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Evaluation of Autoclave Induced Expansion of Some Iowa Carbonate Rocks

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Evaluation of Autoclave Induced Expansion of Some Iowa Carbonate Rocks[1]

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Abstract. An autoclave environment of 110°C and a steam pressure of 25 psi. was used to develop a rapid method of determining the expansive characteristics of some carbonate rocks. The current procedure for such determinations requires several weeks treatment of the aggregate sample in a 1N NaOH solution at room conditions.

Application of the autoclave method indicates that the rate of expansion is greatly increased but that the actual mechanism of expansion is not changed. Only those samples placed in an alkaline solution in the autoclave expanded. Control samples in distilled water showed negligible expansion.

The expansion data obtained by the autoclave method is correlative with that obtained by standard procedures, but the test time required is considerably less. Therefore, this rapid autoclave method is suggested as a valid and applicable test in the study of carbonate aggregates.

Previous research, Swenson and Gillott (1960)), Hadley (1961), Newlon and Sherwood (1962), and Lemish and Moore (1964), has indicated that the deleterious behavior of some carbonate rocks, when used as coarse aggregate in highway concrete is related to the expansive character of the rock. The expansive behavior of these rocks, which are highly reactive in an alkaline environment, has been related to growth of concrete by Swenson and Gillot (1960) and Newlon and Sherwood (1962).

The work of Sherwood and Gillot (1960), which initiated research of the carbonate aggregate expansion phenomena, was followed by contributions from Hadley (1961) and Newlon and Sherwood (1962). In 1963 Moore (unpublished MS thesis, Iowa State University) presented an improved sample preparation technique, whereby cylindrical-shaped cores two inches long and one-half inch in diameter were used. In all expansion studies to date, the experimental procedure has been to store the rock sample in a sodium hydroxide solution at room temperature for a length of time in excess of 150 days. Periodically the total length of the sample is measured and the percentage length-change determined.

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An autoclave environment is proposed in an attempt to develop a rapid method of testing carbonate aggregates for excessive expansive behavior. This paper is presented to evaluate the validity of autoclave induced expansion as compared to expansion obtained at room temperature.

Method

Twelve different rocks from quarries in eastern Iowa, with known service records and whose expansion behavior at room temperature has been previously studied, were used in this study. The carbonate aggregate cores were cored normal to the bedding from rock slabs two inches thick with a diamond bit, press drill. The cores were tapered on a carborundum grinder to a 45° taper to fit micrometer end plugs, washed in distilled water, and placed in a NaOH solution or distilled water. Six cores from each different rock were used for the actual expansion determinations in NaOH, three being kept in the autoclave environment and three at room temperatures. Control cores in distilled water were also kept in each environment.

For each expansion change determination, four micrometer readings were taken by rotating the core through 90° steps in the micrometer and averaging the four readings. The autoclave conditions were maintained at 110°C. and 25 psi. throughout the study. Expansion measurements were made with a bench-mounted micrometer with end plugs which accepted a 45° taper. The micrometer was read to 0.001″ and estimated to 0.0001″.

Cores with the same letter designation are all from the same rock slab.

Results

Figure 1 shows the effects of autoclave expansion of cores in a NaOH solution versus distilled water. The rate and degree of expansion of autoclaved cores in NaOH are several times greater than that of cores reacted in NaOH at room temperature. However, the increased expansion is absent in autoclaved control cores in distilled water. This phenomenon was observed in all rocks studied, and implies that the alkaline solution is a necessary component of the autoclave induced expansion and that the elevated temperature alone is insufficient.

The greatest expansion observed in any autoclaved control core in distilled water was 0.04% by core 9GL16, figure 1. Since cores in distilled water at room temperature contract less than —0.05%, the maximum amount of expansion attributable to the autoclave environment would be less than 0.1%. However, this

