

# LINSEED OIL FORMULATIONS AS CURING AND ANTISCALING COMPOUNDS FOR CONCRETE

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An extensive testing program was undertaken to determine the effectiveness of linseed oil in mineral spirits both as a curing compound and as an anti-scaling compound for inadequately air-entrained portland cement concrete. The testing program is discussed in light of the immediate and residual effectiveness and the value of such linseed oil formulation treatments.

•LABORATORY tests were conducted to determine the effectiveness of linseed oil in a mineral spirits solution as a curing and antiscaling compound for concrete. A discussion of the test materials, facilities and equipment, procedures, and results follows.

## MATERIALS

### Concrete

A single lot of Type I portland cement (ASTM C 150) was used. The entire supply of cement was blended into steel drums that were then sealed with gasketed lids to protect the cement from atmospheric water and carbon dioxide until time of use.

The coarse aggregate used was crushed limestone from a quarry in Waubunsee County, Kansas. It was graded within the limits for No. 67 in ASTM C 33 (sieve size:  $\frac{3}{4}$  in. to No. 4). Its measured absorption capacity was 1.38 percent of oven-dry weight.

The fine aggregate used was from a deposit in the Kansas River. This material is produced according to the specifications for state road and bridge construction (section J1-3) of the Kansas State Highway Commission.

Supplies of coarse and fine aggregates were individually tempered to the saturated, surface-dry condition and stored in 30-gal (114 litre) steel drums with gasketed lids. This was done to preserve the moisture condition until time of use when a given drum was emptied and the contents were thoroughly blended prior to batching to redistribute any possible moisture concentration that may have occurred during storage.

The air-entraining agent was a commercial neutralized vinsol resin (ASTM C 494). Tap water was used for mixing.

### Curing and Antiscaling

Boiled linseed oil, mineral spirits, and resin-based and wax-based white-pigmented curing compounds (ASTM C 309) were obtained from a commercial supplier in lots sufficient for the entire project.

Boiled linseed oil emulsion developed by the Northern Laboratory was obtained from a commercial producer.

All other curing and antiscaling materials, including the National Flaxseed water-soluble curing compound, were supplied in quantity by the Northern Laboratory.

## FACILITIES AND EQUIPMENT

All work was carried out in the Department of Applied Mechanics laboratories, which house a full complement of standard concrete fabrication and testing equipment.

The surface abrasion apparatus was built according to drawings and instructions from the California Highway Commission. We followed the California testing procedure and call the results California abrasion loss, which may be interpreted as a relative measure of surface strength.

## PROCEDURES

Standards

Applicable portions of current ASTM specifications for concrete testing were adhered to throughout the project with the following exceptions:

Our old automatic freeze-thaw equipment conforms with the requirements of ASTM C 666 except for the requirement that a nominal cycle shall not exceed 4 hours. With a full load of 50 specimens in brine in individual polyethylene containers, the nominal cycle in our machine is 6 hours. Photography was used instead of weight loss and durability factor to record performance.

Abrasion resistance was determined by the California method rather than by ASTM C 418.

Concrete Mix Proportions

A single concrete mixture was proportioned by trial to yield a standard compressive strength of 4,000 to 5,000 psi (27.58 to 34.47 MPa) and 5 to 6 percent entrained air (by volume). (The prescribed amount of entrained air is inadequate because it is less than the average of 7 percent recommended by ACI for concrete containing  $\frac{3}{4}$ -in. maximum aggregate.) Properties of the resulting concrete were slump,  $3\frac{1}{2}$  in. (8.9 cm); unit weight, 140.1 lb/ft<sup>3</sup> (2244 kg/m<sup>3</sup>); air content, 5.5 percent (by volume); water/cement ratio, 6.2 gal/sack (0.55); cement factor, 5.1 sacks/yd<sup>3</sup> (284 kg/m<sup>3</sup>); and standard compressive strength, 4,530 psi (3123 kPa). Mixture proportioning was not an element of the study, therefore mix-determined proportions were used for making all test specimens.

