

Surface Popouts Caused by Alkali-Aggregate Reaction

by Robert Landgren and David W. Hadley



R E S E A R C H & D E V E L O P M E N T B U L L E T I N R D 1 2 1

Abstract: Studies of an alkali-aggregate reaction which causes small popouts in the surfaces of hard-troweled slabs are described. Silica-rich shale particles present in certain Midwest sands are involved in the reactions. Alkali contributed by the cement is the other reactant.

The popouts are a surface phenomenon and their severity appears to be a function of the alkali content at the slab surface. Alkali content at the surface depends on the portion of the cement alkali that is readily soluble, and the amount of alkali that is brought to the surface during drying of the concrete. Migration of alkali during drying can increase the alkali concentration of the surface layer as much as 10-fold. Alkali migration towards the surface of a concrete slab is increased during placing and curing by such factors as high temperature, high wind velocity, low humidity, thick slabs, and long periods of drying during finishing and the first stages of curing.

Inappropriate curing procedures aggravate popouts. Preliminary studies indicate that thorough curing of concrete, using excess water, can completely eliminate popouts from occurring in the surfaces of slabs cast with high-alkali cement and cured at 38°C (100°F). Recommended curing procedures are the ponding of slabs or the use of wet burlap, soil, or sand.

Other approaches to solve the problem, such as the use of chemical admixtures or pre-reaction of deleterious aggregate particles, are also discussed.

Keywords: Alkali-aggregate reactions, alkali content; climate, concrete durability, curing, discoloration, drying, durability, fly ash, moist curing, popouts, sand (material), shales, surface finishes (fresh concrete).

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EDITOR'S FOREWORD

This report was drafted in July 1968 as part of the authors' activities in the Research and Development Laboratories of the Portland Cement Association. For unknown reasons, the report was never completed, but draft copies have been found in various archives. Finalization of this report was undertaken 32 years later to make the information available to the concrete industry. The research remains sound.

Between 1968 and 2002, many advancements in publication technology occurred. The draft manuscripts were typewritten in 1968; the current document was scanned from those documents, reformatted, and submitted for review by concrete technologists. During these reviews, it was noted that editorial revisions were needed. These modifications are not intended to change the original meaning, but rather to improve readability of the document. The authors themselves could not approve the changes, so it seemed appropriate to inform the reader that their words had been edited.

Likewise the layout and formatting of the document has been revised to fit modern conventions. In no case was data altered or conclusions redrawn. In addition, the original work was done in US customary units; these units have been retained in parentheses, but SI units (converted) have been made primary.

Popouts still occur occasionally in concrete. By offering recommendations for minimizing popout potential, it is hoped that publication of this report will serve to improve the quality and visual appearance of concrete.

Paul D. Tennis
July 2002

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Portland Cement Association

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Cover Photo: ASR-induced popouts on a slab (not a specimen of research detailed in this report) (51117).

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TABLE OF CONTENTS

Introduction	1
Nature of the Deleterious Shale Particles	1
Preliminary Investigation of the Mechanism of Popout Formation	1
Theoretical Considerations Affecting the Formation of Popouts or Discoloration	3
Scope of Concrete Investigations	4
Materials and Test Procedures	4
Fine Aggregates	4
Coarse Aggregate	4
Portland Cement	4
Admixtures	4
Concrete Mixes	4
Finishing	7
Curing	7
Test Results and Discussion	7
Effect of Variation in Cement-Alkali Content	7
Alkali Migration and the Effect of the Total Alkali Content of the Concrete	7
Effect of Severe Temperature and Humidity Conditions	7
Effect of Slab Thickness	7
Effect of Drying Prior to Final Finishing	9
Effect of Cement Content	9
Effect of Finishing Techniques	9
Effect of Air Entrainment	9
Effect of Curing Procedures	9
Evaluation of Possible Remedial Measures	11
Summary	12
Recommendations	12
Acknowledgements	13
References	13

INTRODUCTION

For a number of years, the use of local river sands in scattered locations in South Dakota, Iowa, and Minnesota has resulted in a spotty surface discoloration or, more commonly, numerous small popouts, with a maximum diameter of about 6 mm ($\frac{1}{4}$ inch) and depth of about 3 mm ($\frac{1}{8}$ inch), on concrete flatwork. Although the surface discoloration is of minor consequence, reportedly being readily "scuffed off" by foot traffic, the closely spaced popouts are of some concern to users of portland cement concrete in the affected areas.

These popouts, which are normally most abundant on hard-troweled surfaces, are extremely unusual in that they often appear within only a few hours after the concrete is finished. In most cases, popouts have been reported to occur within the first few weeks. Merrill, Handy, and Fox (1967) do, however, report instances of popout formation at later ages. These latter occurrences are apparently the result of sealing the slab surface with paint or tile-setting cement.

Field studies have shown that the popouts are caused by shale particles in the local sands. These sands are largely glacial or glacio-fluvial in origin and the shale particles are believed to be derived from the extensive cretaceous shales exposed in the northern great plains. Although sands of this origin are widely distributed throughout the Minnesota-Iowa-South Dakota region, concrete distress is normally confined to a relatively small number of locations. During the summer of 1966, which was unusually hot and dry, the problem appeared to be both more severe and significantly more widespread.

Field evidence indicates that in addition to the weather conditions during construction, the alkali content of the cement, finishing techniques, and curing techniques all influence the frequency and severity of popout formation.

The purposes of the research described in this report are as follows:

1. To determine the mechanism of popout formation,
2. To evaluate those factors believed to influence the formation of popouts, and
3. To develop and evaluate remedial techniques.

NATURE OF THE DELETERIOUS SHALE PARTICLES

Samples of sand with bad service records were obtained from a number of sources, and the shale particles in the sands were examined petrographically. All were found to consist of a predominantly montmorillonitic clay and to contain abundant opaline microfossils or fossil debris. Fig. 1 is a photomicrograph of a typical shale particle showing large numbers of opaline microfossils.

The shale particles were found to normally constitute less than 5% of the total sand by particle count and to be present in all of the sand sizes.

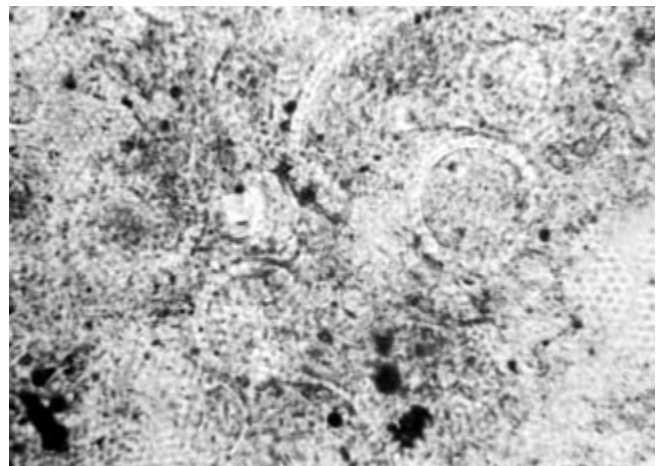


Figure 1. Photomicrograph of a thin section of a shale particle showing alkali reactive opaline micro-fossils (field width: approximately 0.5 mm).

