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## Autoclave Method for Determining Susceptibility of Carbonate Aggregates To Silicification

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# Autoclave Method for Determining Susceptibility of Carbonate Aggregates To Silicification

By JOHN LEMISH and RAMON E. BISQUE

*Abstract.* Autoclave treatment of concrete bars made from argillaceous dolomitic carbonate aggregates at a temperature of  $420^{\circ} \pm 3^{\circ}$  F. and a steam pressure of  $295 \pm 10$  lbs. for 3 to 9 hours caused the progressive development of silicified reaction shells on the coarse carbonate aggregate similar to those found in distressed concrete. Similar treatment of concrete bars made from pure limestone aggregates induced no reaction shell growth. The susceptibility of carbonate aggregates to silicification could be rapidly determined by the autoclave method.

Previous research has demonstrated that certain carbonate aggregates develop reaction shells in concrete due to the introduction of silica derived from the cement paste (Bisque and Lemish, 1959). A rapid method of identifying carbonate rocks which are susceptible to this type of "silicification," believed to be deleterious to concrete, is described in this report.

Distressed concrete containing coarse aggregate produced from the Rapid member of the Devonian Cedar Valley formation in the Glory Quarry (Black Hawk County, Iowa) was characterized by dark colored reaction shells in the carbonate particles. Subsequent petrographic (Lemish et al., 1958) and chemical studies (Bisque and Lemish, 1958) demonstrated that these reaction shells result from a selective silicification of the argillaceous dolomitic rocks. Rocks which have a high carbonate and low insoluble residue content (i.e., pure limestones) were not affected. Reaction shells were grown in the laboratory by soaking concrete bars made with reactive aggregates in water at  $125^{\circ}$  F. and also by alternate wetting and drying at  $125^{\circ}$  F. Reaction shells similar to those in distressed concrete were developed in three weeks by either method in argillaceous dolomitic aggregates but not in aggregates consisting of pure limestones. Autoclave treatment of the concrete bars was attempted to determine if reaction shells could be grown more rapidly.

## METHOD

Concrete bars (2 in. x 2 in. x 14 in.) were prepared in the laboratory of the Iowa State Highway Commission according to the following proportions:

Cement (Penn Dixie, Type 1).....	750 gms.
Graded Ottawa sand .....	2,063 gms.
Water .....	400 gms. $\pm$ 50 cc.

Two types of coarse aggregate, acceptable and unacceptable, differentiated on the basis of available service records were used. Argillaceous dolomitic rock from the Rapid member of the Glory quarry was used for the unacceptable aggregate and pure limestone from the Solon member of the Devonian Cedar Valley formation in the Burton Avenue quarry was used for the acceptable aggregate.

The autoclave was the same type required by the ASTM Test C-151-58 entitled "Method of Test for Autoclave Expansion of Portland Cement" (ASTM, 1958). The bars were treated in steam at a pressure of  $295 \pm 10$  lbs. for three hours at a temperature of  $420^\circ \pm 3^\circ$  F. Successive autoclave runs of three hours each were made, and Figure 1 shows the progressive development of reaction shells in argillaceous dolomitic aggregate of a concrete bar which has been cut to expose the aggregate and etched in 3N hydrochloric acid. Figure 2 shows the results of similar treatment of bars made with non-reactive limestone from the Solon member.

Autoclave tests were carried out on bars containing both types of aggregate to see if any expansion occurred. Bars from both types of aggregates showed the same amount of growth (0.1 percent) after three autoclave runs.

#### SUMMARY

If the growth of the reaction shells is considered to be deleterious to the strength of concrete, a rapid method of identifying the susceptibility of carbonate aggregates to this type of silicification has been defined. Although the method does not simulate natural conditions it does serve to differentiate the susceptibility to "silicification."

The expansion of the autoclaved bars was not considered excessive for the length of time the bars were treated. Neat cement paste bars made from the same Penn-Dixie cement used in the concrete bars and treated in the autoclave under the same conditions (ASTM Test C-151-58) showed the same amount of growth. No warping of the concrete bars was noted after nine hours of treatment.