Test Specimens

For each test condition (Table 4), two slabs 16 by 18 by 4 in. (40.6 by 45.7 by 10.2 cm) and three beams 3 by 4 by 16 in. (7.6 by 10.2 by 40.6 cm) were cast from a single concrete batch.

At the conclusion of the prescribed curing and surface treatment, three beams were sawed from one of the slabs, vacuum saturated, and tested in flexure with center-point loading (three values) and in modified-cube compression (six values).

The remaining slab was stored in the laboratory and air controlled at 70 F (21 C) and 50 percent relative humidity pending abrasion testing at 2, 4, and 6 months.

The formed faces of the three beams were coated with boiled linseed oil in mineral spirits to inhibit salt intrusion through these faces; the finished surface of each specimen was the only surface of interest. All beams were exposed to laboratory air at 70 F (21 C) and 50 percent relative humidity for a period of 14 days before being exposed to freezing and thawing in 2 percent sodium chloride solution.

Portions of beam ends from the flexure and modified-cube compression tests were treated on the fracture surface with a 50 percent sulfuric acid solution and were charred in an oven to delineate penetration of the prescribed surface treatment material into the concrete substrate.

## RESULTS

Data for strength in flexure and in modified-cube compression are given in Table 1, and California abrasion loss data for all conditions of test are given in Table 2. Typical durability results through 300 cycles of freezing and thawing in 2 percent NaCl brine

**Table 1. Compressive and flexural strength data.**

Condition	Compressive Strength			Flexural Strength		
	Strength (psi)	$\sigma$ (psi)	V (percent)	Strength (psi)	$\sigma$ (psi)	V (percent)
1100	5,040	290	5.75	810	67	8.29
1200	5,060	211	4.18	873	48	5.51
1300	4,990	211	4.22	882	20	2.28
1400	4,690	110	2.35	698	67	9.61
1500	4,860	73	1.50	843	36	4.26
2102	5,020	99	1.97	890	42	4.75
2201	5,210	130	2.49	785	14	1.82
2202	5,050	315	6.24	860	12	1.41
2301	4,170	178	4.27	574	12	2.12
2302	4,020	113	2.82	541	4	0.69
4001	5,120	193	3.77	785	59	7.46
4002	5,120	247	4.83	815	20	2.49
4003	4,970	120	2.41	849	22	2.63
4004	5,020	112	2.22	734	14	1.96
4005	4,850	195	4.02	647	34	5.21
4006	4,900	268	5.46	658	28	4.27
4007	5,160	223	4.33	886	33	3.77
4008	5,150	190	3.68	948	69	7.33
4009	5,270	292	5.55	909	30	3.35
4010	4,920	472	9.60	645	81	12.59
4011	5,240	107	2.04	679	32	4.66
4012	5,000	238	4.77	699	49	6.99
4013	4,260	159	3.73	537	31	5.72
4014	5,180	378	7.29	791	45	5.73
4015	5,060	306	6.05	781	33	4.25

Note: 1 psi = 6.894 757 kPa.

**Table 2. California abrasion loss data.**

Condition	California Abrasion Loss (grams)		
	2 Months	4 Months	6 Months
1100	29.5	29.7	29.9
1200	26.3	26.5	22.9
1300	29.8	29.4	23.5
1400	20.2	22.9	20.5
1500	23.9	24.6	24.5
2102	26.8	24.9	23.7
2201	35.3	28.3	29.8
2202	23.6	24.2	23.0
2301	33.0	34.3	32.7
2302	22.5	24.8	24.4
4001	26.6	25.4	23.1
4002	28.5	29.4	26.4
4003	25.5	26.6	23.7
4004	31.8	27.2	27.2
4005	26.4	23.0	25.0
4006	32.1	30.4	29.3
4007	27.0	25.7	25.2
4008	28.3	26.9	26.0
4009	27.1	24.6	25.8
4010	22.3	24.8	22.6
4011	24.2	22.5	21.9
4012	26.0	26.4	23.8
4013	22.8	27.0	24.0
4014	31.2	26.8	24.0
4015	33.7	31.1	31.1

