Linseed Oil on Concrete: Penetration of Linseed Oil Compositions Into Sheet- and Liquid Membrane-Cured Concretes[1]

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ABSTRACT

Penetrations of concrete specimens by solutions of boiled linseed oil in mineral spirits and emulsions of boiled linseed oil were measured. Concrete specimens were cured with polyethylene sheet (removable) and wax-based and resin-based compounds prior to penetration tests. Penetration was greatest at 35 days and least at four and seven days after curing. Penetration at 105 days was only slightly less at 35 days. Maximum penetrations were obtained with solutions more dilute than 50 volume per cent of linseed oil in mineral spirits. Penetrations on polyethylene-, wax- or resin-cured specimens varied from 1.5 to 3 mm when 50 volume per cent linseed oil solution or emulsion was spread on the specimen at the rate of 0.16 lb/yd^2.

INTRODUCTION

The deterioration of concrete caused by deicing salts and freeze-thaw cycles greatly concerns highway people, the Portland Cement Association and others (1,2). In searching for a solution to this problem, member companies of the National Flaxseed Processors Association, a number of State Highway Departments and the Northern Laboratory have shown that boiled linseed oil in mineral spirits or as an emulsion in water is one of the most effective agents in reducing damage to concrete caused by freeze-thaw cycles and salt (3-9). These linseed oil antispalling compounds are usually applied to sheet-cured concrete about one month after pouring. Today, liquid membrane-curing compounds containing wax or resins are being used in increasing amounts. These materials leave a coating on the surface that may lower the effectiveness of the linseed antispalling compounds. We believe that some penetration of the boiled linseed oil into the concrete is necessary to obtain protection, but no detailed studies have been carried out to determine the best way to use linseed antispalling compounds on sheet-cured concrete, and no work has been done on whether or not these compounds will penetrate into concrete previously cured with typical commercial pigmented resin and wax compounds. Recent work at our Laboratory and field tests in two southwestern states have shown that properly formulated boiled linseed oil emulsions can be used as curing agents for concrete. After periods of one to five years, no deterioration from freeze-thaw and deicing chemicals is apparent in any of these field tests. Thus, we wanted to know if the linseed emulsion used as a curing agent would penetrate and if LASC would penetrate month-old concrete previously cured with these linseed emulsions. Because of this lack of knowledge, work was undertaken to study the penetration of linseed oil into concrete under a variety of conditions and to correlate this information eventually with companion durability studies.

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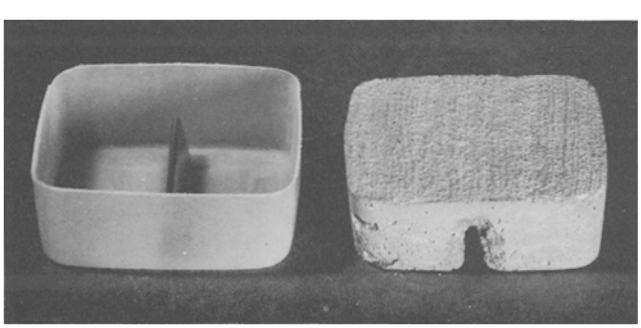

FIG. 1. Left, concrete specimen dish showing stainless-steel wedge in position. Right, a typical unbroken block specimen.