PRELIMINARY INVESTIGATION OF THE MECHANISM OF POPOUT FORMATION

Since the shales had been found to be predominantly montmorillonitic, a possibility existed that the popouts could be attributed to the pressures generated by the absorption of water by the montmorillonite.

Samples of the shale were immersed in water, and in solutions of sodium hydroxide and sodium hydroxide plus calcium hydroxide. Shale particles were measured

with a microscope and stage micrometer. In no case was there any measurable expansion. As a further check, mortar specimens were prepared in which shale particles were incorporated in a matrix of plaster of Paris and then moist cured. No popouts were formed in this exposure. It does not appear, therefore, that swelling of the montmorillonitic clay is a significant factor in popout formation.

Since the particles had been found to contain large amounts of opaline silica, the probability that the popouts may be the result of alkali-silica reactivity was investigated.

Shale particles were hand picked from the aggregate and were imbedded in pats of neat cement paste either of a very high- or low-alkali content. The pats were cured in the moist room overnight. After 18 hours, numerous popouts had formed on the high-alkali pat while the low-alkali pat showed no deterioration. After several months additional moist cure, no popouts had formed on the low-alkali pat and no additional popouts had formed on the pat of high-alkali content. Petrographic examination of the popouts formed in the high-alkali pat showed the aggregate sockets to contain alkali-silica gel.

It would appear, therefore, that the popouts are the result of alkali-silica reactivity involving the opaline microfossils in the shale particles.

The extreme rapidity of popout formation, both in the laboratory tests and in the field concrete, is most probably the result of the extremely high surface area of the opal within the shale particles. Inspection of Fig. 1 will show that the opaline particles are not only very small, but are in many cases quite porous.

Shale particles of this type from the Watertown, South Dakota region were studied by Mather, Buck, and Luke (1964), who found definite evidence of alkali-silica reactivity. As early as 1940, shale popouts in California were attributed to alkali-silica reactivity by Stanton (1940).

The spotty surface discoloration, which is sometimes observed in the field, was found to be the result of exudations of a very fluid alkali-silica gel centered over the reacted shale particles. This gel, which is the reaction product between the opal in the shale and the cement alkalies, may appear on the concrete surface as early as one day after casting. Its passage to the surface often is accomplished without damage to the concrete.

Fig. 2 shows a view of the surface of a laboratory slab that was disfigured by alkali-silica reactions. Two popouts are visible in the picture: a large one at the top and a small one at the bottom right of the picture. Between the two popouts is a mass of hardened froth, which is the dried residue of a very fluid alkali-silica gel.

The spectacular popout on a laboratory slab in Fig. 3 is quite instructive. The top of what looks like a palm tree in the figure is the popout material. The trunk is a fiber of hardened silica gel that was extruded from a reactive aggregate particle. The gel causing this popout had to be quite viscous, since it maintained a reasonably constant

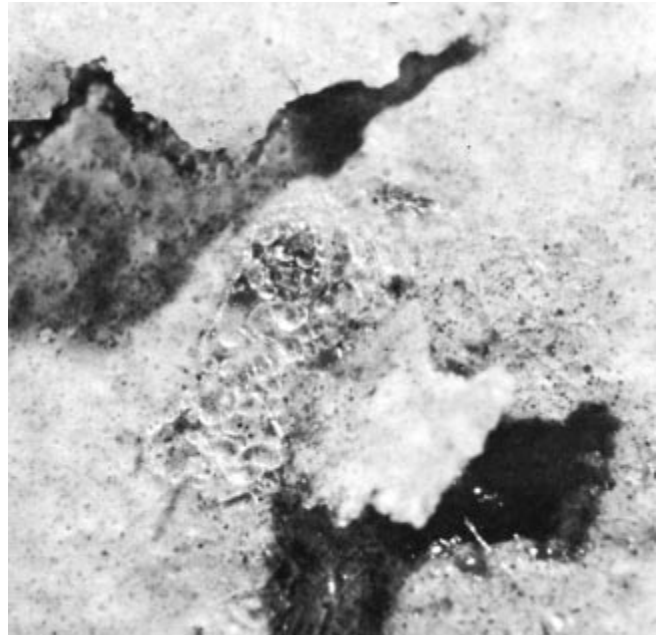


Figure 2. Dried silica gel between two popouts (field width: approximately 1 mm).



Figure 3. Two reaction popouts (field width: approximately 4 mm).

cross-sectional area as it pushed the popout a considerable distance away from its socket. The shadowy finger of this popout points at another popout that soon will be lifted atop another upthrusting column of silica gel.

Although these aggregates have been shown to be reactive, there have been no reported instances of the general deterioration and gross expansions normally associated with alkali-aggregate reactions. In addition, Mather, Buck, and Luke (1964) reported expansions of mortar bars tested to an age of one year by the Corps of Engineers Method CRD-C 123-57 were only 0.025% maximum, although the shale particles showed definite evidence of reaction. The reasons for this atypical behavior are not fully known.

THEORETICAL CONSIDERATIONS AFFECTING THE FORMATION OF POPOUTS OR DISCOLORATION

Both the popouts and gel surface discoloration just described are manifestations of the alkali-silica reaction. By studying the factors believed to influence the formation of these surface blemishes and exploring methods for modifying or eliminating those factors found to be harmful, it may be possible to prevent or limit popouts and gel discoloration. The following discussion describes mechanisms considered responsible for reaction surface distress. It reviews factors known to affect alkali-silica reaction, describes known preventative measures for harmful reactions, and discusses areas of investigation which could ultimately produce ways of obtaining better concrete surfaces in the affected localities.

The reaction between cement alkalis and the reactive shale particles forms an alkali-silica gel at the surface of the reacted particle. Other factors being equal, the quantity of gel produced from a single reactive particle, and the rate at which it forms, will be greatest if the particle surfaces are exposed to high-alkali concentrations. Once formed, the alkali-silica gel may absorb water and swell. Sometimes, the swollen gel can be accommodated without damage to the concrete surface. For example, the swollen gel might occupy available pore space in the concrete. Or, if the swollen gel is sufficiently fluid and the surface paste sufficiently permeable, the gel may force its way through pores in the paste to the surface of the concrete without damaging the mortar. The dried remains of such gel may well produce alkali-silica gel surface discoloration (see the silica-gel froth in Fig. 2).

