Linseed Oil Emulsions for Protecting Concrete[1]

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Abstract

Emulsions of boiled linseed oil have certain physical characteristics suitable for application to concrete both as curing and antispalling agents. This preliminary report gives methods of preparation, physical properties and use of these compositions including application to NRRL sidewalks and parking lots. Emulsions appear to be comparable to solutions of boiled linseed oil in mineral spirits for imparting resistance to spalling. Emulsions create fewer fire hazards than the solutions, and permit easier cleaning of the equipment used in spraying. Tests show that the emulsions are effective in protecting air-entrained concrete from damage that results from the application of deicing materials and concurrent freeze-thaw cycles. Emulsions are effective both as curing and antispalling agents. Linseed oil was applied at the rate of 0.16 to 0.20 lb/sq yd.

Introduction

During the 1920's and 1930's, many states coated their concrete highways with boiled linseed oil to protect them from freeze-thaw damage. Usually the oil was dissolved in an equal volume of a solvent such as turpentine, kerosene, gasoline or naphtha. This dilution made the oil easier to apply and increased the penetration of the oil into the concrete. Some states preferred to have the kerosene solution of linseed oil emulsified in water with soap powder and polyphosphates (6). These emulsions were not stable and were often washed off the pavement if rain fell soon after application.

About 1941, it was learned that air entrainment of sound concrete gave excellent freeze-thaw resistance; consequently, as air-entrained concrete came into common use, linseed oil coatings were no longer deemed necessary (7). However, increased use of deicing chemicals caused serious deterioration (spalling) of air-entrained concrete laid in the field. About 5 years ago the application of boiled linseed oil to prevent this deterioration began and has been increasing ever since (1). The National Flaxseed Processors Association (NFPA) recommends an "antispalling compound" consisting of 50-volume percent of boiled linseed oil in mineral spirits (1). For the past 10 or 15 years, the use of liquid membrane curing compounds to retain water required for hydration of the cement has been increasing (3). This water retention is essential to develop the ultimate strength of the cement. Since linseed oil forms a moisture-resistant film, it seemed reasonable to try to formulate linseed oil into a compound that would act both as a curing compound and subsequently as an antispalling agent (4).

Linseed Oil Emulsions and Emusifiable Oil Compositions

Preparation of Linseed Oil Emulsion

One of the better formulations we have found to date is shown under "emulsion" in Table I. Saturated tallow alcohols were dissolved in boiled linseed oil at 60C. Sodium hydroxide (to neutralize the fatty acids) and dipicolinic acid (to assist in emulsification of metallic driers present in boiled oil) were dissolved in water. The components were emulsified by adding the oil phase slowly to an equal volume of the water phase in a 4-in. Cowles dissolver operating at 2,900 rpm. Finally, the emulsion was pumped through a Manton-Gaulin two-stage homogenizer at 3,500 psi. The emulsion was stable through five freeze-thaw cycles (cycle 70 to 0 to 70F) and remained stable for 2 years.

TABLE I
Composition of NRRL Emulsion and Emulsifiable Oil

Composition	Emulsion, weight %	Emulsifiable oil,[a] weight %
Oil phase (50 vol %)		
Boiled linseed oil	97.0	97.0
Saturated tallow alcohols	3.0	3.0
	100.0	100.0
Water phase (50 vol %)		
Water	99.60	
Sodium hydroxide	0.37	
Dipicolinic acid	0.03	
	100.00	

[a] Emulsifiable oil composition is prepared by mixing with an equal volume of water.

Neutralizing the free fatty acids present in the oil with sodium hydroxide has two advantages. The in situ reaction yields excellent emulsions and forms soaps that are mainly polyunsaturated. Upon drying the soaps may polymerize into the oil film and thus lower its water sensitivity. The dipicolinic acid chelates both the metallic driers in the oil and the calcium and magnesium salts in the water, thus prevents them from breaking the emulsion (5). Also, this acid does not slow the drying of the oil as much as other chelating agents do. Saturated tallow alcohols increase the stability of the emulsion without increasing the water sensitivity of the dried oil film.

