WRG 001121
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PLAINTIFF'S EXHIBIT
WRG-693

REFORMULATION AND EVALUATION OF MONO-KOTE

Project 2659 December 1972

Personnel: J. W. Kraus, R. N. Johnson, T. W. Sappington
Mathematical Dept. - P. J. Press

Project Objectives

1) Assist CPD in developing an asbestos-free Mono-Kote having 100% total equivalency to MK-III, per UL fire-test ratings, for steel decks, beams, and columns;

2) Assist CPD in developing a "Super" Mono-Kote with top UL fire ratings and possessing superior thermal, acoustical, and application properties.

Summary of Months Activities

Response data gathered in a parametric study of MK-IV, while curing and drying in a controlled environment, is being statistically analyzed by Phil Press of the Mathematical Department. He estimates the analyses will be complete by January 15, 1973. Higher priority work has delayed the evaluation.

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Studies of shrinkage and cracking in Mono-Kote were broadened this month to include MK-IV and MK-V. The same four factors - excess water, delayed spraying after mixing, pressurization and curing rate - were examined for their effects on shrink-cracking.

Present results show that MK-III exhibits greater shrinkage than either MK-IV or MK-V. MK-III shrinkage ($\frac{\Delta l}{l}$) ranged from -0.004, -0.006, -0.008, -0.0126 to -0.0191, while MK-IV obtained two values of +0.0029 and -0.0028 and MK-V showed recorded values of +0.0030, -0.0036, and -0.0059. Basis existing data, MK-III also showed the greatest tendency to develop cracks. This month's work has clearly shown that any of the three Mono-Kotes, III, IV, or V, can be made to undergo shrinkage and cracking when certain conditions are met, whether contrived or accidental. Of the four factors tested, the combination of delayed spraying and over watering has a powerful effect and leads to shrink-cracking. As singular effects, over watering and pressurization are comparable in their effect on shrinkage; MK-III $\frac{\Delta l}{l}$ = -0.0079 as a result of excess water and $\frac{\Delta l}{l}$ = -0.0039 and -0.0064 as an effect of pressurizing wet MK-III to 1300 psi.

Pressurization and delayed spraying plus excess water produce denser dried products with harder surfaces, which show recorded penetrometer values of 285-320 psi. Excess water alone

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produces a softer or punkier dried product. As a result of over watering, MK-III obtained a penetrometer reading of 150 psi when dried, MK-IV 91 psi and MK-V 70 psi.

WRC laboratory study of mold growth on MK-IV was reactivated this month. The purpose of this work is to determine the effects of ammonium isobutyrate (AIB) as a fungicide for Mono-Kote and compare its efficiency to sodium propionate (NAP). Fungicide concentrations were set at approximately 500 and 1500 ppm. These same fungicides are being tested for their effects on material strength of Mono-Kote, since CPD indicates a desire to incorporate the fungicide in the Mono-Kote formula.

I. Specific Objective (Target Date - December 1, 1972)

Determine the effect of environmental conditions on curing of MK-IV and MK-V.

Status

Laboratory work on an experimental design to evaluate the effects of environmental conditions on curing and drying behavior of MK-IV were completed in October 1972. The response data were then sent to the Mathematical Department (WRC) for statistical analyses. We were awaiting the outcome of the analyses before preparing a final report.

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Progress This Month (J. W. Kraus, P. J. Press, R. N. Johnson)

Phil Press informed me the statistical analyses was begun about November 20, 1972, but the work was not completed. A higher priority project at CPD has taken most of his attention and time. The recovery of missing data and the checking of questionable data have also caused some delay. He expects to finish these analyses between the 8th and 15th of January 1973.

Conclusions

None to add to or replace those stated in the October Monthly Summary.

Recommendations

None

Work Next Month

Review the results of the statistical analyses and prepare a final report to CPD.

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References

October Monthly Summary (10/31/72), Project 2659

November Monthly Summary (11/30/72), Project 2659

II. Specific Objective (Target Date - January 1, 1972)

Determine the factors that promote shrink-crack.