If the swollen gel is too viscous, or if the permeability of the surface paste becomes too low to permit the gel to pass easily, pressures can be generated that are great enough to disrupt the mortar between the confined gel and the concrete surface, thus forming a popout. The viscosity of the gel appears to be controlled by its alkali concentration and by its water content. Paste permeability is controlled largely by the water-cement ratio and degree of curing of the paste.

From the preceding discussion, it is evident that the various factors that influence the alkali content, porosity, and permeability of concrete slab surfaces should also affect the surface condition of slabs made with a reactive sand.

The portion of alkali in portland cement that is water soluble at early ages is a primary factor in determining the alkali content of the concrete surface layer and, consequently, the extent of surface reaction (Stanton 1940). Therefore, the alkali content in the near-surface region of the concrete is not dependent only upon the "total" alkali content of the cement. Drying of concrete causes alkalis to migrate toward the surface that is being dried (Greening and Landgren 1966), thereby significantly increasing con-

centration of alkalis in the surface layer. Protecting a slab against drying caused by high temperature, high wind, or low humidity should lead to lower surface alkali concentration and reduce popout distress.

A thin slab would, when drying, show less of an increase in alkali content in the surface layer than a thicker slab of the same concrete subjected to the same drying conditions. Similarly, a slab made with a lean concrete mix contains less total alkali than a companion slab made with a rich mixture. Thus the accumulation of surface alkali during drying should be less for a slab cast with lean concrete than for the slab cast with a rich concrete.

It can be assumed that once the reaction between the alkali and shale particle occurs, a popout will result unless the mortar surrounding the aggregate particle can either internally accommodate the swollen gel or permit its passage through to the surface of the slab. It would be expected, therefore, that those factors that influence the porosity and permeability of the paste would also influence the susceptibility of the concrete to popout formation. Cement content and water-cement ratio could have a major influence.

Similarly, hard troweling of the concrete would tend to decrease the permeability of the surface and could therefore render the surface more susceptible to popout damage than a relatively porous, permeable surface that has not been troweled.

A number of other factors could confine the alkali-silica gel in concrete to the extent that the concrete ruptures and a popout is formed. The surface outlets of concrete pores may be sealed against gel exudation, inducing popouts. One example of this might be the use of sprayed-on membrane compounds to cure the concrete. Such compounds may seal gel exudate as well as water for hydration in fresh concrete. Similarly, gel already exuded to the surface of the slab may dry and harden, thereby sealing escape channels for gel against further exudations. It is quite possible that the nature of the alkali-silica gel inside the concrete pores may change from a relatively fluid to a more highly viscous condition as the surface of the concrete dries, thereby inducing popouts in the concrete surface.

There are three procedures that may be used to limit or eliminate expansion caused by the alkali-silica reaction. The first is to avoid the use of alkali-reactive aggregates. The second is to limit the alkali content of the cement. The third is to use pozzolans to avoid harmful reaction effects. All three of these preventative procedures should also be of some value in decreasing or stopping the surface popout and discoloration.

The old curing procedure of ponding a slab with excess water might also reduce popout distress. This curing procedure could be of value primarily because the alkali-silica reaction involving the opaline shale sand particles progresses much faster than the conventional expansive-type alkali-aggregate reactions described by Stanton

(1940) and others. An abundance of curing water applied to the slab surface immediately after troweling might lessen popout damage because (1) excess surface water would dilute alkali concentrations present at the slab surface and stop concentration of excess alkalis at the slab surface caused by drying, (2) possible increases in gel viscosity due to drying would not occur if the slab were kept wet for a period of days after casting, and (3) gel migrating to the surface of the slab would dissolve in the excess curing water, thereby avoiding hardened concentrations of air-dried silica gel which might seal gel escape passages and eventually cause popouts.

Pre-treatment of the aggregate with alkali before the aggregate is used in concrete might also eliminate the problems of popouts and gel surface discoloration.

It also may be possible to treat the reactive sands to remove the offending shale particles. For example, Merrillo, Handy, and Fox (1967) suggest that heavy media separation of reactive sands to remove particles having specific gravities less than 2.4 might improve the surfaces of concrete made with the sand. It should be realized, however, that unless beneficiation were almost total, some popout formation would occur. No aggregate beneficiation studies were made for the work reported here.

SCOPE OF CONCRETE INVESTIGATIONS

To investigate the preceding considerations, and, if possible, to devise means for avoiding popout damage to concrete surfaces, various laboratory tests were performed. In these tests, slabs, usually 30 cm (12 in.) square by 7.6 cm (3 in.) deep were used. The slabs were examined periodically, and the number of popouts and concrete surface quality recorded. A description of these slabs, 89 in all, is given in Table 1.

The work was directed to the following items:

1. *The significance of alkali content.* Studies involved the influence of the alkali content of the cement and the importance of alkali migration to the surface due to excessive drying caused by simulated severe climatic conditions. Total available alkali effects resulting from variation in concrete cement content and slab thickness were also investigated.
2. *Concrete properties affecting the flow of gel to the finished surface.* Among those properties investigated were cement content, finishing techniques, air entrainment, and curing techniques such as membrane curing and ponding.
3. *Remedial or preventative measures.* Studies were made of the possibility of reducing or eliminating popouts by means of limiting the cement-alkali content, using pozzolanic replacement, the pre-reaction of aggregates with alkali before they are used in the concrete, and the significance of curing techniques.

MATERIALS AND TEST PROCEDURES

Fine Aggregates

A reactive sand from central Iowa (PCA Lot No. 20700) was used in the preliminary testing. Later tests were run using a similar sand from southeastern Minnesota (PCA Lot No. 20701).

Coarse Aggregate

A non-reactive glacial gravel from Elgin, Illinois, was used in all of the tests.

Portland Cement

Two cements available in the Chicago area were used in this investigation. One was a cement of high total alkali content and the other a low total alkali content cement as shown in Table 2.

Table 2. Alkali Contents of Cements in this Study

PCA Lot No.	% Na ₂ O	% K ₂ O	% Total Alkalies (Na ₂ O equivalent)	% Water Soluble Alkali (Na ₂ O equivalent)*
20686	0.12	0.11	0.19	0.05
20687	0.16	1.30	1.02	0.70

*Per ASTM C 114.

Cements of intermediate alkali content were made by blending appropriate amounts of these two cements.

Admixtures

Neutralized Vinsol resin was the admixture used to entrain air. A fly ash from the Chicago area (PCA Lot No. 19683) was used as a pozzolan.

Concrete Mixes

Except where otherwise noted, concrete was made with a 19 mm (¾ in.) maximum size aggregate, a nominal cement content of 307 kg/m³ (517 lb/yd³), and a net water-cement ratio of approximately 0.49. Slump was generally maintained in the range of 75 to 100 mm (3 to 4 in.) and net air content (pressure method) in the range of 4½ to 5½%. Sand constituted 42% of the total aggregate volume.