EXPERIMENTAL PROCEDURES

Concrete test specimens were prepared according to the

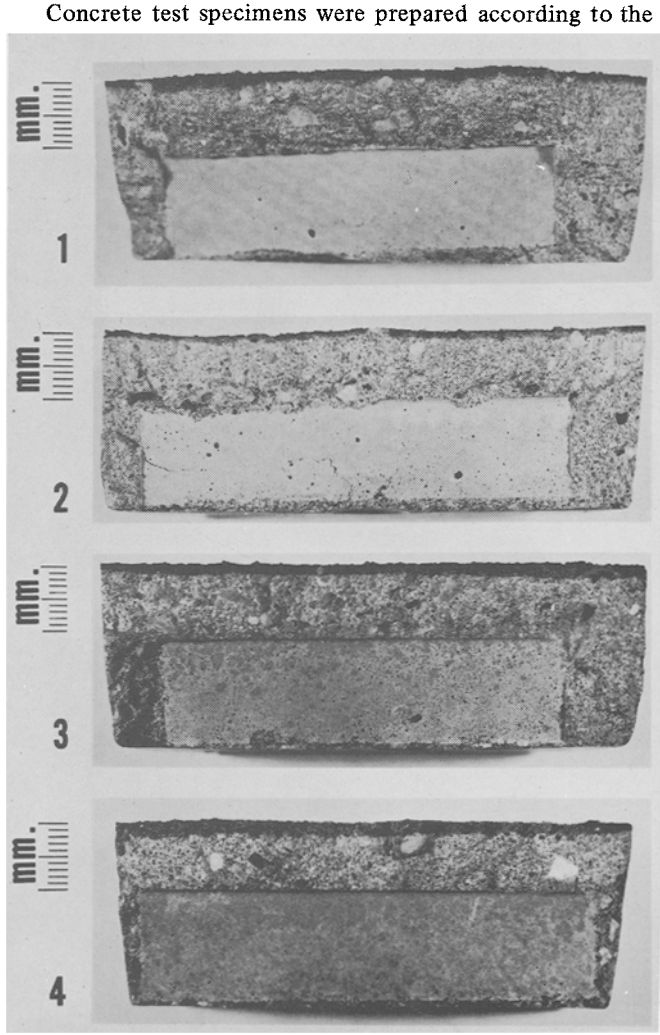

FIG. 2. Concrete test specimens showing penetration of 50% boiled linseed oil in mineral spirits (LASC) into polyethylene-cured concrete between 1 and 35 days old. (1) LASC applied at one day, stored indoors; (2) LASC applied at seven days, stored indoors; (3) LASC applied at 35 days, stored indoors; (4) LASC applied at 35 days, stored indoors seven days then outdoors 28 days.

TABLE I

Penetration of Linseed Oil Compositions Into Sheet- and Liquid Membrane-Cured Concrete Treated Between 1 and 105 Days After Casting (Measured as Depth of Penetration in Millimeters)

Age of concrete,[a] days	Polyethylene cured		Pigmented wax cured		Pigmented resin cured	
	LASC[b]	LSOE[b]	LASC	LSOE	LASC	LSOE
1	1-2	---	1-1.5	1-1.5	---	---
4	0.5-1	---	0.5-1	0.5	---	---
7	0.5-1	---	0.5-1	0.5	1.0	---
14	1-2	---	1-1.5	1-1.5	1-1.5	---
35	2-2.5	2.0	2.0	1.5-2	2-3	1.5
35[c]	2-2.5	2-3	2-2.5	2.0	3.0	2.0
105	1-2	2.0	---	---	---	---

[a]When treated with linseed oil compositions at 78 F and stored indoors.

[b]LASC, 50 volume per cent boiled linseed oil in mineral spirits. LSOE, water emulsion containing 50 volume per cent boiled linseed oil. Application rate, 0.16 lb/yd^2.

[c]When treated with linseed oil compositions at 78 F and stored outdoors.

following formula: Sand and gravel (all particles <1/4 in. in diameter), 3 parts, 4800 g; cement, 1 part, 1600 g; water, 0.5 part, 800 g; air entraining agent (Darex), 1.2 ml.

