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Microscopic Investigation of Scaled and Unscaled Concrete

INVESTIGATION CONDUCTED BY THE MICHIGAN STATE HIGHWAY DEPARTMENT

In Co-operation with

THE ENGINEERING EXPERIMENT STATION OF MICHIGAN STATE COLLEGE

By

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MICHIGAN ENGINEERING EXPERIMENT STATION

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CONTENTS

	Page
Introduction	5
Concrete Pavement Scale	6
Physical and Chemical Properties of Scale	7
Microscopic Study of Scale	8
Comparison of Scaled and Unscaled Concrete	10
Solubility of Calcium Hydroxide in Calcium Chloride Solution	13
Concentration of Calcium Chloride on Highway Surfaces	16
Depth of Penetration of Calcium Chloride	17
Summary of Microscopic Studies	. 18
Bibliography	19

ILLUSTRATIONS

			Page
Fig.	1.	Enlarged view of scale showing its resemblance to con- crete mortar	8
Fig.	2.	Photomicrograph of scale showing crystalline calcium hydroxide	9
Fig.	3.	Photomicrograph of calcium carbonate deposits in cracks	10
Fig.	4A.	Photomicrograph of section of scale surface showing cracks penetrating from surface	11
Fig.	4B.	Photomicrograph showing crystalline calcium hydroxide in cracks	11
Fig.	5.	Photomicrograph of scaled surface showing crystalline calcium hydroxide in cracks	12
Fig.	б.	Photomicrograph showing concentration of crystalline calcium hydroxide near surface of pavement	13
Fig.	7.	Photomicrograph showing formation of calcium hydrox- ide crystals on neat cement plate	14
Fig.	8A.	Photomicrograph showing calcium oxychloride crystals on neat cement plate	14
Fig.	8B.	Synthetic calcium oxychloride crystals from pure com- ponents of calcium hydroxide and calcium chloride in water	14
Fig.	9.	Graph showing solubility characteristics of calcium hy- droxide in water and calcium chloride solution at dif- ferent temperatures	15
Fig.	10.	Graph showing penetration characteristics of calcium chloride in concrete pavements	17

FOREWORD

This bulletin presents a report of a special study of scaled and unscaled concrete which was performed in conjunction with an investigation on the durability of concrete being conducted by the Michigan State Highway Department.

The preparation of the thin sections and subsequent microscopic analysis are the work of John A. Young, Petrologist, Geology and Geography Department, Michigan State College. The physical and chemical phase of the work was conducted by Richard Thurm and George T. Bauer, Research Chemists of the Michigan State Highway Department. The investigation was under the general supervision of the author and J. W. Kushing, former Research Engineer of the Michigan State Highway Department.

The Research Laboratory of the Michigan State Highway Department, of which Charles M. Ziegler is State Highway Commissioner, is located in the R. E. Olds Hall of Engineering on the Michigan State College Campus, East Lansing, Michigan.

Microscopic Investigation of Scaled and Unscaled Concrete

By E. A. FINNEY

INTRODUCTION

One form of concrete deterioration prevalent in Michigan is surface scaling and subsequent disintegration of the pavement from the surface downward. In 1939 a survey of concrete pavements on state trunk line highways in Michigan revealed that approximately 6.5 percent of the total pavement surface had scaled in varying degrees as follows: 6.3 percent light to medium surface scale, and 0.2 percent heavy scale to progressive disintegration. Other states in corresponding climatic zones have experienced similar scaling conditions. This condition has become more apparent as the use of chemical salts has increased for the removal of ice from pavements. The unsightliness of scaled areas, the necessary subsequent maintenance and the added possibility of further deterioration are of immediate concern to the highway engineer.

Consequently in 1939, an investigation of scaling, to determine its causes and methods of prevention, was incorporated into the research program of the Michigan State Highway Department as a definite project under the title "Durability of Concrete". The investigation was dedicated to the establishment of certain fundamental principles in concrete construction and to the correlation of certain laboratory studies with construction methods in order to develop more durable concrete pavements. To obtain this end, the investigation was divided into two separate studies: first, a laboratory study of concrete durability as related to scaling; and second, the construction of a special pavement for field observations which is widely known as the durability project of the Michigan Test Road.

The natural approach to the scaling problem has been to find ways and means of improving the quality of the concrete. In this respect the problem resolves itself into several phases: first, the acquisition of certain knowledge concerning the phenomenon of scaling; second, the improvement of the quality of the concrete constituents; third, improvement of workmanship as related to the finished product; fourth, the improvement of concrete by changing its character; and fifth, by the application of surface treatments. All of these factors have been considered to some degree in the scope of the research program pertaining to the durability of concrete for pavements.