Before mixing, the air-dried aggregates were immersed in water for 18 hours. Small laboratory pan-type mixers were used. Concrete was mixed for 2½ minutes in a laboratory maintained at 23°C (73°F) and 50% R.H. After

Table 1. Description of Slabs, Curing Procedures, and Popout Development

Slab No.	Cement Alkali*, %	Pre-trowel Cover	Cover First Day	Cure Temp, °C (°F)	Total Popouts	Remarks
Sand Lot No. 20700; Slab dimensions: 61 cm x 61 cm x 3.8 cm (2 ft x 2 ft x 1.5 in.) Except where noted						
1	1.0	Poly	Moist	23 (73)	0	Membrane back and sides at 3d.
2	1.0	Poly	Membrane	23 (73)	10	
3	1.0	Poly	Poly	23 (73)	0	
4	0.8	Poly	Moist	23 (73)	0	Membrane back and sides at 3d.
5	0.8	Poly	Membrane	23 (73)	8	
6	0.8	Poly	Poly	23 (73)	0	
7	0.6	Poly	Moist	23 (73)	0	Membrane back and sides at 3d.
8	0.6	Poly	Membrane	23 (73)	0	
9	0.6	Poly	Poly	23 (73)	1	
10	0.2	Poly	Moist	23 (73)	0	Membrane back and sides at 3d.
11	0.2	Poly	Membrane	23 (73)	0	
12	0.2	Poly	Poly	23 (73)	0	
13	1.0	None	None	23 (73)	52	Slab 30 cm x 30 cm x 15 cm (1' x 1' x 6")
14	1.0	None	None	23 (73)	30	Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
15	1.0	Poly	Poly	23 (73)	15	Slab 30 cm x 30 cm x 15 cm (1' x 1' x 6")
16	1.0	Poly	Poly	23 (73)	17	Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
17	1.0	Poly	Poly	23 (73)	0	Wood screed only Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
18	1.0	Poly	Poly	23 (73)	0	Screed and magnes. float Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
19	1.0	Poly	Poly	23 (73)	2	Early steel trowel Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
20	1.0	Poly	Poly	23 (73)	16	Correct trowel time Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
21	1.0	Poly	Poly	23 (73)	10	Late trowel Slab 30 cm x 30 cm x 7.6 cm (1' x 1' x 3")
Sand Lot No. 20700; Slab dimensions: 30 cm x 30 cm x 7.5 cm (1 ft x 1 ft x 3 in.) Except where noted						
22	1.0	Poly	Poly	23 (73)	5	Slab 30 cm x 30 cm x 15 cm (1' x 1' x 6")
23	1.0	Poly	Poly	23 (73)	28	Slab 30 cm x 30 cm x 15 cm (1' x 1' x 6")
24	1.0	Conf	Poly	23 (73)	0	
25	1.0	Conf.	Poly	23 (73)	5	
26	1.0	None	Poly	23 (73)	11	8% Fly ash replacement
27	1.0	Poly	Poly	23 (73)	14	8% Fly ash replacement
28	1.0	Poly	Moist	23 (73)	0	8% Fly ash replacement
29	1.0	None	Poly	23 (73)	14	15% Fly ash replacement
30	1.0	Poly	Poly	23 (73)	14	15% Fly ash replacement
31	1.0	Poly	Moist	23 (73)	0	15% Fly ash replacement
32	1.0	None	Poly	23 (73)	1	Cement content: 223 kg/m ³ (376 lb/yd ³ – 4-bag mix)
33	1.0	Poly	Poly	23 (73)	0	Cement content: 223 kg/m ³ (376 lb/yd ³ – 4-bag mix)
34	1.0	Poly	Moist	23 (73)	0	Cement content: 223 kg/m ³ (376 lb/yd ³ – 4-bag mix)
35	1.0	None	Poly	23 (73)	5	Cement content: 390 kg/m ³ (658 lb/yd ³ – 7-bag mix)
36	1.0	Poly	Poly	23 (73)	4	Cement content: 390 kg/m ³ (658 lb/yd ³ – 7-bag mix)
37	1.0	Poly	Moist	23 (73)	0	Cement content: 390 kg/m ³ (658 lb/yd ³ – 7-bag mix)
38	1.0	Poly	Poly	38 (100)	30	
39	1.0	None	Poly	38 (100)	56	
40	1.0	None	4 hr. None; Poly	38 (100)	24	
41	0.6	Poly	Poly	38 (100)	10	
42	0.6	None	Poly	38 (100)	83	
43	0.6	None	4 hr. None; Poly	38 (100)	40	
44	1.0	None	Poly	23 (73)	15	20% Fly ash replacement
45	1.0	Poly	Poly	23 (73)	14	20% Fly ash replacement
46	1.0	Poly	Moist	23 (73)	0	20% Fly ash replacement

Table 1. Description of Slabs, Curing Procedures, and Popout Development (cont'd.)

