

# Proceedings of the Iowa Academy of Science

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Volume 74 | Annual Issue

Article 21

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1967

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### Recommended Citation

Elwell, James H. and Lemish, John (1967) "Pop-outs in Hematite Colored Concrete Floor," *Proceedings of the Iowa Academy of Science*: Vol. 74: No. 1 , Article 21.  
Available at: <http://scholarworks.uni.edu/pias/vol74/iss1/21>

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# Pop-outs in Hematite Colored Concrete Floor

JAMES H. ELWELL<sup>1</sup> AND JOHN LEMISH<sup>2</sup>

*Abstract.* Pop-outs found in a hematite colored basement floor subjected to wetting and drying action were attributed to shale introduced into the concrete as fine aggregate. The shale particles in pop-outs were characterized by clear gel rims associated with the alkali-silica reaction as well as red-brown hydrated iron oxide gel caused by oxidation of indigenous iron. Fresh shale particles from the same fine aggregate source used in the concrete under investigation were placed in sodium hydroxide which developed reaction zones characteristic of the concrete reaction and were interpreted as evidence supporting the instability of this material in alkaline environment. The hematite color which marked the original particle surfaces, indicated that reaction rims were developed entirely within the shale particle. Further study of the rim gel is needed to explain the pathway and interactions between the iron oxidation reactions and the alkali-silica reactions.

Pop-outs are small pieces of a concrete surface that became detached through chemical or physical expansion processes leaving unattractive holes. A newly placed red-colored concrete floor in a building under construction on the Iowa State University campus developed pop-outs after it was flooded during a heavy summer rain storm. Because the floor obviously had not been subjected to freezing and thawing action, pop-out formation was attributed to flooding which permitted water to cover the floor and to presence of a reactive lithology that induced the well-known alkali-silica reaction. This reaction is known to depend on the presence of alkali ions, water and the reactivity of the silicate mineral structure. Cristobalite, tridymite, and opal are possible reactive structural forms of silica; quartz is considered unreactive. Since the alkali-silica reaction causes expansion and disruption of concrete by imbibing water to form a volume increasing gel, recognition and subsequent elimination of the reactive lithology was a direct approach to the pop-out problem. The overall engineering aspects of the problem were handled by Mr. Derwin C. Merrill under the direction of Dr. Richard L. Handy of the Iowa State University Civil Engineering Department and at their request our laboratory worked on the problem of detection of the reactive lithology and source of such deleterious material. Possible sources included the fine aggregate sand fraction of the concrete and the silica sand component of the hematite coloring material.

This case study is of general geologic interest because of the recognition of an associated indigenous iron reaction in the reactive particles and of how the geologic concept of time can be applied to practical problems.

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INVESTIGATIONS

Pop-outs were studied using a reflected light microscope. In each case the reactive pop-out particle was described as having a gray argillaceous lithology. In order to determine the source of such deleterious material both the fine aggregate source material and hematite dye sand particles were investigated. Shale particles from the fine aggregate material were placed in 1 N NaOH to confirm their reactivity in an alkaline environment.

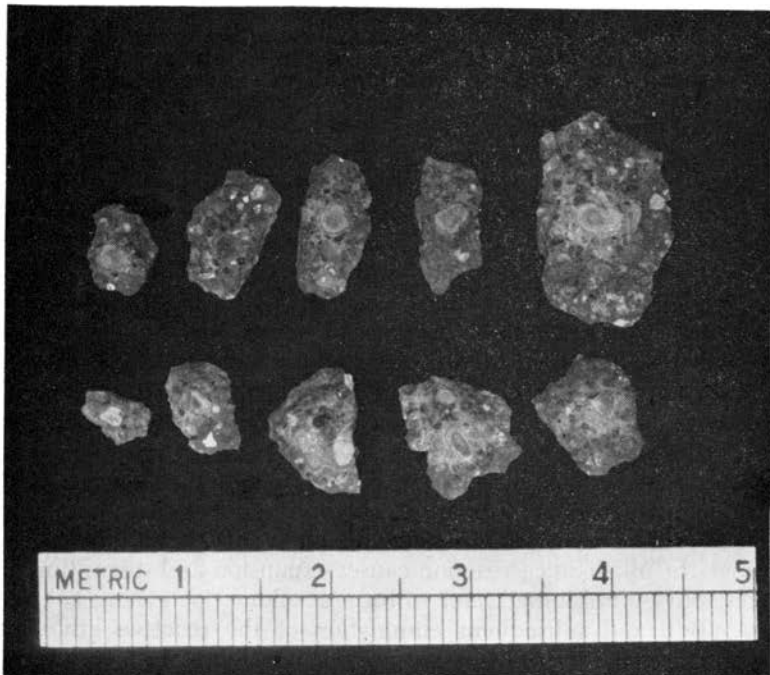


Figure 1. Pop-outs from a hematite-colored concrete floor.