Concrete blocks were prepared in polyethylene dishes (4 x 4 x1 1/8 in.) by a method developed by A. Arters of Sherwin-Williams Company, Cleveland, Ohio (Fig. 1). A stainless-steel wedge (4 x 3/4 in.) is placed in the center of each dish, and the dish overfilled slightly with concrete. The wedge produces a weak area along which the block later can be easily broken. As soon as free water disappears, the excess concrete is screeded off. After about 2 hr, the surface is finished with a wooden block and a stiff paint brush (1 x 1 7/8 in., angle-cut, beveled, hog bristle) to simulate a bridge deck or highway surface. The blocks are immediately sprayed with a curing compound at the recommended rate (usually 200 ft^2/gal) or the surface of the block is covered with polyethylene. In addition to polyethylene and wax and resin curing compounds, boiled linseed oil emulsion (LSOE) or emulsions containing mixtures of boiled and bodied linseed oils were used as curing agents. The blocks are cured for seven days at room temperature indoors, removed from the dishes and then stored in the laboratory or outdoors on the laboratory roof until coated with linseed antispalling compound. In a few tests, linseed antispalling compounds were applied when the concrete was one and four days old but curing in the dish was always continued for a total of seven days.

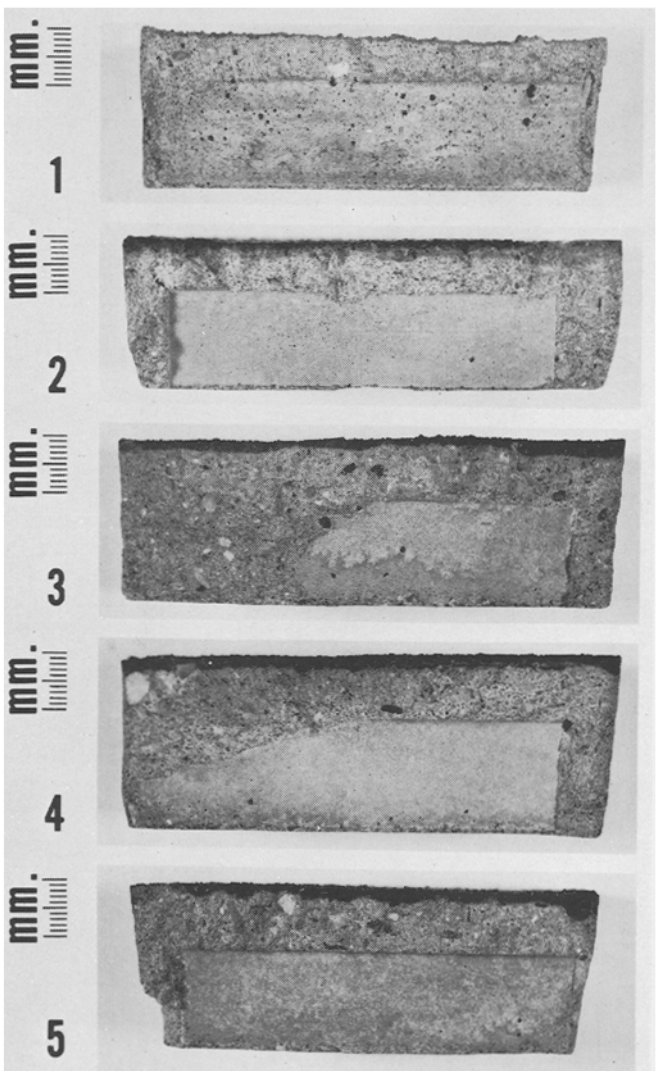

FIG. 3. Concrete test specimens showing penetration of LASC and an emulsion of 50% boiled linseed oil in water (LSOE) into liquid membrane-cured concrete. (1) A wax-curing compound; (2) LASC through a wax compound; (3) LASC through a resin-curing compound; (4) LSOE through a resin-curing compound; (5) LASC through a pigmented resin-curing compound.

Boiled linseed oil in mineral spirits (LASC) or LSOE were used as antispalling compounds. They were sprayed on at the rate of 0.16 lb/yd^2 unless otherwise indicated. The term 50% LASC denotes a solution of 50 volume per cent boiled linseed oil in mineral spirits and 50% LSOE is an emulsion of 50 volume per cent boiled linseed oil in water (10). In some tests, concentrations of boiled linseed oil other than 50% in mineral spirits or emulsions were used, and these compositions are specified.