Accelerated hydration of the cement in the matrix which generally results from autoclave treatment did not "fix" the available silica in the concrete matrix of the bars. Enough silica was available to silicify the reactive aggregates.

#### ACKNOWLEDGMENTS

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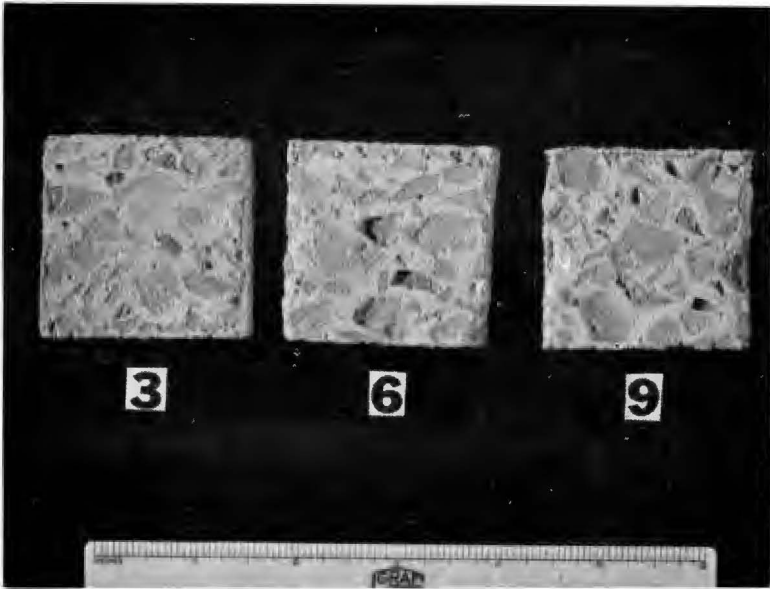


Figure 1. Acid etched slices of a concrete bar showing the progressive growth of reaction shells on argillaceous dolomitic aggregate after 3, 6, and 9 hours of autoclave treatment.

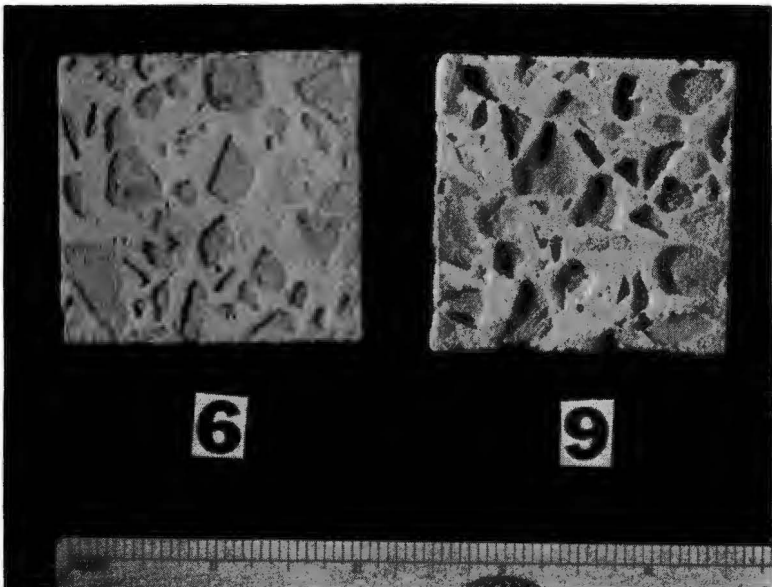


Figure 2. Acid etched slices of a concrete bar showing the lack of reactive shells on pure limestone aggregates after 6 and 9 hours of autoclave treatment.

Johnson, Laboratory Chief, and Laverne E. Huckstadt are thanked for their assistance and cooperation. Mr. Bert Myers, materials engineer, and Mr. Ted Welp, geologist, of the Iowa State Highway Commission, suggested the use of the autoclave.

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**Method Referred To**

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