Status

Shrinkage and subsequent cracking of MK-III have been frequently found in field applications. Recent development of MK-IV and MK-V has raised similar questions about their potential cracking behavior under similar conditions. Within the last 30 days, one severe case of shrink-cracking was observed in MK-IV at a New York City job site. CPD personnel requested WRC laboratory to undertake a quantitative study of the factors believed to cause such cracking. First, it was necessary to devise suitable sample configurations to demonstrate both shrinkage and cracking in Mono-Kote, which was done. Then in October, it was shown that excess water was a significant factor in causing shrink-cracking in MK-III, but not necessarily MK-IV and MK-V. Later in November, it was reported:

1. A combination of delayed spraying after mixing and excess water interact to cause large shrinkage and severe cracking in MK-III on drying.

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2. MK-III subjected to high pressure, 1300 psi, will exhibit shrinkage and cracking on drying. CPD asked that a comparable study be done with MK-V.

Progress This Month (J. W. Kraus, R. N. Johnson, and T. W. Sappington)

Procedures of preparation and Mono-Kote treatment, such as delayed spraying, pressurization, or differences in curing rate are described in the November Summary.

Table I (revised 12/15/72) contains all data from the November Summary, some corrected, and recent shrinkage and cracking observations.

First, let us inspect the data for MK-III in Table I. The part we are primarily interested in is found in Columns 12 through 18, inclusive. Here it is found that shrinkage and cracking can be generated in MK-III by either excess water (at a water ratio of $\geqslant 2.3:1$) or delayed spraying until after the mortar sets. Excess hydraulic pressure (1300 psi) is also a contributory factor. This was disclosed in the November Summary so it is not new data. However, when last reported the samples were not fully dried so cracking had not fully developed and shrinkage measurements were not finished.

Of the four factors examined, a combination of delayed spraying and excess water (R_{H_2O} = 2.6:1) appears to cause the greatest shrinkage, where $\frac{\Delta l}{l}$ equaled -0.0126 and -0.0191 at 40 days curing and drying. Cracking was also most severe in this instance. In fact, shrinkage was so large as to cause severe map cracking of the Mono-Kote on flat galvanized steel sheets; fissures 1/8" width developed when the Mono-Kote was still in the curing stage.

Excess water is the most important single effect in promoting shrink-cracking. A shrinkage value ($\frac{\Delta l}{l}$) of -0.003 was recorded at 30 days. The Mono-Kote completely separated from the angle iron, at the interface, less than 20 hours after removal from the curing chamber.

High pressure is the second most important single effect in causing shrink-cracking. Subjecting Mono-Kote to a high pressure of 1300 psi, is comparable to piston pumping the wet mortar 40 stories height. This appears sufficient to bring about the undesired effect. Observations revealed the cracking phenomenon along the axis of the angle iron was less severe and map cracking on the flat plate was as hair lines. Measured shrinkage values equaled -0.004 and -0.006. This small amount of shrinkage was obviously sufficient to cause cracking of the MK-III.

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A comparison of Runs 28, 29, 30, and 31 shows within each pair, i.e., 28 and 29 or 30 and 31, the differences in shrinkage are small compared to differences between sample pairs. This shows positively that the major effects are produced by over watering, delayed spraying, and pressurization and the curing conditions have only a minor effect on shrink-cracking.

A. M. Rosenberg (CPD) telephoned me on November 21, 1972 to discuss shrink-cracking in Mono-Kote. A job site prepared with MK-IV from the Trenton plant (N.J.) had exhibited severe cracking and the Mono-Kote was found to set in 1 to 1-1/2 hours, too short a set time for easy handling. CPD has reserved a sample for WRC to examine. Meanwhile, WRC undertook a preliminary study with MK-IV from the New Castle, Pa. plant. The first factor tested was that of delayed spraying. Wet mortar was prepared at a water/Mono-Kote = 2.0:1 (see Run 34, Table I). After the mortar set (3.8 hrs.), water was added to refluidize the Mono-Kote which resulted in a final water ratio of 2.4:1. Naturally, such a high water level brought about a condition of over watering, but it was needed to make the Mono-Kote sprayable.