Slab No.	Cement Alkali*, %	Pre-trowel Cover	Cover First Day	Cure Temp, °C (°F)	Total Popouts	Remarks
Sand Lot No. 20700; Slab dimensions: 30 cm x 30 cm x 7.5 cm (1 ft x 1 ft x 3 in.)						
47	0.8	Poly	Poly	38 (100)	275	
48	0.8	None	Poly	38 (100)	394	
49	0.8	None	4 hr. none; Poly	38 (100)	380	
50	1.0	None	Poly	23 (73)	1	30% Fly ash replacement
51	1.0	Poly	Poly	23 (73)	1	30% Fly ash replacement
52	1.0	Poly	Moist	23 (73)	0	30% Fly ash replacement
Sand Lot No. 20701; Slab Dimensions: 30 cm x 30 cm x 7.5 cm (1 ft x 1 ft x 3 in.)						
53	1.0	Poly	Moist	23 (73)	0	
54	1.0	None	Poly	38 (100)	156	
55	1.0	None	4 hr. None; Poly	38 (100)	210	
56	1.0	None	Poly	38 (100)	209	
57	1.0	None	See remarks	38 (100)	124	Wash with water at 8 hr – Poly
58	1.0	None	See remarks	38 (100)	122	Wash with Ca(OH) ₂ at 8 hr – Poly
59	1.0	None	Poly	38 (100)	228	5% Ca(OH) ₂ addition
60	1.0	None	See remarks	38 (100)	217	Wash with water at 8 hr – Poly
61	1.0	None	Poly	23 (73)	0	5% Ca(OH) ₂ addition
62	1.0	None	Poly	38 (100)	5	4-bag mix: 223 kg/m ³ (376 lb/yd ³)
63	1.0	None	Poly	38 (100)	159	5½ -bag mix: 307 kg/m ³ (517 lb/yd ³)
64	1.0	None	Poly	38 (100)	167	7-bag mix: 390 kg/m ³ (658 lb/yd ³)
65	1.0	None	Poly	38 (100)	20	Agg. pre-reacted 18 hr – 3% NaOH
66	1.0	None	Poly	38 (100)	6	Agg. pre-reacted 1.5 d. – 3% NaOH
67	1.0	None	Poly	38 (100)	0	Agg. pre-reacted 2.5 d. – 3% NaOH
68	1.0	None	Poly	38 (100)	133	
69	1.0	None	Pond ¼" water	38 (100)	0	Water not changed
70	1.0	None	Pond ¼" water	38 (100)	0	Water changed morning and night
71	1.0	None	Loose Poly	38 (100)	25	Spec. washed morning and night
72	1.0	None	Cover ½" sand	38 (100)	0	Sand kept wet
73	1.0	None	Poly	38 (100)	143	10% Fly ash replacement
74	1.0	None	Poly	38 (100)	134	20% Fly ash replacement
75	1.0	None	Poly	38 (100)	62	30% Fly ash replacement
76	0.4	None	Poly	38 (100)	13	0.4% alkali (as Na ₂ O equivalent) in cement
77	0.2	None	Poly	38 (100)	0	0.2% alkali (as Na ₂ O equivalent) in cement
78	1.0	None	Poly	38 (100)	330	Non-air-entrained concrete
79	1.0	None	Poly	38 (100)	302	High entrained air content (8½%)
80	1.0	None	None	38 (100)	53	Slab covered with fluid, wet clay which was dried and scraped off
81	1.0	None	Wet burlap	38 (100)	0	Slab covered with wet burlap
82	1.0	None	Pond ¼" solution	38 (100)	23	Slab ponded with 2 molar NaOH solution
83	1.0	None	See remarks	38 (100)	13	Slab "cured" by spreading flake CaCl ₂ on finished surface
84	1.0	None	Poly	38 (100)	8	Control – no ponding
85	1.0	None	See remarks	38 (100)	0	Ponded just after finishing
86	1.0	None	See remarks	38 (100)	0	Slab finished, Poly for 6 hr., pond 3d
87	1.0	None	See remarks	38 (100)	0	Slab finished, Poly for 11 hr., pond 3d
88	1.0	None	See remarks	38 (100)	30	Slab finished, Poly for 23 hr., pond 3d
89	1.0	None	See remarks	38 (100)	41	Slab finished, Poly for 45 hr., pond 3d

"Poly" — Slab covered with polyethylene film.

"Moist" — Slab stored in moist room.

"Membrane" — Slab sprayed with resin membrane cure compound.

"Conf" — Slab sprayed with "Confilm."

*Total alkalis, expressed as % Na₂O equivalent.

measuring concrete slump, air content, and unit weight, the concrete was placed in watertight Plypreg or sheet metal molds. Specimen sizes are shown in Table I. Concrete was placed in two lifts and consolidated on a table vibrator. Immediately after consolidation, the concrete was struck off with a wooden straightedge.

Finishing

In most cases, shortly after strike-off the concrete surface was smoothed with a magnesium float. Initial and final steel troweling was performed by a skilled technician. In most instances, this technician did all of the finishing so that this critical operation could be performed as consistently as possible.

Curing

A detailed compilation of the various curing techniques utilized is given in Table I.

TEST RESULTS AND DISCUSSION

Effect of Variation in Cement-Alkali Content

Stanton (1940) established that the severity of the concrete deterioration caused by alkali-silica reactivity of the general type is a function of cement-alkali content. Field evidence would also indicate that there is a general relationship between cement-alkali content and the occurrence of popouts. A series of test slabs were made with cements, ranging in alkali contents from 0.2% to 1.0%, expressed as Na_2O equivalent.

The slabs were cast at 23°C (73°F), exposed to 38°C (100°F) and 25% R.H. air until troweling, and cured at 38°C (100°F) under polyethylene for 3 days. This procedure was designed to simulate field conditions encountered in poorly controlled concrete work during the hottest and driest days of the summer. These slabs (Numbered 39, 42, 48, 76, and 77 in Table 1) are shown in Fig. 4.

Inspection of this figure will show that although there is a general decrease in both the size and number of popouts with decreasing alkali content, popout formation occurred even with cements of "low" total alkali content.

It would therefore appear that any reasonable limitation of cement-alkali content would not insure the satisfactory performance of these aggregates under the most adverse summer field conditions.

Alkali Migration and the Effect of the Total Alkali Content of the Concrete

The increase of surface alkali content caused by alkali migration to the drying face of the concrete has already been discussed. This alkali migration was demonstrated by an electron probe analysis of a slab made with a cement of high alkali content (1.02% as Na_2O equivalent). Prior to troweling, the surface was exposed to an environment of 38°C (100°F) and 25% R.H. After troweling, the slab was cured under polyethylene for 3 days at that temperature, followed by drying in air at 23°C (73°F) and 50% R.H. This analysis showed the concentration of potassium at the troweled surface to be about 10 times greater than that in the interior of the slab.

Three test procedures were used to evaluate the effects of alkali migration on popout frequency. The results were as follows:

Effect of Severe Temperature and Humidity Conditions

Since it had been reported that the severity of the problem was greatly accentuated during the unusually hot and dry summer of 1966, a series of slabs was cast and cured in different environments, then dried at 23°C (73°F) and 50% R.H. Typical results (Slab No. 35 and 64, Table 1) for mixtures made with 390 kg of cement per cubic metre of concrete (658 lb of cement per cubic yard of concrete) and similar sands are given below.

Table 3. Effect of Severe Temperature and Humidity Conditions on Popout Formation

Finishing and Curing Environment	Number of Popouts
Unprotected at 23° (73°F) and 50% R.H., troweled then cured under polyethylene 3 days	5
Unprotected at 38°C (100°F) and 25% R.H., troweled, then cured under polyethylene 3 days	167

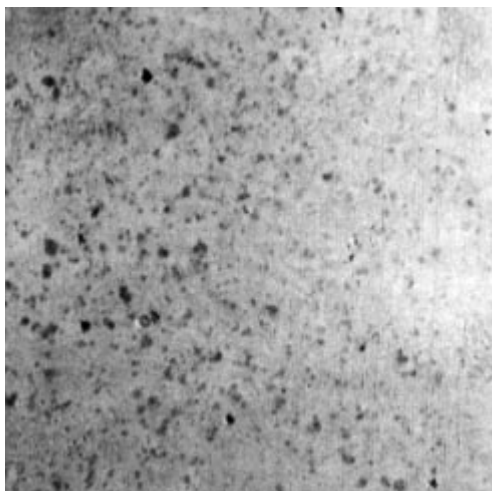
The increase in popout formation may be mostly due to the increased migration of alkali to the surface of the slab exposed to the higher temperature and lower humidity prior to finishing and curing.

