Effectiveness of Corrosion Inhibitors and Their Influence on the Physical Properties of Portland Cement Mortars

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This paper presents the results of laboratory studies using some of the various chemical corrosive admixtures in order to determine their action on physical properties of cement mortar mixes. The compression and splitting tensile strength tests as well as the workability of these mixes were used to determine the influence of the chemical additives on the cement mortar mixes. The ability of these chemical admixtures to prevent corrosion in reinforced concrete was investigated by 2 methods: anodic polarization and visual tests. In these tests, small reinforcing bars were embedded in cement mortar and examined at a later date. Calcium chloride was added in varying amounts along with rust inhibitors to observe their effect on the physical and corrosive protection properties.

•THE PROBLEM OF CORROSION in reinforced concrete has been given considerable attention in the past few years. There have been cases of corrosion that have led to expensive maintenance and repair. The California Division of Highways has had specific problems with bridges near a marine environment. The San Mateo-Hayward Bridge is a good example of corrosion of reinforcing steel in a marine environment. The stirrups in some places have completely disintegrated. There is spalling of the concrete because of the action of corrosion. Where there are cracks in the concrete, rust stains appear on the concrete surface. There have been similar deteriorations in structures located in Florida (1), Texas, Malaya, and South Africa (9).

The presence of calcium chloride has also caused severe corrosion in reinforced concrete, especially under steam-cured conditions. The use of chloride salts as deicers in the northern states has caused deterioration in bridge decks. The salts have penetrated the protective coatings of some bridges and caused corrosion of the reinforcing steel.

The review of literature (3, 15 through 34), which was limited to the addition of inhibitors to portland cement as protective measures, yielded a list of chemicals that in previous studies gave indication of being effective in reducing corrosion attack. The recommended compound groups were chromates, phosphates, hypophosphites, alkalies, and fluorides. The chemicals used in past studies that showed good results in resisting corrosion in reinforced concrete were barium, strontium, lead, and potassium chromates; calcium and silicon fluorides; sodium nitrite; sodium benzoate and sodium metasilicate; ethyl aniline and calcium lignosulfonate; aluminum acetate; chrome oxide; chrome carbides; chlorate; thirourea; and calgon and mercaptobenzothiazole. The review of literature indicated a lack of knowledge concerning the effects of these chemical inhibitors on the physical properties of the mortar or concrete. From this list of chemicals, sodium nitrite, sodium benzoate, and potassium chromate were chosen for this study.

MATERIALS

The basic mortar mix used in this study consisted of 3 different components: cement, Ottawa sand, and water. The corrosion inhibiting chemicals that were added to the

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basic mortar mix in varying proportions were selected on the basis of past work by researchers.

Cement

A type 1 normal portland cement that came from the same clinker batch was used in this investigation. This precaution was to ensure that the cement had the same chemical composition. Thus a difference in the physical properties could not be related to a difference in the chemical composition of the cement. The chemical and physical properties of the cement used were not investigated.

Chemicals

The selection of chemicals that serve as rust inhibitors had to be carefully made. The chemical inhibitors used in this study must act as rust inhibitors in cement paste, must cover the different types of inhibitors that can be used, and must be practical and easy to use in field operations. The selection was based on work that had been performed by other investigators studying the effectiveness of chemical rust inhibitors. The chemicals selected for this investigation were as follows:

1. Sodium nitrite. This chemical is one in the nitrite salt group that acts as an anodic rust inhibitor. It has been recommended by many researchers as a very effective chemical rust inhibitor. Sodium nitrite showed passivation at low concentrations. These concentrations were approximately 1 percent by weight of cement.
2. Potassium chromate. This chemical represents the chromates, the other main type of salt, that are effective as anodic rust inhibitors. The effective concentration of this salt is approximately twice that of the sodium nitrite.
3. Sodium benzoate. This chemical, which is one in the anodic inhibitor group, required large concentrations to have a beneficial effect in passivation.
4. Calcium chloride. This salt is a very widely used accelerator that is added tc concrete. It has been found by some investigators to cause corrosion of reinforcing steel in concrete. This chemical was added in varying amounts with the rust inhibitors, and its effect on the physical properties of the paste was observed.

