

LABORATORY EVALUATION OF LINSEED OIL EMULSION AS A CURING AGENT
FOR
PORTLAND CEMENT CONCRETE

By

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ABSTRACT

This investigation involves the evaluation of two linseed oil emulsions as membrane curing compounds and surface sealers with respect to use by the Texas Highway Department. The effects of these emulsions upon surface hardness and flexural and compressive strengths, and their surface penetration qualities are also included in the investigation. Comparison tests were run concurrently with materials satisfactorily being used on Texas Highway projects.

SUMMARY

This investigation was limited to evaluating the two submitted linseed oil emulsion products as both curing materials and as surface treatments for concrete under the same laboratory conditions all materials proposed for these uses are tested for compliance. Additional physical tests were performed on both L.O.E. samples along with known acceptable materials of other types to determine any possible deleterious effects to the concrete. The data obtained supports the conclusions that neither of the linseed oil emulsion products satisfies the standard requirements of the Texas Highway Department for the purposes intended and produces apparent detrimental effects not found with presently used compounds.

IMPLEMENTATION

On the basis of the results of this investigation, the use of linseed oil emulsions for curing of concrete in Texas Highway projects is not recommended.

I. PURPOSE

The purpose of this investigational project is to evaluate the use and effectiveness of emulsified linseed oil as a curing medium for portland cement concrete and as a possible surface sealer.

II. CONCLUSIONS

Based upon laboratory performance of the linseed oil emulsion samples submitted to this Department as representative of production material the following conclusions were determined.

1. Linseed oil emulsions will not satisfy the moisture retention requirements at 24 or 72 hours for membrane curing compounds as set forth by Texas Highway Department Standard Specifications.
2. Linseed oil emulsions will also not meet the run and sag requirements of the same specifications. In fact, their moisture retention properties are lessened when applied to a simulated crown or curve of 5° whereas other materials currently in use are not affected.
3. Linseed oil emulsions leave the surface soft and friable for an extended period after application.
4. Water permeability is higher through the linseed oil emulsions than all other materials tested. It appears that when compared to blank specimens with no coating applied that permeability is actually augmented.
5. So long as the membrane curing compound surface is intact, neither linseed oil-mineral spirits surface treatment nor linseed oil emulsions will penetrate into the concrete surface.

III. RECOMMENDATIONS

In accordance with the findings of this study the following recommendations are made:

1. That linseed oil emulsions not be used for the curing of concrete on Texas Highway Department projects.
2. That linseed oil-mineral spirits surface treatments not be applied to surfaces where membrane curing compound is still intact.

IV. MATERIALS

Laboratory No. R3-71-218: United States Department of Agriculture sample of boiled linseed oil emulsion (L.O.E.), 6325-39-2.

Laboratory No. R3-71-372: Boiled linseed oil emulsion (L.O.E.), Protecto Coat No. 66, Nu Pro, Inc., Oklahoma City, Oklahoma.

Laboratory No. R3-71-442: A concrete surface treatment consisting of a blend of 50 percent linseed oil and 50 percent mineral spirits (50/50 L.O./M.S.) meeting Texas Highway Department Special Specification Item 1825, "Concrete Surface Treatment."

Laboratory No. R3-71-443: Texas Highway Department Standard Specifications, Item 531, Type 1, wax base membrane curing compound.

Laboratory No. R3-71-444: Texas Highway Department Standard Specifications, Item 531, Type 2, white pigmented membrane curing compound.

Laboratory No. R3-71-445: Texas Highway Department Standard Specifications, Item 531, Type 1, resin base, membrane curing compound.

Note: For ease in handling, only the last three digits are used to identify these materials throughout the report.

V. TEST METHODS AND EQUIPMENT

Infrared Study: A study of the infrared spectra of both linseed oil emulsions was made with the Perkin-Elmer Infrared Spectrophotometer, Model 521. The results of this comparative study along with other pertinent information concerning these emulsions is included in the Appendix of this report.

Drying Time: The time for the material to dry "to touch" was determined according to ASTM Designation C 309 with the compound applied at the rate of 180 sq. ft. per gal.