Effect of Slab Thickness

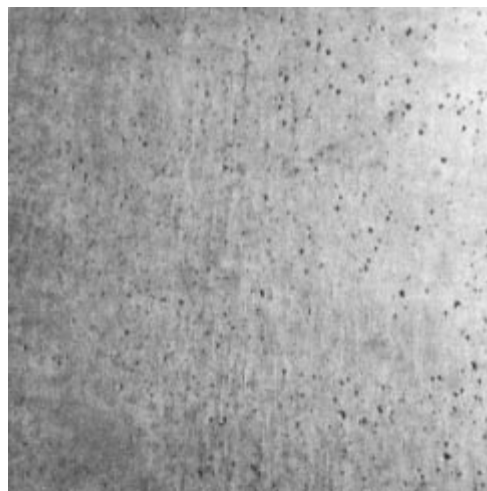
To evaluate the influence of this variable, two slabs (Slabs No. 13 and 14) varying only in thickness were cast, exposed to air at 23°C (73°F) and 50% R.H. for 18 hours, then cured under polyethylene for 3 days. Results were as follows:

Table 4. Effect of Slab Thickness on Popout Formation.

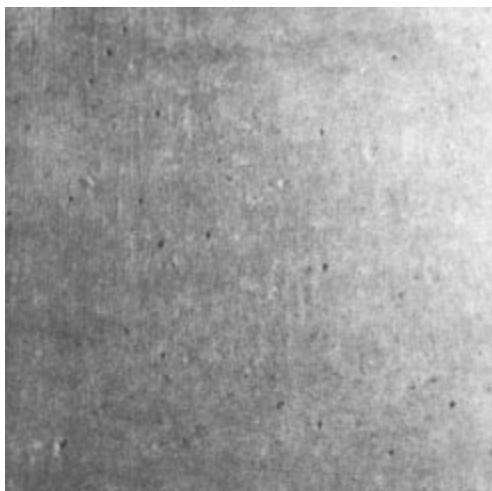
Slab Thickness, inches	Number of Popouts
6	52
3	30



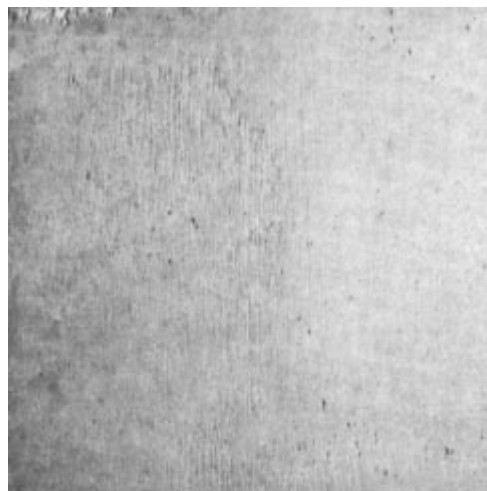
4a. Slab 39, 1.0% alkali



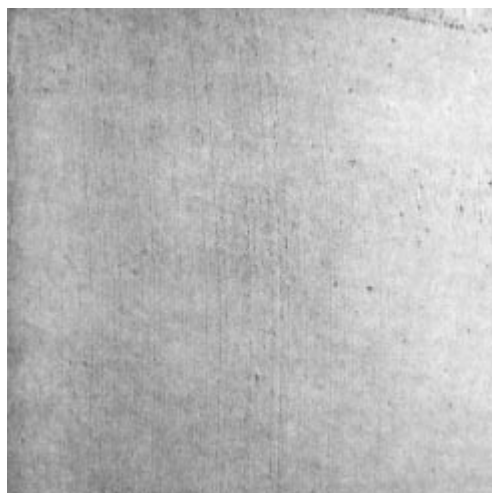
4b. Slab 48, 0.8% alkali



4c. Slab 42, 0.6% alkali



4d. Slab 76, 0.4% alkali



4e. Slab 77, 0.2% alkali

Figure 4. Effects of cement-alkali content (as Na_2O) on popout frequency in a 307 kg/m^3 (517 lb/yd^3) concrete mixture [slabs are 930 cm^2 (1 ft^2)].

These results suggest that thick slabs, with their higher total alkali content for a unit area of drying surface, are more susceptible to popout damage than thin slabs.

Effect of Drying Prior to Final Finishing

This was found to be one of the most significant variables. Slabs that were protected from drying with a polyethylene cover prior to troweling were found to develop significantly fewer popouts than slabs allowed to dry during this waiting period (Slabs No. 15 and 13). Comparative data are listed in Table 5.

Table 5. Effect of Polyethylene Covering on Popout Formation

Polyethylene Cover	Number of Popouts
Yes	15
No	52

This demonstrates that drying migration of alkalis to the slab surface increases popout frequency. Covering the slab during the interval between casting and final troweling should decrease alkali migration and thus the number of popouts.

These three examples of the effects of severe temperature and humidity conditions, slab thickness, and drying prior to troweling indicate that the number and size of the popouts formed on the exposed surface of concretes made with the reactive sands under investigation is a function not only of initial alkali content of cement, but also of the total alkali content of the concrete and the extent to which these alkalis are redistributed and concentrated.

Effect of Cement Content

To study the effect of variations in cement content, slabs were made from three mixes, containing 223, 307, and 390 kg of cement per cubic meter of concrete (376, 517, and 658 lb of cement per cubic yard of concrete, respectively). The slabs were cast at 23°C (73°F), moved to a room held at 38°C (100°F), and left exposed until troweling. Following troweling, the slabs were covered with polyethylene for 3 days of curing, then moved back to 23°C (73°F) to air-dry at 50% R.H.

Photographs of these slabs (No. 62, 63, and 64) are shown in Fig. 5. The number of popouts that developed are tabulated in Table 6.

Table 6. Effect of Cement Content on Popout Formation

Cement Content of Mix, kg/m ³ (lb/yd ³)	Number of Popouts
223 (376)	5
307 (517)	159
390 (658)	167

Not only the number, but also the size, of the popouts tended to increase with increasing cement content. An increase in popout frequency with the higher cement content may be due to two causes. High cement content concretes have greater total alkali contents than lean mixtures and could be expected to have greater alkali migration toward the drying surface than lean mixtures. The permeability and porosity of high cement content concrete is low. Such concrete would have limited internal pore space to accommodate swelling gel and might offer such a high resistance to flow of gel to the surface that popout occurrence would be likely.

