

A NEW, NOVEL WELL-CEMENTING POLYMER-CONCRETE COMPOSITE

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Synopsis: The feasibility of using the products of free-radical copolymerization of cyclic and linear organosiloxanes in the formation of polymer concrete (PC) composites for use in the completion of geothermal wells has been demonstrated. The PC contained a mixture of tetramethylvinylcyclotetrasiloxane and polydimethylsiloxane used in conjunction with aggregate materials such as silica flour and portland cement. The use of these compounds resulted in composites with high strength and with thermal and hydrolytic stability.

Thermogravimetric analyses and compression strength tests at elevated temperatures have been used to determine the thermal stability of the composites. The results from these studies indicate that over the temperature range 25° to 350°C, the compressive strength is essentially constant at a value of ~72 MPa and there is also a relatively low weight loss of polymer (~1.0 wt%).

The hydrolytic stability of the composites was determined by using infrared spectroscopy on a variety of free and bonded OH functional groups before and after the samples were exposed to a 25% brine solution at 300°C. These results showed that the inclusion of various additives such as Ca or Mg compounds in the inorganic phase affects the hydrothermal stability.

Pumpability tests were also performed, and the results indicated that a PC slurry containing 35.5 wt% organosiloxane mixed with 64.5 wt% silica flour and cement as an aggregate did not change viscosity at temperatures of 150° to 165°C and a pressure of 36.5 MPa for at least 4.5 hr. Increasing the temperature to 205°C resulted in increased viscosity after 4 hr. The results from these studies indicated that this system can be used as a geothermal well-completion material.

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Neal R. Carciello is a Chemistry Associate in the Geothermal Materials Group, Process Sciences Division, Department of Energy and Environment at Brookhaven National Laboratory. He received his A.A.S. at the State University of New York at Farmingdale in 1961. Present research is in the field of polymer chemistry with the objective to find a high temperature polymer system that can be used to cement geothermal wells.

INTRODUCTION

The availability of durable and economic construction materials for handling hot brine and steam is a serious problem in the development of geothermal energy. To date, corrosion and scale incrustations have been encountered in all geothermal plants and, to various degrees, have adversely affected plant life times and power output (1).

The durability of the cementing materials used in the completion of geothermal wells is often the controlling factor in the operating life of a geothermal well. Therefore, the economics of geothermal power could be improved significantly if better well-cementing materials were developed.

In an attempt to solve this problem, a research program to develop high-temperature polymer cements (PC) for use in well-completion systems has been in progress at Brookhaven National Laboratory since 1976. The goals of the program are to develop thermally and hydrolytically stable PC systems with low permeability (<0.1 millidarcy) to water, and having minimum compressive strengths and shear bond strengths to steel well casing of 6.9 MPa and 69 kPa, respectively, at an age of 24 hr.

PC consists of a solid phase filler mixed with a liquid phase binder. After curing, high-strength durable composites are produced. To date, polymer binding materials such as polyester, polyurethane, polymethylmethacrylate, and polystyrene have generally been used in PC (2-5). Copolymers formed from mixtures of monomers such as styrene, acrylonitrile, acrylamide, divinylbenzene, and trimethylolpropane trimethacrylate have also been used.

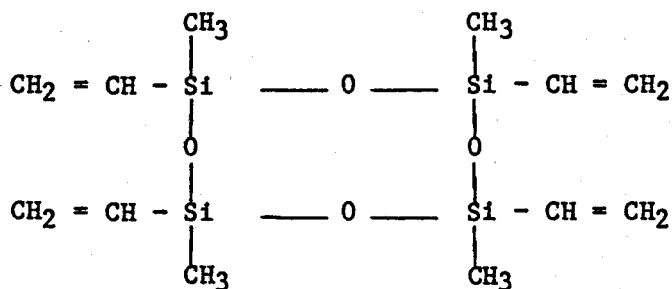
The network of all these components consists of C-C bonds. Since cleavage of these bonds occurs at temperatures near 250°C, the use of organic binders in PC is limited to temperatures below 250°C. Above this temperature the specimens become soft and crack or swell because of the evolution of gas.

In this paper, experiments with semi-inorganic polymer compositions (siloxanes) are described. These materials contain a network of Si-O-Si bonds. The dissociation energy for these bonds at 25°C is 190.9 ± 2 kcal/mol compared with 145.5 kcal/mol for C-C bonds. In addition, the molecular chains of siloxanes are more rigid than those of organic polymers (6).

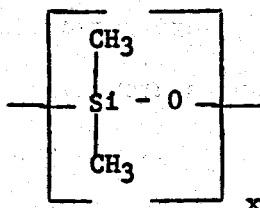
CHEMICAL SYSTEMS

Siloxanes are a class of high-temperature polymers that appear to have important chemical and physical properties which can be utilized in PCs for geothermal environments.

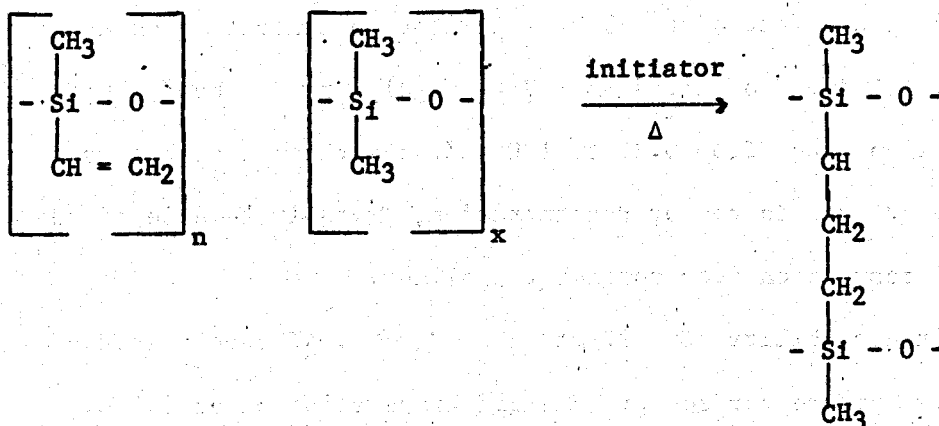
Mixtures of siloxanes having unsaturated and saturated organic groups in their molecular chains were evaluated. One material, tetramethyltetravinylcyclotetrasiloxane (RZ), has a cyclic structure and contains one vinyl group for each silicon unit. This structure is shown below.



The second monomer used in this study was polydimethylsiloxane (V-47). Its structure is as follows:



The cross-linking of vinyl-substituted cyclic siloxanes with V-47 occurs through the vinyl and methyl groups via the formation of a trimethylene bridge as shown below (6).



Polymerization of the materials was initiated using di-tert-butyl peroxide (DTBP).

EXPERIMENTAL RESULTS

A mixture of silica flour sand and portland cement was selected as the filler material for the organosiloxane polymer concrete (OSPC). Procedures for mixing and placement of the OSPC and the test methods for property measurements were published earlier (4).

Optimization of Monomer Composition

Various copolymers were made and tested for thermal stability. Thermogravimetric analysis measurements were performed in nitrogen gas at a heating rate of 10°C/min and the results are given in Figures 1 and 2. Infrared analysis data are given in Figure 3. As can be seen in Figure 1, when the cyclic siloxane RZ was mixed with the linear siloxane V-47, maximum stability occurred at an RZ concentration in the range of 97 wt%. All data points on the curves represent the averages from four test samples.

In Figure 2, the effect of the DTBP initiator concentration on the thermal stability at 300°C of three RZ - V-47 copolymers is shown. Over a DTBP concentration range from 0.25 to 1.0 wt%, the thermal stability increased with increased initiator concentration, probably because of the higher degree of conversion from monomer to polymer.

The hydrolytic stability of a 97 wt% RZ - 3 wt% V-47 copolymer was determined after exposure for 360 hr to a 25% brine solution at 250°C. No appreciable weight loss or deterioration was noted. Infrared (IR) analyses before and after exposure (Figure 3) showed the formation of free OH groups, indicating some hydrolysis. IR peaks at 1650 to 1600 cm^{-1} which correspond to CH groups, decreased after exposure to brine. Both changes were very small and can be reduced essentially to zero during the formation of OSPC.