Initial work with over watered MK-IV produced a net expansion and no cracking, as is described in Table I, Run 19. In this test (Run 34) for the effects of delayed spraying, the

material was cured 7 days at 95% R.H. and 75°F without cracks appearing in the Mono-Kote. Two days after removal from the curing chamber, while drying, a long crack appeared in the Mono-Kote parallel (||) to the axis of the angle iron. Subsequently, cracks ⊥ to the axis showed. Although shrinkage was small (-0.0098), it was sufficient to cause cracking. This is obviously not a clear cut case for the effects of delayed spraying because there is interaction with excess water as well. A test for the singular effect has been devised.

The results of two tests prepared with MK-V, Runs 32 and 32A, can be found in Table I. Both were prepared to test for the effects of delayed spraying.

Previous work (Run 24) with MK-V, blended at WRC, showed no cracking effects as a result of over watering even at a water ratio of 2.5:1. Measurements did show a net expansion of MK-on-steel plate, as given in Columns 13 and 14 ($\Delta 1/1 = +.003$). This is no doubt an effect of air entrainment which caused the Mono-Kote to expand the first 30 minutes after spray placement. Runs 32 and 32A may have been prepared with a slight excess of water ($R_{H_2O} = 2.3:1$). However, the previous Run 24 tolerated a water ratio of 2.5:1. The

noticeable feature of this material was the ease with which the Mono-Kote could be refluidized by gentle stirring without the use of more water. The initial mixer Vicat number was 37 mm, which remained the same after set and stirring. The wet mix sprayed well. Both samples of MK-on-angle iron cracked after curing while drying. Curing conditions had little apparent effect on the course of shrinkage and cracking. A slightly greater shrinkage ($\frac{\Delta l}{l}$ = -0.0059) was obtained for the sample cured at 55% R.H. than for that ($\frac{\Delta l}{l}$ = -0.0036) cured at 95% R.H.

The results of penetrometer tests, surface hardness, are given in Column 11, Table I. These may be compared with wet and dry density values in Columns 9 and 10. MK-III data indicate that delayed spraying plus excess water or pressurization produce a denser, sprayed product of 64-75 pcf. compared to normal densities of 48-51 pcf. The dried products have correspondingly harder surfaces.

Conclusions

1. Excess water is a critical factor in causing MK-III to develop shrinkage and cracking. The critical level of water will no doubt vary for MK-III from plant to plant.

2. MK-IV and MK-V, basis singular tests, do not develop shrink-cracking as a result of over watering alone.

3. Over watering (excess water) Mono-Kote, as a single factor, produces a softer dried product.

4. Delayed spraying after mixing and excess water interact to produce severe shrinkage and cracking of any MK-on-steel, flat or angle iron.

5. Pressurization of wet MK-III, to 1300 psi, before spraying is sufficient abuse to cause shrinkage and cracking while drying.

6. Pressurization densifies the sprayed product and results in a harder, dry surface.

7. Curing conditions the first 7 days after placement will modify, but not override, the main effects of over watering, delayed spraying, or pressurization.

Recommendations

None

Work Next Month

Continue with the study of factors that cause shrink-cracking in MK-IV and MK-V. Check the effects of over watering with a plant blended MK-V. Examine the effects of delayed spraying,

without over watering. This can be accomplished by using a normal water ratio of 2.0:1, allowing the wet mortar to stand for intervals of one hour or two hours then spraying it into place.

References

Notebook No. 8245

October Monthly Summary (1972)

November Monthly Summary (1972)

III. Specific Objective (Target Date - February 1, 1972)

Determine the effects that gypsum manufacturing techniques have on Mono-Kote properties to include retarders, stabilizers, and aridizers.

Status

Work in this area has been delayed until more pressing problems, such as shrink-cracking and mold growth, are resolved. A shortage of trained personnel has also contributed to the delay.