**Table 3. Penetration data.**

Condition	Depth of Penetration of Surface Treatment Formulation (mm)	Condition	Depth of Penetration of Surface Treatment Formulation (mm)
1100	n/a	4004	1/2 to 1
1200	2	4005	<1/2
1300	1/2	4006	1/2 to 1
1400	1 to 2	4007	<1/2
1500	1/2 to 1	4008	<1/2
2102	1 to 2	4009	<1/2
2201	Not measurable	4010	<1/2
2202	2	4011	1/2 to 1
2301	Not measurable	4012	1/2 to 1
2302	5	4013	1/2
4001	<1/2	4014	1 to 2
4002	<1/2	4015	1/2 to 1
4003	<1/2		

are shown in Figure 1, and data from penetration of surface treatments into the concrete substrate are given in Table 3.

## DISCUSSION OF RESULTS

This test was designed to answer certain questions about inadequately air-entrained portland cement concrete. Testing was done within the constraints of particular conditions (Table 4).

### Question 1

Is boiled linseed oil in mineral spirits an effective antiscaling compound for concrete exposed to freezing and thawing in the presence of deicing salts?

Test conditions 1100 through 1500 (Fig. 1) are used to answer this question. Condition 1100 (control) shows evidence of surface scaling after only 20 cycles, with serious surface deterioration setting in after about 40 cycles. Conditions 1300 and 1500 held up better than the control through 20 cycles but performed much like the control thereafter. Condition 1200 held up well through about 80 cycles, and condition 1400 through more than 100 cycles. Therefore, 1400 (75 percent linseed oil in mineral spirits) is superior to 1200 (50 percent linseed oil in mineral spirits), which is superior to 1300 (25 percent linseed oil in mineral spirits), which is roughly equal to 1100 (control) in inhibiting surface deterioration under the prescribed test conditions. Recoating condition 1300 at the same coating rate after 24 hours (producing condition 1500) did not improve performance significantly.

Within these test constraints, boiled linseed oil in mineral spirits is an effective antiscaling agent roughly in proportion to the concentration of oil and therefore in proportion to the oil treatment rate for a one-shot treatment. A one-shot treatment is markedly superior to a two-shot treatment, which totals the same oil treatment rate.

### Question 2

Is the effectiveness of boiled linseed oil in mineral spirits as an antiscaling compound impaired by the presence of a wax-based or a resin-based curing compound?

Pertinent test conditions for answering this question are 2201 through 2302.

The performances of conditions 2201 (wax-cured) and 2301 (resin-cured) were very similar to that of condition 1100 (control), whereas the performance of condition 2202 (wax-cured plus treatment with linseed oil in mineral spirits) and 2302 (resin-cured plus treatment with linseed oil in mineral spirits) was very similar to that of 1200 (standard-cured plus treatment with linseed oil in mineral spirits), with perhaps a slight edge to condition 2202.

Table 3 gives the depth of penetration of oil through the curing membranes to a depth equal to or greater than the penetration depth in the absence of curing membranes. Figure 2a shows the resin-cured specimen, and Figure 2b shows an oil-treated, resin-cured specimen before sulfuric acid treatment. The light streak at as much as 5 mm below the treated surface is pigment from the curing compound carried into the concrete substrate by the boiled linseed oil in mineral spirits.

Within these test conditions the presence of a wax-based or a resin-based curing compound does not impair the effectiveness of boiled linseed oil in mineral spirits as an antiscaling compound.

### Question 3

Are any of several other linseed oil formulations effective as antiscaling compounds?

Test conditions 2102, 4001 to 4003, 4007 to 4010, and 4013 are used to answer this question.

The performance of blends of Z8-bodied linseed oil and boiled linseed oil emulsion (4001 to 4003) was roughly equivalent to the performance of boiled linseed oil in mineral spirits (1200) through 100 cycles of freezing and thawing. Condition 4001 (15 percent Z8 and 85 percent emulsion) was roughly equal to 4002 (20 percent Z8 and 80 percent emulsion), which was superior to 4003 (25 percent Z8 and 75 percent emulsion), which

Figure 1. Durability results.