Substituting other bases such as KOH or NH_4OH for the NaOH formed less stable emulsions. Using lauryl or linseed alcohols instead of tallow alcohol also lowered the stability of the emulsion.

Preparation of Emulsifiable Linseed Oil Composition

To avoid the cost of shipping water, an emulsifiable boiled linseed oil was formulated as shown in the last column of Table I. This formulation contains only saturated tallow alcohols and boiled linseed oil. The emulsifiable oil composition was prepared by mixing this formulation with an equal volume of water immediately before use. A paddle-type stirrer was employed. This emulsifiable oil composition was stable for about 24 hr. However, two of the five commercially available boiled linseed oils studied failed to give satisfactory emulsions when formulated as described in Table I. Apparently, differences in processing account for this behavior. The addition of 0.6% 2-amino-2-methyl-l-propanol to the oil phase made all oils examined emulsifiable in this composition.

Rate of Dry

To test the rate of dry, a 5-mil film of the formulation being tested was drawn on a glass plate and the percentage change in weight with time measured.

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[2] No. Utiliz. Res. Dev. Div., ARS, USDA.

TABLE II
Drying Times of Linseed Oil Films—Hours Required to Gain Given Percentages of Original Weight

Films from:	Percent weight gain			
	2.5	5	7.5	10
	Hours required			
Mineral spirits solution	8	13	21	26
Emulsifiable oil	6	9	15	21
Emulsion	9	16	44	....
Emulsifiable oil with 2-amino-2-methyl-l-propanol	9	15	21	40
Emulsifiable oil with mono- or triethanol amine	42–47	....	....	....

The minimum weight of each film was used as the base for the percentage calculations. The initial drop in weight comes from the loss of mineral spirits or water.

The films from boiled linseed oil-mineral spirits solution and the emulsifiable oil gained weight faster than the other formulations tried. With the boiled oils that did not emulsify readily, the addition of amino alcohol made them emulsifiable. The films from emulsifiable oils containing 2-amino-2-methyl-l-propanol gained weight fairly rapidly but the films from oils containing mono- and triethanol amine did not. Data are given in Table II.

Properties of Antispalling Agents

The properties of the NFPA antispalling compound and the Northern Regional Research Laboratory (NRRL) emulsion are shown in Table III. See Table II for information on drying rates.

Tests of Performance as Antispalling Agents

In 1962 boiled linseed oil was applied to a new parking lot at NRRL. Selected areas received 1 or 2 coats of emulsion or of NFPA antispalling compound at coating rates of 0.16 lb of oil phase per square yard. After 3 years, no apparent damage has occurred in the coated areas. The uncoated area has shown some deterioration although the only salt applied was that carried in by traffic. Fig. 1 shows part of the uncoated area about 2 years after coating.

Coefficients of Friction

The coefficient of friction between rubber and concrete was determined in selected areas of the parking lot by drawing a box over the surface by means of weights and pulleys. The bottom of the box was covered with a piece of gasket rubber ¼ in. thick, 5¼ in. wide and 8 in. long. Coefficients were measured at 16 different places in each area (4 times at each of 4 weights). The weights of the box were 666 (empty), 1,166, 1,666, and 2,166 g. Measurements were made before and after wetting the surfaces with a thin film of water. Visual observation of the surfaces suggests that the differences in friction are more likely due to variations in roughness than to any effect of the oil.

Resistance measurements with the British Portable Tester were deliberately made in areas where traffic had somewhat worn away the rough surface. These data suggest that the friction measurements of the various treated areas show no significant differences between themselves or the uncoated area. A marked difference in friction existed between dry and wet concrete. Skid resistance of all concrete surfaces measured is sufficient to be considered safe according to the criteria set up by the Road Research Laboratory (7).