Aggregates

To ensure that the aggregate introduced no variation in the results, standard grade natural silica sand from Ottawa, Illinois, that met ASTM Specification C 109 was used. This sand was used in both the compressive and splitting tensile mortar specimens.

Water

The water used in the cement mortar mix was distilled water obtained from the Sanitary Engineering Laboratories. The temperature of the mixing water was kept between 68 and 72 F. The amount of water put into a mortar mix was kept constant. The chemicals were dissolved in the water before the proper amounts were measured.

OUTLINE OF INVESTIGATION

The object of this study was to determine the influence of certain chemical rust inhibitors on some physical properties of cement mortar mixes that had varying amounts of calcium chloride present. These properties were workability, compressive strength, and splitting tensile strength. Also under investigation was the effectiveness of the admixtures to serve as chemical rust inhibitors. There has been some concern recently with the corrosion of steel in reinforced concrete. Investigations have been directed toward finding different chemical admixtures that will prevent corrosion, but there has been very little effort expended on what would happen to the properties of the hardened paste when these admixtures are added.

In the field, the presence of calcium chloride at times has proved to be a cause in corrosion of reinforcing steel in concrete. Because this chemical is widely used in field practice, various percentages were used in this investigation.

To obtain a true picture of the behavior of the strength properties and workability, an experimental model was set up. The amounts and types of chemicals, materials, procedures, and apparatus were decided. The order in which the specimens were to be cast and tested was developed in a somewhat random procedure. A random mathematical table was used to decrease the day-to-day personal differences in the procedure. The specimens that were to be cast were divided into 3 series. These series, based on the amount of time for curing, were 3-day, 7-day, and 28-day tests. In each series, different combinations of chemical corrosive admixtures were prepared. These 30 different combinations were picked in a random manner from a table of random numbers. Because of the time element in preparing the specimens, this was the extent of randomization of specimens. In each of these 30 combinations, 6 specimens were made: 3 compressive strength specimens and 3 splitting tensile strength specimens. During each casting, 6 different combinations could be made.

It was desirable to evaluate workability of the various combinations because of the necessity of knowing whether the various mixtures could be placed in the field. The flow table was used for measuring the workability of the mixes. This method seemed to give a good indication of this parameter.

There has been some difference of opinion on whether chemical rust inhibitor admixtures produced a passivation of the steel. These opinions were (a) that chemical rust inhibitors would prevent corrosion, (b) that the effectiveness of the inhibitors would be lost after a few years because of leaching, and (c) that the presence of the inhibitors would have no effect. The effectiveness of the 3 chemicals for passivation of the reinforcing steel was investigated by 2 different methods: anodic polarization and visual observation of the piece of reinforcing steel in the split specimen. The ages at which these were to be tested were 4 days for the anodic polarization and 270 days for the visual observations.

APPARATUS AND PROCEDURE

Preparation of Mortar Mixtures

The cement mortar mix proportions remained constant throughout this study. The mix proportions of water, cement, and aggregate were 1:2:5.5 by weight. This gave a good workable mix for all of the chemicals used in this study. The aggregate-cement ratio was taken from ASTM Specification C 109. The amount of water was determined on a trial-and-error basis by flow table measurements prescribed by ASTM Designation C 230. The flow for a nil batch was appromixately 80 percent. The chemicals were added to the mixing water before the water was measured.

The quantities of water, cement, and aggregate were measured to the nearest gram. The chemicals added to the mix were measured to the nearest tenth of a gram. Each batch-day consisted of the making of 36 specimens. That was 6 combinations of 3 compressive and 3 tensile strength specimens. The mixing and casting room temperature was controlled at 68 to 72 F. The relative humidity fluctuated from 50 to 70 percent.

The mechanical mixer used was a Hobart model with a 250 cu in. capacity that conformed to ASTM Designation C 305-64T. The mixing procedure that was adopted for all specimens was the one prescribed in this specification.

Each mix batch contained enough for all compressive and splitting tensile specimens for that particular combination in its series. Flow table measurements were taken immediately after mixing. These measurements were a means for determining the consistency of the mixes; they were also used as a means for determining the workability of the mixes. The flow table conformed to ASTM Designation C 230-61T.

