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Publisher's version / Version de l'éditeur:

[https://doi.org/10.1016/0008-8846\(72\)90054-3](https://doi.org/10.1016/0008-8846(72)90054-3)

Cement and Concrete Research, 2, July 4, pp. 375-386, 1972-07-01

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ANALYZED

FACTORS AFFECTING THE YOUNG'S MODULUS - POROSITY RELATION OF
HYDRATED PORTLAND CEMENT COMPACTS

by
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Reprinted from

CEMENT AND CONCRETE RESEARCH

An International Journal

Vol. 2, No. 4, July 1972

p. 375

48451

Research Paper No. 519
of the
Division of Building Research

OTTAWA
July 1972

Price 25 cents

NRCC 12584

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ANALYZED

FACTORS AFFECTING YOUNG'S MODULUS -
POROSITY RELATION OF HYDRATED PORTLAND
CEMENT COMPACTS

by

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(Communicated by P. J. Sereda)

ABSTRACT

Two series of compacts are studied, one d-dried before and one after compaction. Measurements of absolute density, helium flow characteristics and Young's modulus indicate that high pressures can force the layers of d-dried material closer together, giving a Young's modulus double that displayed when no interlayer water is present and identical to that when spaces are occupied by water molecules. Water can re-enter between the layers, however, and on redrying Young's modulus is reduced by 50 per cent to the normal value for the d-dried material. The interlayer water must be regarded as part of the solid.

SOMMAIRE

Deux séries de compacts sont étudiées, l'une "d-dried" avant le compactage et l'autre après. Les calculs de densité absolue, les caractéristiques d'écoulement à l'hélium et le Modulus Young indiquent que les hautes pressions peuvent forcer les couches de matériaux "d-dried" ensemble donnant ainsi un Modulus Young double de celui obtenu quand aucune eau est présente dans les entrecouches et identique à celui qui est obtenu quand les espaces sont occupés par des molécules d'eau. L'eau peut ré-entrer entre les couches, cependant et sur reséchage, le Modulus Young est réduit par 50 p. 100, à la valeur normale, pour le matériau "d-dried". L'eau d'entrecouche peut être considérée comme faisant partie du corps solide.

Introduction

Porosity is the major factor governing Young's modulus of elasticity of porous bodies (1, 2), and previous work on hydrated portland cement has shown that this material is no exception (3, 4, 5). Curves of porosity versus Young's modulus for hydrated portland cement specimens prepared by paste hydration or by compaction of bottle hydrated cement approximately coincide (5).

It has also been shown (3) that it is possible to increase the Young's modulus at a given porosity by allowing complete re-entrance of interlayer water. It is recognized (6, 7, 8) that interlayer space has special properties, in contrast with other pores, and a less arbitrary definition of porosity is required if it is to be used in correlation with other properties. Recently it has been demonstrated (9) that porosity of hydrated portland cement can be measured by helium pycnometry and that interlayer space can be excluded by this technique.

This work has involved a study of porosity: Young's modulus relation of compacts using various techniques whereby the interlayer space configuration may be modified; the widely used exponential mathematical expression was applied in this work for correlating the data.

Experimental

Materials

Hydrated cement was prepared by hydrating a Type I cement in a rotating bottle at a water/cement ratio of 5.0. Hydration was continued for up to 28 months and was found to be sensibly complete. When mixing was completed the hydrated cement was filtered, dried at 32% RH over a saturated solution of CaCl_2 and then screened through a 100-mesh sieve. The remaining fraction (mostly $\text{Ca}(\text{OH})_2$ crystals) was ground and remixed with the sieved material.

Compaction

Two series of samples were made in this work:

Series A: Compacts were made from the above powdered materials, which had been dried at 85°C for three hours in a vacuum chamber. This had

previously (7) been found to approximate the d-dried condition. Compacts were fabricated at nine pressures: 285, 570, 1140, 2850, 4270, 5700, 8540, 11,400 and 14,350 kg/cm² in a gloved box conditioned at less than 1% RH. The specimens were discs 32mm in diameter and 1.25mm thick. Details of the compaction procedure have been published elsewhere (3). Twelve compacts were fabricated at each pressure except the two highest, where nine compacts were fabricated. At a later date but using the same material, a similar series was prepared consisting of ten compacts each at 570, 5700 and 14,350 kg/cm².

Series B: A series of compacts was prepared as above, with the hydrated cement dried to only the 32% RH level. After compaction these samples were dried by heating at 85°C until the weight loss indicated that the samples were adequately dried with respect to powdered material. Again at a later date and using the same material, a repeat series of 10 compacts was made at 5700 kg/cm².