Moisture Loss: The moisture loss in each instance was determined according to Test Method Tex-219-F using 180 sq. ft. per gal. as the rate of application, unless otherwise specified. A copy of Test Method Tex-219-F is included in the Appendix.

Sand Blast: The test specimen surface was sand blasted with the equipment and procedure similar to that given in ASTM Designation C 418.

Water Permeability: The water permeability meter consisted of a calibrated vertical column of water sealed to the surface of the specimens. The test value was the measurement of the drop in this column of water after exposure to 30 psi air pressure for 5 minutes.

Flexural Strength and Compressive Strength: The test procedures for determining both the flexural and compressive strengths are to be found in the Appendix.

Maximum Depth of Penetration: This test consisted of measuring with a

graduated scale the maximum depth of penetration of the linseed oil into the mortar specimen as indicated by a darkened area resulting from treating the broken face of the specimen with a 50 percent aqueous solution of sulfuric acid dried for approximately 30 minutes at approximately 130 C (266F).

VI. PROCEDURE

Work began on the project with the mixing of mortar consisting of Graded Ottawa Silica Sand (ASTM Designation C 109), Type III cement, and water, and the molding of specimens (12"x6"x2-1/8") 1 through 12 (See Table I) in accordance with Test Method Tex-219-F. After two hours of initial set in the constant temperature-humidity cabinet, the specimens 1 through 6 were sprayed with the Type 1, wax base, compound and specimens 7 through 12 were sprayed with Type 1, resin base, compound, both at the rate of 180 sq. ft. per gal. Specimens 1 through 3 and 7 through 9 were sprayed in a horizontal position. Specimens 4 through 6 and 10 through 12 were sprayed in a position 5° inclined from the horizontal. These specimens remained in this inclined position until tested. This was to simulate deviations of the pavement from level or horizontal, such as crown, super-elevation and grade.

The moisture retention at 24 hours and 72 hours of each specimen was determined as described in Test Method Tex-219-F and the average of each set of three recorded in Table I opposite the first specimen in each series. (See values for specimens 1, 4, 7 and 10).

Spraying the specimens on Monday afternoon resulted in removing the specimens from the temperature-humidity cabinet, after the 72 hour moisture retention test, on Thursday afternoon. The next morning specimens 2, 5, 8 and 11 were

sprayed with the U.S. Agriculture Department linseed oil emulsion (L.O.E., 218) at the rate of 360 sq. ft. per gal. During the same spraying period, specimens 3, 6, 9 and 12 were sprayed with the 50 percent linseed oil, 50 percent mineral spirits (50/50 L.O./M.S., 442) concrete surface treatment. All of these specimens were then placed on a shelf and left undisturbed at room temperature and humidity until further testing.

The same procedure just described was followed with the Type 2, white pigmented compound (444) and specimens 13 through 18.

As previously mentioned, the Nu Pro, Inc. emulsified linseed oil sample (372) arrived after the project was underway. Since a simple numerical sequence was used for identifying each specimen, the specimen numbers allotted to the test specimens treated with this emulsion necessarily were out of sequence with others in their group as recorded in Table I. Hence, specimen number 49 follows specimen number 3. Specimen 49 was fabricated, treated, and handled in a manner identical to the other three specimens in its group, all being cured for 72 hours with Type 1, was base, curing compound prior to the application of the linseed oil products. (Specimen number 1 of each series received no linseed oil treatment.) This procedure applies to the other apparently out-of-sequence specimens 50, 51, 52, 53 and 54. Each was treated identical to the others in its group and the only possible significant difference in specimens 49 through 54 being that they were made at a later date and not a part of their original groups. Since they were not part of the original groups, the moisture loss of these individual specimens is recorded for comparison with the earlier tested specimens.

Specimens 19 through 24 were similarly fabricated and placed in the temperature-humidity cabinet for the 72 hour moisture retention test. These specimens were not sprayed at that time with any type of curing or treatment compound to yield a blank or untreated surface for delayed application of LO/MS material. After sealing, specimens 22 through 24 were inclined 5° until further testing. The average moisture loss at 24 hours and 72 hours for each group of three is recorded in Table I. After 72 hours in the cabinet, the specimens were removed, and, following the sequence previously described, specimens 19 through 24 were sprayed with the linseed oil/mineral spirits blend (442). These were also shelved under room conditions until further tests were due.