Optimization of Filler Composition

Sand Size--It was shown earlier (7) that the particle size of sand has an effect on the properties of PC and is dependent upon the viscosity of the monomer composition. For organic binders with viscosities close

to the viscosity of water, the best results were obtained using a mixture of sand sizes (3,7). In these cases, the low-viscosity monomers could easily be mixed with the filler to wet the particles and to fill the voids in the aggregate.

It has also been shown (8) that as the viscosity of the monomer mixture is increased, the percentage of filler that the monomer wets decreases. This results in nonuniform polymer loading which decreases the PC strength and durability.

Similar studies have been performed with organosiloxane materials (7). These results indicated that maximum strength was obtained when silica flour sand passing a 30- μ m sieve was used in conjunction with portland cement.

Cement Type--It is well known that the inclusion of portland cement in PC improves the strength and durability. As a result, studies were performed to determine the effects of concentration and cement type on the properties of OSPC.

Four commercially available cements were tested as partial constituents of the aggregate system for OSPC. The compositions of these cements are given in Table 1.

The properties of OSPC as a function of cement type are summarized in Table 2 and Figure 4. The results indicated that OSPC samples containing Type II portland cement had the highest compressive strength and that a cement - silica flour sand ratio in the 9:1 range was optimum. Stress-strain curves (Figure 5) indicated that OSPC samples containing Type I or II cement were more brittle than specimens containing Type III or V. The reason for these variations may be the differences in the chemical composition of the cements.

Figures 6 to 9 summarize test data from OSPC after exposure in brine at 300°C for 60 days. Samples containing silica sand and Type III cement mixed in a ratio of 9:1 had the highest strength and lowest water absorption.

Sand-Cement Ratio--Studies were performed to determine the optimum ratio of sand to cement that should be used in the formation of OSPC composites. The results from compressive strength tests made before and after exposure of samples to brine at temperatures of 275° and 300°C are summarized in Table 3 and Figure 10. The data indicated that OSPCs containing cement up to a concentration of ~15% are stable after exposure to 275°C brine. At 300°C, only samples containing up to 10% cement were stable.

Electron microscope techniques were used to study the fracture surfaces of the OSPC before and after brine exposure. No changes in structure or composition were noted. OSPC samples after exposure for 30 days to 300°C brine are shown in Figure 11.

Strength test results from OSPC samples as a function of time, cement type, and cement content, before and after 300°C brine exposure, are summarized on Figure 12. The results indicated that a system consisting of 97 wt% RZ - 3 wt% V-47, in conjunction with silica flour - type III portland cement in the ratio of 9:1, had the greatest hydrothermal stability.

Additives--Based upon the results from the studies described above, work was performed to make further improvements in the mechanical properties of OSPC and to reduce the small amount of hydrolysis that occurred. In this work, various additives were included in the inorganic

phase and their effects on the properties determined. These results, given in Figure 13, indicate that significant improvements in the hydrothermal stability can be obtained by the addition of $MgSiO_3$, $CaSiO_3$, and Fe_2O_3 .

WELL-CEMENTING PROPERTIES

In addition to strength and durability, there are other equally important properties that a well cement must exhibit. These include low permeability, adequate bond strength to steel casing, and a curing rate which allows the cement to be pumped into a well. Tests to measure these characteristics were performed.

Permeability--The OSPC formulation given below was selected for use in permeability tests:

37 wt% monomer (97 wt% RZ - 3 wt% V-47) - 63 wt% filler
(90 wt% silica sand - 10 wt% Type III portland cement).

The tests were conducted in accordance with the American Petroleum Institute's (API) recommended practice for testing oil-well cements and cement additives (9). Samples were evaluated before and after exposure to brine at 300°C for 120 days. After 48 hr in the permeability test equipment with a pressure differential of 344.5 kPa, no water penetration was measured. Earlier data for organic PCs indicated permeabilities in the range 0.001 to 0.02 millidarcy.

Bond Strength to Steel--Currently there are no ASTM standards for measuring the shear bond strength of PC materials. The procedure used at BNL was as follows:

The mold for preparing the bonding strength samples consisted of a base made of wood, carbon steel pipe measuring 42 mm x 100 mm long, and a glass tube measuring 58 mm x 100 mm. The steel pipe was placed inside the glass tube and both were sealed to the base with silicone rubber cement. PC slurry was poured into the annulus between the glass and the steel. The filled mold was placed in an oven at the required curing temperature to form PC composite bonded to the outer surface of the pipe.

The bonding strength test was performed by placing a metal ring on the PC portion of the test sample and applying a load on the pipe in a compression tester. The load was recorded and the stress was calculated by taking the area of the pipe surface that was covered with PC and dividing it into the load.

Bond strengths for the OSPC ranged between 150 and 200 kPa, values greater than those specified by the design criteria, but considerably below the 3,000 to 4,000 kPa normally exhibited by organic PC.

Pumpability Tests--Two types of tests were performed to determine the pumpability of the OSPC formulation. Before initiating these tests, work was conducted to reduce the viscosity of the formulation to a value of 20 to 40 units of consistency (cP), the approximate initial viscosity of inorganic cement slurries used in the completion of oil and geothermal wells. This was accomplished by increasing the monomer concentration in the OSPC formulation to 35.5 wt%. To meet the pumpability requirements, it is essential that the viscosity of the slurry remain <100 units of consistency for ~4 hr at the placement temperature and pressure conditions.

Preliminary tests were performed in a pressure vessel equipped with a stirrer, to obtain data on the curing time as a function of pressure and temperature. These results indicated little change in viscosity after 4 hr at 149°C and 68 MPa.

On the basis of these data, API pumpability tests were performed using a high-pressure consistometer at the Dowell Division of Dow Chemical U.S.A. The results from these tests, given in Figures 14 and 15, indicated that without an initiator the viscosity remained constant at temperatures in the range 149° to 163°C and a pressure of 36.5 MPa for at least 4.5 hr. Increasing the temperature to 204°C resulted in a viscosity change after 4 hr.

CONCLUSIONS

A number of monomers, organic and semi-inorganic, in conjunction with varying types of cement and sand particle sizes have been studied for their potential as geothermal well cementing materials. The goal of this study was to develop thermally and chemically stable composites that can withstand highly reactive geothermal environments and are also pumpable. Two years of laboratory study indicate that PC consisting of a mixture of high molecular weight siloxane copolymer as a binder and silica sand - cement mixtures as a filler, meet the design criteria.

Major emphasis in this study was to determine the monomer ratio based on the reactivity of each component in the monomer mixture as well as the sand - cement ratio. The objective of these experiments was to optimize the conditions when the combination of polymer and filler forms thermally and hydrolytically stable composites with three-dimensional structures.

From the laboratory evaluation, an inorganic PC formulation based on 97 wt% RZ - 3 wt% V-47, in combination with a mixture of sand flour and cement at a ratio of 9:1, appears suitable for use as a well-cementing material for high-temperature applications (~300°C). Continued evaluation is in progress at the National Bureau of Standards.

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Table 1

Compound Composition of Portland Cements

Compound Composition, wt%

Cement	Compound Composition, wt%						Loss on ignition, %	Calculated compounds ^a			
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃		C ₃ S	C ₂ S	C ₃ A	C ₄ AF
Type I	19.9	7.31	2.72	62.8	2.9	3.0	1.3	43.9	22.4	14.4	8.2
Type II	21.3	4.18	3.43	61.8	4.2	2.5	1.5	49.4	23.9	5.3	10.4
Type III	19.1	7.11	2.65	61.8	2.8	3.7	1.4	44.7	20.8	4.4	8.1
Type V	23.4	3.40	3.45	64.4	1.6	1.9	1.4	51.6	27.0	3.2	10.5

^aIn the abbreviated formula, C = CaO; S = SiO₂; A = Al₂O₃; F = Fe₂O₃.