TABLE III
Some Characteristics of Antispalling Formulations

Material	Density		Oil	Brookfield viscosity at 25C, 60 rpm	pH
	g/cc	lb/gal	lb/gal		
NFPA solution	0.854	7.12	3.85	9	.....
NRRL emulsion	0.958	7.98	3.85	11	11.5

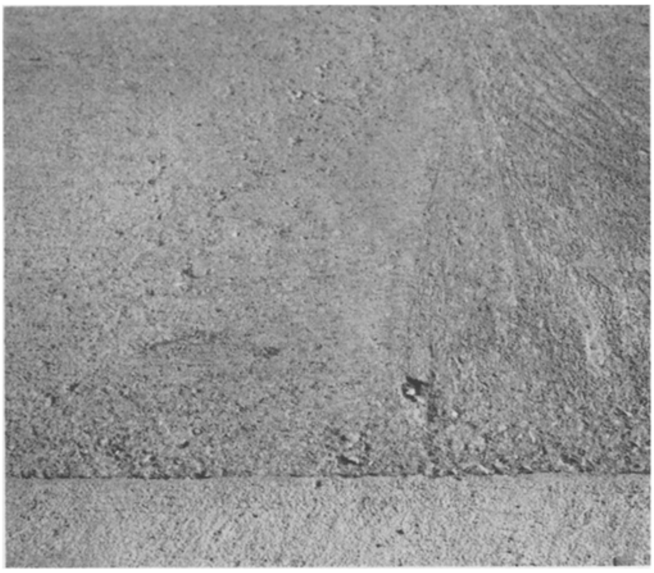

FIG. 1. Photograph of uncoated area of NRRL parking lot showing erroded areas.

Tests of Linseed Oil as a Curing/Antispalling Agent

Although linseed oil has been applied for many years as an effective antispalling agent, its possible use as a membrane-forming agent in the curing of concrete appears to have been overlooked. To demonstrate that linseed oil could form a film on freshly laid concrete, linseed oil emulsions were sprayed on

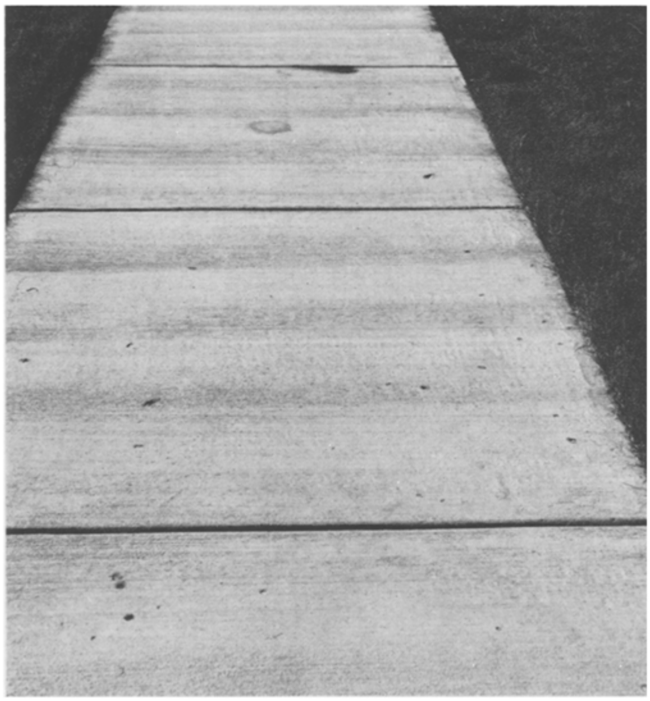

FIG. 2. Photograph of uncoated NRRL sidewalk showing many popouts. Circular area at top is a plug where core was removed for compressive strength measurement.

TABLE IV
Number of Popouts obtained in Concrete Cured with Linseed Oil Emulsions and Other Agents

Method of cure	Number of Popouts/100 sq ft between		
	1–2–64	4–17–64	4–16–65
None	10.0	140.0	222
Polyethylene	7.2	100.0	157
Wax-resin	8.0	104.0	152
NRRL LSO emulsion	0.1	6.5	20
NRRL emulsifiable oil compositions	0.1	21.4	55
Total number of salt applications	3	13	39

