

## Innovations Deserving Exploratory Analysis Programs

**Highway IDEA Program** 

## **Waterproofing Concrete Highways**

Final Report for Highway IDEA Project 85

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# INNOVATIONS DESERVING EXPLORATORY ANALYSIS (IDEA) PROGRAMS MANAGED BY THE TRANSPORTATION RESEARCH BOARD (TRB)

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#### **Executive Summary**

This project tested the concept of permanently waterproofing concrete by incorporating a waterborne dispersion of soybean oil phosphate ester polyol (SOPEP) into the concrete at the time of mixing. Rebar corrosion caused by water and chloride permeation through capillaries in concrete leads to loss of reinforcement of the concrete, which leads to highway surface deterioration and potholes. Various additives have been used to waterproof concrete including polymers and latexes, oils and fatty acids, silicone oils and polymers, asphalt and pitch, and colloidal clay. "Leakage" of the oils and mobile organics from the concrete has been a problem. It was postulated that the water dispersible ionomeric soybean oil phosphate ester would initially act as a plasticizer/air entrainment admix. Then as the cement hydrated, SOPEP would be concentrated in the capillaries and the phosphate portion of the oil would adhere to the concrete matrix of the capillary walls and "lock" the oil in the cement capillaries to give permanent waterproofing so that oil would not leach out to the surface causing traction problems. The oil bound in the concrete capillaries would minimize trapped water and not only minimize freeze-thaw damage, but also slow the permeation of water and chloride into the concrete and give longer concrete highway service life. The idea of waterproofing the concrete from within the concrete capillaries should be possible, without affecting the skid resistance of the surface. The scope of this project was to test the hypothesis and evaluate reduced water permeation in treated concrete.

In concrete tests, soy phosphate ester polyol did increase fresh concrete slump as expected, but it was not as effective as commercial super plasticizers. It also retarded cure as expected for a phosphate. SOPEP did adhere strongly to the concrete matrix. Unfortunately, it bonded so strongly and so soon, that it did not accumulate in the capillaries as much as expected. At 5% by weight of the cement, SOPEP reduced water absorption by only about 15%. Compressive strength of the concrete at 28 days cure was reduced greater than the 10% reduction considered acceptable. Epoxidized soybean oil (ESBO), neutral hydrolyzable oil, was evaluated as a possible replacement for SOPEP that would not initially adhere to the concrete matrix so strongly or so fast and would later hydrolyze to the polyol, which would hydrogen bond to the capillary walls for reduced migration. At 10% by weight of the cement, ESBO reduced water absorption by 48%. Compressive strength of the concrete was reduced below acceptable levels after a 28-day cure. SOPEP was also sprayed on fresh concrete and evaluated as a possible curing agent to retard drying of concrete—it was marginally effective. SOPEP is being evaluated as a bound lubricant for rebar to evaluate the corrosion resistance of a rebar phosphate layer protected by an integral permanent monomolecular layer of bound oil. This test is ongoing and, if successful, could extend to several years of testing.

#### **BODY**

#### IDEA PRODUCT

Rebars in concrete highways corrode due to permeation of moisture and chloride ions through concrete. The IDEA product is an admix to economically waterproof concrete highways so that the concrete will repel salt water incursion, minimize rebar corrosion, and prolong the life of concrete highways without affecting automobile skid resistance. The Principal Investigator found that an aqueous dispersion of soybean oil phosphate ester polyol (SOPEP, Structure 1) can be added to plaster of paris to obtain cured products that contain 5% soybean oil phosphate ester. The oil is locked into the cured plaster by the phosphate group during cure and the surface of the plaster is not oily. The specific admixture product proposed was a 50% dispersion of soybean oil phosphate ester polyol.

#### Structure 1. SOPEP

SOPEP is a promising new material being developed for paint and coating applications where it appears to have a significant cost-performance advantage in many applications. Up to about nine phosphate groups could be added to the soybean oil molecule. The common lab product contains 1% phosphate and samples have been made with up to 5% phosphate. Borate and other esters can also be made. Rebar could also be pre-coated with SOPEP at the factory or in the field to minimize corrosion. The phosphate portion of the SOPEP molecule would be expected to react with the metal so the oil would stick to the rebar for additional corrosion protection before and after use in concrete. SOPEP is free of volatile organic compounds; biodegradable if spilled in the environment, and represents a sustainable technology derived from crop-based products envisioned by Vision 2020.

Theoretically, SOPEP water dispersions should do interesting and complex chemistry with hydrating cement because it is an ionomeric polymer. In this project we proposed to determine the optimum level of soybean oil in concrete, demonstrate and quantify the waterproofing effect, demonstrate the long life of the waterproofing effect, and demonstrate that the oil does not migrate to the surface to reduce normal tire friction on the concrete road surface.