Effect of Finishing Techniques

To evaluate the effect of finishing techniques on popout formation, otherwise identical slabs were given various finishes which included (1) a simple wood screeding, (2) wood screeding and magnesium floating, (3) wood screeding and magnesium floating followed by early steel troweling, (4) troweling when the surface was at the proper consistency, and (5) late troweling. Popouts were observed to form only on the steel-troweled surfaces. This is probably the result of a decrease in both the permeability and porosity of the paste at the surface as a result of hard troweling, thereby decreasing the space available to accommodate the alkali-silica gel as it is formed and making it more difficult for the swelling gel to move to the surface of the concrete slab.

It is quite likely that slight variations in finishing were a major cause of differences in popout frequency noted in Table 1 for similar slabs nominally given the same treatment. Early finishing tended to produce few popouts and late finishing tended to produce many.

Effect of Air Entrainment

Air entrainment increases the space available to accommodate gel formed. However, air entrainment did not seem to affect popout frequency appreciably. Data for Slabs No. 78 and 79 are listed in Table 7.

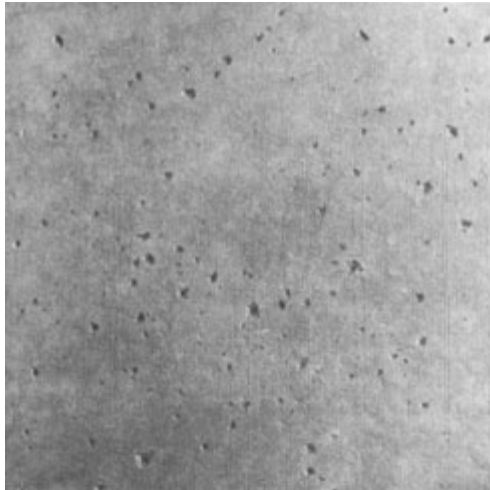
Table 7. Effect of Air Entrainment on Popout Formation

Entrained Air Content, %	Number of Popouts
1.5	330
8.5	302

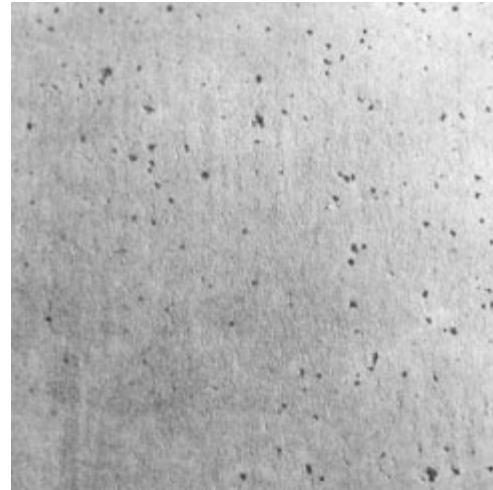
The availability of additional space provided by the entrained air may have been offset by the lower water-cement ratio and hence lower permeability.

Effect of Curing Procedures

One of the most significant findings of this investigation is that certain commonly recommended curing procedures,



5a. Slab 64, 390 kg/m³



5b. Slab 63, 307 kg/m³



5c. Slab 62, 223 kg/m³

Figure 5. Effects of concrete cement content on popout frequency [slabs are 930 cm² (1 ft²)].

while adequate for most concretes, are definitely inappropriate for slabs incorporating the reactive sand. The data suggest that curing a slab by applying a polyethylene film cover after troweling will produce as many, if not more, popouts than if the slab were not cured at all. Membrane curing definitely caused more popouts than polyethylene curing. Neither of these curing procedures prevents the early migration of alkali to the finished surface during the interval of drying between casting and troweling. Both retain water in the concrete after alkali concentration has taken place, thereby possibly promoting the alkali-silica reaction as well as hydration of the cement. Membrane curing sealed in all gel until popouts started to form. This kind of cure probably was responsible for the high popout frequency noted.

A number of slabs were cured with excess water. Soon after troweling, these slabs were either ponded with 6 mm (¼ inch) of water or covered with wet burlap or sand. The ponding or wet coverings were maintained for at least 3 days. Such treatments completely eliminated both popouts and gel exudations.

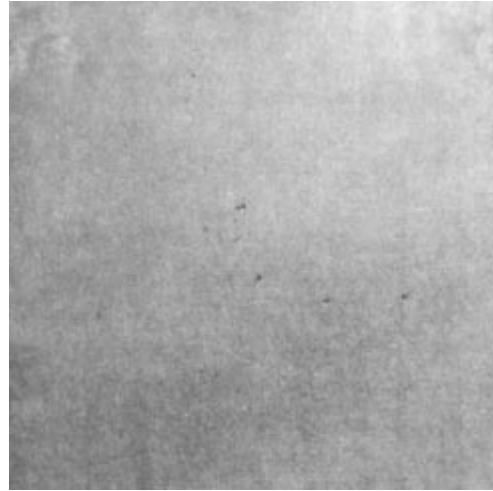
These wet curing techniques are apparently effective through the leaching of both the cement alkalies and, most probably, of the alkali-silica gel formed near the concrete surface. Fig. 6 shows the effect of various curing techniques on the formation of popouts on Slabs No. 68, 69, 70, 71, and 72.

It is extremely important that these wet curing procedures be initiated soon after troweling. If such procedures are delayed too long, they are no longer effective in pre-

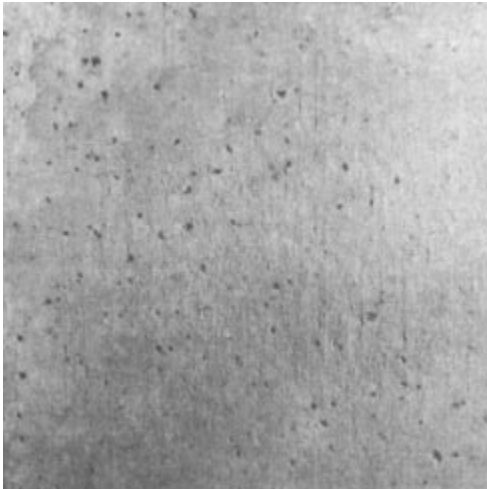
venting popouts. In a test series designed to evaluate the time delay factor, it was found that if the wet cure was delayed up to 11 hours, no popouts formed, but the slab surface was badly discolored by gel residue. Delaying wet curing by a day or more caused more popouts than if the slab had not been ponded at all. To minimize popouts, positive curing should be applied as early as possible, and the slab surface should be soaked as long as possible.