Casting and Curing of Specimens

After it was mixed, the mortar was then cast into molds. The molds used for the compressive strength specimens conformed to the standard 2-in. cube molds of ASTM Specification C 109. The splitting tensile test specimens were formed in 2-in. diameter by 4-in. high cylindrical molds. The placing and compacting were accomplished according to ASTM Specification C 109.

The specimens used in the study of the anodic polarization effectiveness were mixed in the same manner as the compression and splitting tensile specimens. The pieces of steel that were set in the mortar mix had 2 paraffin coats that were placed 2 in. apart to set up a known area of bar surface exposed to the cement mortar. After 24 hours, the specimens were stripped of the paper cup molds and divided into 3 groups. Each group consisted of 1 specimen of each combination as previously described. One group was left exposed to the air at 72 F and 50 to 70 percent relative humidity. The other 2 groups were placed in tap water. After 9 months, one group that was in the water was then subjected to a saltwater bath. The other 2 groups were tested at this time.

Compressive and Splitting Tensile Tests

The compressive and splitting tensile strength measurements were made according to ASTM Specification C 109. Careful alignments were made in placing the specimens in the correct position under the floating head of the testing machine. For the splitting tensile specimens, ¼- by 1- by 6-in. pressed masonite was used for the bearing strips.

The compressive strengths were determined by

$$\sigma_{comp} = \sum \frac{P}{A}$$

where

σ_{comp} = compressive strength, psi;
P = sum of failure loads of the specific combination; and
A = sum of the cross-sectional areas of the specimens, in^2.

The splitting tensile strengths were determined by

$$T = \sum \frac{2P}{ldn}$$

where

T = splitting tensile strength, psi;
P = sum of the failure loads of a specific combination;
l = length, in.;
d = diameter, in.; and
n = number of specimens.

Effectiveness Studies of the Admixtures in Mortar Mix

The specimens were placed in water saturated with calcium hydroxide. An external voltage was applied to the steel reinforcing bar that was in the specimen and was acting as an anode and to a platinum electrode that was acting as a cathode. The potential of the steel was measured relative to a saturated calomel electrode by means of a vacuum tube voltmeter. This apparatus was used for the specimens that were 7 days old.

The anodic effectiveness of the rust inhibitors was performed by measuring the steel potential as a function of the polarization current density per unit area of anode. Definite current values were established between the steel anode and the platinum cathode, starting with the open circuit or zero current value, and the resulting potentials of the anode relative to the calomel electrode were measured. After a potential measurement, the current was increased to the next definite value, and the potential was read again after 5 minutes. This procedure was continued for 7 to 10 five-minute intervals. This procedure is shown in Figure 1 and is the same procedure that was used in the Portland Cement Association study (15).

A visual test was performed on the remaining specimens to determine the effectiveness of the various chemical additives in retarding corrosive attack after 9 months

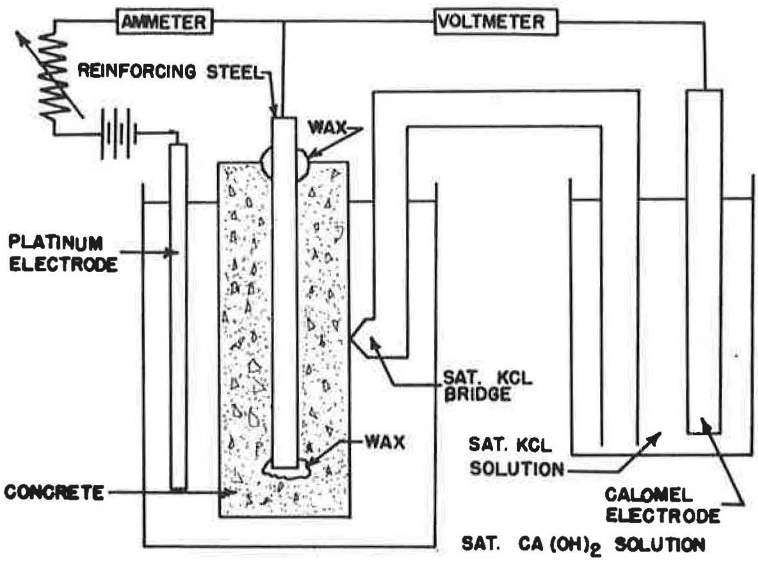

Figure 1. Circuit diagram for polarization measurements (15).