Admixtures are ingredients other than portland cement, water, and aggregates that are added to the concrete mixture immediately before or during mixing. They are used to modify certain properties of the concrete and can be classified according to their function:

- 1. Air-entraining admixtures.
- 2. Water-reducing admixtures.
- 3. Retarding admixtures.
- 4. Accelerating admixtures.
- 5. Cementing agents.
- 6. Workability agents.
- 7. Miscellaneous agents for bonding, damp proofing, and permeability reducing.

Because of its chemical structure, SOPEP could potentially act as five of the above admixtures.

#### CONCEPT AND INNOVATION

Concrete is a mixture of portland cement, water, aggregates, and in some cases, admixtures. The cement and water form a paste that hardens (hydrates) and bonds the aggregates together. Concrete quality is directly related to the amount and properties of the materials used, and the way that it is placed, finished, and cured. Concrete is a versatile construction material, adaptable to a variety of agricultural and residential uses. With proper materials and techniques, it can withstand many acids, silage, milk, manure, fertilizers, water, fire, and abrasion. Concrete can be finished to produce surfaces ranging from glass-smooth to coarsely textured, and it can be colored with pigments or painted. Concrete has substantial strength in compression, but is weak in tension. Most structural uses, such as beams, slats, and manure tank lids, involve reinforced concrete, which depends on concrete's strength in compression and steel's strength in tension.

The counter-ion used to make SOPEP water dispersible can also be used to affect the chemistry of concrete.

This project tested the concept of permanently waterproofing concrete by incorporating a low cost waterborne dispersion of soybean oil phosphate ester polyol (SOPEP) into the concrete at the time of mixing. Rebar corrosion caused by water and chloride permeation through concrete leads to loss of reinforcement of the concrete, which leads to highway surface deterioration and potholes. Various additives used to waterproof concrete include super absorbent polymers (1), latexes (2, 3), oils and fatty acids (4), siloxane compositions (5), asphalt and pitch (6), and colloidal clay (7). Leakage" of the oils and mobile organics from the concrete has been a problem.

Ren reported that a silicone admixture added to concrete before setting made the entire concrete block water repellent.(5) Water absorption of the pressed concrete was reduced by up to 90% by adding 0.05% of the admixture into the concrete. Efflorescence of the concrete was also significantly decreased due to the low water absorption. Both compressive and flexural strength was significantly increased with the process used. The organic silicone resin reportedly lines the capillary walls of the concrete to impart water repellency resulting in the limitation of water penetration through the capillaries. This new admixture apparently overcomes the limitations inherent in traditional non-reactive admixtures.

Similarly, it was postulated that water dispersible ionomeric soy phosphate ester polyol oil would locate in the capillaries of cured concrete and "lock" the oil into the cement matrix to give permanent waterproofing that would not leach out to the surface causing traction problems. We also postulated that the soy phosphate ester polyol molecule would initially act as an air entrainment and slump admix. Then the phosphate portion of the oil will be incorporated into the mineral structure and trap the oil in the cement capillaries. The oil bound in the concrete capillaries was expected to minimize trapped water and not only minimize freeze-thaw damage, but also slow the permeation of water and chloride into the concrete and give longer concrete highway service life. The idea of waterproofing the concrete from within the concrete capillaries with a low cost "green" admix should therefore be possible, without affecting the traction efficiency of the surface. The scope of this project was to prove the expected permanent benefit in reducing water permeation.

#### INVESTIGATION

The goals of this project were:

- 1. Make SOPEP and aqueous dispersions.
- 2. Determine upper limit of SOPEP compatibility with concrete.
- 3. Evaluate the effect of phosphate content and counter ion on compatibility.
- 4. Evaluate the curing characteristics of the SOPEP concrete.
- 5. Evaluate the water absorption of the SOPEP concrete.
- 6. Evaluate the strength of the concrete.

If successful in accomplishing the above goals then:

- 7. Evaluate the corrosion resistance of SOPEP modified concrete.
- 8. Evaluate the effect of SOPEP on setting of concrete under freezing conditions.
- 9. Make pothole repair material with SOPEP to evaluate the adhesion of new concrete to old concrete.

