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A Review of Properties of Polymer Impregnated Concrete*

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A Review of Properties of Polymer Impregnated Concrete

Allan Auskern

I. Introduction

A joint effort on polymer-concrete materials at Brookhaven National Laboratory, Upton, N. Y., and the United States Bureau of Reclamation, Denver, Colorado, has been supported mainly by the U. S. Atomic Energy Commission, Division of Isotope Development, and the Office of Saline Water of the U. S. Department of Interior. This program grew out of research on wood-polymer, where radiation is used to polymerize monomer filled wood. It was observed that wood-polymer combinations had improved strength and durability properties. About four years ago the suggestion was made to impregnate a piece of concrete with a fluid monomer, and then polymerize the monomer in-situ; i.e., to make the concrete analog of wood polymer. The experiment was done, and some strength properties of the polymer impregnated concrete were measured. The results were so encouraging that the present joint program was undertaken.

It is the purpose of this report to review some of the important properties of polymer impregnated concrete (PIC), discuss factors affecting its properties, outline techniques for producing the materials, and briefly explore its potential

and limitations. Much of the information to be presented has appeared in four annual reports ^(1, 2, 3, 4), in internal reports, and in research articles which have been appearing more and more frequently in scientific and engineering journals.

II. Preparation of Polymer Impregnated Concrete (PIC)

The preparation of PIC requires first the manufacture of the concrete item, then filling the pores with a monomer, followed by polymerizing the monomer. Early work ⁽¹⁾ demonstrated that the amount of monomer filling the concrete was strongly dependent on the degree of drying. Since the mechanical properties of the PIC improved with greater polymer loadings, thorough drying of the concrete is considered routine, and drying temperatures as high as 125°C (255°F) are used. A typical walk-in dryer used for a large laboratory operation is shown in Fig. 1, where pieces of concrete pipe are being processed.

Monomer impregnation involves soaking the dried specimens in a liquid monomer for a time sufficiently long so that all the accessible pores will become filled. It has been found that the soaking time can be appreciably shortened if the dried concrete is evacuated in the soaking vessel, prior to the introduction of the monomer. It is also common laboratory practice

to apply an overpressure of about 25 psig during soaking. A great many factors influence the rate of monomer filling. For example, the distance to be penetrated, the total porosity of the concrete, the pore size, monomer viscosity, whether or not vacuum and/or pressure is used will all affect the filling rate. The amount of monomer absorbed at saturation is, of course, a function of the total porosity of the concrete.

No systematic set of experiments involving all these parameters has been undertaken, but some of the individual variables have been studied.

As an example of impregnation requirements, complete impregnation of 6x12 in. cylinders of nominal 28 day strength of 5000 psi requires (after thorough drying at 110°C) one hour vacuum (~2mm Hg) followed by 1-2 hour soaking in monomer (viscosity ~1 centipoise) with an overpressure of 25 psig.

The polymer loading of the concrete pipe of Fig. 1 is shown being carried out in an early laboratory impregnator in Figs. 2 and 3.

After the sample is monomer filled, the excess monomer is removed from the system and the monomer is polymerized

quickly to prevent surface losses from monomer drainage and evaporation. Note that both evaporation and drainage are minimized by having a small pore size, but small pore size requires longer times for monomer filling.

Monomer polymerization is either by the thermal-catalytic process (i.e. heat and a catalyst are used to promote polymerization), or by gamma radiation. In modern practice thermal-catalytic polymerization is done either under hot water, where the water will minimize evaporation, but not necessarily drainage, or in the monomer impregnation vessel where an atmosphere of monomer vapor can be introduced to reduce evaporation. Another process to reduce evaporation and drainage is to dip the monomer filled concrete in a viscous polymer solution of low volatility to produce a stable coating over the concrete. In some processes the concrete is wrapped in aluminum foil or polyethylene film to minimize evaporation prior to radiation or thermal treatment. An example of pipe being wrapped is in Fig. 4.