Table 2

Properties of OSPC as a Function of Cement Type

Aggregate composition, wt%	Cement	Cement type	Boiling H ₂ O absorption, %	Compressive strength, MPa	Modulus of elasticity, MPa
95	5	I	0.06	81	—
		II	0.09	111.2	—
		III	0.1	70.5	—
		V	0.09	49.1	—
90	10	I	0.07	106.9	6826
		II	0.08	101.6	—
		III	0.05	52.9	6791
		V	0.11	74.39	7526
85	15	I	0.11	86.1	—
		II	0.08	102.9	—
		III	0.08	72.2	—
		V	0.13	70.85	—
80	20	I	0.22	47.5	—
		II	0.12	71.6	—
		III	0.19	72.7	—
		V	0.14	68.2	—

^a, Particle size, sieve opening <30 μ m
 Monomer composition, 97 wt% RZ - 3.0 wt% V-47
 Curing agent, 1.0 wt% di-tert-butyl peroxide
 Curing condition, 110°C 18 hr; 135°C 3 hr; 170°C 2 hr
 Monomer concentration, 35 wt%

Table 3

Sand-Cement Ratio for System RZ - V-47 (Ratio 97:3)

Aggregate ratio, wt%		Boiling water absorption, wt%	Compressive strength, MPa, after 30 days			Modulus of elasticity after exposure to hot brine for 30 days at 300°C
Sand ^a	Cement ^b		boiling water	in autoclave at 275°C	in autoclave at 300°C	
100	0	0.02	49.9	48.3	—	
95	5	0.08	74.2	55.9	79.6	
90	10	0.07	90.2	69.7	57.1	
85	15	0.04	36.2	40.6	—	
80	20	0.54	15.2	weak	—	
70	30	0.69	34.2	weak	—	
95 ^c	5	0.09	89.1	48.5	60.9	
90 ^c	10	0.12	46.4	107.2	62.8	

Initiator 1/2 wt% DTBP; 1/2 wt% Silane A-174

Curing condition: 125°C, 16 hr;
150°C, 3 hr;
180°C, 3 hr.

^aParticle size, sieve opening <30µm.

^bPortland cement Type III.

^cThese samples were made by adding a wetting agent.
Monomer concentration, 35 wt%.

FIGURE CAPTIONS

- Figure 1. The effect of various ratios of RZ and V-47 organosiloxanes on the thermal stability of the polymer at 300°C.
- Figure 2. The effect of initiator concentration on the thermal stability of RZ and V-47 organosiloxanes at 300°C.
- Figure 3. Infrared spectra of polymer before and after exposure to 250°C brine. Organosiloxane mixture of 97 wt% RZ - 3 wt% V-47. a. after exposure; b. before exposure.
- Figure 4. Compressive strength and boiling water absorption as a function of cement type. Monomer composition, 97 wt% RZ - 3 wt% V-47. Sand-cement ratio: (1) 95:5; (2) 90:10; (3) 85:15.
- Figure 5. Compressive stress-strain curves for OSPC after preparation as a function of cement type. Monomer composition 97 wt% RZ - 3 wt% V-47. Aggregate composition, 90 wt% flour sand - 10 wt% cement. (1) Type I cement; (2) Type II Cement; (3) Type III Cement; (4) Type V Cement.
- Figure 6. Compressive strength and absorption of OSPC after exposure to brine at 300°C. OS composition, 97% RZ - 3% V-47. Aggregate composition, 95% sand - 5% cement; (I) Type I cement; (II) Type II cement; (III) Type III cement; (V) Type V cement.
- Figure 7. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition, 90% sand - 10% cement; Monomer composition and type of portland cement same as in Figure 6.
- Figure 8. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition 85% sand - 15% cement. Monomer composition and type of portland cement same as in Figure 6.
- Figure 9. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition, 80% sand - 20% cement. Monomer composition and type of portland cement same as in Figure 6.
- Figure 10. Compressive strength as a function of sand-cement ratio. Monomer composition: 97 wt% RZ - 3 wt% V-47; Aggregate composition: sand-sieve opening <30 μ m: cement-type III. (1) after boiling water; (2) after exposure to brine at 275°C; and (3) after exposure to brine at 300°C.
- Figure 11. Organosiloxane PC samples from RZ and V-47 after exposure in brine at 300°C for 30 days.

- Figure 12. Compressive strengths of OSPCs, after exposure to brine at 300°C as a function of cement type and concentration. Summary of results.
- Figure 13. The effects of additives on the hydrolytic stability of OSPCs after exposure to brine at 300°C for 30 days.
- Figure 14. API thickening time test. Monomer composition 97 wt% RZ - 3 wt% V-47; temperature 190°C.
- Figure 15. API thickening time test. Monomer composition 97 wt% RZ - 3 wt% V-47; temperature 204°C.

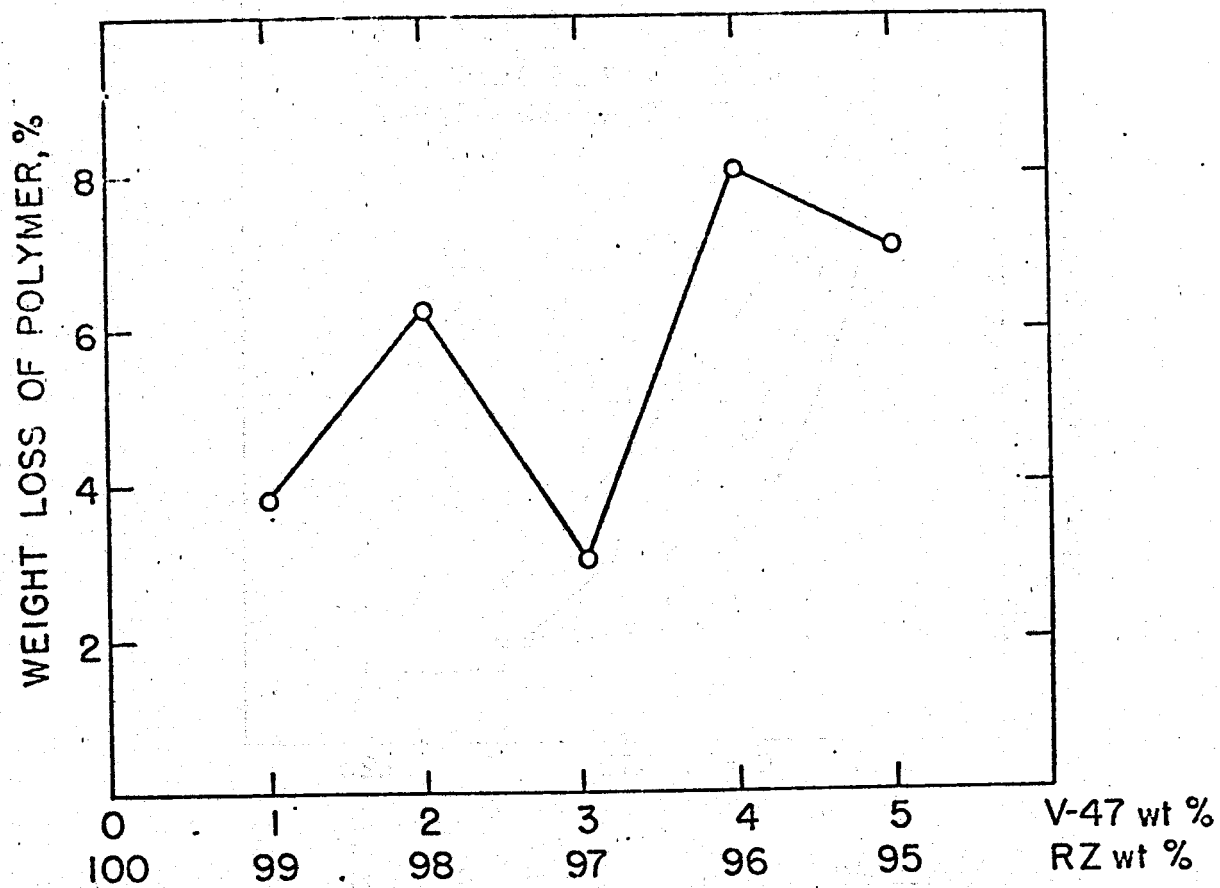


Figure 1. The effect of various ratios of RZ and V-47 organosiloxanes on the thermal stability of the polymer at 300°C.