This report presents the results of a study pertaining to the acquisition of certain knowledge associated with the phenomenon of scaling. The study was undertaken in connection with the investigation on durability of concrete in an attempt to discover any visible chemical or physical properties associated with scaling.

The study includes a review of the scaling phenomenon as observed to take place on concrete pavements, the physical and chemical properties of scale material, microscopic analysis of scale and its composition, microscopic comparison of scaled and unscaled concrete, solubility characteristic of calcium hydroxide in calcium chloride solution, the concentration of calcium chloride on pavement surfaces and depth of penetration of calcium chloride into the concrete surface.

Studies were made of thin sections cut out of pavement cores taken from scaled and unscaled pavements as well as from fragments of scale which had come from scaled portions of the highway. The thin sections were made according to the technique commonly used in petrography and described in most petrographic reference books. The thin sections were then examined in polarized light under the petrographic microscope.

CONCRETE PAVEMENT SCALE

Scale as related to concrete pavements may be pictured as a thin heterogeneous lamina approximately 1/16 to 3/8 inch thick appearing sporadically on the surface, in general composed of cement particles and components of hydrated cement in combination with very fine aggregate, and possessing physical and chemical properties analogous to those of the parent concrete.

The process of scaling is generally understood to include: first, the formation of scale; second, the deterioration of scale by physical and chemical processes, and third, the subsequent displacement of the scale from the pavement surface.

The scaling phenomenon is caused by the sequence of several events, the first of which is the deposition of a thin layer of heterogeneous material on the pavement surface, owing to the redistribution of the concrete components. The redistribution of the various components occurs because of the differences in specific gravities and sizes of the various components, causing a migration of both light and small particles towards the surface. This condition is accelerated by the presence of excess mixing water and by excessive manipulation of the concrete mass during placing and finishing operations. The hardened scale is analogous in character to the parent concrete. Second, because of its inherent properties, scale disintegrates under the influence of internal and external chemical and physical forces. In this connection the products of hydration are subject to hydrolysis and decomposition through the action of water and carbon dioxide in the air; also by contact with chemically active materials applied to the surface. Scale disintegrates physically, as manifested by cracking of the scale structure owing to abnormal physical forces incurred by crystal growths and excessive volume changes caused by fluctuations in temperature and moisture conditions. Finally, further breakdown and displacement of the scale from the pavement surface occurs under the influence of freezing and thawing action and abrasion by traffic.

Physical and Chemical Properties of Scale

Analysis of various samples of scale revealed certain definite generalities with respect to chemical and physical properties. Chemically, the scale has essentially the same composition as the mortar in the parent concrete. However, the proportions of the various components have been somewhat altered owing to segregation of the ingredients of the concrete. Determination by chemical analysis of cement content in scale samples by various methods shows that the scale contains more cement than the underlying concrete. For example, analysis showed that the average cement content of the underlying concrete was between 17 and 21 percent, while the cement content of the scale ranged from 19 to 25 percent.

On the other hand, it was observed that the cement content of the mortar paste in the parent concrete contained more cement than the scale. The cement content of the mortar paste ranged from 25 to 30 percent.

It is to be noted that the analyses of the parent concrete were made of material taken from immediately below the scale area. Consequently, it is only natural to expect that the cement content at that point will be somewhat higher than the design calculated value which amounts to 13 and 30 percent in the concrete and mortar, respectively. This variation in cement content is believed to be due to the natural migration of the fine cement particles towards the surface at the time of manipulation of the concrete. Thus it is evident that the scale differs in composition from that of the pavement concrete and mortar paste.



Fig. 1. Enlarged view of scale showing its resemblance to concrete mortar.

From a physical standpoint, the scale was found to be structurally weak and friable, showing incipient cracking and great porosity. In addition, scale possesses a different coefficient of expansion under the influence of temperature and moisture variation than that of the parent concrete.

An examination of scale and parent concrete immediately under the scale showed a marked difference in specific gravity, pore volume, water absorption and permeability. In most cases the scale material had values different than those of the parent concrete. There was a difference in thermal coefficients between wet and dry specimens, and in different temperature ranges. The data in Table 1 show the variation in thermal coefficients ranging from approximately 8×10^{-6} to 10×10^{-6} per degree centigrade for a temperature range between 24° and 50° C. The change in length from wet to dry state was of the order 20×10^{-5} to 30×10^{-5} inches per inch at 24° C. The coefficients were determined by a Chevenard Thermo Analyzer requiring a specimen $\frac{1}{8}$ " x $\frac{1}{4}$ " x 2" in length.