Time for polymerization is a strong function of monomer type, catalyst, catalyst concentration, temperature and for the radiation process, the radiation intensity and size of

specimen. (1) For example, the thermal catalytic polymerization of 10 ml methyl methacrylate samples at 75°F (in a water bath) using 1% benzoyl peroxide catalyst requires $1\frac{1}{2}$ hours for complete polymerization. Styrene under the same conditions requires 8 hours. These times may vary depending on sample size, inhibitor concentration, thermal environment, and catalyst.

Radiation polymerization requires no catalyst. Rates are a function of radiation intensity, and the time for complete polymerization is also dependent on inhibitor concentration. At a dose rate of 1×10^5 rads/hour cobalt-60 radiation methyl methacrylate requires a total dose of 2.1×10^6 rads (21 hours); at a dose rate of 5.3×10^5 rads, a total dose of 4.8×10^6 rads is required (0.1 hours). (1) These values assume normal inhibitor concentrations. Dose requirements will vary depending on inhibitor concentration, and references (1, 2) should be consulted for specific details.

There is ample evidence that after monomer filling and polymerization the concrete is filled with polymer. The concrete has gained weight; the weight gain being a function of the porosity of the concrete. In Fig. 5 the variation in polymer content with concrete density is illustrated for a

single batch of 5000 psi concrete, 28 day fog cure. Note that the polymer content is indicated as wt.% loading, which is the weight of polymer (W_p) divided by the dry weight of concrete (W_c) $\times 100$. This unit is adequate when comparing concretes of comparable density and for estimating monomer requirements. For many uses the volume concentration of polymer is more desirable (since it is the pore volume that is being filled). Use of the volume concentration permits polymer requirements of concretes of different densities to be compared. The volume concentration is obtained from the weight loading by multiplying the weight loading by the ratio of concrete density to polymer density. For example in Fig. 5 the highest polymer loading is 7.9 wt.% for a concrete of density 131 lb/ft³ (2.1gm/cc). The volume concentration is $7.9 \times \frac{2.1}{1.17} = 14.2\%$. For the densest material, the volume concentration of polymer is $6.1 \times \frac{2.23}{1.17} = 11.6\%$.

Further evidence of loading is indicated by color changes on polished sections. This is illustrated in Fig. 6 where the outer one-half inch of a 3 in. dia x 6 in. long cylinder has been impregnated. The presence of polymer darkens the concrete. In Fig. 7 a completely impregnated section is shown, where there has been some monomer loss in the outer surface.

On closer examination of cut and polished sections, polymer can often be seen filling or lining entrapped air voids. An example of this is shown in Fig. 8. There are also nondestructive indications that significant changes have occurred to the concrete. Rebound numbers of the Schmidt impact hammer are 20-35% greater for polymer filled concrete. (3) Also, the sonic velocity of polymer filled concrete is significantly greater than that of dried or monomer filled concrete. (2)

III. Mechanical Properties of Polymer Impregnated Concrete

A. Stress-Strain Relations in PIC

Perhaps the most dramatic demonstration that a fundamental change in the nature of concrete after polymer impregnation takes place is to observe a PIC specimen fail in compression. Examples of failed normal concrete and PIC are shown in Fig. 9. The normal concrete on failure exhibited a few cracks but essentially remained in one piece. The PIC completely shattered on fracture. In the figure the pieces are held together by tape. What is most significant about the fracture is that the cracks pass through, not around, the large aggregate. This suggests that the role of the polymer must be, at least in part, to significantly strengthen

the bond between mortar and aggregate. The strengthening mechanism of the polymer in PIC has been discussed elsewhere. (5)

In Fig. 10 the stress-strain curves for the concrete cylinders of Fig. 9 are presented. The normal concrete shows typical nonlinear stress-strain behavior from nearly the start of loading; at fracture, the stress-strain curve is nearly horizontal. The deviation from linearity is to a large extent attributed to progressive failure of the aggregate cement bond. (6) The PIC sample behaves much differently. The stress-strain curve is linear until, in this case, greater than 78% of the fracture load has been applied; at fracture there is relatively little deviation from linearity. PIC behaves essentially elastically. The linearity of the stress-strain curve supports the suggestion that part of the role the polymer plays is to improve the mortar-aggregate bond. (5)

Besides the nature of the stress-strain curves for PIC and normal concrete being different, the most important result is that the compressive strength and the elastic modulus of PIC are significantly greater than those for the normal concrete. In the example shown here the PIC compressive strength is 17,000 psi and the elastic modulus is 5.5×10^6 psi. These values are significantly greater than those of the corresponding normal concrete. The compressive strength of PIC will be discussed in more detail later.