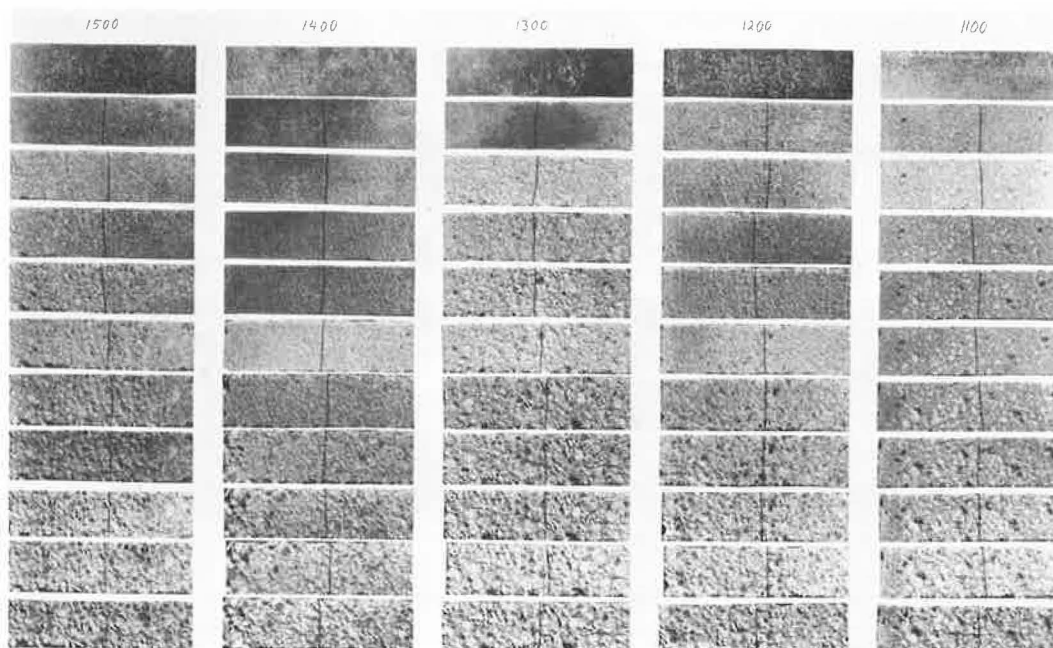


Table 4. Test conditions.

Condition	Description
1100	28-day moist cure; 2 weeks at 50 percent relative humidity, 70 F (21 C) (control)
1200	28-day moist cure; 2 weeks at 50 percent relative humidity, 70 F (21 C); 50-50 linseed oil in mineral spirits; 7 days at 50 percent relative humidity, 70 F (21 C)
1300	Same as 1200, except 25-75 linseed oil in mineral spirits
1400	Same as 1200, except 75-25 linseed oil in mineral spirits
1500	Same as 1300, plus second coating of linseed oil in mineral spirits 24 hours after first coating
2101	Same as 1100 (control)
2102	Same as 1200, except linseed oil in mineral spirits replaced by linseed oil emulsion
2201	Same as 1100, except wax cure
2202	Same as 1200, except wax cure
2301	Same as 1100, except resin cure
2302	Same as 1200, except resin cure
4001	15 percent Z8-bodied linseed oil and 85 percent boiled linseed oil emulsion <sup>a</sup> , evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4002	20 percent Z8-bodied linseed oil and 80 percent boiled linseed oil emulsion <sup>a</sup> , evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4003	25 percent Z8-bodied linseed oil and 75 percent boiled linseed oil emulsion <sup>a</sup> , evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4004	Same as 4001, evaluated as curing compound at 200 ft <sup>2</sup> /gal (4.9 m <sup>2</sup> /litre)
4005	Same as 4002, evaluated as curing compound at 200 ft <sup>2</sup> /gal (4.9 m <sup>2</sup> /litre)
4006	Same as 4003, evaluated as curing compound at 200 ft <sup>2</sup> /gal (4.9 m <sup>2</sup> /litre)
4007	50 percent solution of linseed fatty acids in mineral spirits, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4008	50 percent solution of dimer acids (Emery 1016) in mineral spirits, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4009	50 percent solution of 50-50 mix of linseed-dimer acids in mineral spirits, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4010	50 percent solution of tall oil fatty acids in mineral spirits, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4011	50 percent boiled linseed oil emulsion, evaluated as curing compound at 175 ft <sup>2</sup> /gal (4.3 m <sup>2</sup> /litre)
4012	National Flaxseed water-soluble curing compound, evaluated according to recommendations on container
4013	50 percent solution of 50-50 mix of linseed fatty acids and boiled linseed oil in mineral spirits, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4014	28-day moist cure; 50 percent solution of boiled linseed oil in mineral spirits, recoated periodically with boiled linseed oil solution through 300 freeze-thaw cycles, evaluated as antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)
4015	Boiled linseed oil emulsion cure subjected to freeze-thaw tests, then 50 percent solution of boiled linseed in mineral spirits applied when deterioration starts, evaluated as curing compound at 175 ft <sup>2</sup> /gal (4.3 m <sup>2</sup> /litre) and antiscaling agent at 225 ft <sup>2</sup> /gal (5.5 m <sup>2</sup> /litre)