RESULTS

Observations of pop-outs indicated that each was related to a particle with an argillaceous lithology coated with dried gel. Figure 1 shows a sequence of several pop-out samples with the typical flat failure cone associated with alkali reactive particles. The conical pop-outs penetrate through the surfacing application and the deleterious particles lie within the near surface portion of the concrete. The light colored area surrounding reactive particles represents dried gel on the hematite colored surface. An enlargement of the middle particle in the row furthest from the scale is shown in Figure 2. Three zones are visible in reacted particles: a central region surrounded by a light colored area followed by an outermost colorless boundary zone (appears as clear

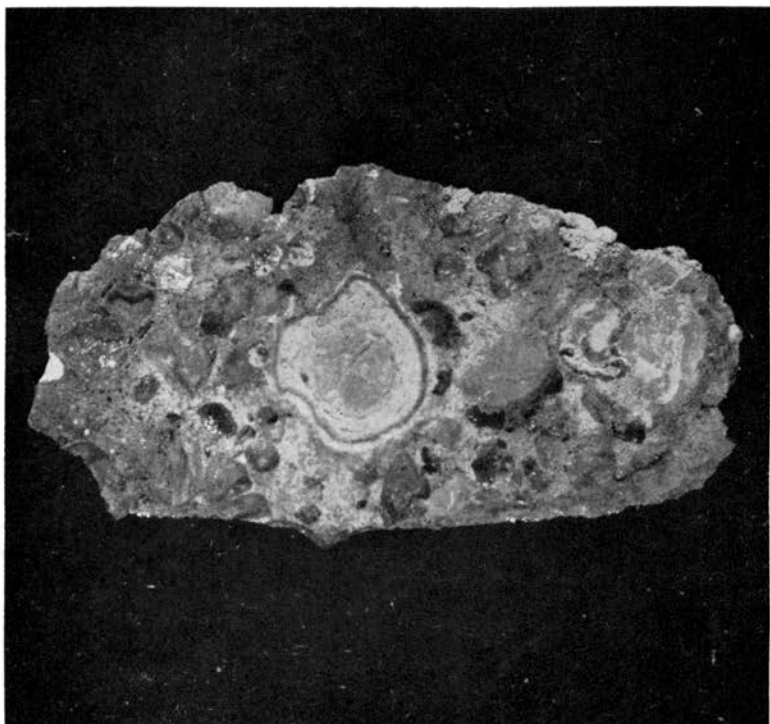


Figure 2. Reaction zones in reactive pop-out lithology.

dark outer band in Figure 2). Central, intermediate, and outer zones occurred in most pop-out samples observed, but in some cases reaction proceeded to the extent that the central zone was absent. In Figure 2 cracking sub-parallel to the particle boundary occurs in the intermediate zone and radial cracks traverse the entire grain. The lighter color of the intermediate zone is caused by change in oxidation state of indigenous iron material and presence of dried gel. Minute specs of reddish brown color are disseminated throughout the intermediate zone.

The sand particles isolated from the hematite dye were sub-angular quartz grains and could not be lithologically related to unaltered pop-out material. However, a soft argillaceous material was found in the fine aggregate sample which was identified as shale or claystone and matched the gray central zone in pop-out material (1). Several such shale particles were broken for examination and found to consist of a uniform gray lithology throughout which indicated that they were unreacted.

The fine aggregate shale particles which had been placed in a

oxidation state of its iron material resulted in a brown stain similar to that of the intermediate zone of pop-out particles. Some of the iron material was transferred from shale particles giving the NaOH solution the color of hydrated ferric oxide. In a comparable test of hematite coated quartz grains in 1 N NaOH, the solution remained clear indicating that hematite powder was stable in this environment. This eliminated the quartz grains and the hematite dye from further consideration as a potential source of trouble.

#### DISCUSSION

The hematite dye covered surfaces of all particles in the concrete and formed a red border which in effect provided a time and spatial reference line for detection of subsequent events such as gel flow resulting from post coloring reactions. Since hematite was previously shown to be immobile in an alkaline environment and was absent in the outer zone of pop-out particles, it was concluded that the outer zone developed within the original boundary of the particle as defined by the red border. Since a brown color zone with characteristics comparable to the intermediate zone of pop-out particles was developed in shale particles immersed in sodium hydroxide solution, this zone, defined by the brown color of oxidized iron, was considered a result of reaction in the alkaline environment of concrete.

The inner zone of the pop-out particle, which has a gray color resembling shale particles found in fine aggregate source material, is interpreted as the least altered zone of an incompletely reacted pop-out. The lighter colored brown intermediate zone acts as the zone of reaction while the clear outer zone resists flow of gel by sealing the surface.

Observation of change in the oxidation state of iron, induced by wetting and drying of the concrete, is important because the color change of the iron defines the presence of an oxidation reaction during the substitutional alkali-silica reaction. Since the increased volume of the ferric oxide hydration product of clay ironstones as discussed by Mielenz (2) has been thought to cause pop-outs, the hydrated ferric oxide product of this study which is related in time to the alkali-silica reaction may be considered partly responsible for the present pop-outs. Formation of ferrous hydroxide followed by oxidation to hydrated ferric oxide is thought to be the path for this iron-gel reaction. There is no doubt that the mechanism of expansion in this case should be classified as an alkali-silica reaction (1) and that a causal relationship between the iron-gel reaction and structure sensitive alkali-silica reaction has not yet been shown. However, because these reactions are associated in time and space, the possibility

of their interaction has now been recognized and the question of the role of the iron reaction in expansion mechanisms is raised.

#### CONCLUSIONS

1. Elimination of shale particles from the fine aggregate fraction would prevent pop-outs. Sand-sized shale particles present in fluvial deposits over much of the north-central states area and their removal will require elimination by some means prior to use in concrete.

2. The inert hematite stain may be useful laboratory technique to mark initial surfaces for subsequent alkali reaction studies.

3. Further investigation of the iron oxidation reaction, mobility of iron gel and interactions between iron and alkali-silica reactions is needed to explain the mechanism of expansion of this material.

#### Literature Cited

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