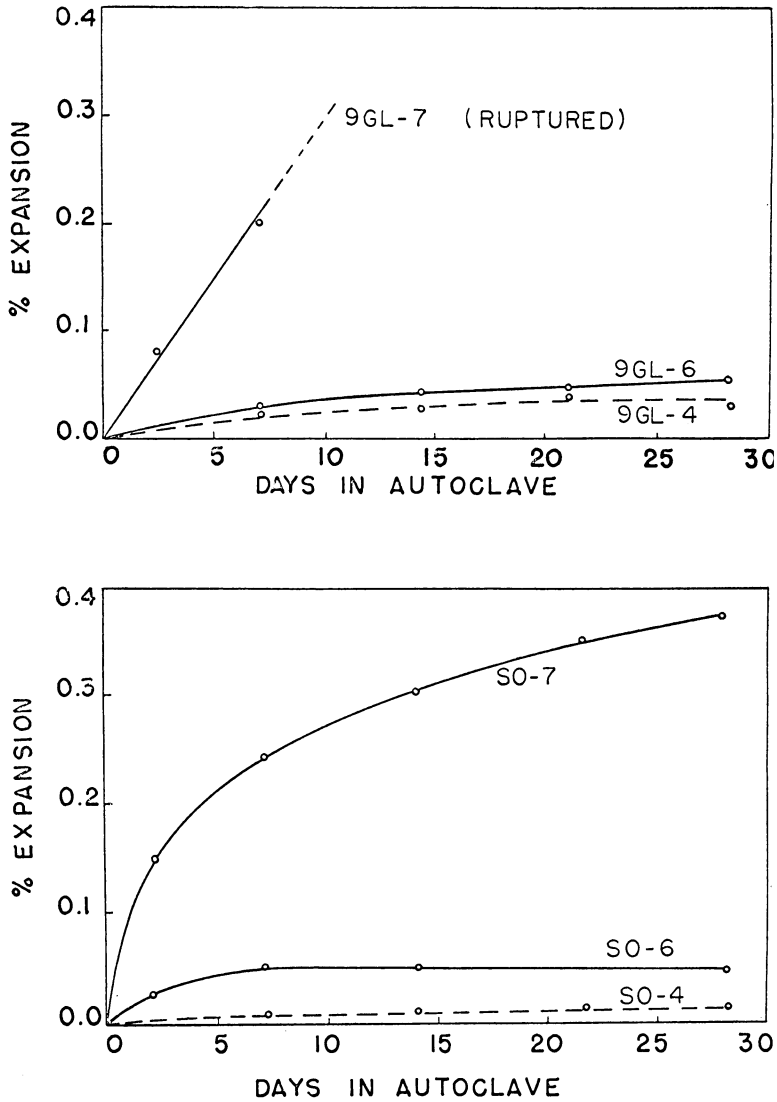

Figure 1. Comparison of autoclave induced expansion rates of samples 9GL-7 and S0-7 in NaOH, and samples 9GL-6 and S0-6 in distilled water. Samples 9GL-4 and S0-4 were kept in NaOH at room temperatures.

amount is observed only in highly expansive rocks. A rock which contracts in NaOH at room temperature will expand less than 0.01% when autoclaved in distilled water.

The autoclave induced expansion is particularly useful for expansive rocks such as 9GL. Figure 1 shows that after 21 days the autoclaved core (9GL-7) has ruptured, whereas, the core

treated at room temperature (9GL-4) has expanded less than 0.05%. However, after several weeks reaction, core 9GL-4 will have expanded greater than 0.5%. This trend was observed for all rocks used in this study, i.e., the autoclave induced expansion proceeded at an accelerated rate compared to expansion at room temperature.

To further demonstrate the applicability of the rapid autoclave method and to establish its independence of increased thermal conditions, control core 9GL-6 (discussed above) was

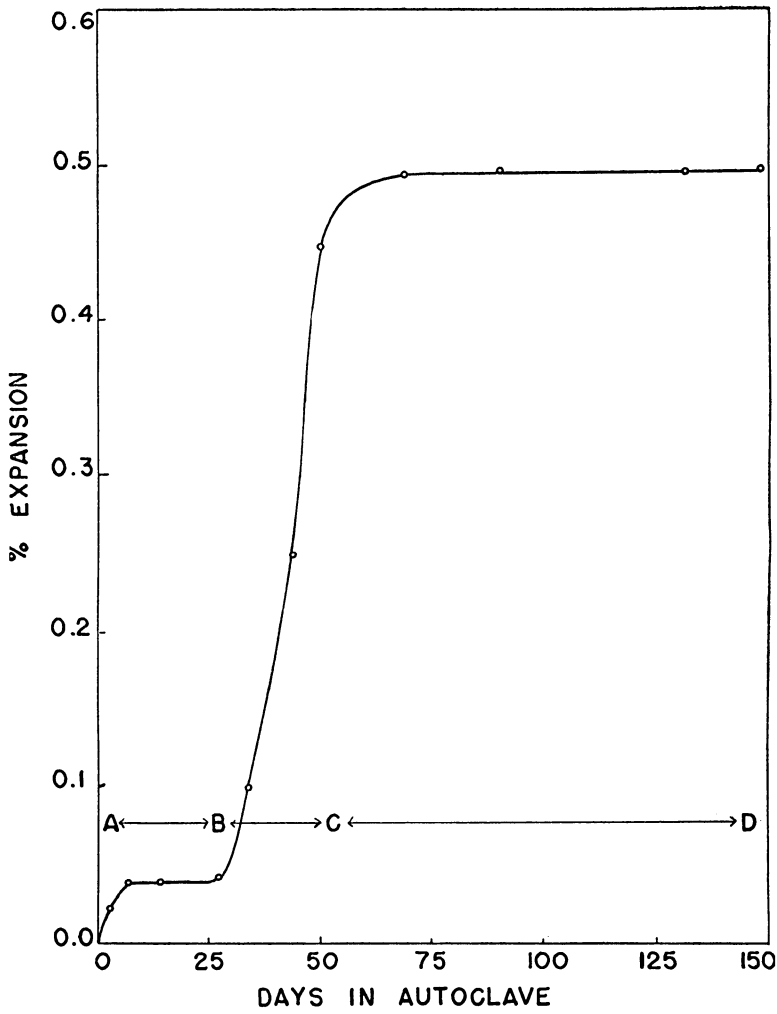

Figure 2. Effect of alternating an autoclaved expansive rock from distilled water to a NaOH solution and back to distilled water.

removed from the distilled water and placed in a 1N NaOH solution at the end of 28 days. It was then autoclaved for 21 days, at which time it was placed in distilled water and autoclaved for approximately 100 days. The graph in figure 2 shows the effect of alternating the reaction solution of a rock in an autoclave environment. The slope of curve segment BC in figure 2 is similar to that of sample 9GL-7 in figure 1 (NaOH was used in both cases) whereas, segment CD can be considered as merely an elevated extension of the original 28-day curve segment AB (reacted in distilled water).