<sup>a</sup>These formulations are mixtures of 50 percent emulsions, e.g., No. 4001 contains 15 percent Z8-bodied oil emulsion containing 50 percent oil and 85 percent boiled oil emulsion containing 50 percent oil.

Figure 2. (a) Resin-cured specimen, and (b) oil-treated, resin-cured specimen.

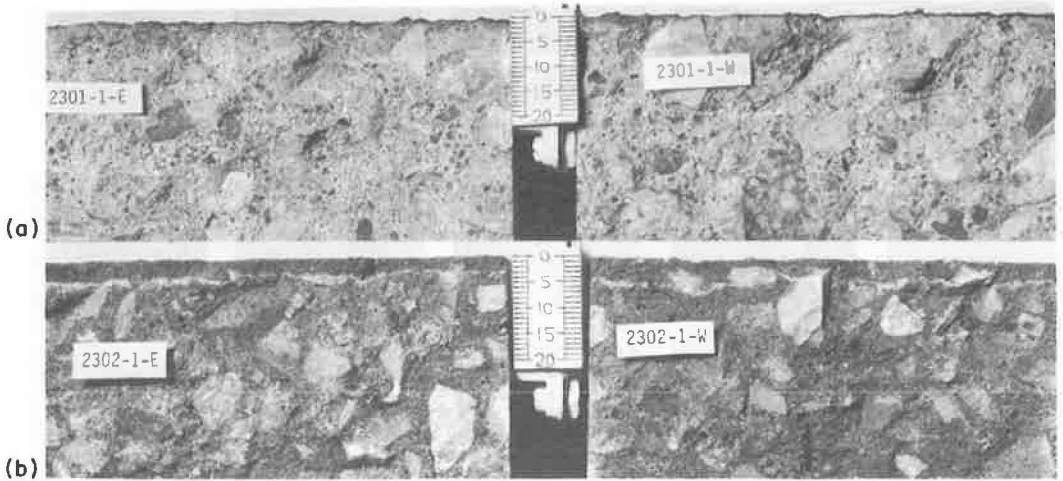


Table 5. Pertinent strength data relating to linseed oil formulations as curing compounds.

Condition	Modified-Cube Compression				Flexure			
	Mean (psi)	Difference in Means (psi)	t (df = 10)	Significance Level	Mean (psi)	Difference in Means (psi)	t (df = 4)	Significance Level
1100	5040	20	0.16	n.s.	810	75	1.90	0.10
4004	5020	20	0.16	n.s.	735	75	1.90	0.10
4005	4850	190	1.33	n.s.	645	165	3.80	0.01
4006	4900	140	0.87	n.s.	660	150	3.58	0.01
4011	5240	200	1.59	n.s.	680	130	3.03	0.025
4012	5000	40	0.26	n.s.	700	110	2.30	0.05

Note:  $t = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{\frac{\sum(x_1 - \bar{x}_1)^2 + \sum(x_2 - \bar{x}_2)^2}{(N_1 - 1) + (N_2 - 1)} \left( \frac{N_1 + N_2}{N_1 N_2} \right)}}$

was roughly equal to 1200 (50-50 linseed oil in mineral spirits). There were only nominal differences in performance among conditions 2102, 4007, 4008, 4009, 4010, and 4013, and none of them showed any significant advantage over 1200.