Evaluation of Possible Remedial Measures

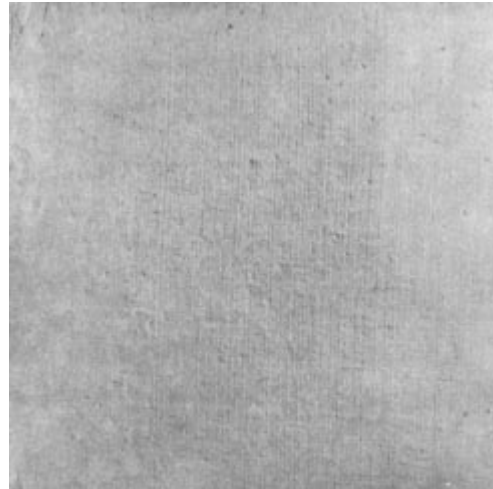
Since the observed concrete deterioration had been shown to be the result of an alkali-silica reaction, a series of tests was made to evaluate remedial techniques aimed at preventing or moderating the chemical reaction involved.



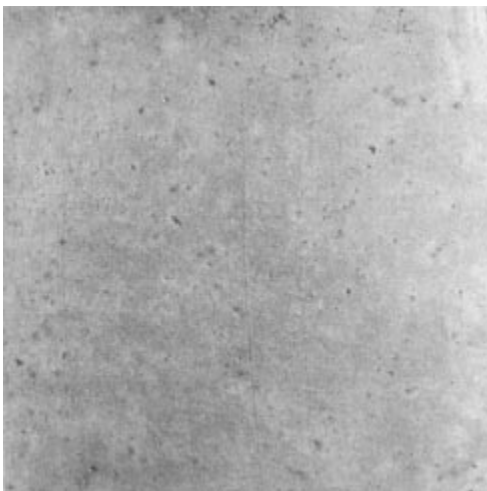
6c. Slab 69, ponded 3 days.



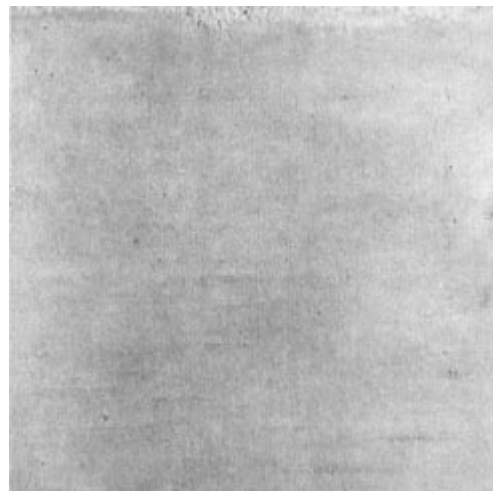
6a. Slab 68, covered with polyethylene film for 3 days.



6d. Slab 70, ponded 3 days (water changed).



6b. Slab 71, covered with polyethylene and rinsed twice daily for 3 days.



6e. Slab 72, covered with wet sand 3 days.

Figure 6. Effects of curing procedure on popout frequency [slabs are 930 cm² (1 ft²)].

Effect of Pozzolanic Replacements. The addition of a pozzolan to a concrete mix is a conventional way of tying up available alkalis and decreasing alkali-aggregate reactivity. Calcined shales are efficient pozzolans. Probably the most readily available pozzolan in the Midwest is fly ash. Fly ash 19683 was used in these tests on Slabs No. 13, 44, 50, 73, 74, 75, and 79 with the results shown in Table 8.

Table 8. Effect of Fly Ash Replacement and Curing Temperature on Popout Formation

Fly Ash Replacement, % by weight of cement	Number of Popouts	
	Cured at 23°C (73°F)	Cured at 38°C (100°F)
0	30	30
10	*	148
20	15	134
30	1	62

*No data collected.

These data show that at moderate temperatures, a 30% replacement with fly ash is effective in preventing popout formation. However, at the elevated temperatures generally encountered during the summer in the affected area, fly ash replacements at even this level were found to be inadequate.

Effect of Sodium Hydroxide Pretreatment. Studies were made of the possibility of reacting the alkali-reactive sands before their use in concrete. Three batches of sand (for Slabs No. 65, 66, and 67) were reacted with a 3% solution of sodium hydroxide for various periods of time before they were used in concrete. One batch was soaked for ½ hour in the alkali solution before the solution was decanted from the sand, and was used in concrete 18 hours after soaking. The second batch was similarly soaked and decanted, but was not used in concrete for 1½ days. The third batch was kept under an excess of alkali solution for 3 days before it was decanted and then immediately used in concrete. Even the lightest pretreatment caused significant reductions in the number of popouts in the standard 38°C (100°F) concrete mix. No popouts were evident with the sand that was given the most thorough pretreatment.

However, alkali treatment has certain disadvantages such as extra costs and safety hazards. The excess alkali carried into the mix with the sand contributed to the following problems: (a) the slab surfaces were markedly discolored with streaks and beads of silica gels although foot traffic might scuff this off readily, and (b) the excess sodium hydroxide greatly accelerated the set of the concrete, indicating problems of practical concrete control

with treated sands. It might also contribute to siliceous or alkali-carbonate reactions with other constituents of the aggregate, causing excessive expansions normally encountered with such reactions.

SUMMARY

Shale particles in glacially derived sands have caused surface discoloration and popout formation on hard-troweled concrete flatwork in portions of Iowa, Minnesota, and South Dakota.

Laboratory studies have shown that both the discoloration and the popouts are the result of alkali-silica reactions involving opaline materials in the shale.

A series of laboratory concrete tests was performed to evaluate the factors which influence popout formation, and to test various possible remedial techniques. These tests showed that the problem was aggravated by the migration of alkalis to the drying surface both during and after the finishing operation, and that the temperature-humidity conditions also had a large effect. Commonly used curing techniques, such as membrane cure and curing under tarps or polyethylene sheeting, were also found to increase the number of popouts. Although there was some relationship between the size and number of popouts and cement-alkali content, it was found that popouts would form in significant numbers even with cements of 0.4% alkali content when the concrete was cured under high temperature-low humidity conditions which could be encountered during summer construction. Although a 30% replacement with fly ash was found to stop popout formation, such fly ash replacement was not effective in preventing distress under adverse climatic conditions. Pretreatment of the aggregate with alkaline solutions was found to be effective, but might well be impractical

It was found that popout formation and gel residues could be completely eliminated by curing the concrete with an excess of water. This can be done by ponding, or by curing under continually moist sand or burlap. To be effective, however, these curing techniques must be initiated soon after finishing is completed.

RECOMMENDATIONS

On the basis of the experimental work described in this report, the following procedures would seem to offer the best protection against the formation of gel exudations or popouts when reactive sands must be used:

1. If high-alkali cement is used in the hot summer months, wet curing is essential. Wet curing should be initiated as early as possible.
2. The fresh concrete should be protected from drying prior to the final finishing operations.
3. Where possible, hard troweling should be avoided.

ACKNOWLEDGEMENTS

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