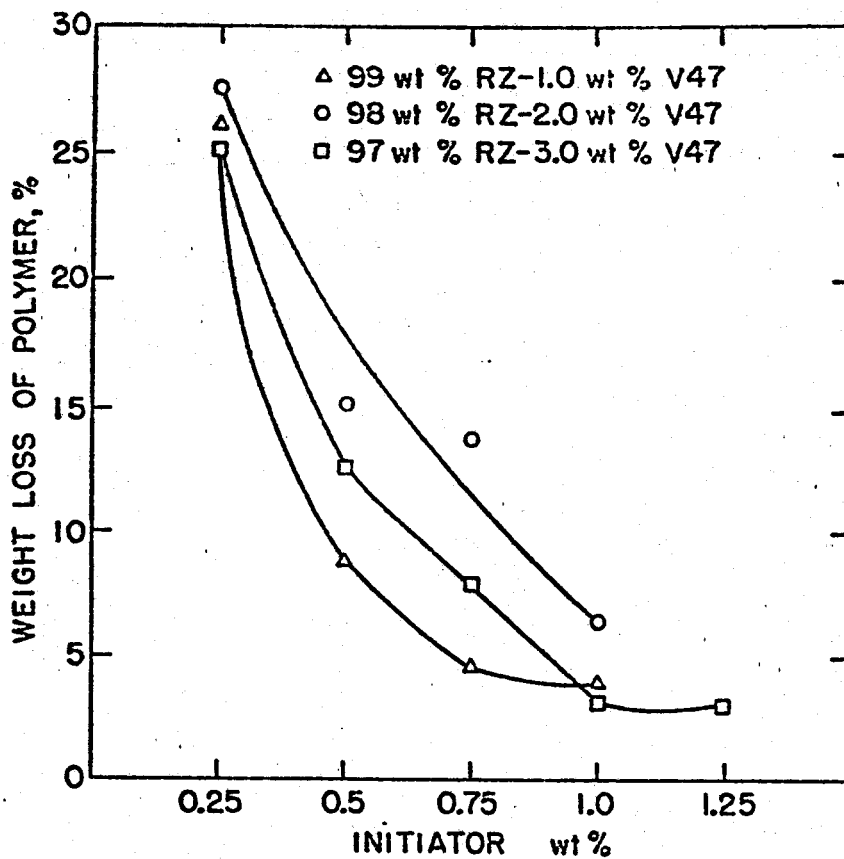


Figure 2. The effect of initiator concentration on the thermal stability of RZ and V-47 organosiloxanes at 300°C.

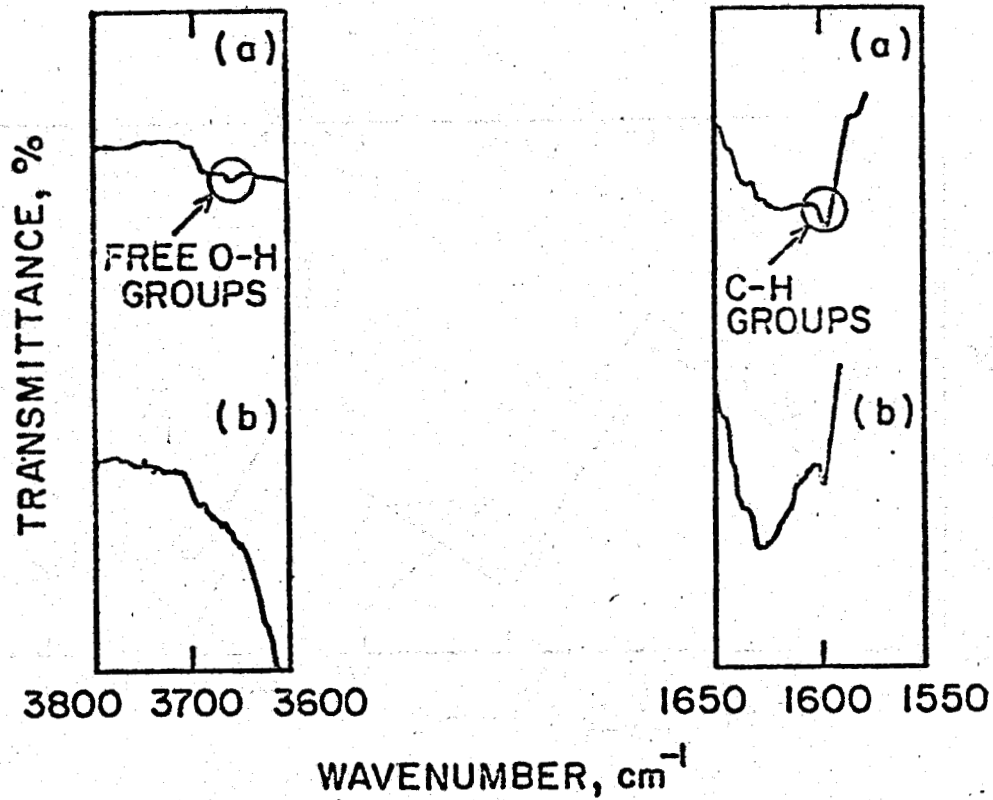


Figure 3. Infrared spectra of polymer before and after exposure to 250°C brine. Organosiloxane mixture of 97 wt% RZ - 3 wt% V-47. a. after exposure; b. before exposure.

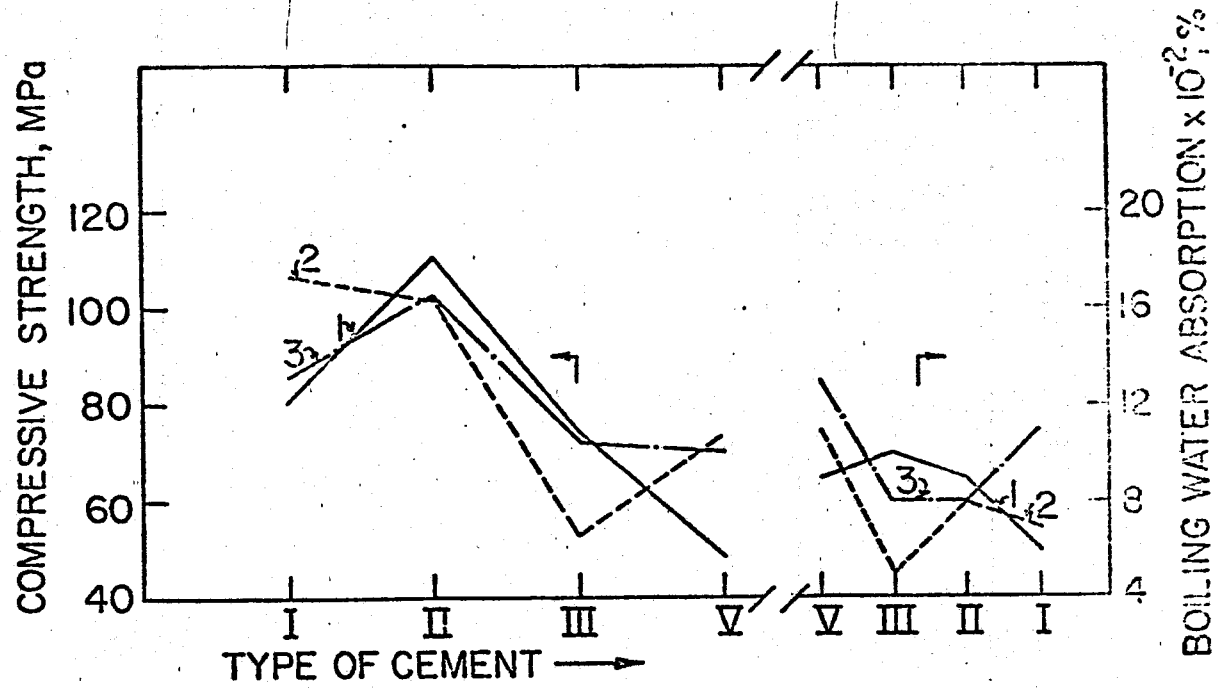


Figure 4. Compressive strength and boiling water absorption as a function of cement type. Monomer composition, 97 wt% RZ - 3 wt% V-47. Sand-cement ratio: (1) 95:5; (2) 90:10; (3) 85:15.