Microscopic Study of Scale

Under the petrographic microscope, scale resembles normal mortar in structure and color except in the size and uniformity of the aggregate particles. An enlarged scale section is illustrated in Fig. 1. There were present in the scale microscopic cracks in which were found

	Scale	Top parent concrete
Apparent specific gravity	2.30	2.43
Pore volume (percent)	6.2	3/1
Absorption (percent)	9.06	4.6
Permeability (inches per 24 hours)	8/32″	3/32″
Thermal coefficient of expansion—X10-6 Dry, 24°C. to 50°C	8.8	10.0
Thermal coefficient of expansion-X10-6 Wet, 24°C, to 50°C	9.6	9.2
Change in length due to moisture-X10-3 at 24°C. (inches per inch)	0.32	0.20

TABLE 1-Comparative data on physical properties of scale and parent concrete

deposits of crystals in considerable amounts. Among the crystals identified were calcium carbonate and calcium hydroxide, with a trace of unhydrated dicalcium silicate and tricalcium silicate. Scale containing crystals of calcium hydroxide is illustrated in Fig. 2.



Fig. 2. Photomicrograph of section of scale showing crystalline calcium hydroxide dispersed throughout the gel structure and concentrated in the cracks as designated by white areas. Crossed nicols, 80X.

The presence of calcium carbonate in the cracks probably results from a reaction of calcium hydroxide with the carbon dioxide of the air. This conversion of hydroxide into carbonate was duplicated in the laboratory by subjecting cracked neat cement plates to an atmosphere



Fig. 3. Photomicrograph of calcium carbonate deposits in cracks formed in neat cement subjected to an atmosphere of carbon dioxide. 80X.

of carbon dioxide as shown in Fig. 3. The irregular, connected white areas denote the presence of calcium carbonate in the cracks of the neat cement plate which was subjected to carbon dioxide in an accelerated manner.

An examination of the cleavage plane between scale and underlying concrete revealed a preponderance of large calcium hydroxide crystals. In nomal concrete calcium hydroxide is microcrystalline.

Large crystals of calcium hydroxide were also distributed uniformly throughout the scale structure. This is clearly illustrated in Fig. 2. The irregularly shaped white patches

are concentrations of calcium hydroxide crystals. The large, clear white patches are quartz grains in the aggregate.

COMPARISON OF SCALED AND UNSCALED CONCRETE

A microscopic analysis of concrete which had scaled revealed that the concrete near the surface contained many fine cracks penetrating much of the section to a depth of at least $\frac{3}{4}$ inch below the surface, which was the thickness of the specimen under observation. The sections of scaled concrete illustrated in Figs. 4 and 5 show the development of calcium hydroxide crystals in the cracks. Throughout this illustrated area, and especially near the top surface of the pavement, calcium hydroxide was present in moderate amount as defined by the white specks. Two sections of scale collected from the same area, cut parallel to and just below the top surface, showed the calcium hydroxide crystals to be present to a considerable degree, and in some places constituting almost the entire area of the scale section. The occurrence of associated crystal growth has been observed by other investigators (1)



Fig. 4A. Photomicrograph of a section of scaled concrete showing cracks penetrating from surface. Large white areas are aggregate particles. Small white flecks are crystals of C_sS , C_2S ,* or $Ca(OH)_2$. 80X.

Fig. 4B. Same view as A with crossed nicols, 80X. Large crack filled with crystalline calcium hydroxide is visible. The white area extending to right of crack is a large concentration of calcium hydroxide crystals, apparently developed by solutions permeating the porous gel structure.

 $C_aS = 3 CaO$. SiO₉, Tricalcium Silicate

 $C_{p}S = 2 CaO$. SiO₀, Dicalcium Silicate



Fig. 5. Photomicrograph of scaled concrete parallel to surface, crossed nicols, 80 X. Black area to left is region where fragment was broken from section. Note concentration of crystalline calcium hydroxide along small cracks.

(2) (3).* The scale sections were all rather badly cracked, and these cracks were filled with crystals of calcium hydroxide, indicating that solutions of some sort must have passed through these cracks, either during the curing stage or at some later time. The scale sections, especially where abundant calcium hydroxide is present, show only a trace of dicalcium silicate and tricalcium silicate, whereas the amorphous cement $\frac{1}{2}$ inch below the surface shows a trace of tricalcium silicate and a larger amount of dicalcium silicate particles.

An analysis of an unscaled surface showed that, in general, there existed a zone up to approximately 2 mm. in thickness in which there was a considerable amount of crystalline calcium hydroxide. The average size of all of the calcium hydroxide crystals varied from 1 to 2 microns. The cement below this zone contained an abundance of calcium hydroxide.