Specimens were broken four days after treatment with antispalling agent (six days for blocks treated at one day) and penetration of oil into the concrete could be seen as a faint gray-yellow area on the broken side near the cured top

TABLE II

Effect of Compositions Differing in Concentration of Linseed Oil on Penetration Into Concrete 30 and 105 Days Old (Measured as Depth of Penetration in Millimeters)

Antispalling compound	Polyethylene cured		Pigmented resin cured	
	30 Days[a]	105 Days[a]	30 Days	105 Days
25% LASC[b]	6.0	4-5	3-5	4-5
50% LASC	2-3	1-2	2-3	2-4
75% LASC	—	—	1-2	—
2-25% LASC[c]	6-7	5-7	6-7	4-5
25% LSOE	—	1.5	1.0	—
50% LSOE	—	1.5	—	—
2-25% LSOE[c]	—	—	1-2	—

[a]See footnote a, Table I.

[b]One coat of indicated volume per cent of boiled linseed oil in mineral spirits (LASC) or emulsion (LSOE) applied at 0.16 lb/yd^2.

[c]Two coats of indicated volume per cent of boiled linseed oil in mineral spirits (LASC) or emulsion (LSOE) applied at 0.16 lb/yd^2 each.

TABLE III

Effect of Concentration of Oil in Linseed Oil Compositions and of Temperature on Penetration Into Polyethylene- and Pigmented Resin-Cured Concrete 30 Days Old (Measured as Depth of Penetration in Millimeters)

	Curing conditions				
	34 F[b]		78 F	100 F	
Per cent LASC[a]	PE[c]	PR[c]	PE	PE	PR
20	3-6	2-4	1.0	3.5-4	2.5
25	3.0	—	2.0	2.5-3	—
30	3-4	1.5-2.5	1.5-3	1-2	1-2
35	2-2.5	1-2.5	1.0	2.0	1-2
40	2.0	1.5-2	1-2	2.0	1.0
50	2.0	1-2	—	2-2.5	1.0
50 (control, 78 F)	2.0	1-2	1-2	2.0	0.5-1

[a]See footnote b, Table II.

[b]Temperature of concrete when LASC was applied and for five days thereafter.

[c]PE, polyethylene cure; PR, pigmented resin cure.

of the block. However, a better method for determining penetration was to spray the broken side of the block with a 50% solution of sulfuric acid and bake in an oven at 130 C for 30 min. The acid treatment charred the organic material and produced a black area where the oil had penetrated. Figure 2 is a photograph of selected blocks and illustrates the relative ease with which penetration can be seen from the sulfuric acid test. This test was developed in cooperation with members of Sub-Committee V, National Flaxseed Processors Association.

DISCUSSION

In Table I are shown results of tests made to determine the penetration of linseed oil compounds into concrete at time intervals up to 105 days. Thus, 50% LASC at 0.16 lb/yd^2 was applied to polyethylene-cured concrete after 1, 4, 7, 14, 35 and 105 days. The first specimen was cured for one day and then treated with LASC. Penetration occurred to a depth of 1-2 mm. Specimens sprayed with LASC after curing 4 and 7 days showed little penetration while the 14-day treatment gave penetration similar to the 1-day specimen. Specimens treated with LASC at 35 days gave the deepest penetration. No difference in penetration was observed between indoor and outdoor specimens. A specimen treated with LASC after 105 days had a penetration somewhat less than the 35-day specimens but equal to the concrete treated at 14 days. Table I lists the penetration of LSOE applied to indoor and outdoor samples at 35 days and indoors at 105 days of age. Penetration of the emulsion was similar to corresponding specimens treated with LASC.