curing. The specimens containing the reinforcing steel were split longitudinally, and the effectiveness was measured by the percentage of corrosion and the severity of attack.

$$\text{Percentage of corrosion} = \frac{A_c}{A_t}$$

where

A_c = corrosive surface on the reinforcing embedded in concrete; and
A_t = total area embedded in concrete (between 2 wax spots).

The corrosion severity was rated from 1 to 5 as follows:

Condition	Rating
Very thin film	1
Thick film	2
Small pitting	3
Medium pitting	4
Severe pitting	5

DISCUSSION OF TEST RESULTS

Physical Properties Observed During Testing

The chemicals that were utilized in this study produced significant physical changes when added to the mortar mixes. The chemical admixtures when mixed with the mortar gave the surface of the specimens different colored appearances. These color changes were not uniform across the surface. The potassium chromate actually gave the mix a light green cast. Most of the other salts produced a whitish color on the surface. While the specimens were kept under controlled curing conditions, the aqueous salt solutions leached to the outer surface of the specimens and formed a thin scum around t outside.

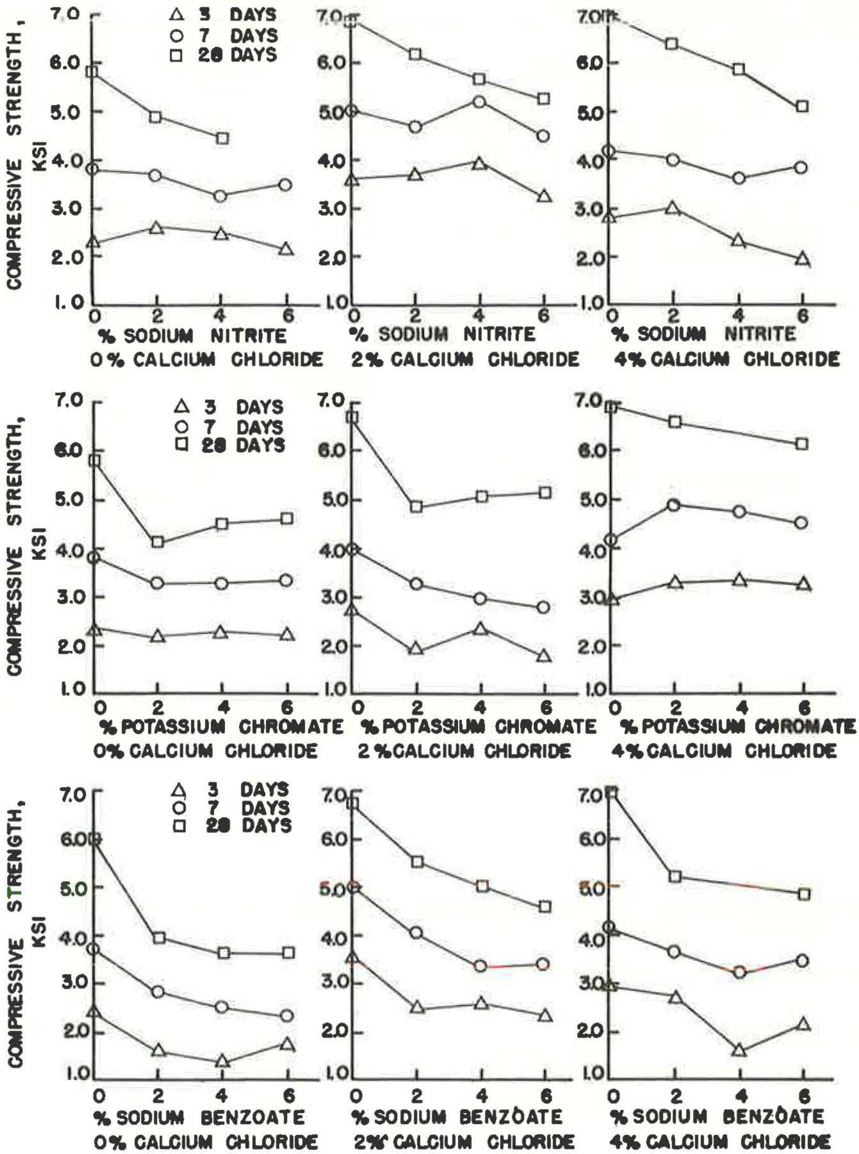

Figure 2. Influence of varying amounts of chemical corrosion inhibitors with specific amounts of calcium chloride on compressive strength of standard mortar cubes.

Compression Strength Test Results

The compressive strength results are shown in Figure 2. Because of the number of combinations and amounts of additives used in this study, compression strength test results will be discussed according to the varying amounts of calcium chloride.

