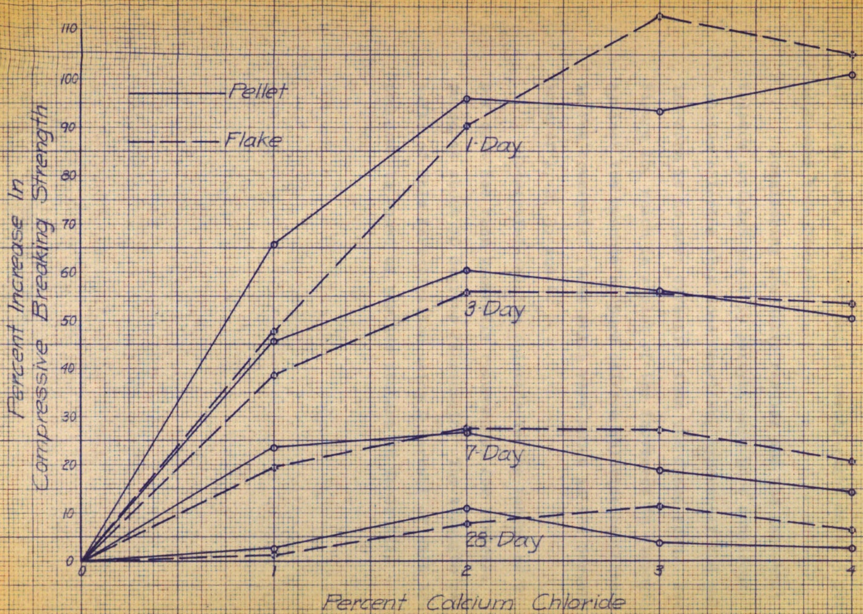

Fig. 15. Percent increase in strength at particular age versus percent flake and pellet calcium chloride

at seven days, no more; and at twenty-eight days, 3 percent more than Dowflake.

Considering the above results and the fact that Peladow contains about 19 percent more pure anhydrous calcium chloride than does Dowflake, apparently the pellet form is no more effective than the flake form. See Figure 16, which is a plot of the increases in strength against the amounts of pure calcium chloride contained in the commercial forms which were added in the percentages of 1, 2, 3, and 4.

Commercial calcium chloride in the amount of 2 per cent has been generally recommended for concrete cured at about 70 degrees Fahrenheit. The strength results of this investigation using 2 percent of salt are compared to those of earlier studies in Table III.

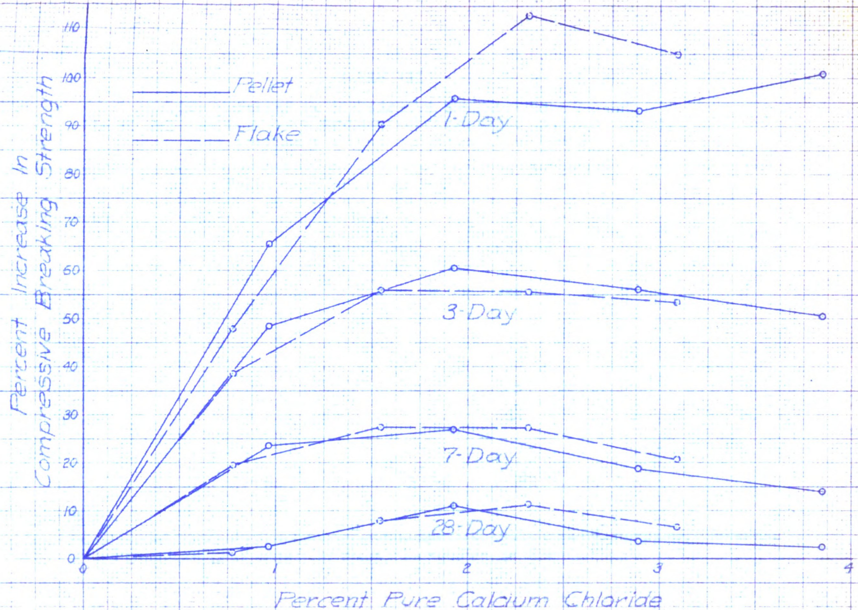

Fig. 16. Percent increase in strength at particular age versus percent pure calcium chloride in flake and pellet forms

TABLE III

PERCENT INCREASE IN COMPRESSIVE STRENGTH OF CONCRETE DUE TO THE ADDITION OF 2 PERCENT OF COMMERCIAL CALCIUM CHLORIDE
(All specimens stored in moist atmosphere)

Age at Test	Tests made prior to 1934*			This test (1951)	
	Portland Cement Assn.	District of Columbia Highway Dept.	National Bureau of Standards	Dowflake	Peladow
1 day		80	114	90	96
2 days	48	52			
3 days		46	62	56	60
7 days	25	21	29	27	27
28 days	7		14	8	11
90 days	7		7		
1 year	12		8		
3 years	9				

* Information from paper by Rapp, reference number 4.

V. CONCLUSIONS

A. Slump--A Measure of Consistency

The addition of certain amounts of either form of calcium chloride had a beneficial effect upon the workability of the concrete.

The maximum average increase in slump for the six Dowflake series occurred with concrete containing 4 percent of the salt. This increase in slump over concrete containing no calcium chloride was about 40 percent.

Apparently, the optimum amount of Peladow was 2 percent. This amount increased the average slump practically 30 percent.

B. Air Content

With the addition of either form of calcium chloride, a small increase in the air content was noted. For both forms, the maximum change in percent of entrained air was about three-tenths.

C. Compressive Breaking Strength

The results seem to justify the following conclusions:

(1) Additions of either form of calcium chloride in any percentage (1 to 4) increased the strength of the

concrete at all ages (one day to twenty-eight days).

(2) The effect of calcium chloride was greatest at one day and least at twenty-eight days.

(3) With one exception, the addition of from 2 to 3 percent of commercial calcium chloride provided the greatest increase in strength at all ages tested. The exception was that at the age of one day concrete containing 4 percent of Peladow provided the greatest increase.

(4) The optimum amount of flake calcium chloride was 3 percent (although 2 percent would be satisfactory). The average breaking strength at one day was more than doubled by the use of 3 percent of Dowflake.

(5) The optimum amount of pellet calcium chloride was definitely 2 percent. This amount increased the breaking strength at one day by approximately 96 percent.

(6) Peladow was equal to or slightly superior to Dowflake.

(7) On the basis of pure anhydrous calcium chloride, one form was about as effective as the other form.

(8) Since the actions of the two forms of calcium chloride were so similar, the economy of manufacture and distribution should be considered in the choice of one form over the other.

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