#### **Milestones:** Timeline for completion of key project activities and tasks.

- 1. Transfer technology for making SOPEP to Atofina
- Work with Atofina to provide gallon size samples
   Test various levels of SOPEP dispersion in concrete.
   Measure effect of SOPEP on cure time of Concrete
   Measure effect of SOPEP on strength of concrete
   Measure water permeability of untreated/SOPEP-treated concrete
   Prepare final report, disclosures on patentable technology, papers

#### Materials

SOPEP1 and epoxidized soybean oil (ESBO, Vikoflex<sup>TM</sup> 7112 from Atofina- Iodine value -1.35, Acid value 0.08mg KOH and percent oxirane oxygen- 7.0) were used as supplied. Superphosphoric acid, diethyleneglycolmonobutyl ether, and dimethylethanolamine amine were from Aldrich Chemical Company. The concrete was based on the Texas Department of Transportation specifications for bridge decks using Colorado River sand, crushed limestone coarse aggregate, and Type I/II cement.

#### **Characterization of Polyols**

Acid values (A.V.), oxirane numbers (O.N.), hydroxyl values (H.V.) of the SOPEP samples were determined by ASTM methods (ASTMD 1639-90, ASTMD 1652-97, and ASTMD 1957-86, respectively). The viscosities of the samples were measured using Brookfield viscometer at 25°C. The molecular weight was determined by Gel Permeation chromatography (GPC) using a polystyrene standard. The SOPEP samples were also characterized with FTIR spectra.

#### **SOPEP Synthesis**

Soybean Oil Phosphate Ester Polyols (SOPEP) were prepared by reaction of ESBO with superphosphoric acid and water, in presence of a polar organic solvent. The process involved addition of superphosphoric acid in solvent, at controlled rate, to the mixture containing ESBO, solvent and water, under mechanical stirring. A series of SOPEP was synthesized using amounts of phosphoric acid varying from 2.0 to 30 % by wt of ESBO. The reaction temperature for synthesis of SOPEP varied from of 25-85 °C. The reaction was monitored by periodic determination of percent oxirane oxygen content and acid value of the sample. Upon completion of the reaction, solvent and residual water were removed using rotary vacuum evaporator. Unreacted phosphoric acid was removed by successive washing the ethereal solution of the product with water.

#### Preparation of Aqueous dispersions of SOPEP

SOPEP was neutralized with calculated amount of amine or ammonia (based on 100% neutralization) under stirring at room temperature. Dimethylethanolamine was used as amine neutralizer. Deionized water was added slowly to the neutralized SOPEP under high-speed disperser, at 2000 rpm. The mixture was stirred for 30 minutes after addition of water was complete. The resultant dispersions were milky to translucent to clear solution depending upon phosphate ester content of the product. All the dispersions were prepared to have 50% SOPEP, with no co-solvent. It was found that the samples between 3.0 to 4% phosphate ester content formed stable emulsions in water, those between 4-10 % were translucent, while those with >10% dissolved in water and gave clear solutions.

#### **Concrete Test Protocol**

Tests were conducted to compare the properties of normal concrete with concrete to which SOPEP was added. Properties examined included (1) compressive strength based on ASTM C109 at 7, 14, and 28-days of curing, both air and moist cured; (2) flow of cement mortar (ASTM C1437), and (3) absorption (ASTM C642-97). Additionally, SOPEP was applied topically to fresh concrete specimens and compared to a commercially available concrete curing compound.

The mortar mixes consisted of cement, water, and sand. The mix was designed for a 1.5L mixer. The water-cement ratio was 0.5 in all cases. The typical mixture proportions, shown in Table 1, were adjusted accordingly for the added water contained in the SOPEP solution. Percent SOPEP numbers reported are for solids not solution.

TABLE 1. Typical Mixture Proportions

	kg
Cement	0.750
Water	0.375
Air	0.000
Sand	2.344

Two-in. mortar cubes were tested in a cube testing machine. The flow test was conducted on a standard flow table per ASTM specifications. The absorption tests were conducted on 2-in. x 2-in. cube specimens. Curing compound tests were conducted on 3-in. x 3-in. x 12-in. beams. The curing compound was sprayed onto the exposed surface, and the specimens were allowed to cure. After curing, the specimens were cracked using loads at third points. The tension face was the face coated with curing compound in order to best examine the effects of the curing compounds.

SOPEP solutions tested consisted of 30% phosphate, 3.5%, 2.5%, 1% phosphate, 1% phosphate plus 12% maleic acid, and no phosphate.

#### Results

#### **SOPEP Synthesis**

Initially, nine soy polyols with various levels of phosphate ester content were synthesized. They were then characterized for various physical properties and chemical parameters (Table 2). Table 3 shows the dispersibility of the SOPEP variants in water with an amine counter-ion.