Figure 6 shows a portion of the upper surface of unscaled concrete. The large, white areas on right and left are sand grains from the aggregates. The irregular, blotchy white mass between these two

^{*(1), (2), (3)} See Bibliography.

consists of numerous concentrations of extremely minute crystals of calcium hydroxide. This zone extends only to a depth of 0.5 mm. below the surface. The amount of calcium hydroxide present at the upper surface is very small.

SOLUBILITY OF CALCIUM HYDROXIDE IN CALCIUM CHLORIDE SOLUTION

The literature shows considerable evidence that calcium chloride is detrimental to concrete pavement when applied to the surface for ice removal purposes. Our laboratory and field studies also show that calcium chloride greatly accelerates scaling. The process by which the chlorides attack the concrete has not been clearly understood.



Fig. 6. Photomicrograph showing concentration of crystalline calcium hydroxide toward surface of pavement. Crossed nicols, 80X.



Fig. 7. Photomicrograph showing formation of calcium hydroxide crystals on a neat cement plate exposed to calcium chloride solution. 80X.

On the basis of the relationship found in our study of scale it is probable that the deteriorating effect of the chloride salts may be due in part to the promotion of crystal growth, resulting in abnormal interstitial pressures, and in part to the disintegrating effect of the chlorides through removal of certain cement constituents.



Fig. 8A. Photomicrograph showing formation of calcium oxychloride crystals on a neat cement plate treated with calcium chloride solution. 80X.



Fig. 8B. Synthetic calcium oxychloride crystals from pure components of calcium hydroxide and calcium chloride in water.

Experiments in the laboratory on neat cement plates exposed to calcium chloride solutions revealed the conversion of minute particles of calcium hydroxide into large crystals of calcium hydroxide, as illustrated in Fig. 7. This confirms the hypothesis made on the basis of microscopic observation in Fig. 2. Further, it can be shown that the presence of calcium chloride in certain concentrations removes the calcium hydroxide from the rigid cement gel and forms new chemical compounds, one of which can be definitely identified in crystalline form as calcium oxychloride (Fig. 8). Available data further indicate the existence of calcium oxychloride in dilute solutions of calcium chloride when in contact with neat cement. Solubility characteristics



Fig. 9. Graph showing solubility characteristics of calcium hydroxide in water and calcium chloride solution at different temperatures. (4)

(4) See Bibliography.

for calcium hydroxide in water and in calcium chloride solutions are shown in Fig. 9. These chemical phenomena are conducive in a variable degree to the ultimate decomposition of the scale material.

The rate and degree of decomposition of the scale as affected by chloride salts are proportionate, to a certain extent, to the concentration of the salts and depth of penetration. The lack of factual data concerning the probable concentrations of salts on pavement surfaces and the depth of penetration under normal conditions has led to a study of these factors.

CONCENTRATION OF CALCIUM CHLORIDE ON HIGHWAY SURFACES

It is generally recommended that calcium chloride should not be applied to concrete pavements for ice removal purposes in amounts sufficient to result in concentrations exceeding 10 percent.

Lack of available data on the subject led to a brief investigation to determine the probable maximum concentration of chemical salts on highway surfaces resulting from continued applications and subsequent evaporation of the water resulting from the melted snow and ice. Certain areas on specially selected trunk lines were chosen for study where it was expected maximum concentration would occur under normal ice control treatments. In order to obtain a truly representative picture, four test areas were established. These four areas included street and trunkline intersections where it was known that frequent applications of calcium chloride would be required, and on a two-lane and four-lane rural highway subjected to normal ice control treatments.

The amount of calcium chloride present on the pavement surface was determined both by analyzing the melted snow and ice and by examination of the dry dust brushed from the pavement surface. The samples were taken from badly scaled surfaces conducive to localized concentrations in depressions and from unscaled pavement surfaces where it was natural to expect that low concentrations would be found because of traffic and drainage conditions.

The results of these analyses as presented in Table 2 show that high concentrations of calcium chloride may occur under favorable conditions even though the average concentration during application for ice control is kept at a low value.

d a



Fig. 10. Graph showing penetration characteristics of calcium chloride in concrete pavements.

DEPTH OF PENETRATION OF CALCIUM CHLORIDE

In conjunction with the concentration studies, measurements for depth penetration of calcium chloride were made. Portions of the pavement slab were removed at intervals of $\frac{1}{8}-\frac{1}{4}-\frac{1}{2}-\frac{3}{4}$ and 1 inch, respectively, below the surface and analyzed. Results from these analyses are illustrated in Fig. 10.