We have also demonstrated that linseed oil penetrates concrete cured with some commercial resin and wax compounds (Fig. 3). Block 1 was treated with wax-curing compound only, broken after 30 days of aging, sprayed with 50% aqueous sulfuric acid and baked to develop the thin black line that represents essentially no penetration. Results were similar when blocks were treated with resin and resin-pigmented curing compounds. Blocks 2-5 were cured with a wax- or resin-curing compound and treated 30 days later with 50% LASC or LSOE. Linseed oil penetrated the concrete through all wax- and resin-cured test specimens whether it was in mineral spirits solution or as an emulsion.

Table I tabulates the penetration by 50% LASC and LSOE of concrete blocks previously cured by a commercial pigmented wax compound. LASC applied one day after pouring penetrated the concrete 1-1.5 mm. When LASC was applied at 4 or 7 days, penetration reached a depth of 0.5 to 1 mm. At 14 days, penetration was equal to the 1-day sample. After 35 days, penetration of LASC was significantly greater than at shorter lengths of time.

Specimens cured with pigmented wax compound and treated with 50% LSOE instead of LASC showed that some penetration occurred at one day and less penetration at four and seven days. At 14 days, the depth of penetration increased and was 1-1.5 mm. Wax-cured specimens sprayed with linseed emulsion at 35 days showed a penetration of 1.5-2 mm for specimens exposed both indoors and outdoors.

Table I also lists a series of tests conducted with a commercial pigmented resin-curing compound instead of wax and subsequently sprayed with LASC or LSOE. Depths of penetration were similar to the wax-cured specimens.

Concrete cured under polyethylene or by spraying with commercial wax- or resin-curing compounds and subsequently treated with 50% of either LASC or LSOE was penetrated. Penetration of antispalling compounds was greatest when the concrete was treated one month after curing. With concrete up to 35 days old and treated with 50% solution or emulsion, all methods of curing tested gave similar penetration patterns with time and suggest that the depth of penetration of these antispalling compounds into new concrete depends more on the age of the concrete at the time of applying the linseed oil compound than it does on the curing method used.

Table II records data on concrete blocks cured under polyethylene or the commercial resin and treated with solutions of LASC or LSOE that contained different amounts of boiled linseed oil, e.g., 25% LASC represents 25% by volume of oil in mineral spirits. With the samples that were 30 days old when treated with LASC solutions, the 50% solution penetrated to about 2.5 mm, whereas the

TABLE IV

Penetration of Linseed Emulsion Curing Agents and Linseed Antispalling Solutions Into Concrete

Curing agent[a]		Antispalling agent,[b] total penetration, mm
Emulsion type, %	Penetration, mm	
50 LSOE	2.0	3.0
20[c]	1.0	3.0
30	0.6	2.2
40	0.4	2.0
50	0.2	0.2

[a]Applied at 200 ft^2/gal.

[b]Fifty per cent LASC applied at 0.16 lb/yd^2 to linseed emulsion-cured concrete 25 days old.

[c]Emulsion of 50 volume per cent mixture of boiled and bodied linseed oils in water. Values given indicate volume per cent of bodied Z8 linseed oil in boiled linseed oil.

25% solution penetrated more than twice as much. If we apply 25% LASC in two coats of 0.16 lb/yd^2 each and allow 24 hr between applications, we obtain slightly deeper penetration than with the single application. No oil film remains on the surface of the block.

A similar study was conducted on concrete specimens cured under a commercial pigmented resin-curing compound instead of polyethylene. Penetration was greatest with 25% LASC and the specimen coated twice with this solution showed the most penetration. In general, resin-cured specimens treated with LASC displayed about the same penetration pattern as the corresponding polyethylene-cured blocks. When resin-cured specimens treated with one or two coats of 25% LSOE were compared with the corresponding LASC blocks, much less penetration was observed.

Table II also lists penetration on blocks treated with linseed oil solutions and emulsions after 105 days. When polyethylene cured LASC-coated blocks treated at 30 and 105 days are compared, concrete 30 days old was penetrated to a greater depth by LASC than concrete 105 days old. When 105-day-old concrete was treated with LSOE, there was no difference in penetration between 25% and 50% emulsions. When the resin-cured blocks treated with LASC at 30 and 105 days are compared, the penetration depth of the 25% and 50% solutions were similar. When 25% LASC was applied in two coats over the resin-curing compound, penetration was deep but uneven.