For 0 percent calcium chloride, very little change in compressive strength of mortars was observed at 3 and 7 days with increasing amounts of sodium nitrite and potassium chromate. Increasing amounts of sodium benzoate showed some decrease in compressive strength at 3 and 7 days. A marked decrease in strength at 28 days for increasing amounts of chemicals was observed. The maximum decrease of 28 day

for potassium chromate, sodium nitrite, and sodium benzoate were approximately 33, 20, and 40 percent respectively.

The second set of mixes was based on 2 percent calcium chloride. The potassium chromate addition decreased the compressive strengths approximately 20 percent for all concentrations. As the amounts of sodium nitrite were increased, compressive strength for the various mixes decreased in a somewhat linear fashion to a maximum value of 25 percent for the 28-day strength. Increasing amounts of sodium benzoate decreased the compressive strength again in a linear fashion to a maximum value of 35 percent for the 28-day strength.

The third set of mixes was based on 4 percent calcium chloride. Increasing amounts of potassium chromate and sodium nitrite both gave a linear decrease in compressive strength amounting to a maximum of 20 percent and 30 percent respectively for the 28-day strengths. Increasing amounts of sodium benzoate resulted in strength decreases but did not follow a linear pattern. The maximum reduction in strength was 35 percent at 28 days.

Splitting Tensile Strength Test Results

The splitting tensile strength results, shown in Figure 3, varied with the type of rust inhibitor employed. Both sodium nitrite and sodium benzoate produced a decrease in