After approximately 3 weeks of autoclaving in a NaOH solution, all rocks studied could be placed into one of three characteristic expansion categories as shown in figure 3. These three categories can be correlated with Lemish and Moore's (1964) expansion categories determined at room temperatures after reaction for 150 days. Table 1 shows a direct comparison of the categories determined by the rapid autoclave method with the categories determined at room temperatures. Table 2 lists the percentage expansion observed in autoclaved cores and room temperature cores after 21 days reaction in NaOH.

Summary

Though objection may be raised as to the validity of considering autoclave induced expansion in a study of carbonate aggregates it has been shown that the autoclave environment

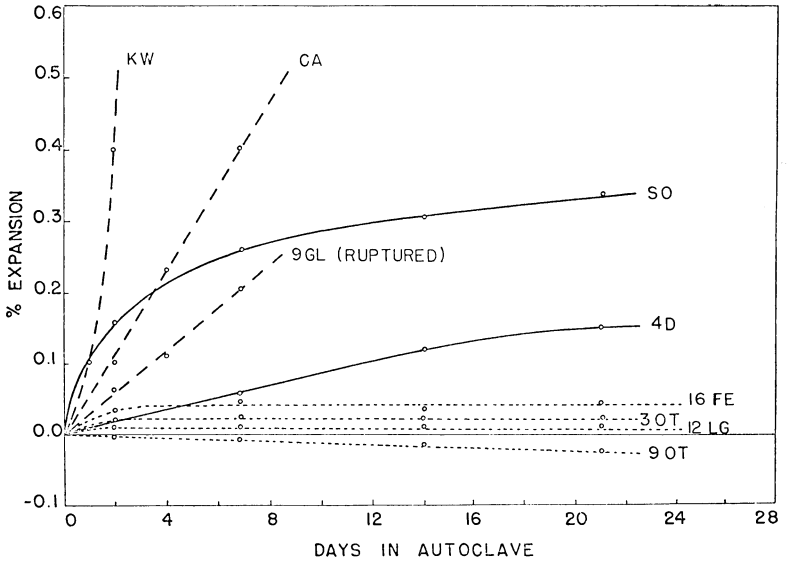

Figure 3. Three categories of autoclave induced expansion: dashed curves, Category A; solid curves, Category B; dotted curves, Category C.

alone will not cause excessive expansion. This indicates that the actual expansion mechanism is independent of the autoclave environment and only the rate of expansion is dependent upon these conditions. Therefore, even though this environment is not typical of that found in highway concrete, its use is applicable in inducing the expansion phenomenon.

The advantage of the rapid autoclave method of determining aggregate expansion is that the highly expansive rocks (greater than 0.5%) can be detected in a few days rather than requiring several weeks at room temperatures. These highly expansive rocks will expand at a constant rate in excess of 1.0% of their total length or rupture within one week.

From the evidence cited and the ability to correlate autoclave induced expansion with that resulting from room temperatures,

Table 1. Categories of autoclave induced expansion as correlated with expansion induced at room conditions.

Expansion Categories after 21 days in NaOH and the autoclave	Expansion Categories after 150 days in NaOH at room conditions
Category A: Samples which expanded greater than 0.5% or which ruptured	Category I: Samples which expanded in excess of 0.5%
Category B: Samples which expanded between 0.5% and 0.15% at a constant rate	Category II: Samples which expanded between 0.0% and 0.5%
Category C: Samples which expanded less than 0.15% with approximately 75% of the expansion occurring within 7 days	Category III: Samples which contracted prior to expanding
	Category IV: Samples which contracted readily

Table 2. Autoclave induced expansion versus room temperature expansion of some carbonate rocks after 21 days in NaOH.

Sample	Room Temperature	Autoclave
Category A:		
KW	0.8	Ruptured
GL	0.05	Ruptured
Category B:		
S0	0.06	0.37
4D	0.05	0.20
Category C:		
12D0	−0.14	0.12
2FE	−0.05	0.10
13FE	−0.08	0.10
16FE	−0.07	0.04
1AL	−0.09	0.10
30T	−0.04	0.03
50T	−0.14	0.08
90T	−0.04	−0.01
12LG	−0.04	0.04

it is suggested that this method is valid and can be considered applicable to the study of carbonate aggregates. Additional experience on a larger variety of rocks of known expansion behavior would be of value, especially the delayed expanding types of carbonate aggregates reported by Dolar-Montauani (1964).

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