The measurements for depth of penetration were made eight weeks after the last salt application. In this interval the rain apparently had reduced the calcium chloride content in the top $\frac{1}{8}$ inch of the pavement to approximately 0.1 percent. The 0.1 percent is somehow tenaciously retained possibly in the form of a double salt. Maximum concentration seemed to occur in the region between $\frac{1}{8}$ and $\frac{1}{2}$ inch below the surface. Only traces of chloride were found at a depth beyond $\frac{3}{4}$ inch from the surface. These traces of calcium chloride evidently were contained in the original concrete and did not diffuse from the surface.

Test area	Concentration of CaCle in slush and water on surface. Percent by weight	Concentration of CaCl ₂ in moisture contained in the sand and dust on surface. Percent by weight
1	$\substack{0.31\\0.42}$	$28.5 \\ 31.6$
n	$\begin{smallmatrix}0&44\\0&41\end{smallmatrix}$	$\begin{array}{c} 42.8\\51.6\end{array}$
Πι	$\begin{array}{c} 0.22\\ 0.51 \end{array}$	20.2 10.8
IV	$\substack{0.55\\0.37}$	$39.7 \\ 42.6$
v	0.23 0.41	$\begin{array}{c} 15.5\\ 49.6\end{array}$

TABLE 2-Concentration of calcium chloride on pavement surface

SUMMARY OF MICROSCOPIC STUDIES

From a physical standpoint, concrete scale was found to be structurally weak and friable, with great porosity. The volume changes of scale were found different from those of parent concrete under the influence of temperature and moisture. These characteristics promote separation of the scale by causing differential expansion and fracture. The high porosity of the material also makes it subject to ready penetration by water and salt solutions, with subsequent disruption due to freezing and chemical action.

The scale has essentially the same composition as the mortar in the parent concrete with slight variations in the proportions of the various components, the average cement content of the scale being somewhat higher than that of the parent concrete.

Microscopic analysis revealed deposits of calcium carbonate and calcium hydroxide in microscopic cracks throughout the scale. Also large crystals of calcium hydroxide were found to be distributed uniformly throughout the scale structure as well as in the cleavage plane between scale and underlying concrete.

Concrete which had scaled contained numerous fine cracks penetrating downward from the surface. Calcium hydroxide crystals were observed in the cracks.

An analysis of unscaled concrete revealed a zone approximately 2 mm. in thickness at the surface which contained considerable crystalline calcium hydroxide. The average size of the calcium hydroxide crystals varied from 1 to 2 microns. The presence of certain concentrations of calcium chloride promotes the crystal growth of calcium hydroxide. It also removes calcium hydroxide from the cement gel by forming new chemical compounds, one of which can be positively identified in crystalline form as calcium oxychloride. Examination of pavement surfaces shows that sufficient concentration of calcium chloride is present to cause these reactions.

The mechanism of these reactions is assumed to be as follows:

1.
$$\frac{3 \text{ CaO} \cdot 2 \text{ SiO}_2 \cdot n \text{ H}_2\text{O} + \text{Ca (OH)}_2}{\text{Hydrated Cement}} + \frac{\text{CaCl}_2 \cdot n \text{ H}_2\text{O}}{\text{Solution}} \longrightarrow$$
$$\frac{3 \text{ CaO} \cdot \text{CaCl}_2 \cdot 15 \text{ H}_2\text{O}}{\text{Calcium Oxychloride}} + \frac{\text{SiO}_2 \cdot n \text{ H}_2\text{O}}{\text{Hydrated Silica}}$$

2. Calcium Oxychloride + Temperature Change + Dilution > $\frac{\text{Ca (OH)}_2}{\text{Crystalline}} + \frac{\text{CaCl}_2 \cdot n H_2O}{\text{Solution}}$

On the basis of the relationships found in this study it is evident that the decomposition of scale material is due in part to the inherent physical characteristics of scale resulting during its early formation and in part to the chemically deteriorating effect of chloride salts which promote crystal growth, resulting in abnormal interstitial pressures and the removal of certain cement constituents from the gel structure.

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The Michigan Engineering Experiment Station was established on January 16, 1924, by resolution of the State Board of Agriculture.

The station is maintained for carrying on investigations and research in engineering, and the publication of the results thereof, for the stimulation of scientific ideals and interests in the engineering students and teachers of the college, for contributing to engineering science and for increasing the contact of the college with the people of the state engaged in engineering industries or in other industries dependent to a greater or less extent upon engineering.

Correspondence is solicited. Address: Engineering Experiment Station, Box 470, East Lansing, Michigan.

*Leave of absence.