Further evidence of a change in the nature of PIC is given in Fig. 11. Here again, the compressive strength of the PIC is much greater than that of the starting concrete (21,000 psi vs 5,000 psi nominal), and the modulus of elasticity is about twice as great as that of the corresponding normal concrete. In this particular experiment the load was cycled 4 times at each of the indicated loads, up to 62% of the failure load. There was no evidence for any gross deviations from linearity as a result of the cycling, and the final curve to failure, which is shown on the figure, is almost identical to the results of earlier cycles.

It appears then, that PIC made under the conditions described results in a new material of construction, based on normal concrete, with high strength, greater stiffness and a completely different mode of fracture.

B. Factors Affecting the Compressive Strength and Elastic Modulus of PIC

Probably the most critical single factor affecting the compressive strength of PIC is the degree that the pores of the concrete are filled. This is illustrated in Fig. 12 where the compressive strength is plotted against the polymer loading. There is a very strong dependence of strength on

polymer loading. For any PIC system, the maximum strength is obtained with the maximum polymer loading. In the same figure two other variables are indicated. They are the polymer type and polymerization technique. The results for thermal catalytically polymerized styrene are very poor, while the strength results for radiation cured styrene are comparable to those for thermal catalytically cured methyl methacrylate. Radiation cured methyl methacrylate PIC strengths appear to be 10-15% higher than thermal catalytically cured MMA.

A summary of the compressive strength results for a number of different polymer systems is in Table 1.⁽²⁾ These data were obtained on a nominal 5000 psi concrete, fog cured for 28 days. Three inch diameter by six inch long cylinders were used for compression testing. The polymer cure is indicated. In all cases the radiation cured samples had higher strengths. For styrene and acrylonitrile the differences are very great, and suggest that the thermal cure may cause a harmful interaction between polymer and concrete. It is not clear why radiation curing produces consistently higher strengths with this concrete.

It is also obvious from the table, that for either polymer cure, MMA, and MMA + 10% TMPTMA produce the highest compressive strengths. The origin of all these polymer differences is not

apparent. All except acrylonitrile (ACN) form rigid, glassy polymers. ACN polymer is insoluble in the monomer, so it forms as a powder. Other monomers may chemically interact to various degrees with the concrete during polymerization. Since all the monomers decrease in volume on polymerization, the volume of concrete pores that is monomer filled is greater than the pore volume that is polymer filled. However MMA, which has a high volume shrinkage on polymerization (~20%) produces one of the highest strength PIC materials.

Probably the second most important factor affecting the strength of PIC is the concrete itself. Fig. 13 is a compilation of test results showing the final, polymer filled strength as a function of the strength of the unimpregnated concrete. Unimpregnated strengths range from 2500 psi to 9500 psi. With the exception of the two BPR-L-3 series, the plot, although there is considerable scatter, indicates that the final strength is independent of the initial strength. Other, more limited experiments indicate that the greater the initial concrete strength, the greater the PIC strength, although the relative increase is greater for weaker concretes. (3, 7) Fig. 13 also suggests that the maximum PIC compressive strength is around 23,000 psi. More recent work on concretes cured by high pressure steam achieved compressive strengths greater than

30,000 psi for polymer loadings around 7 wt.%.⁽⁸⁾ These concretes were made with the same cement and aggregates used for much of the work in Fig. 13. Other unpublished work indicates that steam curing of concrete leads to higher PIC compressive strengths than water or fog curing. Porosimetry measurements show that the average pore size of steam cured cements is greater than that of water cured cements.⁽³⁾ This may lead to more efficient polymer loading. It is quite likely that at the present time the ultimate strength for PIC has not been reached. One reason for this is that a complete understanding of the interrelations between concrete, porosity, and polymer has not been achieved.