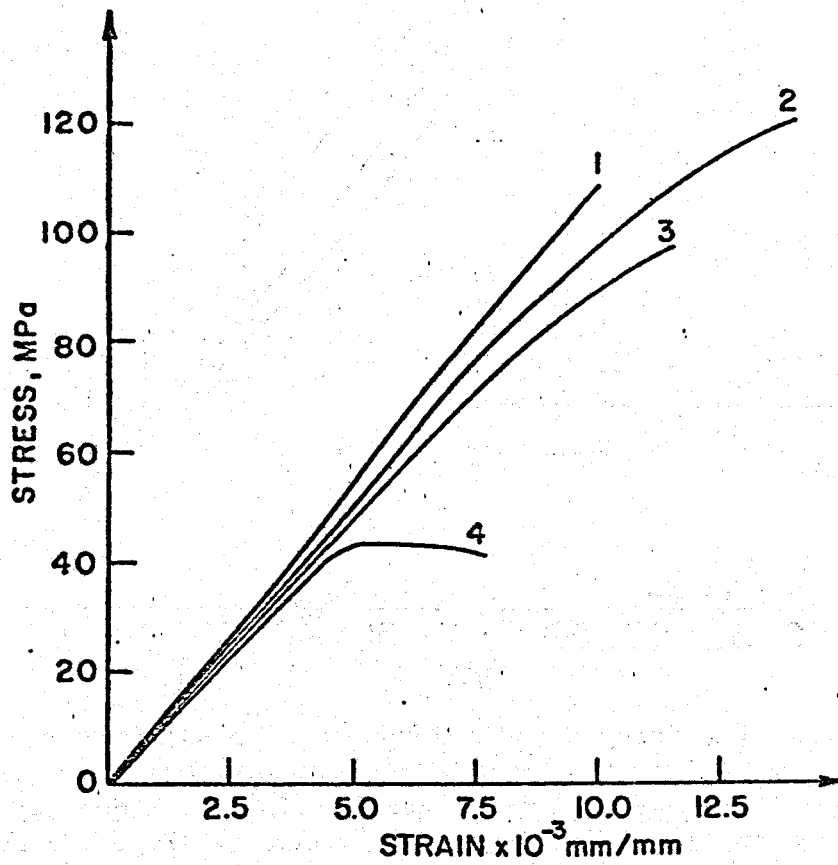


Figure 5. Compressive stress-strain curves for OSPC after preparation as a function of cement type. Monomer composition 97 wt% RZ - 3 wt% V-47. Aggregate composition, 90 wt% flour sand - 10 wt% cement. (1) Type I cement; (2) Type II Cement; (3) Type III Cement; (4) Type V Cement.

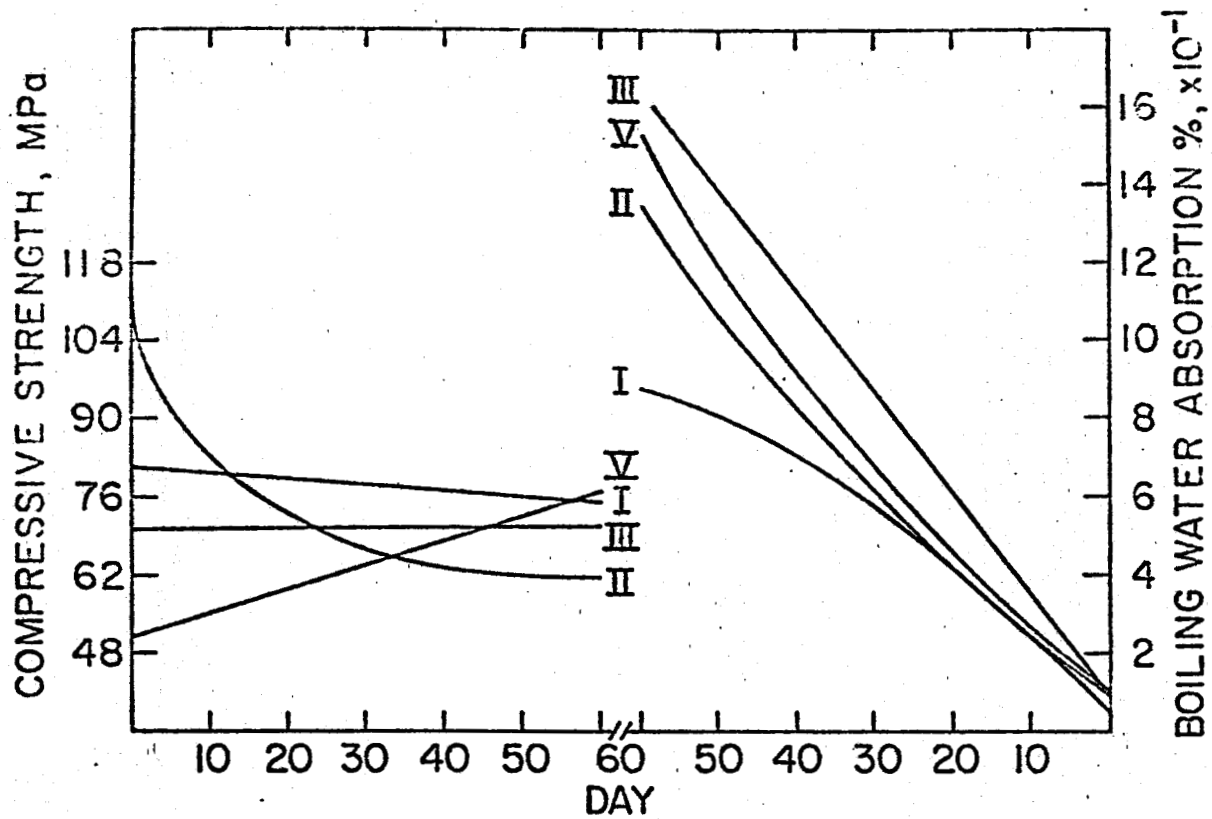


Figure 6. Compressive strength and absorption of OSPC after exposure to brine at 300°C. OS composition, 97% RZ - 3% V-47. Aggregate composition, 95% sand - 5% cement; (I) Type I cement; (II) Type II cement; (III) Type III cement; (V) Type V cement.