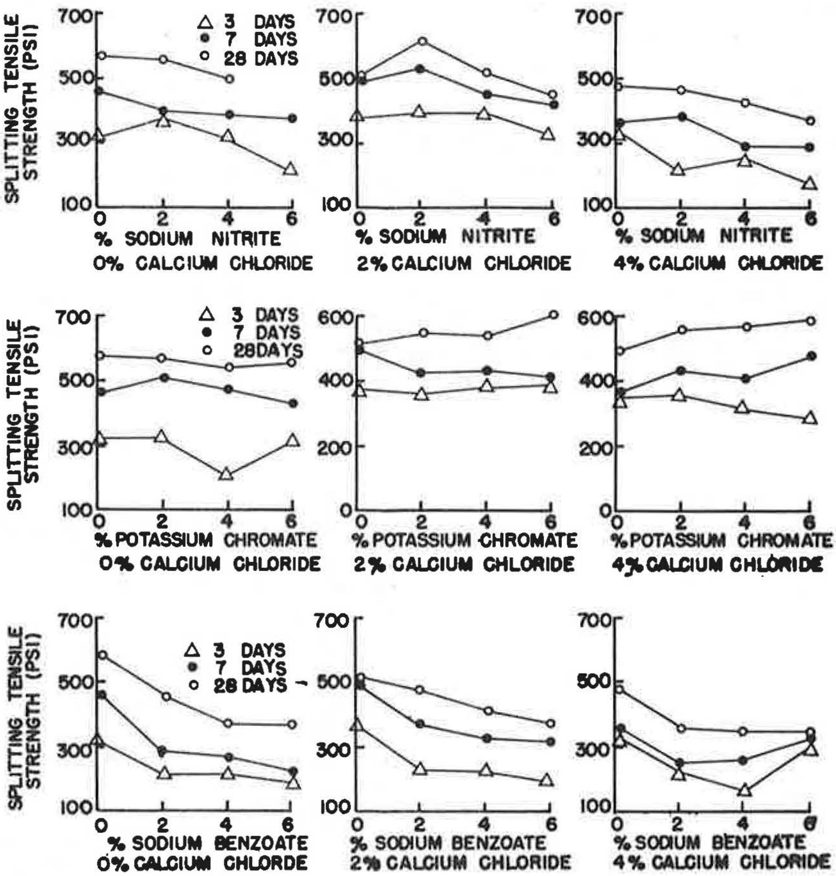

Figure 3. Influence of varying amounts of chemical corrosion inhibitors with specific amounts of calcium chloride on splitting tensile strength of mortar cylinders.

TABLE 1

VISUAL OBSERVATIONS OF REINFORCED MORTAR SPECIMENS FOR ABILITY OF MIXTURES TO PREVENT CORROSION

CaCl (percent)	Chemical		Corrosion—Percentage Exposed Surface						Rating
	Compound	Percent	0	20	40	60	80	100	
0			o -						—
2				o -					4
4					o -				24
0	$NaNO_2$	2	o -						—
0	$NaNO_2$	4	o -						—
2	$NaNO_2$	2	o -						—
2	$NaNO_2$	4	o -						—
0	$Na_2C_7H_5O_2$	4	o -						—
0	$Na_2C_7H_5O_2$	6	o -						—
2	$Na_2C_7H_5O_2$	4			o			-	52
2	$Na_2C_7H_5O_2$	6			-			o	5
0	K_2CrO_4	2	o -						—
0	K_2CrO_4	4	o -						—
2	K_2CrO_4	2			o -				5
2	K_2CrO_4	4		-	o				15

Note: o = specimens cured in air; - = specimens cured in water.

the splitting tensile strength with an increase in the amount of rust inhibitor. This held true for the 0, 2, and 4 percent calcium chloride groups. The maximum reduction was between 20 and 30 percent.

For the case of increasing amounts of potassium chromate with 0 percent calcium chloride, very little change in splitting tensile strength was observed at 3, 7, and 28 days. For increasing amounts of potassium chromate with 2 percent calcium chloride, very little change in strength was observed for 3 and 7 days. At 28 days a slight increase in splitting tensile strength was obtained. With 4 percent calcium chloride, . creasing amounts of potassium chromate showed very little change in strength at 3 days, but showed an increase in strength at 7 and 28 days.

Effectiveness of Inhibitors in Reducing Corrosion

In the potential current measurements for the cement mortar mixes, passivity was indicated by a sudden sharp rise in potential at low current densities. The use of the anodic inhibitors, sodium benzoate and potassium chromate, resulted in passivity for

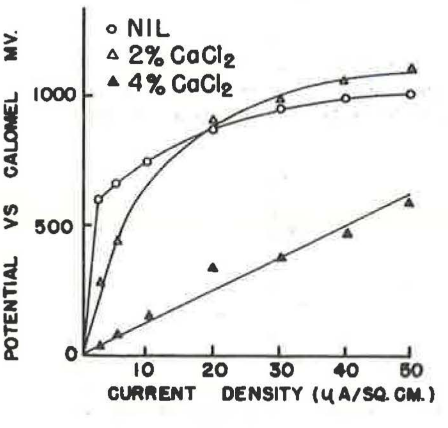

Figure 4. Effect of calcium chloride on anodic polarization of steel in reinforced concrete.

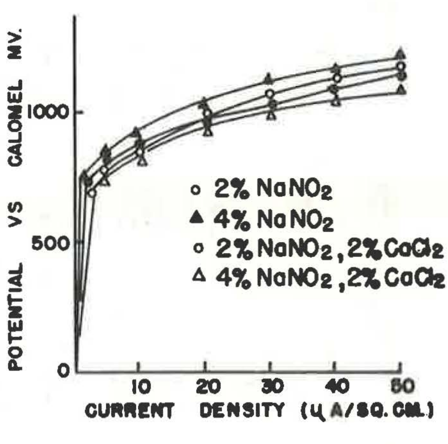

Figure 5. Effect of sodium nitrite on anodic polarization of steel in reinforced concrete.