Probably the factors that affect the compressive strength of PIC affect the modulus of elasticity in the same manner. It was pointed out earlier that the elastic modulus of PIC was greater than that of normal concrete. The increase in modulus follows approximately a square root relationship with the compressive strength, much like normal concrete does. One could predict, based on these relationships, an elastic modulus of about 10×10^6 psi for a 30,000 psi PIC. Moduli greater than 8×10^6 psi have been observed.

C. Tensile and Flexural Strength of PIC

Figure 14 shows the tensile strength of PIC as a function of polymer loading. The improvement is much like that observed for the compressive strength. At full polymer loading, about 6 wt.%, the tensile strength is about four times the normal concrete tensile strength. The ratio of compressive strength to tensile strength is about the same for both PIC and normal concrete (for this concrete).

The modulus of rupture of PIC is also about four times greater than that of the concrete from which it was made, and is strongly dependent on polymer loading, dependent on polymer type, and on polymer cure. This is illustrated in Fig. 15.

Both the tensile and modulus of rupture testing were performed on the same 5000 psi strength, 28 day fog cure concrete upon which most of the compressive testing was done. It is anticipated that the same factors that affect the compressive strength of PIC will affect the tensile strength and modulus of rupture.

IV. Durability Properties

A. Freeze-Thaw

Good quality concrete can be expected to withstand 500 standard freeze-thaw cycles before failure (25% weight loss). (9)

Since for PIC, the concrete pores are almost completely filled, the amount of water that can enter the structure and do damage is limited. Fig. 16 shows some freeze-thaw results on four PIC samples and two control samples. The weight loss and weight loss per cycle are significantly lower for the PIC samples. Testing on the PIC samples designated CP-1B-3A and 3B has been carried out to a few thousand cycles with no significant weight loss. Fig. 17 shows a normal and impregnated concrete sample after 490 cycles. The normal concrete has failed, while the PIC appears almost unaffected.

Although PIC has shown very little freeze-thaw damage by weight loss testing, sonic techniques for evaluating freeze-thaw durability indicate that there is a continuous reduction in sonic modulus, although at a rate much lower than normal concrete. Compressive testing of PIC samples that have been subjected to ~1000 freeze-thaw cycles without significant weight loss show a considerable decrease in strength. Most of the freeze-thaw work has not been published and more quantitative data is not yet available. However the work so far does indicate that the freeze-thaw cycle damages PIC but at a rate much slower than that of normal concrete.

B. Sulfate Resistance

Sulfate attack on concrete is primarily caused by the reaction of soluble sulfates with calcium hydroxide, producing reaction products that occupy a greater volume than the reactants. The reaction causes expansion and cracking of the concrete.

The sulfate resistance test consists of a 24 hour cycle in which the concrete is soaked in 2.1% sodium sulfate solution for 16 hours, and then dried in hot air for 8 hours. A net expansion of 0.2% is considered a failure.

For the same reasons that freeze-thaw behavior of PIC is improved, sulfate resistance of PIC should be greater than that of normal concrete. In Fig. 18 some results for PIC and controls are shown. The polymer impregnated concretes indicate expansion of about one-tenth that of the normal concretes. (The CP-3 samples have no polymer.) Fig. 19 is a photograph of PIC and normal concrete after 450 test cycles. The normal concrete is quite cracked, and expanded 0.5% compared to 0.02% for PIC.