For these test conditions, only blends of Z8-bodied linseed oil and boiled linseed oil emulsion, among the several formulations tested, are superior to boiled linseed oil in mineral spirits as antiscaling compounds (only beyond about 80 cycles of freezing and thawing). The advantage disappears when the proportion of Z8-bodied oil exceeds 20 percent (by volume).

#### Question 4

Do wax-based and resin-based curing compounds have residual antiscaling value?

Pertinent test conditions for answering this question are 2201 and 2301.

The performance of the wax-based curing compound (2201) and of the resin-based curing compound (2301) is clearly inferior to the control (1100). Wax-based and resin-based curing compounds have no residual antiscaling value.

#### Question 5

Do any of the linseed oil formulations used as curing compounds have residual antiscaling value and, if so, are they effective as curing compounds?

Test conditions used are 4004 through 4006, 4011, and 4012 (Table 2).

Performance of blends of Z8-bodied linseed oil and boiled linseed oil emulsion (4004 through 4006) was much like that of the emulsion alone (4011) through about 140 cycles of freezing and thawing. Thereafter, performance improved with increasing percentages of Z8-bodied oil. Northern Laboratory's emulsion alone (4011) and the National Flaxseed emulsion (4012) showed roughly equal performance with perhaps some edge over the control.

Northern Laboratory's emulsion and the National Flaxseed emulsion used as curing compounds may have residual antiscaling value. Blends of Z8-bodied oil and Northern Laboratory's emulsion used as curing compounds do have a residual antiscaling value that increases with an increasing proportion of Z8-bodied oil up to 20 percent Z8.

Pertinent strength data from Table 1 are given in Table 5 for answering the second part of question 5.

None of the linseed oil formulations used as curing compounds had compressive strengths significantly different from that of the control (1100), but all of them had flexural strengths significantly less than that of the control.

The five linseed oil formulations tested as curing compounds yield compressive strengths comparable with the control but result in a moderately significant decrease in flexural strength. (This does not negate their potential usefulness as curing compounds because it is not uncommon for compound-cured concrete to suffer by comparison with standard-cured concrete.)

#### Question 6

Is periodic retreatment with boiled linseed oil in mineral spirits worthwhile?

Pertinent test conditions for answering this question are 4014 and 4015. Note that 4014 is essentially 1200 retreated after 80 cycles and again after 260 cycles of freezing and thawing. Condition 4015 is essentially 4011 treated with boiled linseed oil in mineral spirits at the conclusion of the curing regime and again after 80 cycles of freezing and thawing. There was remarkable improvement in antiscaling performance in both cases. For 4015, scaling was arrested through 260 cycles; for 4014, scaling was arrested through the full 300 cycles.

Under these test conditions, periodic retreatment with boiled linseed oil in mineral spirits arrests salt-scaling indefinitely.

#### Question 7

Is the concrete surface permanently softened by any of these treatments?

At 2 months, only conditions 4004, 4006, 4014, and 4015 among the oil-treated

samples showed higher California abrasion losses (lower resistance to abrasion) than the control (1100) (Table 3). At 4 months, only 4006 and 4015 showed higher abrasion losses, and 4015 alone showed higher abrasion loss than the control at 6 months. In all of these cases the loss difference is nominal, and a difference of only 1.2 grams is of doubtful significance.

By contrast, many of the oil-treated samples showed marked reductions in abrasion loss, whereas the resin-based curing compound (2301) resulted in a substantial increase in abrasion loss at all three test ages.

There is no apparent permanent softening of the concrete surface (as measured by resistance to abrasion by the California method) because of the linseed formulations tested. Most of them improved abrasion resistance.

#### ACKNOWLEDGMENT

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