TABLE 2. SOPEP Physical Properties

Percent Phosphate In SOPEP 4 5 6 8 10 20 30 2 3 0.01 0.07 0.06 0.04 0.03 0.04 0.03 0.02 0.02 Oxirane. (%) Acid Value. 65.9 89.2 104 132 176 26.4 32 59.7 (mgKOH) 11.8 Hydroxyl Value 244 255 263 259 256 279 253 260 (mgKOH) 258 4790 4410 3160 2860 2690 2310 3140 3610 4350 Viscosity (cps) 2717 Mw 3770 6813 7125 7917 7285 3918 2 1.7 1.8 2.7 2.8 2.7 2.8 Mw/Mn

TABLE 3. Dispersibility vs. Acid Number of SOPEP

		Counter	<u>Dispersible in </u>	
% H3PO4	Acid number	Ion	Concrete	Water
1	2.5	RNH3+	NO	NO
1-12*	65	NH4+	-	YES
2	8.5	46	-	NO
4	32		-	YES
6	56	۲,	-	YES
8	69	٠,	-	YES
10	106	٠,	-	YES
20	132	((	-	YES
30	176	((	-	YES
0**	0	SDS	-	YES

<sup>\* 1%</sup> H3PO4 and 12% maleic acid adduct. \*\* Epoxidized soybean oil.

SOPEP3.5 had surprising corrosion resistance in coatings of metal substrates in another project and was evaluated for this waterproofing application. Sample 1-12 in Table 2 contained 1% Phosphate and 12% maleic anhydride for water dispersibility. ESBO is the oil sample with zero phosphate.

#### **Concrete Tests**

#### Soy Phosphate Ester as a Workability Additive

The results indicate that soy phosphate ester polyols function as air-entraining, water-reducing, retarding, and workability additives as expected. Flow of mortar increased for polyol containing 1% phosphate as the percentage of solids increased (Figures 1-2 and Table 4). Slump was reduced about 42% by 0.5% soy phosphate/maleic additive by weight; 2.5% oil gave a 69% increase in slump; but increasing to 5% SOPEP1-12 was counter-productive and gave the same slump as the untreated mortar (Figure 1). The ESBO treated fresh concrete became moister and creamier as the level increased. The 3.5% soy phosphate treated fresh concrete was very creamy at 0.5% oil, but at 5% and 10% was significantly drier and less workable indicating significant rapid interaction of the phosphate groups with the cement. Soy phosphate is a plasticizer at concentrations around 0.1-2%, but not at levels of 5-30%. It is not as effective as commercial superplasticizers. At 15% soy additive with 1% PO4 added as the pure oil (not dispersed in water), the oil separated from the fresh concrete.