C. Acid Resistance

For an evaluation of the acid resistance of PIC, samples and controls are immersed in a 15% hydrochloric acid or sulfuric acid solution, and removed periodically

for weighing. Some results for HCl attack are shown in Fig. 20. The presence of polymer in the pores does reduce the weight loss rate for PIC but the improvement is not nearly as good as that against sulfate attack or freeze-thaw. Attack by 15% sulfuric acid is more severe than HCl. Thermally cured MMA filled PIC suffers 12% weight loss (3 in. dia x 6 in. long cylinders) in 80 days; in the same time period HCl attack results in a 3½% weight loss. Considerable improvement in the acid resistance of PIC results when the polymer impregnated concrete is given two surface coatings of polyester-styrene. (10) Some PIC pipe tested in this manner indicated 0-1% weight loss in ~100 days under test. Uncoated comparable PIC pipe had weight loss of ~50% (extrapolated).

D. Resistance to Hot Distilled Water

Tests to determine the resistance of PIC to hot distilled water were performed in autoclaves by dripping hot condensate onto the sample surface. The results are usually reported in % weight loss, but depth of attack is more meaningful, since the effect takes place at the surface. The most important variables in this test are the temperature, polymer type, and condensate drip rate. Usually if the pure bulk polymer is stable in water at the test temperature, the PIC will be relatively stable also. Extensive testing of high

temperature polymers is limited to two polymers. They are 90% diallylphthalate + 10% methyl methacrylate (Dap-MMA), and 60 styrene + 40% TMPTMA* (stryene-TMPTMA). They have been tested at 250°, 290°, and 350°F, with a distilled water drip rate of between 1 and 1½ pounds of water per hour.

Some results of attack rates at the three temperatures are shown in Table 2. The results are based on weight loss after 75 days condensate drip, and are converted to erosion rates in units of mm/year. These rates should be considered as only estimates since most of the erosion results from the fallout of relatively large aggregate particles after the cement phase is first removed. It should be noted that for the styrene-TMPTMA PIC, there was no aggregate fallout: the cement-polymer phase did not appear to be attached. The erosion appears to be due to attack on the aggregate.

A photograph of some test samples after exposure at 350°F for 97 days is shown in Fig. 21.

V. Creep

Creep of PIC is usually 5-10 times lower than creep under the same load for unimpregnated concrete. However, some PIC samples prepared with methyl methacrylate have indicated a "negative" creep, that is, a net change in length in the direction opposite that of the load. Thus samples in compression get longer

* trimethylpropane trimethacrylate

and samples under tension get shorter. This phenomenon has only been observed under compressive loads of 800 psi and tensile loads of 178 and 345 psi. At compressive loads greater than 800 psi creep becomes normal (positive). Also, negative creep has not been observed for all samples. Some creep results are shown in Fig. 22.

The origin of negative creep is not clearly understood. Some speculation on a negative creep mechanism has been made.⁽¹¹⁾ However experiments designed to demonstrate negative creep have not been completely successful.

VI. Polymer Impregnated Wire Reinforced Mortars

The polymer impregnation process is of course not limited to the impregnation of normal concretes. Significant property improvement has been observed on impregnated lightweight concretes,⁽²⁾ cement block,⁽²⁾ ferro cement, and mortars.⁽¹⁾ Some interesting results have recently been published on polymer impregnated steel fiber reinforced mortars.⁽¹²⁾ These results are shown in Fig. 23 for a mortar with 2% by volume of 3/4" by .015" dia steel fibers. The results show an increase by about 5 times in maximum load and deflection for the steel fiber reinforced mortar over the plain mortar. When polymer is added to the fiber reinforced mortar, another factor of 5 increase results, and the failure mode changes. Now the steel fibers themselves break, so that the

presence of the polymer permits the full utilization of the strength of the steel. Failure is no longer sudden, as in polymer impregnated mortar; the material behaves in a plastic manner, and can absorb about 50 times as much energy at failure as the polymer filled mortar.

VII Conclusions

Incorporating a relatively small amount of a polymer in cured concrete or mortar alters the nature of the original material dramatically. A new and different structural material results with properties only remotely related to the starting materials. The properties reported here were developed from a very limited number of concretes. As work progresses on understanding the basic interactions between the cement, aggregate, and polymer phases, it should be possible to formulate polymer impregnated concretes with specific properties, just as it is now possible to formulate normal concretes with specific properties.