Progress This Month (J. W. Kraus)

None

Conclusions and Recommendations

None

Work Next Month

Progress to be prepared and submitted to CPD.

References

None

IV. Specific Objective (Target Date - November 1, 1972)

Determine the cause of mold growth on MK-IV, the host for mold growth and the means for arresting growth.

Status

CPD requested WRC assistance to investigate the growth of mold on wet MK-IV. WRC responded by carrying out a laboratory study and arranged for a field trial. Laboratory results disclosed that sodium propionate (450 ppm) and Hyamine 2389 (800 ppm conc.) were promising candidate fungicides. Later field tests confirmed the laboratory results. Photographs were taken before and 4 days after the field sites were sprayed. Since mold spores are air-borne, there is little that can be done to prevent their contact with MK-IV.

However, adequate ventilation is one of the best preventives against mold growth. In those instances where ventilation is poor or non-exist, fungicide may be supplied as an integral part of the Mono-Kote formula or sprayed on the observed mold site. An attempt was made to test for the effects of sodium propionate (500 ppm) on Mono-Kote material strength. The test results were inconclusive and should be repeated or evaluated at CPD by the Korenberg Furnace test.

Progress This Month (J. W. Kraus and R. N. Johnson)

Mr. T. G. Gibian suggested that ammonium isobutyrate (AIB) might be a suitable fungicide for Mono-Kote. Other independent work at WRC has shown AIB protects silage from mold growth during storage, up to one year duration, at a conc. of 1.75%. This chemical is a by-product from a W. R. Grace plant, Hatco Division, Fords, N.J. It is available as liquid (60% active ingredient) or as dry solids with 5% clay. In bulk quantity, AIB can be obtained in a truck load or less at a cost of $0.20/lb. We have followed Mr. Gibian's suggestions and re-opened the study of mold growth inhibitors with AIB.

A set of MK-IV control samples without fungicide, two sets of Mono-Kote plus AIB and two sets of Mono-Kote plus sodium propionate (NAP) were prepared. Sprayed samples were placed in a high humidity chamber to develop mold growth (95% R.H. at 75°F). Tests were begun on December 12, 1972 and are expected to continue through January 15, 1973, with daily observations made for detection of mold growth.

A. M. Rosenberg informed us that CPD wants to add fungicide directly to dry blended Mono-Kote. Past results with Mono-Kote and chemical additives indicated that small amounts can alter the bonding properties of Mono-Kote drastically. The first attempt to learn of the effects of NAP on material strength were inconclusive. We have taken this opportunity to conduct simultaneously with the mold work a study of bond properties as affected by the fungicides. To this end, samples for fire cup tests, tensile adhesive and tensile strength tests, and adhesive shear tests were prepared. AIB concentration was set at 550 ppm and 1000 ppm, basis the water added to the Mono-Kote. NAP concentrations were set at 500 and 1500 ppm.

Results to date show mold growth on the control Mono-Kote, which appeared 7 days after preparation. AIB and NAP treated samples show no mold activity, yet.

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Conclusions and Recommendations

None

Work Next Month

Continue daily observations for mold growth. After 14 days remove four control samples and spray two with 500 ppm solution of AIB and two with 500 ppm NAP solution. Observe daily for kill of the mold mycelium. Test each set of samples for bond strength properties.

References

Notebook No. 8212

Memo: J. W. Kraus to A. M. Rosenberg (10/12/72)

October Monthly Summary (1972)

November Monthly Summary (1972)

John W. Kraus — Date

JWK:des

Attachment (Table I)

cc: C. C. Legal
L. V. Triggiani
A. M. Rosenberg
R. C. Ericson
R. Rettew
I. Odler
K. Maity

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Table

Kote Shrinkage and Cracking Study
(Revised 12/15/72)

