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G. G. Wheat

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STUDIES IN THE SOLUBILITY OF PORTLAND CEMENT CONTINUED FROM 1908.

BY G. G. WHEAT.

In the year 1908 we presented a brief paper before the Academy of Science, calling attention to certain uses of portland cement concrete for farm drainage and city sewer purposes and in that connection raised the question of the fitness of the material for the use made of it. The importance of the question has greatly increased, as the spirit of improvement and progress is running like wildfire over our state.

Approximately \$300,000,000 will be spent in reclaiming wet lands of the Wisconsin Drift region of Iowa. Of this perhaps \$30,000,000 will be open drainage ditches, the remaining \$270,000,000 being the cost of drain tile and the cost of laying.

Perhaps as much more will be spent in the remaining sixty-nine counties. The rapid advance of our cities and villages in sewerage systems will call for an expenditure of fully one-tenth as much in sewer systems, the grand total being not less than between six and seven hundred millions.*

When compared with the cost of the Panama Canal, quite accurately estimated at from three hundred seventy-five to four hundred millions, its importance becomes more manifest. In view of the fact that all construction is underground and most of it impossible to inspect, the necessity of perfect materials is obvious. Since the crops upon the millions of acres to be drained will depend upon the free circulation of ground waters through the tile systems, the importance of the nature of the materials, as related to our future food supply, asserts itself, and perhaps more important than all, the health of our citizens in our villages and cities depends upon pure water for the household and a perfect elimination of all excretia from the city and from its soil.

In this last collection the recent reports from the city of LeMars are of interest. Sewage contamination of the city water led to the examination of the sewer system. A break in the sewer manhole connections

*Note.—These calculations are based upon the actual amount expended to date in thirty counties, the actual acreage cost and the acres remaining yet to be done. These records were personally taken from the county records by the writer.

was repaired and it was expected that the trouble would cease. Later the writer visited the city, inspected a number of manholes and found that considerable of the cement mortar in the bottom of manholes is in various stages of decay, some crumbles readily and is easily removed. This condition should it prevail in the mortar joints of pipe lines, offers an outlet in the ground water in the gravel beds which supply the city water. The matter has gone into the courts, meanwhile the people's health is at the mercy of escaping sewage and poisoned household water.

A continuation of the studies suggested two years ago called for a definite inquiry as to the exact power of pure distilled water to act upon the substance of portland cement. Mr. Orin L. G. Kipp, now assistant professor at Ames, carried out tests in the school year of 1908-9.

Samples were taken from a number of twelve-inch concrete pipe. These were chosen to study two conditions of pipe. Some of these pipe rang clear and strong under the testing hammer and when submitted to the crushing test carried a load of far more than the required strength. The broken section showed dense, close concrete with plenty of cement. These pipe carried a load equal to 77% of first class vitrified pipe tested on the same machine and the same method of contact. These samples were chosen as being representative of the better quality of concrete now being manufactured in the state of Iowa.

In preparing a sample for testing, a piece about an inch square was broken off and all the loose pebbles or sand brushed from it. The sample was then heated in an oven at about one hundred degrees C. until constant weight was obtained. After weighing the sample, it was placed in a Gooch crucible which was lined with a filter cup, and this was suspended at the lower end of the inverted condenser projecting into a Soxhlet extractor. The bowl of this extractor was filled about two-thirds full of distilled water, and the flame lighted below it.

As this method of producing a flow of water over the concrete might be subject to criticism on account of the fact that the water flowing over the concrete was hot, a piece of special apparatus was constructed which allowed the water to flow over the concrete at normal temperature. This special apparatus is shown in the figure below, a slight study of which will make its working clear. The steam passes from the bowl of the tube to the right and into the condenser from which the cold water flows over the piece of concrete, "A," and then flows back to the bowl at intervals as the receptacle below fills and syphons over.

In order that a fair comparison of the action of cold and hot water might be obtained, two samples as nearly alike as possible were taken from the same piece of concrete, and after being prepared in the usual

manner, one was placed in the special apparatus and the other in the ordinary extractor. The per cents dissolved under the two conditions I will dwell upon later.

After an extractor had run for a period, the length of which was generally chosen at 120 hours, the bowl and its contents were replaced by another bowl containing a fresh supply of distilled water. The dissolving process was then continued for another period.