It is doubtful that the ultimate properties of PIC have been achieved. If, as seems likely, there will be further improvements in the mechanical properties of PIC, we will achieve a material derived from concrete, but without concrete's most serious limitation, poor tensile strength.

At present attempts to develop techniques for incorporating the monomer or polymer in the concrete mix rather than the cast concrete have been unsuccessful. Also, there has been little work done in impregnating existing concrete structures. So for the present, PIC is limited to precast concrete shapes.

In the decision of a concrete manufacturer to produce PIC, he must carefully evaluate the additional expense of installing the necessary monomer handling, impregnation, and polymerization equipment against the potential market for a new material derived from concrete with unique structural and durability properties.

Table 1 (2)

<u>Polymer</u>	<u>Wt. Loading, %</u> (range)	<u>Compressive Strength, psi</u>	
		<u>Thermal Cure</u>	<u>Radiation Cure</u>
MMA	4.2-6.7	18,200	20,300
Styrene	4.2-6.0	8,800	14,100
MMA + 10% TMPTMA*	5.5-7.6	19,000	21,600
Acrylonitrile	3.2-6.0	10,750	14,410
Chlorostyrene	4.9-6.9	14,400	16,100

* TMPTMA = Trimethylopropane trimethacrylate

Table 2

Distilled Water Erosion of PIC
(mm/year)

<u>Temperature</u>	<u>Control</u>	<u>PIC (Dap-MMA)</u>	<u>PIC (Sty-TMPTMA)</u>
250° F	30	12	6
290°	40	25	8
350°	60-70	30	10

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Fig. 1 - Walk-in type resistance heated furnace
with concrete pipe specimens drying.

Fig. 2 - Typical assembly of a 3-ft high by 2-ft diameter concrete pipe specimen in the impregnator. Note the insert used to minimize the free volume.

Fig. 3 - Impregnator and ancillary equipment.

Fig. 4 - Wrapping a monomer saturated concrete pipe specimen with aluminum foil to reduce evaporation losses prior to polymerization.

Fig.5 - Variation of polymer content with density for a single batch of concrete.

Fig. 6 - A polished section through the center of a partially impregnated 3 in. dia. x 6 in. long concrete cylinder. The polymer is in the outer half inch.

Fig. 7 - A polished section through the center of a fully impregnated 3 in. dia. x 6 in. long concrete cylinder. Some polymer loss from the surface has occurred.

Fig. 8 - A void in polymer impregnated
concrete lined with polymer.

Fig.9 - Fracture of normal concrete and polymer impregnated concrete.

Fig.10 - Comparison of compressive stress-strain relationship for normal and polymer impregnated concrete.

Fig. 11 - Stress-strain relationship for polymer impregnated concrete after cycling.

Fig.12 - Compressive strength as a function of polymer filling.

Fig. 13 - Strength of polymer impregnated concretes as a function of strength of the corresponding unimpregnated (normal) concrete.

Fig. 14 - Tensile strength of polymer impregnated concrete as a function of polymer filling.

Fig. 15 - Flexural strength of polymer impregnated concrete as a function of polymer filling.

Fig. 16 - Freeze-thaw durability of polymer impregnated concrete.

**Fig. 17 - Normal and polymer impregnated concrete
after 490 freeze-thaw cycles.**

Fig. 18 - Sulfate resistance of polymer impregnated concrete.

Fig.19 - Comparison of sulfate resistance of normal and polymer impregnated concrete after 450 days in test. Left sample is normal concrete and shows cracking and 0.5% expansion. Polymer impregnated sample shows expansion of 0.02%.

Fig. 20 - Acid resistance - 15% HCl, of normal concrete and polymer impregnated concrete.

Fig. 21 - Condensate drip on (90-10) DAP-MMA PIC (14), control (24) and (60-40) styrene - TMPTMA (17), after 97 days at 350°F.

Fig. 22 - Creep in PIC, showing negative creep at 800 psi.

Fig. 23 - Load-Deflection curve for plain mortar, steel fiber reinforced mortar, polymer impregnated steel fiber reinforced mortar, and polymer impregnated mortar.