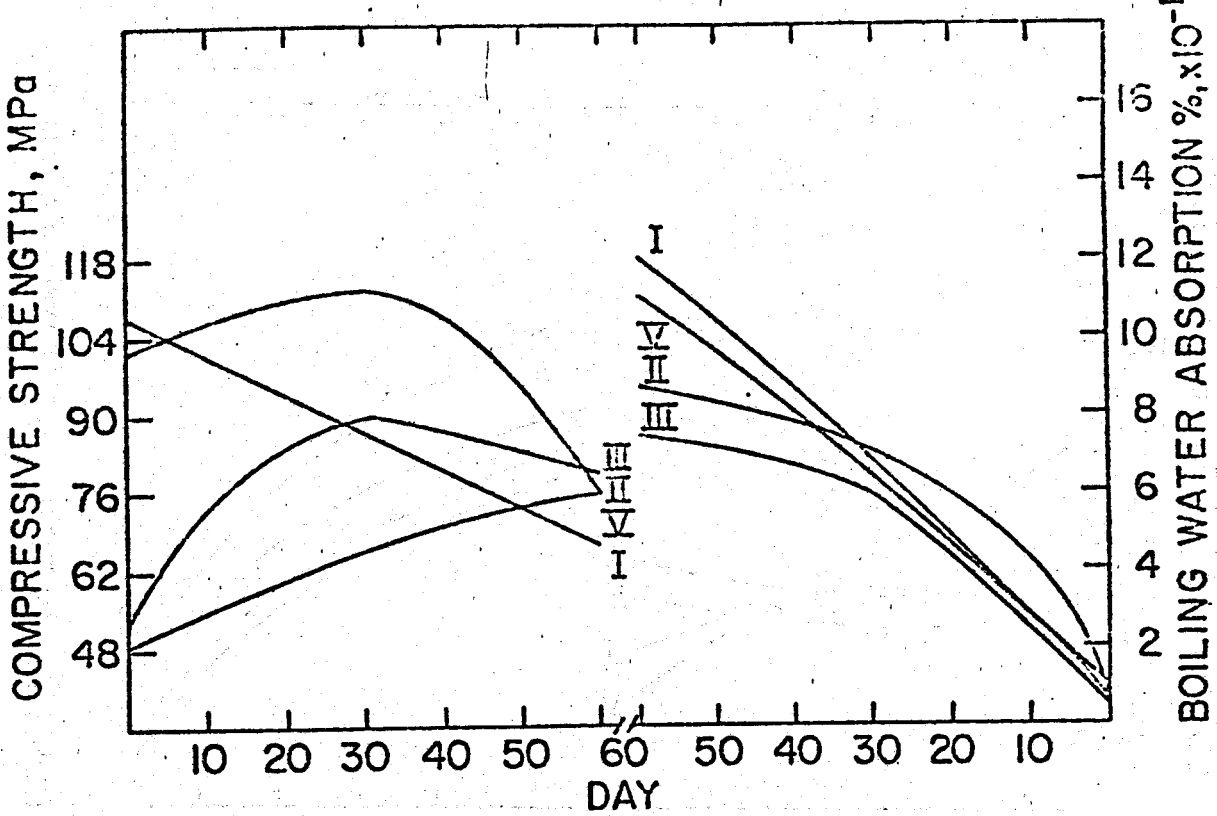


Figure 7. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition, 90% sand - 10% cement; Monomer composition and type of portland cement same as in Figure 6.

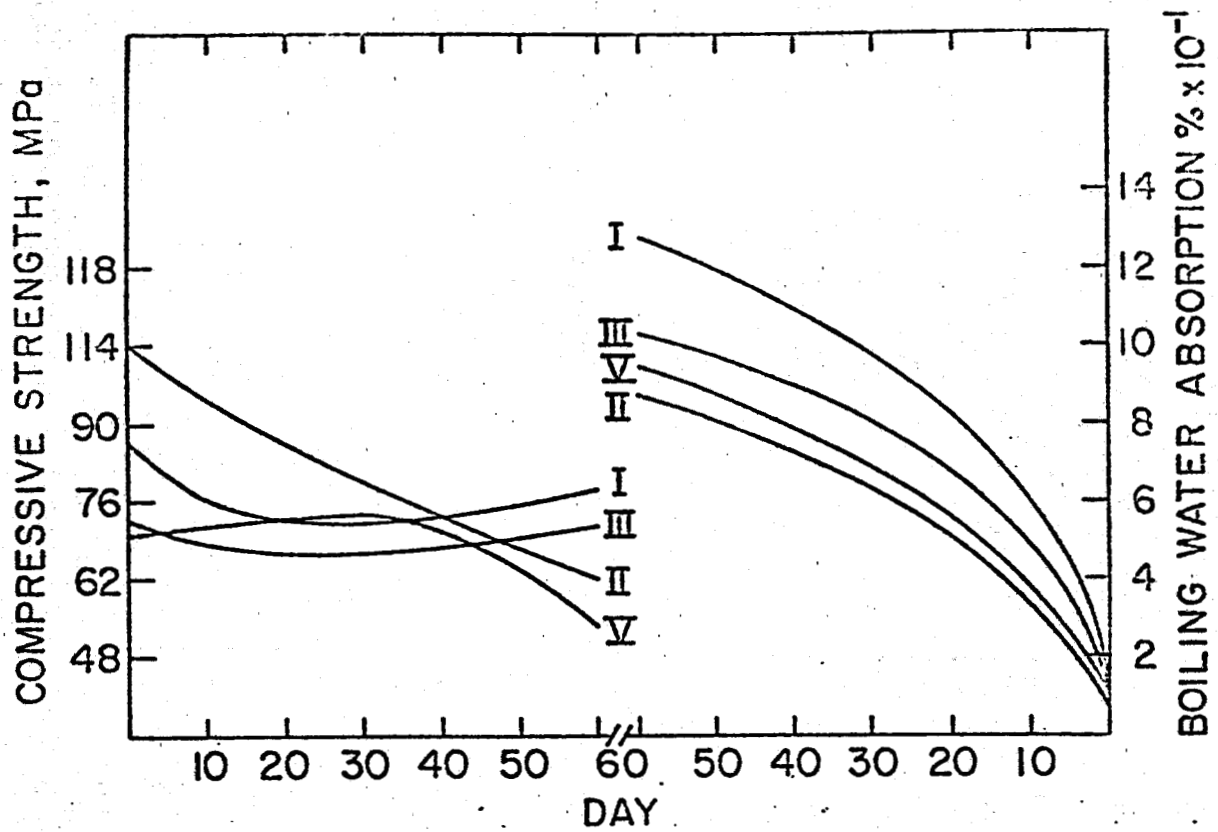


Figure 8. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition 85% sand - 15% cement. Monomer composition and type of portland cement same as in Figure 6.

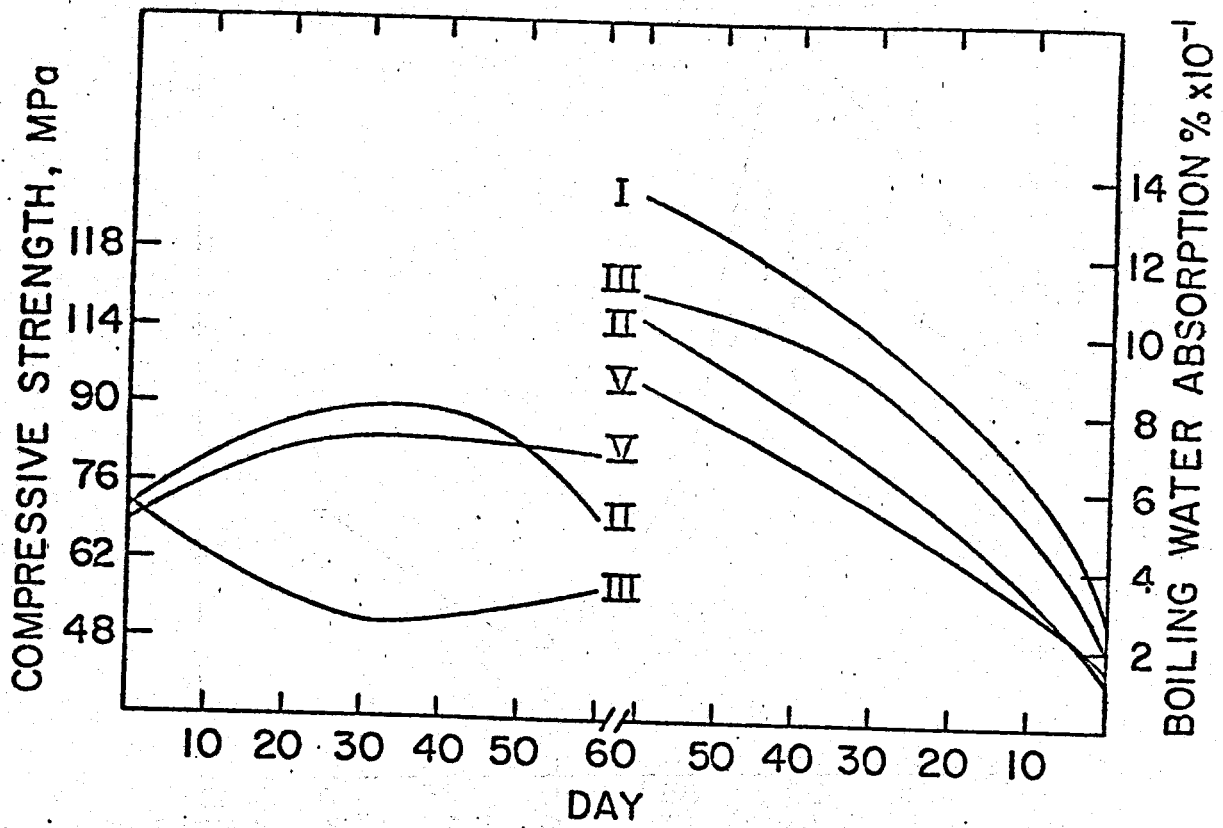


Figure 9. Compressive strength and absorption of OSPC after exposure to brine at 300°C. Aggregate composition, 80% sand - 20% cement. Monomer composition and type of Portland cement same as in Figure 6.