The next portion of the investigation was to test the linseed oil emulsions as curing compounds, spraying the specimens with the emulsions after the initial 2 hour set period in the curing cabinet. Specimens 25 through 36 were sprayed, as recorded in Table I, at the rate of 180 sq. ft. per gal. Specimens 37 through 48 were sprayed with their respective emulsions at the rate of 150 sq. ft. per gal. As indicated in Table I, each linseed oil emulsion group of six specimens contains three specimens sprayed horizontally and three sprayed in the inclined position. The average moisture loss of each set of three specimens is recorded in the table. After the 72 hour test period, following the outlined standard procedure, the specimens were shelved under room conditions for further tests.

The last set of specimens was a set of three blank specimens tested for moisture retention and then shelved in room conditions until further tested.

No coating of any kind was sprayed on these specimens. The moisture loss at 24 hours and 72 hours is recorded in Table I and compares favorably with the moisture loss of the blank specimens 19 through 24.

The drying time test was run independent of the other tests and followed the procedure in ASTM Designation C 309. The materials were sprayed at the rate of 180 sq. ft. per gal. on horizontal 6" x 6" x 1" damp concrete blocks. The times for the materials to dry to touch are recorded in Table I. Neither linseed oil emulsion dried to the point where it was judged "dry to touch." After seven days under the conditions prescribed in ASTM Designation C 309 the emulsions remained tacky to the touch. The emulsions still feel very tender and tacky at this time. Under laboratory conditions they do not satisfy the prevailing specifications.

In order to establish a time schedule for testing, the first groups of specimens (1 through 12) were observed carefully to determine how long it would take for the linseed oil emulsions to "cure out" and the surfaces of the specimens become dry enough to be handled. This schedule was then followed for specimens 1-24 (49-54). After eleven days in room conditions the surfaces were judged dry enough for careful handling. On the eleventh day after spraying with the linseed oil emulsions the specimens were very carefully removed from the pans, the sealing material scraped from the edges of the specimens, and each specimen sawed into smaller test specimens. The 12" x 6" specimens were sawed with a dry blade so that the surfaces would remain unsaturated and as undisturbed as possible. The specimens were cut lengthwise down the middle yielding two approximately 12" x 3" test specimens. One of these halves was used for the flexural and compressive strength

tests, the other half was cut in half again providing two approximately 6" x 3" test specimens for the sand blast, water permeability and depth of penetration tests.

On the same day of the sawing the flexural strength tests were performed. Two flexural breaks were made in accordance with the procedure included in the Appendix. The three pieces of specimen resulting from these breaks were sliced into cubes, capped and tested in compression on the thirteenth day after spraying (fourteen days after the completion of the 72 hour moisture retention test). This procedure is also found in the Appendix.

The sand blast and water permeability testing was done on the twelfth day after spraying with the emulsions, thirteen days after the completion of the 72 hour moisture retention test.

Two sand blast tests were made on each 6" x 3" specimen. The test, using an apparatus similar to that described in ASTM Designation C 418, consisted of sand blasting the surface of the specimen with 600 grams of "Standard Ottawa 20-30 Sand," ASTM Designation C 190, at 60 psi air pressure. The weight of the specimen was determined prior to the testing and after each sand blasting. The percent loss recorded in Table I is the total percent loss in weight of the specimen after these two sand blastings.

The water permeability test was made to determine the susceptibility of the surface of each specimen to the penetration of water. The moisture retention test is a measure of the water leaving a specimen. The

permeability test is intended to measure the water entering a specimen. The "permeability meter" consisted of a vertical glass tube with a six inch scale, calibrated in tenths of an inch, encased in a metal housing with a water inlet valve and air valve at the top and an o-ring seal on the bottom. The housing was clamped on the surface of the 6" x 3" specimen, the tube filled with water, and the surface exposed to the water for 5 minutes at 30 psi air pressure. The area exposed to the water is a 1-7/8" diameter circle. The value recorded in Table I for each specimen is the drop of the column of water measured in inches, measuring the amount of water that is presumed to have uniformly permeated the exposed surface.