FIGURE 1. Concrete Slump with SOPEP1-12

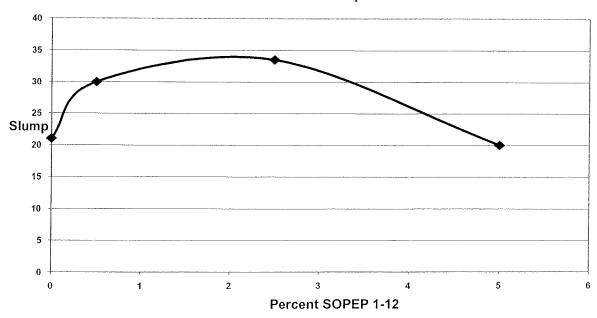


FIGURE 2. Effect of oil phosphate on flow

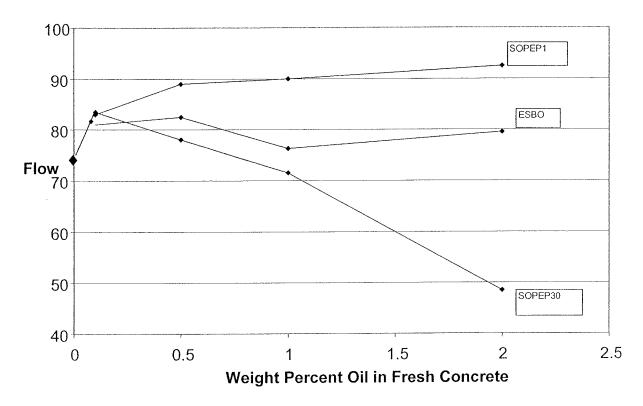


TABLE 4. Compressive Strength and Flow Results

		7 Day		14 Day		28 Day	
% Solids	Flow	Air Cured psi	Moist Cured psi	Air Cured psi	Moist Cured psi	Air Cured psi	Moist Cured psi
0	73	5026	4921	5384	5586	5921	5745
0.1	81 82.5	5233	5250 5003	5591 5801	5921 5776	5848 6128	6135 6273
1	76.3	4384	5134	4863	5374	5750	5655
2	79.5	4058	4416	4351	4885	5121	5385
0.1 0.5 1	83 89 90	4988 3943 4298	5156 4664 4135	5881 4368 4686	5769 4941 4700	5543 5631 5413	6106 5631 5775
2	92.5	3545	3946	4145	4570	4576	4941
0.5	83.5 78 71.5 48.5	4839 4253 2778 1328	4866 3970 3496 2409	5145 4840 3093 1638	4871 4713 4480 3048	5575 5495 3658 1994	6009 4915 4658 3915
	0.1 0.5 1 2 0.1 0.5 1 2 0.1 0.5	Solids         Flow           0         73           0.1         81           0.5         82.5           1         76.3           2         79.5           0.1         83           0.5         89           1         90           2         92.5           0.1         83.5           0.5         78           1         71.5	%         Air Cured psi           0         73         5026           0.1         81         5233           0.5         82.5         4818           1         76.3         4384           2         79.5         4058           0.1         83         4988           0.5         89         3943           1         90         4298           2         92.5         3545           0.1         83.5         4839           0.5         78         4253           1         71.5         2778	%         Air Cured psi         Moist Cured psi           0         73         5026         4921           0.1         81         5233         5250           0.5         82.5         4818         5003           1         76.3         4384         5134           2         79.5         4058         4416           0.1         83         4988         5156           0.5         89         3943         4664           1         90         4298         4135           2         92.5         3545         3946           0.1         83.5         4839         4866           0.5         78         4253         3970           1         71.5         2778         3496	%         Cured Solids         Flow Psi Psi Psi         Moist Cured Psi Psi Psi         Air Cured Psi Psi Psi           0         73         5026         4921         5384           0.1         81         5233         5250         5591           0.5         82.5         4818         5003         5801           1         76.3         4384         5134         4863           2         79.5         4058         4416         4351           0.1         83         4988         5156         5881           0.5         89         3943         4664         4368           1         90         4298         4135         4686           2         92.5         3545         3946         4145           0.1         83.5         4839         4866         5145           0.5         78         4253         3970         4840           1         71.5         2778         3496         3093	%         Cured Solids         Moist Plow Psi         Moist Psi         Air Cured Psi         Moist Psi         Psi	%         Cured psi         Moist Cured psi         Air Cured psi         Moist Cured psi         Moist Cured psi         Air Cured psi         Moist Cured psi         Air Cured psi           0         73         5026         4921         5384         5586         5921           0.1         81         5233         5250         5591         5921         5848           0.5         82.5         4818         5003         5801         5776         6128           1         76.3         4384         5134         4863         5374         5750           2         79.5         4058         4416         4351         4885         5121           0.1         83         4988         5156         5881         5769         5543           0.5         89         3943         4664         4368         4941         5631           1         90         4298         4135         4686         4700         5413           2         92.5         3545         3946         4145         4570         4576           0.1         83.5         4839         4866         5145         4871         5575           0.5         78

7

#### **Effect of Oil Content on Compressive Strength**

Except for SOPEP30, the oils at the 0.1% to 1% level, gave similar results at 28 days of cure as the control (Table 4 and Figures 3-4). In most cases the compressive strength of the specimens containing more than 2% SOPEP were significantly less than the control (Table 5 and Figures 3-6. Even neutral epoxidized soybean oil at 2% by weight of the cement, reduced compressive strength significantly (Table 4 and Figure 3). Soy phosphate ester polyols when added at 2% by weight of cement reduced compressive strength by 14% or more and ESBO at 2% reduced compressive strength by about 6%. These results probably limit the use of SOPEP in concrete to less than 2% by weight, and ESBO to somewhere around 2-5%.