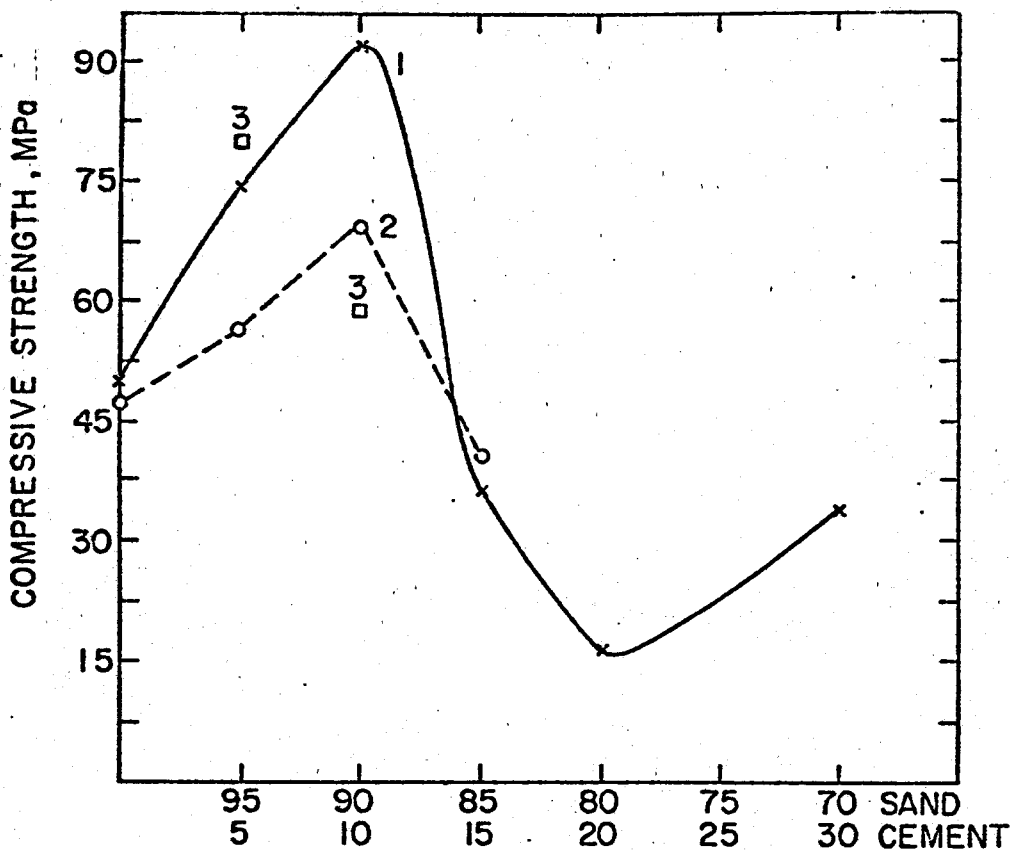


Figure 10. Compressive strength as a function of sand-cement ratio. Monomer composition: 97 wt% RZ - 3 wt% V-47; Aggregate composition: sand-sieve opening <math><30 \mu\text{m}</math>; cement-type III. (1) after boiling water; (2) after exposure to brine at

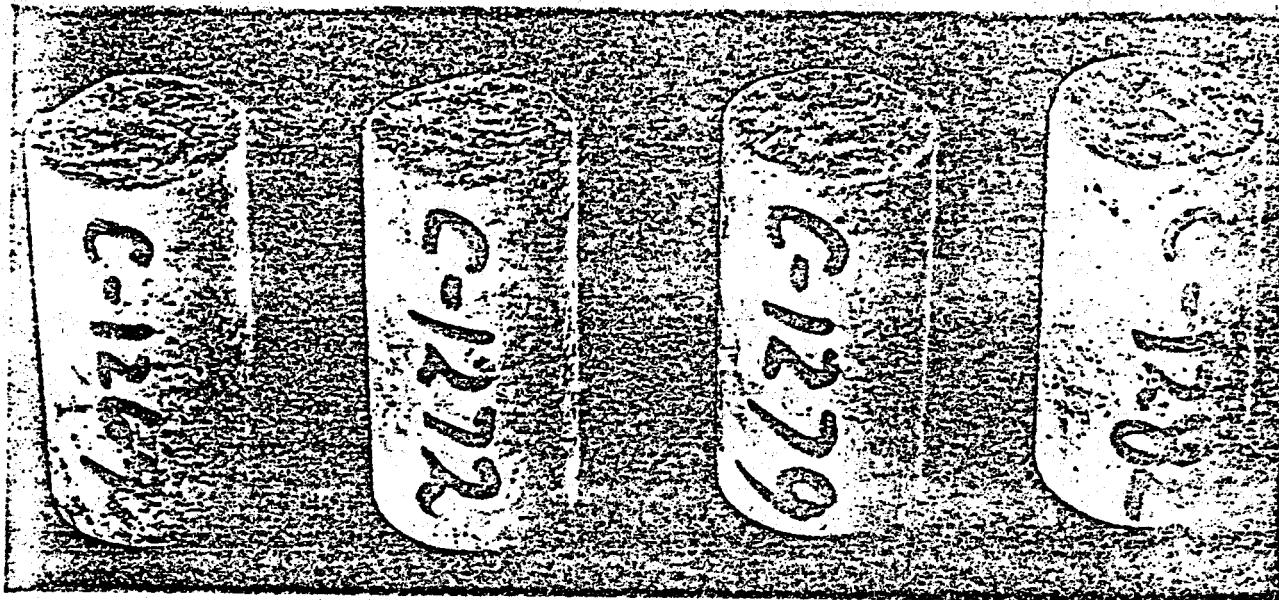


Figure 11. Organosiloxane PC samples from RZ and V-47 after exposure in brine at 300°C for 30 days.