The last test was performed on the thirteenth day after spraying (fourteen days after the 72 hour moisture retention test) to determine the depth to which the linseed oil emulsion had penetrated the surface of the test specimen. The 6" x 3" specimen used for the water permeability test was broken in half with a sharp tap of a hammer. This exposed two fresh broken faces of each specimen which were dipped in a 50 percent aqueous sulfuric acid solution and dried for approximately 30 minutes in an oven at approximately 130 C (266 F). Penetration is indicated by a gray colored area beneath the surface of the specimens and in each instance the edge of this area was an irregular, wavy line. The value recorded in Table I is the greatest depth of penetration of the linseed oil emulsion noticeable across the broken face, measured in inches, hence the term "maximum depth of penetration." In almost every case the value recorded represents one point of the wavy irregular line.

The above timetable was applied to specimens 25-48 (55-57) although delayed spraying of L.O.E. materials was not included.

TABLE I

| <u>Identification</u> | <u>Drying Time</u> | <u>Moisture Loss</u> (%By Wt.) | | <u>Sand Blast</u> | <u>Water Perm.</u> | <u>Flexural Strength</u> | <u>Compr. Strength</u> | <u>Maximum Depth of Penetration</u> |
|---|--------------------|-----------------------------------|-----------|--------------------------|--------------------|--------------------------|-------------------------|-------------------------------------|
| | | <u>24 Hrs.</u> | <u>72</u> | <u>Loss</u> (%By Wt.) | <u>(in.)</u> | <u>(Psi)</u> 12 Days | <u>(Psi)</u> 14 Days | <u>(in.)</u> |
| 1. 443 - Type 1, Wax Base | 210min. | 0.7 | 1.0 | 0.8 | 0.8 | 625 | 9,990 | 0 |
| 2. 443 - Type 1 + L.O.E.(218) | | | | 0.7 | 0.8 | 624 | 9,840 | 0 |
| 3. 443 - Type 1 + 50/50 L.O./M.S.(442) | | | | 0.8 | 0.8 | 637 | 9,870 | 0 |
| 49. 443 - Type 1 + L.O.E.(372) | | 0.6 | 0.9 | 0.6 | 0.4 | 805 | 7,890 | 0 |
| 4. 443 - 5° incline | | 1.0 | 1.2 | 0.8 | 0.8 | 642 | 9,830 | 0 |
| 5. 443 - Type 1 + L.O.E.(218) | | | | 0.7 | 0.6 | 657 | 9,700 | 0 |
| 6. 443 - Type 1 + 50/50 L.O./M.S.(442) | | | | 0.8 | 0.8 | 648 | 9,750 | 0 |
| 50. 443 - Type 1 + L.O.E.(372) | | 1.2 | 1.7 | 0.5 | 0.3 | 700 | 7,340 | 0 |
| 7. 445 - Type 1, Resin Base | 170min. | 1.6 | 2.5 | 0.6 | 1.7 | 567 | 9,880 | 0 |
| 8. 445 - Type 1 + L.O.E.(218) | | | | 0.6 | 0.5 | 622 | 9,990 | 0 |
| 9. 445 - Type 1 + 50/50 L.O./M.S.(442) | | | | 0.7 | 0.9 | 606 | 10,080 | 0 |
| 51. 445 - Type 1 + L.O.E.(372) | | 1.4 | 2.3 | 0.6 | 0.8 | 710 | 8,260 | 0 |
| 10. 445 - 5° incline | | 1.6 | 2.8 | 0.7 | 1.0 | 596 | 10,190 | 0 |
| 11. 445 - Type 1 + L.O.E.(218) | | | | 0.7 | 0.7 | 644 | 9,740 | 0 |
| 12. 445 - Type 1 + 50/50 L.O./M.S.(442) | | | | 0.8 | 0.9 | 541 | 9,450 | 0 |
| 52. 445 - Type 1 + L.O.E.(372) | | 1.5 | 2.8 | 0.5 | 0.6 | 715 | 8,400 | 0 |
| 13. 444 - Type 2 | 170min. | 0.8 | 1.3 | 0.7 | 0.8 | 879 | 10,460 | 0 |
| 14. 444 - Type 2 + L.O.E.(218) | | | | 0.6 | 0.6 | 842 | 10,070 | 0 |
| 15. 444 - Type 2 + 50/50 L.O./M.S.(442) | | | | 0.7 | 0.8 | 883 | 10,430 | 0 |
| 53. 444 - Type 2 + L.O.E.(372) | | 0.9 | 1.2 | 0.5 | 0.4 | 850 | 7,260 | 0 |
| 16. 444 - 5° incline | | 0.7 | 1.1 | 0.6 | 1.0 | 809 | 10,220 | 0 |
| 17. 444 - Type 2 + L.O.E.(218) | | | | 0.5 | 0.7 | 841 | 9,530 | 0 |
| 18. 444 - Type 2 + 50/50 L.O./M.S.(442) | | | | 0.6 | 0.6 | 798 | 9,960 | 0 |
| 54. 444 - Type 2 + L.O.E.(372) | | 0.9 | 1.1 | 0.5 | 0.6 | 855 | 7,380 | 0 |
| 19. 442 - 50/50 L.O./M.S. Sprayed on | | 11.4 | 13.4 | 0.7 | 1.5 | 797 | 8,640 | 3/32 |
| 20. Blank specimen after 72 hr. curing | | | | 0.7 | 1.5 | 789 | 8,580 | 3/32 |
| 21. In temp./humidity cabinet | | | | 0.6 | 1.8 | 766 | 8,350 | 3/32 |
| 22. 442 - Same as 19, 20 & 21 except | | 12.2 | 13.9 | 0.7 | 1.7 | 766 | 8,260 | 3/32 |
| 23. Specimens at 5° incline after | | | | 0.6 | 1.7 | 723 | 8,260 | 3/32 |
| 24. Sealing | | | | 0.7 | 1.9 | 716 | 8,310 | 3/32 |