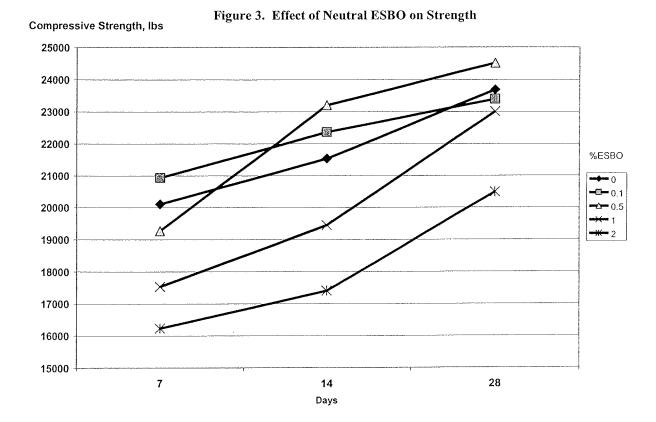


FIGURE 4. 7-Day Compressive strength vs. percent phosphate-solids

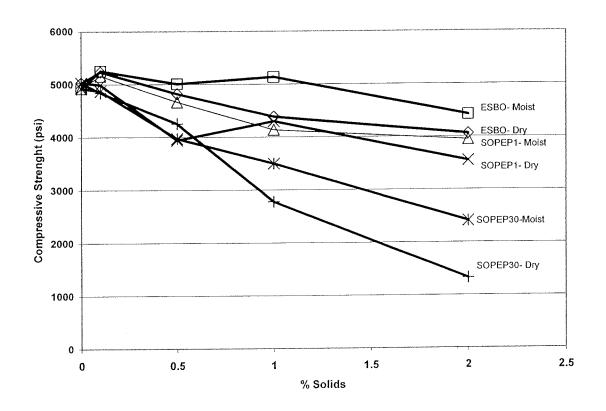


FIGURE 5. 14-Day Compressive strength vs. percent phosphate solids

14 Day Strength vs % Solids

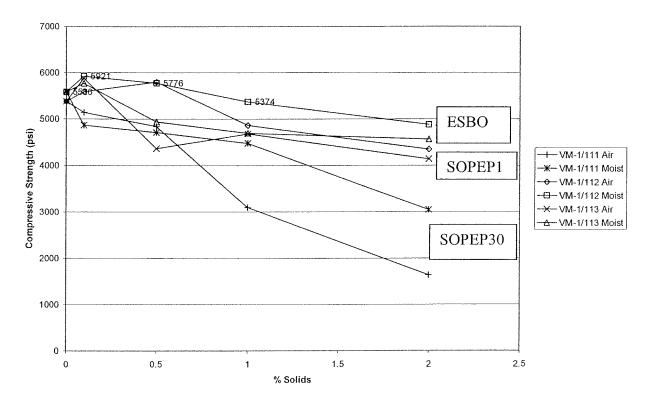
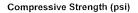
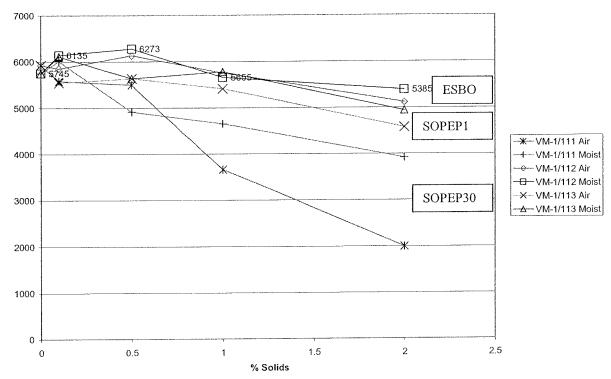


FIGURE 6. 28-Day Compressive strength vs. percent phosphate solids





#### **Effect of Phosphate Content**

Soy phosphate ester polyol containing 1% PO4 increased slump by 13% at 0.1% in the fresh concrete. Slump was 26% improved at 1% in the concrete (Figure 1). SOPEP1-12 gave a 42% reduction in slump at 0.5% concentration. Compressive strengths were essentially unchanged at up to 1% soy phosphate additive, but deteriorated significantly at 2% soy additive. Soy phosphate additive with 30% PO4 had small effects on slump and compressive strength at 0.5%, but at higher levels it negatively impacted slump and compressive strength. Extremely high levels of PO4 in the soy additive have negative effects on concrete performance. Epoxidized soybean oil (Zero PO4) dispersed in water with surfactant and added to concrete at 0.1% by weight reduced slump by about 11%. The effect on compressive strength was negligible.

#### Effect of Soy Additive on Concrete Water Absorption

The addition of 2% SOPEP1 in concrete reduced water absorption by only 6.6%. This was considerably less than the siloxane 90% water absorption reduction discussed earlier. This is much less than we had expected. It is suspected that the free acid of the SOPEP reacted sooner and more thoroughly with the cement than anticipated from preliminary studies with fast setting plaster of paris. We then tested ESBO with no phosphate (Table 5) and SOPEP3.5 (Table 6). SOPEP3.5 was used to see if this somewhat higher molecular weight material (compared to soy polyol with 1% PO4) would act as a polymer concrete additive. Water absorption of the concrete made with ESBO decreased linearly with additional increments of ESBO and was reduced by 57% at 10% ESBO in the concrete (Figure 7). This was still considerably less than that of the siloxane admixture. For concrete made with 5% SOPEP3.5, water absorption decreased 24%, but increased at the 10% loading in one experiment. Clearly, neither of these low Mw products acts like high Mw latex does in polymer concrete.