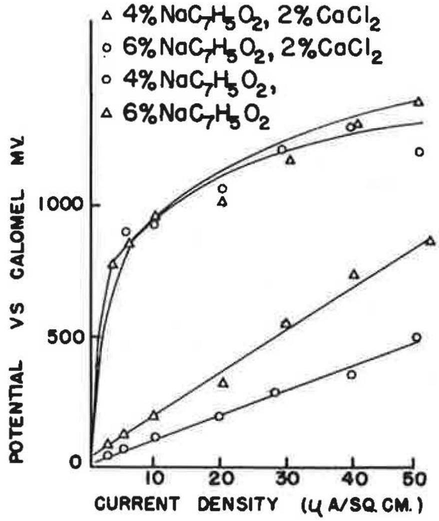

Figure 6. Effect of sodium benzoate on anodic polarization of steel in reinforced concrete.

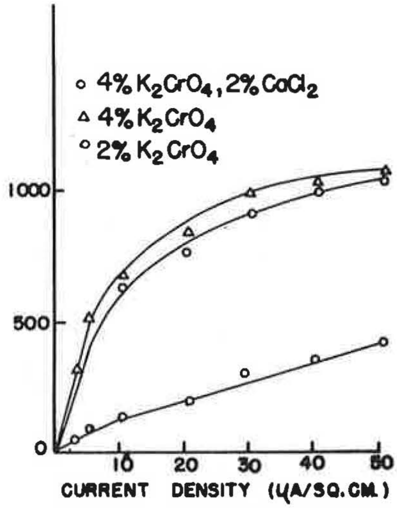

Figure 7. Effect of potassium chromate on anodic polarization of steel in reinforced concrete.

specimens with no calcium chloride in the mix. These same anodic inhibitors displayed no passivity with 2 percent calcium chloride. The sodium nitrite gave indications of being the only anodic inhibitor studied to have passivity when combined with calcium chloride in the mix. The 0 and 2 percent calcium chloride when combined with sodium nitrite gave indications of being passive (Figs. 4, 5, 6, and 7).

When split and examined visually, specimens that were exposed in air and water for 10 months gave similar results to those of the anodic polarization study. From the data given in Table 1, all specimens with 0 percent calcium chloride showed no corrosive action on the reinforcing bar. The only chemical rust inhibitor that showed passivation with calcium chloride was the sodium nitrite inhibitor. The remaining chemical rust inhibitors and calcium chloride showed 10 to 40 percent corrosion area except for the sodium benzoate, which showed 40 to 100 percent. The nil specimens as predicted showed no corrosion because of the pH of the mortar mix. As the percentage of calcium chloride increased in the specimens, the corrosive surface area increased. Similar results were observed for both the air- and water-cured specimens.

Workability of Mixes

The flow table measurements, although a measure of consistency of the mixes, will be used to present a relative picture of the effect of the various salts on the workability. These measurements seemed to give a good indication of the amount of work necessary to place the mortar in the molds (Fig. 8).

The calcium chloride mortar mixes gave a maximum flow of 93 percent at 2 percent calcium chloride and an identical flow of 74 percent at 0 and 4 percent calcium chloride.

The maximum increase in flow was 10 to 25 percent and occurred at 2 percent salt additions. Increased amounts of anodic salts decreased the flow value of the resulting mixes from these maximum values.

The combined mixes of 2 percent calcium chloride and anodic salts decreased the flow of the 2 percent calcium chloride. The flow values for the different salts remain almost constant for the different concentrations of salts except for the 2 percent sodium benzoate whose flow was approximately the same as that of the 2 percent calcium chloride.