NRRL sidewalks about 2 hr after concrete was poured. To check the antispalling activity of treatment, popouts were counted periodically. A popout is a piece of concrete that has been forced out of the surface. Apparent cause is the expansion of a piece of porous aggregate beneath the concrete. The expansion probably results from water absorbed in the aggregate. The water freezes during a cooling cycle and the resultant pressure pops a piece of concrete out of the surface. Size of these popouts varies depending in part on the size of the piece of water-absorbing aggregate. The data in Table IV show the advantage of using linseed oil emulsion in preventing popouts in concrete when compared to other methods of treatment. The linseed oil treatments reduced the number of popouts in comparison to wax resins and polyethylene sheet treatments. Fig. 2 is a photograph of an uncoated, untreated sidewalk area taken May 8, 1964. The sidewalk was laid June 4, 1963. An average of 140 popouts per 100 sq ft were counted after 13 applications of salt during the

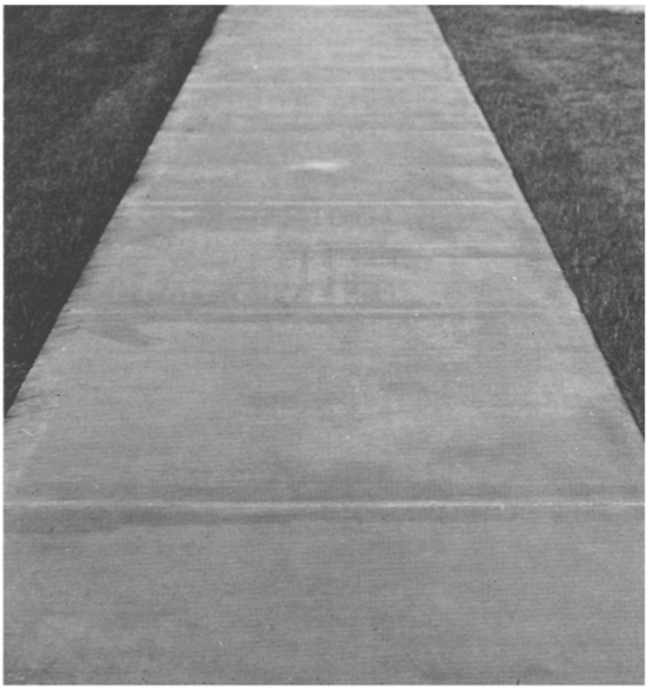

FIG. 3. Photograph of NRRL sidewalk coated with linseed oil emulsion showing relative absence of popouts.

TABLE V
Linseed Oil-Water Emulsions for Curing of Concrete. Water-Loss Test

Emulsion[a]	Coating rate		Water loss
	Surface area	Lb oil/ sq yd	Mg/cm²/3 days
	Sq/ft/gal emulsion		
None	......	......	258
LSO/W (50%)	200	0.18	47
	150	0.24	28
LSO/W (45%)	200	0.20	44
	150	0.26	24
LSO + TA/ Min. Spirits (50%)	200	0.18	79
	150	0.24	55

[a] LSO, linseed oil; W, water; TA, tallow alcohols.

intervening winter. Fig. 3 shows a sidewalk laid May 31, 1963, cured with our boiled linseed oil emulsion at the rate of 0.20 lb of oil per sq yd and photographed a year later. An average of only 6.5 popouts per 100 sq ft were counted after 13 applications of salt during the winter. Emulsifiable linseed oil compositions (Table I) gave a somewhat higher rate of popouts.

Cores were removed from each area and tested. The compressive strength of the cores was 5,000–6,000 psi, and no apparent differences in strength were detected corresponding to differences in treatments given.

Water-Retention Efficiency of Boiled Linseed Oil

It is generally accepted that membranes permitting minimum loss of water result in concrete of highest strength. Water-retention efficiency is usually measured by ASTM C-156. This test did not give reproducible results when used to evaluate boiled linseed oil formulations (2). In general, results have been borderline with some tests passing and others failing the specification of a maximum loss of 55 mg/cm² of surface per 72 hr. In other tests carried out by the U.S. Army Corps of Engineers following their own procedure (CRD-C-302-63), the linseed oil treatment did not meet their specification of 31 mg/cm² in 7 days (8). However, Table V shows that increasing the coating weight decreases the water loss and also that the emulsions are somewhat more efficient than a solution of linseed oil in mineral spirits. Efforts are continuing to develop a linseed oil coating that will meet Army specifications.

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