Since 10% by weight of neutral, but hydrolyzable, ESBO blocked 48% of water absorption in concrete, but reduced concrete strength somewhat, it could be polymerized and that polymer investigated as an inexpensive ingredient for latex concrete. At normal levels of 15% used in latex modified concrete it would be expected to reduce water absorption further and give a stronger concrete. Initial attempts to make the desired ESBO polymer were not successful before the contract expired.

Table 5: Results of Water Absorption Tests with ESBO (No Phosphate)

(140 I nospitate)						
VM-1-112	No Phosph	ates	ESBO	1		
		Dry	Immersed			
		Mass		Absorption		
Control	Specimen	(g)	Mass (g)	%	Avg	
	1	277.1	298.4	7.69	7.69	
	2	274.9	296	7.68		
	3	280.7	301.9	7.55		
	4	269.2	290.1	7.76		
	5	268	288.9	7.80		
	6	269.8	290.4	7.64		
0.50%						
	1	270.9	291.5	7.60	7.44	
	2	273.6	293.3	7.20		
	3	269.5	290.2	7.68		
	4	268.8	288.8	7.44		
	5	269	289.3	7.55		
	6	272.1	291.6	7.17		
5%			•			
	1	270.1	285.3	5.63	5.21	
	2	269.8	285	5.63		
	3	270.9	285.8	5.50		
	4	270.3	282.8	4.62		
	5	269.7	282.7	4.82		
	6	269.6	283.3	5.08		
10%						
	1	269.9	280.2	3.82	3.27	
	2	269.4	280	3.93		
	3	269.6	280.4	4.01		
	4	266.8	273.6	2.55		
	5	268.6	276	2.76		
	6	268.7	275.5	2.53		

TABLE 6. Results of Water Absorption Tests with SOPEP3.5 (3.5% Phosphate)

VM-1-142 3.5% P		sphates	SOPEP3.5	-	
		Dry	Immersed		
		Mass		Absorption	
0.50%	Specimen	(g)	Mass (g)	%	Avg
	1	259.7	279.3	7.55	7.60
	2	256.6	276.1	7.60	
	3	257.5	277.2	7.65	
	4	257	277.1	7.82	
	5	260.2	279.2	7.30	
	6	258.6	278.4	7.66	
5%					
	1	263.6	279.1	5.88	5.74
	2	263.5	279.3	6.00	
	3	261.8	275.7	5.31	
	4	263	278.9	6.05	
	5	262.4	278.4	6.10	
	6	262.9	276.4	5.14	
10%		1			
	1	244.3	265.6	8.72	8.15
	2	244.7	260.4	6.42	
	3	245.4	265.8	8.31	
	4	247.4	265.9	7.48	
	5	244.4	264.6	8.27	
	6	245.8	269.7	9.72	

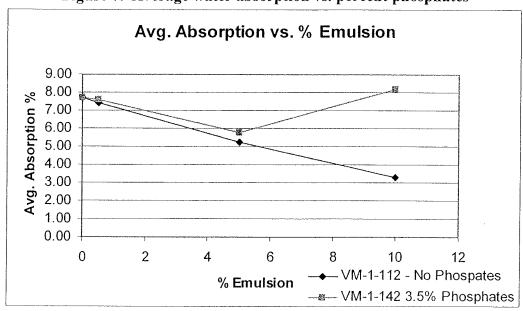


Figure 7. Average water absorption vs. percent phosphates

#### Soy Polyol as Concrete Curing Agent

Concrete cures over time and gains strength with age as long as any unhydrated cementing materials remain, provided the concrete remains moist or has a relative humidity above 80% and the concrete temperature remains favorable. When the relative humidity within the concrete drops to about 80% or the temperature of the concrete drops below freezing, hydration and strength gain virtually stop. It is best to moist-cure concrete continuously from the time it is placed until it has reached the desired properties because concrete is difficult to resaturate.

A curing agent is a coating to retard evaporation of water so that the concrete retains enough water through out the cure to develop maximum strength. This would eliminate the need for frequent wetting of the surface of curing concrete in dry climates. The soy phosphate ester polyol dispersion was expected to be sucked into surface capillaries during the drying process and stick to the walls as originally expected from the mix. SOPEP3.5 was tested as curing agent for concrete (Table 7). Table 7 shows that 50% aqueous dispersion of SOPEP3.5 gave results somewhat better than air drying. The commercial curing agent evaluated as a standard gave results worse than the air dried concrete.