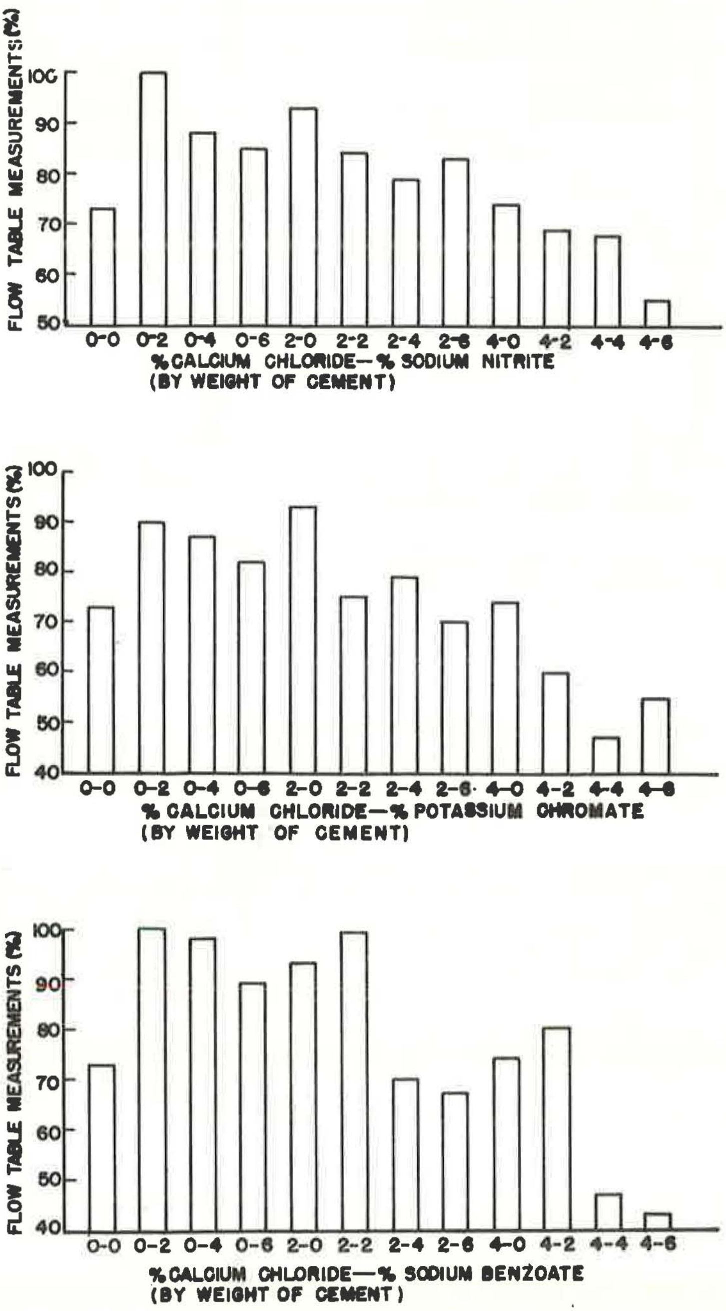

Figure 8. Influence of varying amounts of chemical corrosion inhibitors and calcium chloride on flow table measurements of mortar mix.

The 4 percent calcium chloride and anodic salt combinations behaved somewhat in the same manner as the 2 percent calcium chloride and anodic salt combinations. The flows were all decreased for the 4 percent calcium chloride sets except for the 2 percent sodium benzoate.

CONCLUSIONS

The results of this investigation showed no general trend for all chemical corrosion reducing admixtures under investigation in this study. The results of each individual admixture were presented in the foregoing sections, and only general conclusions will be drawn in this section. Based on this stipulation, the following statements concerning the effects of the various admixtures on the physical properties of portland cement mortars are presented.

1. Sodium nitrite was found to be the only effective chemical reducing admixture of the three that were under investigation.
2. The 28-day compressive strength of mortar mixes is decreased when the anodic rust inhibitors were added to the mix.
3. The presence of the chemical rust inhibitors in the cement mortar mixes increased the fluidity of these mixes. The maximum increase in fluidity occurred with the additions of 2 percent of the inhibitors.
4. The addition of calcium chloride to the cement mortar mixes with the chemical corrosion reducing admixtures decreased the gain in the fluidity obtained when the inhibitors were used by themselves.
5. Calcium chloride present in a cement mortar mix creates a corrosive environment for the reinforcing bar.
6. The mode of corrosion of the reinforcing steel rated from mild to severe pitting as opposed to the desired thin film corrosion when the various chemical admixtures were present in the cement mortar mix.

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