TABLE I - CONTINUED

| <u>Identification</u> | <u>Drying Time</u> | <u>Moisture Loss</u> (%By Wt.) | | <u>Sand Blast Loss</u> (%By Wt.) | <u>Water Perm.</u> (in.) | <u>Flexural Strength</u> (Psi) 12 Days | <u>Compr. Strength</u> (Psi) 14 Days | <u>Maximum Depth of Penetration</u> (in.) |
|----------------------------------|--------------------|-----------------------------------|----------------|-------------------------------------|-----------------------------|--|--|--|
| | | <u>24</u> | <u>Hrs. 72</u> | | | | | |
| 25. 218 - L.O.E. 180 sq.ft./gal. | 7days+ | 4.5 | 6.3 | 1.0 | 1.9 | 859 | 10,070 | 1/16 |
| 26. 218 - L.O.E. 180 sq.ft./gal. | | | | 1.0 | 2.2 | 801 | 9,500 | 1/16 |
| 27. 218 - L.O.E. 180 sq.ft./gal. | | | | 1.0 | 2.7 | 856 | 9,100 | 1/16 |
| 28. 218 - 5° incline | | 4.6 | 6.4 | 1.0 | 2.7 | 854 | 9,190 | 1/16 |
| 29. 218 - 5° incline | | | | 1.0 | 3.1 | 773 | 9,500 | 1/16 |
| 30. 218 - 5° incline | | | | 0.9 | 3.1 | 777 | 8,720 | 1/16 |
| 31. 372 - L.O.E. 180 sq.ft./gal. | 7days+ | 4.1 | 6.0 | 1.1 | 1.8 | 797 | 9,300 | 1/16 |
| 32. 372 - L.O.E. 180 sq.ft./gal. | | | | 1.1 | 2.4 | 711 | 8,630 | 1/16 |
| 33. 372 - L.O.E. 180 sq.ft./gal. | | | | 1.1 | 2.6 | 745 | 8,880 | 1/16 |
| 34. 372 - 5° incline | | 4.8 | 6.5 | 1.0 | 2.8 | 755 | 9,637 | 1/16 |
| 35. 372 - 5° incline | | | | 1.1 | 2.7 | 752 | 9,040 | 1/16 |
| 36. 372 - 5° incline | | | | 1.2 | 2.8 | 759 | 9,017 | 1/16 |
| 37. 218 - L.O.E. 150 sq.ft./gal. | | 3.5 | 5.3 | 1.2 | 1.9 | 1,045 | 8,940 | 1/16 |
| 38. 218 - L.O.E. 150 sq.ft./gal. | | | | 1.1 | 2.1 | 960 | 8,800 | 1/16 |
| 39. 218 - L.O.E. 150 sq.ft./gal. | | | | 1.1 | 2.3 | 1,035 | 9,060 | 1/16 |
| 40. 218 - 5° incline | | 4.8 | 6.1 | 1.0 | 2.5 | 935 | 8,720 | 1/16 |
| 41. 218 - 5° incline | | | | 1.0 | 2.5 | 1,090 | 8,670 | 1/16 |
| 42. 218 - 5° incline | | | | 1.0 | 2.8 | 855 | 8,650 | 1/16 |
| 43. 372 - L.O.E. 150 sq.ft./gal. | | 3.6 | 5.0 | 1.2 | 1.9 | 1,035 | 7,650 | 1/16 |
| 44. 372 - L.O.E. 150 sq.ft./gal. | | | | 1.2 | 2.7 | 895 | 8,540 | 3/32 |
| 45. 372 - L.O.E. 150 sq.ft./gal. | | | | 1.1 | 2.1 | 1,115 | 8,860 | 1/16 |
| 46. 372 - 5° incline | | 4.4 | 5.8 | 1.1 | 2.6 | 1,035 | 8,510 | 1/16 |
| 47. 372 - 5° incline | | | | 1.1 | 2.7 | 960 | 8,890 | 3/32 |
| 48. 372 - 5° incline | | | | 1.0 | 3.2 | 880 | 8,520 | 3/32 |