As soon as a bowl was removed the analysis of its contents was begun. The bowl was placed in a sand bath and the water evaporated to dryness. It was then heated in an oven and cooled in a bell jar. Even where this method was followed, some difficulty was experienced in obtaining the exact weight, as the contents of the bowl gathered moisture as soon as exposed to the air. After weighing, the bowl was filled with dilute HCl and kept on a sand bath for about three days or until the deposit which adhered to the glass could be rubbed loose with a "Police-man." By this process the greater portion of the contents could be removed to an evaporating dish and evaporated to dryness. It generally happened, however, that upon drying, the bowl still showed a white film adhering in spots. To recover this, the bowl was filled with Ammonium Hydrate and again placed in the sand bath for a time. This second amount was likewise evaporated to dryness, then made slightly acid with HCl and added to the first. The bowl was weighed after being dried in an oven and cooled in a bell jar. The total amount removed was then treated to a regular dolomite analysis.

The results of these analyses, together with the per cent that was dissolved out each time, the total per cent dissolved from the piece, the length of the individual running and the total time run are all given in tables I, II, III and IV.

Tables I and II are especially interesting since in them are summarized the results obtained in running the two samples of the same piece of concrete at different temperatures. From the results obtained it appears that the cement is fully as soluble in the cold water as in the hot. We may, therefore, conclude that results obtained when the water was hot are as fair as though the water had been cold in every case. We purposely chose Sample No. 1 for this comparative test because it seemed to be much the best of any of our samples. That it was the best of any we ran is shown by results obtained from Nos. 2, 3, 4 and 5. All of these latter samples were either partially or wholly disintegrated at the end of the last running, yet, while No. 1 was run in both hot and

cold water much longer than any of these, in neither case had it wholly disintegrated, though it was much reduced in size.

In order to ascertain the exact nature of those quantities given in the foregoing tables under the head "Insoluble," a number of these precipitates were fused in a platinum crucible with Na_2CO_3 . The results of the analyses which were made following the fusion are shown in Table V. These analyses showed a very large per cent as still insoluble. To assure myself fully that this part was SiO_2 , I placed several of these precipitates in platinum crucibles and moistened with sulphuric acid and then hydrofluoric acid was added and the whole volatilized. This process left only a slight discoloration on the platinum. Suspecting that this was largely iron, I tested it with potassium ferro-cyanide and obtained the blue color which this gives with iron. The per cent given as SiO_2 , while it is practically all this, really includes, therefore, a small trace of iron.

TABLE I (NORMAL).

NO. 1: TEMPERATURE OF WATER, 98° C.

	First Running	Second Running	Third Running	Fourth Running	Fifth Running
Weight of piece.....	7.1120	6.3376	5.8701	5.5690	5.1410
Insoluble2428	.2086	.1795	.1857	.1880
Al_2O_3 and Fe_2O_30617	.0427	.1040	.0353	.0224
Ca CO_34655	.2148	.0044	.2014	.1412
Mg CO_30044	.0014	.0132	.0056	.0059
Per cent dissolved	10.89	7.38	5.13	7.69	6.95
Total per cent dissolved..	10.89	17.46	21.69	27.71	32.74
Time run	120 hrs.	120 hrs.	120 hrs.	120 hrs.	120 hrs.
Total time	120 hrs.	240 hrs.	360 hrs.	480 hrs.	600 hrs.

TABLE II (NORMAL).

NO. 1: TEMPERATURE OF WATER, 17° C.

	First Running	Second Running	Third Running	Fourth Running	Fifth Running
Weight of piece.....	6.8130	6.2704	5.7063	5.2784	4.8122
Insoluble3555	.3960	.3040	.3335	.3460
Al_2O_3 and Fe_2O_30406	.0203	.0435	.0260	.0182
Ca CO_31416	.1446	.0787	.1638	.1184
Mg CO_30048	.0032	.0017	.0029	.0034
Per cent dissolved	7.96	8.99	7.50	8.83	10.10
Total per cent dissolved..	7.96	16.24	22.52	29.37	36.50
Time run	120 hrs.	120 hrs.	120 hrs.	120 hrs.	120 hrs.
Total time	120 hrs.	240 hrs.	360 hrs.	480 hrs.	600 hrs.

TABLE III.

TEMPERATURE OF WATER 98°C., IN EACH CASE.

	Sample No. 2			Sample No. 3		
	First Runni'g	Second Runni'g	Third Runni'g	First Runni'g	Second Runni'g	Third Runni'g
Weight of piece.....	9.0395	7.9221	7.2923	10.1065	9.1416	8.2156
Insoluble2600	.2817	.1530	.2628	.3430	.3015
Al ₂ O ₃ and Fe ₂ O ₃0373	.0493	.0941	.0483	.0695	.2138
Ca C O ₃7946	.2934	.0500	.6479	.5110	.0125
Mg C O ₃0165	.0054	.0064	.0049	.0025	.0212
Per cent dissolved	12.36	7.95	4.13	9.54	10.13	6.68
Total per cent dissolved...	12.36	19.33	22.68	9.54	18.70	24.13
Time run	120 hrs.	120 hrs.	120 hrs.	120 hrs.	120 hrs.	120 hrs.
Total time	120 hrs.	240 hrs.	360 hrs.	120 hrs.	240 hrs.	360 hrs.