TABLE 7. Soy Polyol as Curing Additive

28-Day Flexural Strength Results on 3x3x12 Beams

·	Specimen	Load (k)	Modulus of Rupture (psi)	Avg. (psi)
Control	1	1.604	594	
Air Cured	2	1.5065	558	576
	3*	1.9201	711	
Commercial	1	1.477	547	
<b>Curing Compound</b>	2	1.3293	492	536
	3	1.5361	569	
SOPEP	1	1.62476	602	
50% Solids	2	1.62475	602	591
	3	1.53614	569	

**Ongoing Research** 

Coating rebar with SOPEP would give a monomolecular layer of iron phosphate-soy polyol bonded to the rebar. This layer would be a corrosion resistant semi-permanent lubricant layer. If effective in retarding corrosion in concrete this would be a low cost, durable, non-chipping, non-cracking method to retard corrosion of rebar in service. This test is in progress, but the results will not be determined until after this contract expires.

#### **Plans for Implementation**

To implement this concept product, the performance must be improved. Possible ways to improve the concept product are:

ADMIX- 1) Use calcium salts of SOPEP so that the SOPEP is not captured by the cement too soon.

2) Increase the molecular weight of the SOPEP so it performs like latex resin, but at a lower cost.

CURING AGENT- Use a dilute solution of SOPEP>10. SOPEP>10 appears to be soluble in water and gives a clear solution; indicating that any oil droplets are smaller than the wavelength of light. These smaller particles should readily penetrate the concrete and lock the oil into the surface layers of the concrete, thus preventing the rapid evaporation of water.

#### **CONCLUSIONS**

SOPEP phosphate-amine complex reacts too soon and too completely with cement particles before the capillaries in concrete are formed. The SOPEP is not concentrated in the capillaries as expected and water absorption is too high for practical benefits. Neutral ESBO at 10% in the concrete does appear to collect in the capillaries as expected and does reduce water absorption by 48%; but at this level of oil, the concrete compressive strength is reduced too much. A higher molecular weight of ESBO or SOPEP with a calcium or sodium counter-ion would be expected to deliver better waterproofing performance and improve concrete physical properties.

SOPEP as a curing agent gave modest improvements in compressive strength over the air cured concrete. A water soluble SOPEP might give improved performance in this application.

Admixtures are used to modify certain properties of the concrete and can be classified according to their function. SOPEP as an admix behaved as follows:

- 1. Air-entraining admixtures. SOPEP made creamy fresh concrete.
- 2. Water-reducing admixtures. SOPEP will allow reduced water in fresh concrete for fixed fresh concrete flow.
- 3. Retarding admixtures. SOPEP retards concrete cure.
- 4. Workability agents. SOPEP increases workability.
- 5. Miscellaneous agents for bonding, damp proofing, and permeability reducing. SOPEP gave marginal reductions in permeability at modest reductions in compressive strength.

#### INVESTIGATOR PROFILE

Dr. John L. Massingill, Jr. has an extensive background in academic and industrial research, development, and pilot plants for resins and monomers, as well as determination of market value and market introduction of plaster additives with the Dow Chemical Company. Dr. Massingill is the Director of the Center for Coatings and Bio-Based Technology at Texas State University at San Marcos. He was Director of the Coatings Research Institute and Acting-Director of the National Science Foundation Industry/University Cooperative Research Center at Eastern Michigan University before returning to his home state.

Dr. David W. Fowler is the Joe J. King Chair in Engineering No. 2 at The University of Texas at Austin where he teaches courses in structures, materials, forensic engineering and repair. Dr. Fowler also performs research in concrete polymer materials and other special concretes, aggregates, building systems, and recycled materials. Dr. Fowler serves as Director of the International Center for Aggregates Research which is operated jointly by UT Austin and Texas A & M University. Dr. Fowler currently served as chair of the ACI Concrete Research Council and is a member of the National Academy of Engineers. He is a Fellow of the American Concrete Institute and of the American Society of Civil Engineers. He is past president of the International Congress on Polymers in Concrete and serves on many national and international committees. Dr. Fowler is a registered professional engineer in Texas.

#### **GLOSSARY**

ESBO- Epoxidized soybean oil.

Concrete- A mixture of cement, aggregate, admixes, and water.

SOPEP- Soybean oil phosphate ester polyol. The first number following SOPEP indicates the amount of phosphate in the oil.

Slump- A measure of the viscosity and ability of the concrete to flow. An indication of how well forms will be filled by the concrete.

Water absorption- The amount of water that concrete absorbs on immersion in water. A measure of the permeability of the cured concrete.

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