| 55. Blank specimen | | 13.8 | 15.6 | 0.5 | 2.4 | 855 | 7,700 | |
| 56. Blank specimen | | | | 0.5 | 1.6 | 840 | 6,740 | |
| 57. Blank specimen | | | | 0.4 | 2.1 | 855 | 7,090 | |

VII. DISCUSSION

Much has been said and been written concerning the use of boiled linseed oil emulsions as curing compounds and surface sealers for portland cement concrete. This investigation is somewhat repetitious of what others have done plus some additional parameters. The major point of difference between this work and that of others is that the resulting data is examined with reference to the Texas Highway Department Standard Specifications and with respect to any benefits that may accrue from the use of these materials by the Texas Highway Department.

This investigation developed from a memorandum from Mr. Wayne Henneberger, Engineer of Bridge Design, of the Texas Highway Department Bridge Division, requesting that a sample of emulsified linseed oil from Mr. William Kubie of the United States Department of Agriculture be tested "for its effectiveness as a curing medium." After the project was well underway, a second sample of emulsified linseed oil ("Protecto Coat No. 66" from Nu Pro, Inc. of Oklahoma City, Oklahoma) was submitted to be included in this investigation. This investigation consists of the evaluation of both linseed oil emulsions as curing compounds and surface sealers by themselves and in conjunction with Standard Specifications Item 531, Type 1 and Type 2, membrane curing compounds plus their effect on other physical properties of the concrete mortar being treated.

Considerable information has been circulated on the possibility of an additional advantage of this material being able to act as a linseed oil treatment, or sealer, to a concrete surface. It was decided to broaden this investigation to include this purported capability.

Examination of the data in Table I indicates clearly the moisture retention qualities of the linseed oil emulsions as determined in the laboratory. When tested according to Test Method Tex-219-F, the moisture loss of the emulsified linseed oil treated specimens (specimens 25 through 48) was excessive in every case, even when the rate of spray was increased to 150 sq. ft. per gal. Both emulsion materials fail to satisfy the primary specification requirement for membrane curing compounds.