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Dry Density (pcf)	Hardness Soil Pen. (⊕ X da.)psi	Initial Length of Steel Plate l_o (cm)	Dimensional Change (⊕ X da.) (-) Δl (cm)	$\frac{\Delta l}{l}$	Cracking Observations: Angle ⊥, ∥ or 0	Interface MK - Steel + or -	Plate (Map Cracking) + or -	Transverse to Long Axis + or -
23.7	(7 da.) 0 Oven Dry-150	30.48	(9 da.) - 0.210 (30 da.) - 0.243	- .0068 - .0079	0	+ *	-	-
22.2	(13 da.) 275 Oven Dry-285	30.47	(12 da.) - 0.448 (42 da.) - 0.582	- .0146 - .0191	∥	+	+	+ ‡
21.3	(10 da.) 320 Oven Dry-285	30.57	(9 da.) - 0.207 (38 da.) - 0.383	- .0057 - .0126	⊥ ∦ ∥	+	+	+ ‡
25.9	(9 da.) 315 Oven Dry-295	30.43	(8 da.) - 0.112 (37 da.) - 0.120	- .0036 - .0039	∥	+	-	-
26.3	(8 da.) 55 Oven Dry-320	30.44	(8 da.) - 0.115 (36 da.) - 0.195	- .0024 - .0064	∥	+	-	-
20.7	(7 da.) 0 Oven Dry-91	30.48	(10 da.) + 0.080 (30 da.) + 0.089	+ .0026 + .0029	0	-	-	-
--	(15 da.) 140 Oven Dry-230	30.48	(23 da.) - 0.084	- .0028	⊥ ∦ ∥	+	+	+
18.0	(7 da.) 15 Oven Dry-70	30.50	(7 da.) + 0.065 (30 da.) + 0.092	+ .0021 + .0030	0	-	-	-
--	(24 da.) 120 Oven Dry-190	30.48	(31 da.) - 0.180	- .0059	∥	+	+	-
--	(24 da.) 120 Oven Dry-165	30.48	(31 da.) - 0.111	- .0036	∥	+	+	-

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J. W. Kraus
Supersedes November, 1972
Version of Table I

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Mono-Kote Formula	Source	Run No.	Treatment	Cure Conditions	Set Time Hrs.	R_{H_2O} X:1	Nozzle Vicat (mm)	Nozzle De
III	New Castle, Pa. BF 55 J82AK6-888 Drum No. 96	21	Excess H_2O	7 da. @ 95% R.H. & 75°F	1.5	2.3	15.0	(
III	New Castle, Pa. Drum No. 96	28	Del.-Spray	7 da. @ 95% R.H. & 75°F	2.25	2.0 (2.63)	(22)M 24	(5 6
III	New Castle, Pa. Drum No. 96	29	Del.-Spray	7 da. @ 55% R.H. & 75°F 8.0 MPH A.V.	2.5	2.0 (2.63)	(22)M ?	(5 6
III	New Castle, Pa. Drum No. 96	30	Pressur-ized	7 da. @ 55% R.H. & 75°F 8.0 MPH A.V.	2.5	2.1	3.0	7
III	New Castle, Pa. Drum No. 96	31	Pressur-ized	7 da. @ 95% R.H. & 75°F	2.5	2.1	4.0	75
IV	New Castle, Pa. Drum No. 80	19	Excess H_2O	7 da. @ 95% R.H. & 75°F	4.5	2.1	17.0	61
IV	New Castle, Pa. Drum No. 96	34	Del.-Spray	7 da. @ 95% R.H. & 75°F	3.8	2.0 (2.42)	3.0	69
V	Baltimore, Md. WRC-Lab	24	Excess H_2O	7 da. @ 95% R.H. & 75°F	--	2.5	20.0	62
V	Travelers Rest, S.C.	32	Del.-Spray	7 da. @ 55% R.H. & 75°F 8.0 MPH A.V.	5.5	2.3	37.0	67
V	Travelers Rest, S.C.	32A	Del.-Spray	7 da. @ 95% R.H. & 75°F	5.5	2.3	37.0	67

* Complete separation at interface

⊥ Perpendicular to axis of angle iron

∥ Parallel to axis of angle iron

‡ Longitudinal cracks also