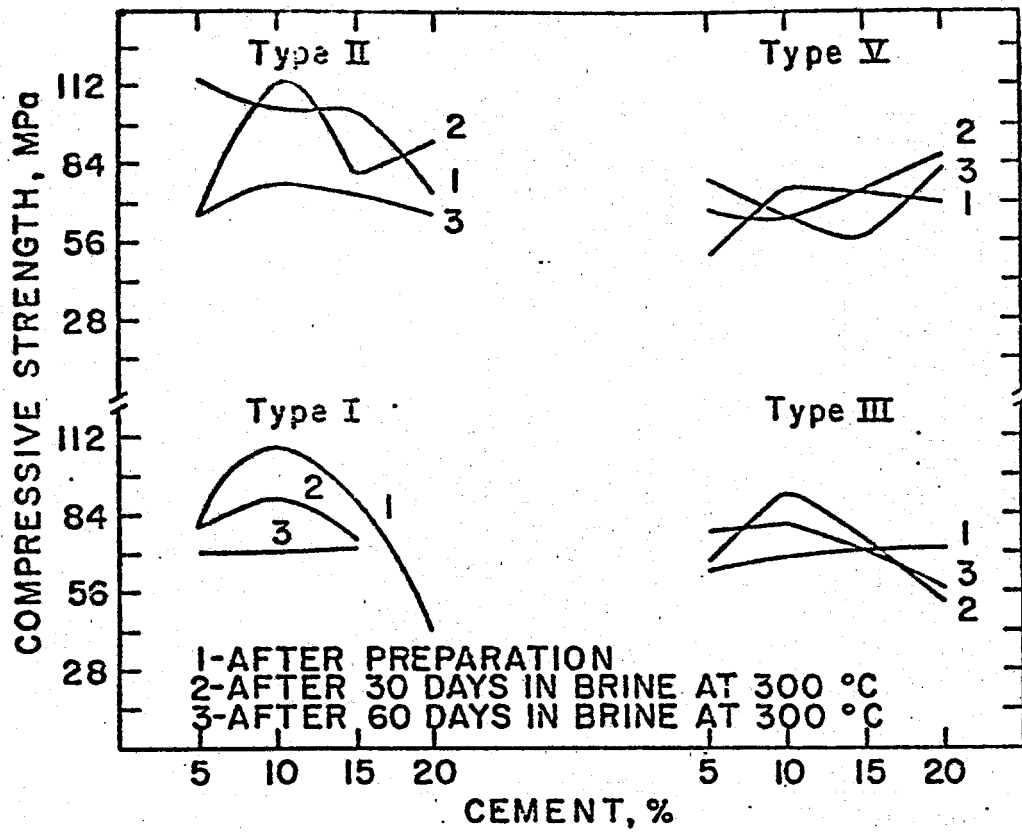


Figure 12. Compressive strengths of OSPCs, after exposure to brine at 300°C as a function of cement type and concentration. Summary of results.

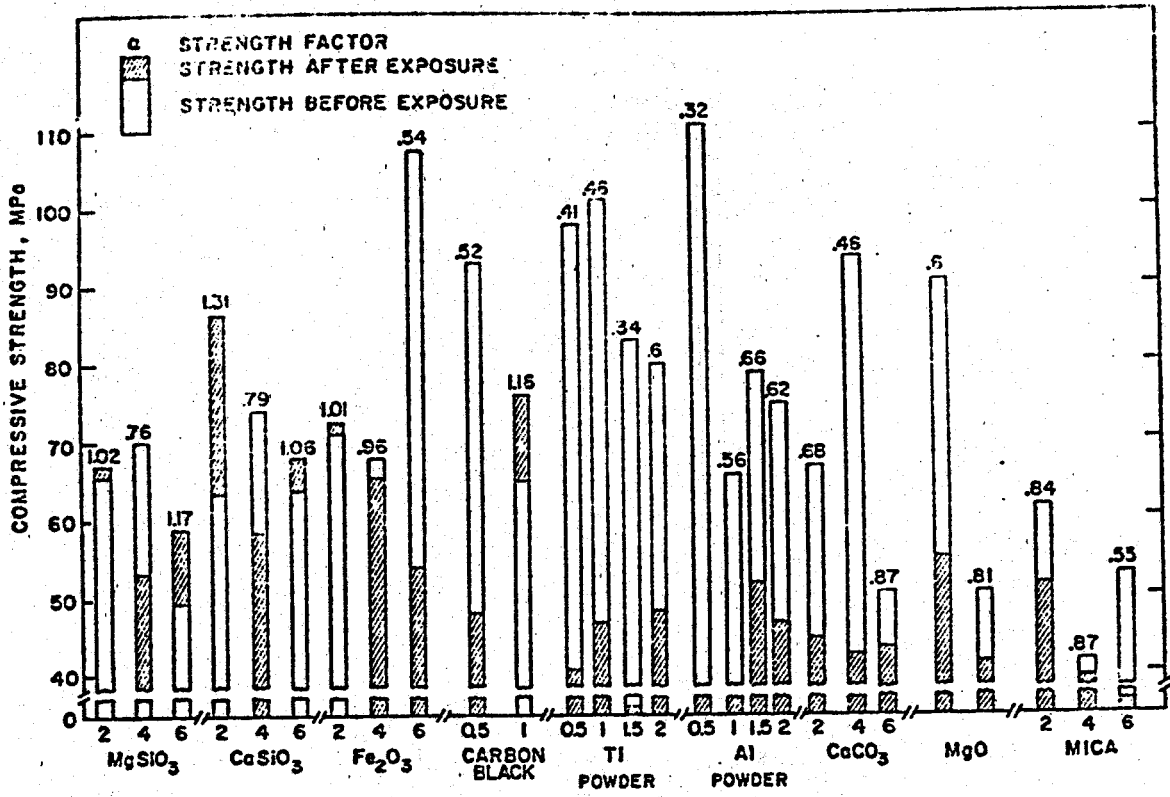


Figure 13. The effects of additives on the hydrolytic stability of CSPCs after exposure to brine at 300°C for 30 days.

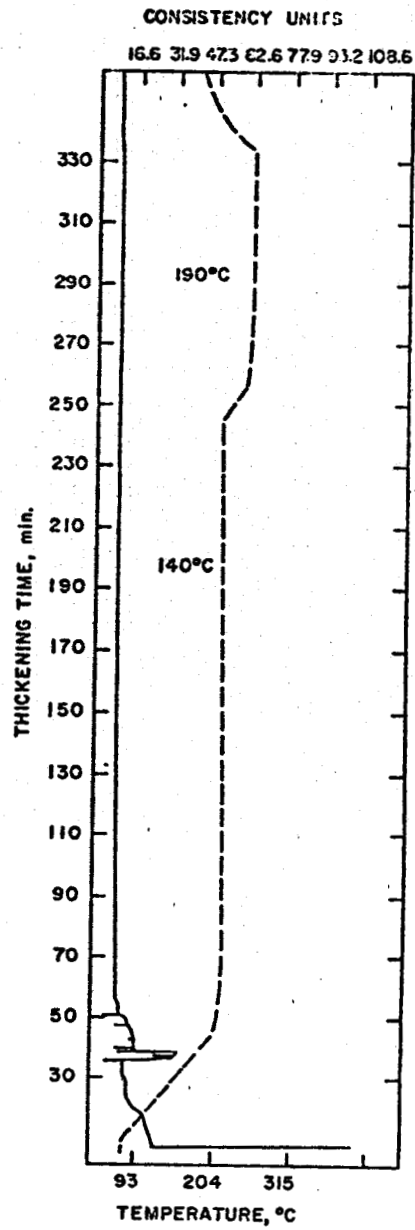


Figure 14. API thickening time test. Monomer composition 97 wt% RZ - 3 wt% V-47; temperature 190°C.

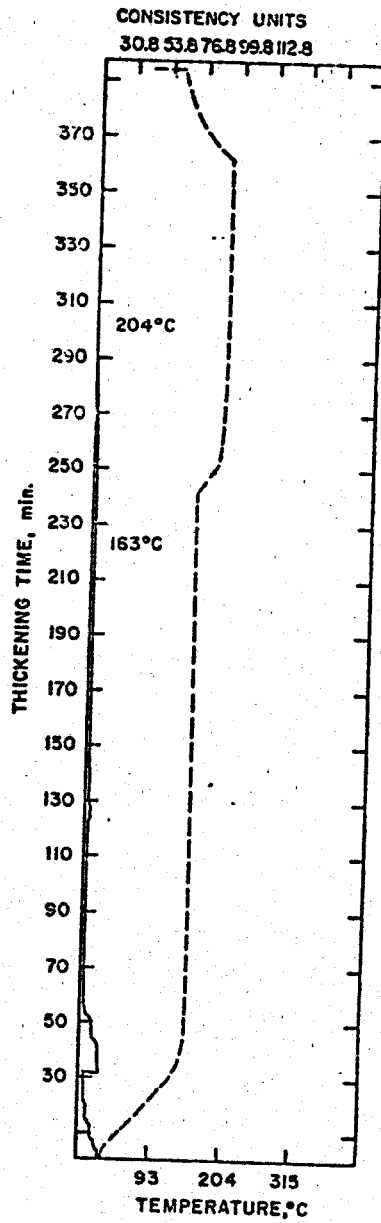


Figure 15. API thickening time test. Monomex composition 97 wt% RZ - 3 wt% V-47; temperature 204°C.