The sand blast test is an indication of surface hardness. Both from close examination of specimen surfaces and from the sand blast test results the detrimental effects of the linseed oil emulsions are obvious. The surfaces of those specimens cured with membrane curing compounds prior to the application of the emulsions were not affected by the emulsions. The linseed oil emulsions did not penetrate the membranes and thus the mortar surfaces were not affected. However, the surfaces of those specimens where the emulsions were applied as curing compounds (specimens 25 through 48) were crumbly, friable and easily abraded with a fingernail. Looking at the sand blast test results for these specimens as a group and comparing them with the results of all the other specimens, the difference in surface hardness is apparent. These two linseed oil emulsions very definitely have a deleterious effect upon the surfaces of these mortar specimens.

From the water permeability (Water Perm.) test results recorded in Table I, it can be concluded that the linseed oil emulsions were poorer seals than the membrane curing compounds and the linseed oil mineral spirits surface treatment. In fact the numerical values indicate that the blank specimens

are less permeable than those cured with the emulsions. It should be pointed out that it is possible that the degraded, crumbly surface of the emulsion specimens (specimens 25 through 48) contributed considerably to the higher water permeability values. Even though the water seal is very tight and absolutely impervious around the base of the gauge, the crumbly surface is a mass of tiny voids possibly creating a layer of porous material on which the water is sealed. Whether or not these surfaces are sealed beneath this layer is indeterminant and is immaterial since either way an objectionable condition exists. Values obtained with the linseed oil-mineral spirits blend indicate that this material does offer some protection when compared to the blank specimens and those treated with the emulsions.

The results of this investigation intimate that either emulsion used as a curing compound and sprayed at the rate of 150 sq. ft. per gal. will produce flexural strengths greater than the other surface applications tested (specimens 37 through 48 in Table I). The companion compressive strengths, however, are less than most of the other tests. Specimens 49, 50, 51, 52, 53 and 54 follow a similar pattern. The emulsion in conjunction with the curing compounds seems to increase the flexural strength while decreasing the compressive strength. The reasons for the development of this pattern are not presently apparent. Normally a loss in strength would be more immediately apparent in the flexure test with the compressive strength remaining more constant. Similar patterns in early strengths have been noted before where test cylinders have been allowed to dry out or lose excessive moisture prior to test, in effect, cutting short the hydration process. It is quite possible also, that the compressive strength was affected by both the poor surface found to exist

with the L.O.E. material and by the flexural test being performed first on the same sample later used for the compression test. The data indicates that the 444, Type 2 membrane curing compound produced the best over-all flexural and compressive strength combinations and that the linseed oil emulsion 218 sprayed at 180 sq. ft. per gal. was a close second.

The depth of penetration test is rather evasive. The maximum depths of penetration recorded in Table I are subject to change since the line of demarcation was indefinite and a question of judgement. The test did reveal that the emulsions will not penetrate a 72 hour cured membrane curing compound. The thinner linseed oil/mineral spirits blend penetrated the deepest into the blank specimens.

Neither of the linseed oil emulsions satisfies the run and sag consistency requirement of ASTM Designation C 309 and Texas Highway Department Standard Specifications. The importance of this requirement is reflected in the moisture loss and water permeability values obtained on specimens inclined at 5° from the horizontal. Specimens in that test condition indicated as much as .8% greater loss in moisture and increased permeability when compared to specimens sprayed and tested in the horizontal position. This was not found to be the case for the Type 1 and 2 membrane curing compounds.

The Texas Highway Department Standard Specifications, Item 531, "Membrane Curing," is based upon laboratory test performance. The linseed oil emulsions will not satisfy this specification. They can not be accepted as concrete curing compounds, they are detrimental to the surfaces of the mortar specimens, and from the results of this investigation their efficiency as surface sealers

is questionable. From a laboratory standpoint, based on the materials investigated, there seems to be no justification for the consideration of emulsified linseed oil as a curing compound and surface sealer for portland cement concrete.