Table III shows data on polyethylene and pigmented resin-cured concretes that had been treated with several concentrations of LASC at 35, 78 and 100 F. Different batches of concrete were used to prepare the blocks for each temperature study, and a control specimen was included as a check on the variability of the concrete. A comparison of the polyethylene-cured controls in Table III with corresponding polyethylene cured blocks treated with 50% LASC shown in Tables I and II illustrates how variability between batches of concrete made in the same way affects penetration. Concrete specimens listed in Table III are somewhat less porous than those listed in the other Tables. Although this variability precludes direct critical comparisons of data in the Tables, some trends can be seen.

Penetration of LASC occurred with all polyethylene-cured specimens at the three temperatures studied (Table III), and greatest penetration was observed with the LASC solutions in the 20-30% range. Since 50% LASC gave moderate penetration in the 34-100 F range, the 50% LASC normally used by highway departments should penetrate sheet-cured concrete under most conditions encountered in field work.

At 34 and 100 F greater penetration of pigmented resin-cured concrete occurred with 20-35% LASC than with solutions in the 40-50% range. When the pigmented resin-cured controls in Table III are compared with the 35-day 50% LASC treated block in Table I, it is evident that tests at 34 and 100 F were done on less porous concrete, particularly the tests at the high temperature. Because of the apparent difference in porosity between 34 and 100 F specimens, we cannot conclude from these data whether there is a difference in penetration between corresponding specimens at 34 and 100 F.

Table IV shows penetration data on concrete specimens cured with 50% LSOE or 50% mixed emulsions that contained Z8 bodied linseed oil in the boiled oil. The curing agent was applied at 200 ft^2/gal and the LASC applied 25 days later at 0.16 lb/yd^2. Clearly, 50% LSOE when used as a curing agent penetrated the concrete. The LASC antispalling agent also penetrated the concrete cured with emulsions where the oil phase of the curing agent contained up to 40 volume per cent of Z8 oil. Penetration of LASC is markedly less into concrete cured with emulsions containing 50% Z8 oil.

We should point out that we tested only a few of the wax- and resin-curing compounds commercially available. Although others might allow more or less penetration of the antispalling compound, we selected curing compounds that, to the best of our knowledge, were representative.

REFERENCES

1. Concrete Information: Protection of Existing Concrete Pavements from Salt and Calcium Chloride, Portland Cement Association, Chicago, Ill.
2. Concrete Information: Effect of Various Substances on Concrete and Protective Treatments Where Required, Portland Cement Association, Chicago, Ill.
3. Kubie, W.L., L.E. Gast and J.C. Cowan, "Linseed Oil for the Preventive Maintenance of Concrete," Highway Research Board, Maintenance Practices 1967, No. 254, p. 61-67, 1968.
4. Morris, C.E., "The Use of Linseed Oil for the Protection of Portland Cement Concrete Surfaces," *American Road Builders* Association Tech. Bull. No. 257, 1965.
5. Kubie, W.L., and J.C. Cowan, JAOCS 44:194-196 (1967).
6. Synder, M.J., "Protective Coatings to Prevent Deterioration of Concrete by Deicing Chemicals," Highway Research Board, National Cooperative Highway Research Program Report 16, 1965.
7. Grieb, W.E., and R. Appleton, "Effect of Linseed Oil Coatings on Resistance of Concrete to Scaling," Public Roads, Vol. 33, No. 1, April 1964.
8. Yamasaki, R.S., J. Paint Technol. 39:394-397 (1967).
9. Walsh, R.J., Civil Eng. (New York), (1956(7):39-41.
10. Kubie, W.L., Sr., U.S. Patent 3,228,777 (1966).

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