TABLE IV.

TEMPERATURE OF WATER, 98° C., IN EACH CASE.

	Sample No. 4			Sample No. 5		
	First Runni'g	Second Runni'g	Third Runni'g	First Runni'g	Second Runni'g	Third Runni'g
Weight of piece.....	10.3076	8.4970	7.9158	10.4800	8.6093	-----
Insoluble6104	.2000	.2510	.6004	.1845	-----
Al ₂ O ₃ and Fe ₂ O ₃0328	.0315	.0405	.0292	.0366	-----
Ca C O ₃	1.1121	.3446	.4610	1.1809	.2777	-----
Mg C O ₃0553	.0051	.0059	.0656	.0023	-----
Per cent dissolved	17.56	6.84	9.58	17.90	4.78	-----
Total per cent dissolved...	17.56	23.20	30.56	17.90	22.68	-----
Time run	96 hrs.	96 hrs.	96 hrs.	96 hrs.	96 hrs.	-----
Total time	96 hrs.	192 hrs.	288 hrs.	96 hrs.	192 hrs.	-----

The power of the water at 17°C. was appreciably greater than at 98°C. The reason for this, as assigned by Dr. W. R. Whitney, President of the American Chemical Society, is that the water at 17°C contained a larger amount of C O₂, dissolved out of the air. We stated, two years ago, that all the results entered into mineralogical literature showed a definite power for C O₂ waters and other acid waters of the soil to act upon all minerals containing calcium, to remove the calcium in solution. Observe the following:

(Clarke, Data of Geochemistry, Bulletin 330, U. S. Geological Survey, p. 165.) "Meteoric waters carrying free carbonic acid are probably the most powerful of agents in the solution of rocks, although their

chemical activity is neither violent nor rapid. Being continually replenished from the storehouses of the atmosphere, their work goes on unceasingly over a large portion of our globe. The calcium which they extract from rocks is carried by rivers to the sea and is finally deposited in the form of limestones. * * * * * Alkaline waters, especially thermal waters of the sodium carbonate class, are also active solvents of mineral substances. Their tendency, however, is opposite to that of the acid waters, for they dissolve silica rather than bases and act as precipitants for magnesia and lime."

The portland cement, being calcium silicate and calcium aluminate, is acted upon readily by the carbonate or other acid waters to dissolve out the calcium, and is also acted upon by the alkaline waters, which replace the calcium and combine with the silica. In either case the integrity of the original silicate has been destroyed and it is no longer a bonding material to hold sand and gravel combined in an artificial stone.

Van Hise' Treatise on Metamorphism, U. S. Geological Survey, gives the factors affecting the alteration of rocks. "Porosity is favorable to rapid change; to contain minerals which are soluble is favorable; to contain water or gas is favorable for rapid alteration." The concrete tile and sewer pipe now manufactured are invariably highly porous. Their mineral content is readily acted upon and dissolved by the free access of solvent acids or alkaline waters to all parts of the porous material. The content of water is large, as the bonding material has derived all its bonding power by a rehydration of the ground cement clinker. Van Hise says: "The greatest destruction of materials occur in the belt of weathering. This belt extends from the surface to the level of ground-water, the greatest destruction occurring at ground-water level." Drain tile is always at groundwater level, for they themselves determine this level. Van Hise assigns the greatest relative importance to CO_2 , as it is everywhere at work upon the surface of the earth and is always active and is immeasurably the most important in the belt of weathering. And again: "The one liquid through which the greater part of all alteration of rocks occurs is water solutions." Kahlenberg and Lincoln, Journal of Physical Chemistry, Vol. 2, p. 1898, holds that "When dilute solutions of silicates are made in groundwaters, the silica exists in the form of colloidal silicic acid." Ostwald divides the bases into the strong, moderate and weak. In the belt of weathering the alkalis, sodium and potassium, are dissolved first, next comes calcium, the magnesia, iron and aluminum dissolving last. Van Hise summarizes the importance of these studies in a paragraph:

“The two great acids of nature are carbonic and silicic and the major contest in the rocks, so far as the acids are concerned, is between the weak carbonic and the very weak silicic. * * * * * The fact of the formation of carbonates and the simultaneous decomposition of the silicates under surface conditions the world over is well known.”

The limits of our space do not permit a more extended presentation of our studies. You will be gratified—following the interest which you so generously displayed two years ago and today—you will be gratified to know that the mining engineering and chemistry departments of several of our colleges and state institutions are pursuing most thorough and careful studies of the action of groundwaters, acid and alkaline, and sewage waters upon the body of cement concrete and we urge again your continued interest and co-operation.