APPENDIX

Infrared Study

Samples of linseed oil emulsions labeled R3-71-218 and R3-71-372 have recently been examined by us and the following information is submitted.

| | R3-71-218 | R3-71-372 |
|---------------|-----------|--------------|
| Gallon weight | 8.08 | 8.03 (split) |
| % Solids | 50.0 | 53.1 |
| pH (5-7-71) | 8.65 | 9.35 |

The above physical constants reflect only relatively minor differences; however, in the case of pH measurement the small difference may be significant when evaluated with other information available. The physical appearance of the two samples is somewhat different. Sample '218 is creamy in appearance and color with a tendency to accumulate a whitish bottom sediment and a 0.3" top oil layer upon standing for periods of more than 72 hours. Sample '372 is light gray in color and with a slightly more buttery or puffy consistency than '218. Sample '372 shows no distinct oil separation upon standing but has a much greater tendency to retain entrained air as evidenced by a 1/2" thick top froth after standing for 72 hours. This quality of air entrapment also made the use of normal techniques for obtaining the gallon weight impossible. The gallon weight for the sample noted on page 1 was obtained by the split weight technique using water as the diluent.

Infrared spectra were obtained for the emulsions as a whole as well as the oils extracted from each sample. Spectra for the emulsions were

run after drying a thin emulsion film at room temperature and under vacuum at 60°. IR spectra for the emulsions indicate that the two materials are very similar but the samples are not identical. The majority of each individual spectrum is indicative of linseed oil (the major constituent other than water); however, at least three distinct bands in the spectrum are not associated with linseed oil and are indicative of both the presence and quantity of some modifying agent or agents. These agents are most probably emulsifying and/or stabilizing agents and as such are necessarily volatile in order to render the membrane film incapable of reemulsification after having been applied for a short period of time. This concept is verified in the infrared spectrum by the disappearance of all bands not associated with linseed oil after drying a thin film at room temperature for approximately 4 hours.

Although the particular materials associated with the infrared bands attributed to emulsifying and stabilizing agents have not been identified, the intensity of these bands indicates a difference between the two emulsion samples. Based on the infrared spectrum, Sample '218 has only half or less the amount of stabilizing or emulsifying agent as is present in Sample '372. This information combined with the separation tendencies and lower pH of Sample '218 would seem to indicate that it is an unstable emulsion through loss or absence of stabilizers or insufficient alkalinity. The effect this condition may have on performance as a curing membrane is not known.

The overall characteristics of these samples would cause them to be classed as similar materials and most like the type produced by the emulsification of linseed oil with water in the presence of sodium hydroxide and high molecular weight alcohols. All analytical information and infrared curves are on file in Section B if needed.

Test Procedure for Flexural Strength of Concrete
(Using Simple Beam with Center-Point Loading)

Apparatus:

Use load-applying and support blocks as described in Figure #1 of ASTM C-293 for 6 inch span.

Test Specimen:

Dry cut a 2-1/8 x 2-1/8 x 12 inch beam from portion of 2-1/8 x 6 x 12 inch mortar slab.

Procedure:

Place top of dry specimen face down and center it on support blocks so that one support is one inch from the end of beam. Bring the load applying block in contact with the beam at the center line between support blocks.

Apply load at the rate of .036 in./min. and measure maximum applied load to the nearest pound force. After test, determine the average width and depth of the specimen at the section of failure, measuring to the nearest 0.001 inch.

$$\text{Flexural Strength psi} = \text{Modulus of Rupture} = R = \frac{3 Pl}{2 bd^2}$$

P = max. load in lbs.; l = span, inches; b = width, in.; d = depth, in.;

Test Procedure for Compressive Strength of Concrete

(Using 2-1/8 x 2-1/8 x 2-1/8 cube specimens)

Test Specimens:

Dry cut 2-1/8 x 2-1/8 x 2-1/8 inch cubes from broken beam specimens cut for flexural strength tests.

Cap top and bottom of specimens with high strength gypsum plaster (ASTM C-617 Section 3.2.1). Measure the average width, length and depth of the cube to the nearest 0.001 inch.

Procedure:

Place top of dry specimen face up and center it in the testing machine. Apply the load continuously and without shock at a constant rate within the range 50 to 75 psi/sec. During application of first of the maximum load a higher rate of loading shall be permitted.

Compressive strength in psi = Max. load \div area.