Porosity, Density and Helium Flow Measurements

Solid volume measurements were made with a helium comparison pycnometer and are described in detail elsewhere (9). Porosity was calculated from measurement of the external geometry and the solid volume; density was calculated from solid volume and weight.

Helium flow measurements also described elsewhere (8) were made on Series A samples compacted at 520, 5700 and 14,350 kg/cm², and on Series B samples compacted at 5700 kg/cm². All density and helium flow measurements were made at the d-dried condition.

Determination of Young's Modulus of Elasticity

Apparatus was used that is described elsewhere (3). The procedure involves the measuring of the deflection of a disc specimen loaded at its centre and supported at three points located at the circumference of a circle 1 in. in diameter. The average of five tests was considered as Young's modulus for any given set of conditions.

Rewetting and Sequence of Experiments

Series A, d-dried before compaction

After compaction, weight and geometry were measured in a gloved dry-box and the samples then transferred to the helium pycnometer where the solid volume and helium flow were measured. The samples were returned to the dry-box and Young's modulus measurements were taken. At this stage the samples were placed in a desiccator maintained at 100% RH for one day, then immersed in saturated Ca(OH)_2 solution for one month. Subsequently, the samples were conditioned in a desiccator over the appropriate salt solution for 42% RH (3) for three months, when the Young's modulus was measured again. Samples compacted at 5700 and 14,350 kg/cm^2 were again d-dried and Young's modulus was measured for the third time. The rewetting procedure previously followed was repeated and Young's modulus was measured for the fourth and last time after conditioning samples to 42% RH.

The solid volume, density, porosity, helium flow characteristics and Young's modulus of the new Series A made at a later date were measured immediately following fabrication of the samples. No rewetting experiments were performed on these samples.

Series B, d-dried after compaction

Solid volume, density, porosity, helium flow characteristics and Young's modulus were all measured at the d-dry condition. The rewetting procedure was also followed with this series, allowing one month of exposure in saturated Ca(OH)_2 solution. Young's modulus measurements were made after samples had been conditioned to 42% RH.

The samples compacted at 5700 kg/cm^2 three years later were only measured for Young's modulus at the d-dry condition.

Results

Porosity

Porosity versus compaction pressures are plotted on Fig. 1 as a semi-log relation. As observed previously, compaction of material conditioned at the higher humidity yields a lower porosity. Porosities are as low as 7% for Series B and 10.5% for Series A. Values are tabulated on Tables I and II. At low pressures, the porosities for the two systems are

quite similar, but the difference widens at higher pressures. Any interlayer space between the collapsed sheets of silicates is not included in this porosity measurement, a point that will be elaborated later.

Density

Variation of density with compaction pressure is shown on Fig. 2. Series A shows a steady increase from 2.274 at 285 kg/cm^2 to 2.395 gm/cc at 14,350 kg/cm^2 . The new Series A varies from 2.293 to 2.408 gm/cc for 570 to 14,350 kg/cm^2 .

FIG. 1

Compaction Pressure vs Porosity

FIG. 2

Density vs Compaction Pressure

The results for Series B show some scatter at the higher pressures, but there appears to be little increase in density beyond 4200 kg/cm^2 , a value between 2.294 and 2.312 at 14,350 kg/cm^2 . Thus the series in which compaction took place after drying shows a much higher absolute density than the series dried after compaction. The difference is not large at lower pressures.

Helium Flow Measurements

The results for Series A, the new series in the d-dried state, are presented in Fig. 3. Helium flow decreases with pressure of compaction.

MODULUS OF ELASTICITY, HYDRATED CEMENT, COMPACTS

TABLE I
YOUNG'S MODULUS DATA FOR SERIES A

Compaction Pressure kg/cm ²	Porosity %	No. of Samples	E (d-dried state) $\times 10^{-4}$ kg/cm ²			E $\times 10^{-4}$ kg/cm ² (rewetted and returned to 42% RH)		
			$\bar{x}$	σ	$(\sigma/\bar{x}) \times 100$	$\bar{x}$	σ	$(\sigma/\bar{x}) \times 100$
284.8	58.96	12	(1)* 1.72	0.22	12.79	(2) 2.09	0.28	13.33
569.6	53.04	12	(1) 2.61	0.31	11.88	(2) 2.88	0.40	13.89
569.6	55.27	10	(1) 2.05	0.11	5.37			
1139.2	46.18	12	(1) 4.33	0.28	6.46	(2) 4.83	1.05	21.69
			(3) 4.27	1.08	25.29			
2848	34.64	12	(1) 10.12	0.33	3.26	(2) 9.43	1.63	17.29
			(3) 9.44	0.92	9.74			
4272	27.90	12	(1) 14.42	0.78	5.41	(2) 13.54	1.52	11.23
5696	23.14	12	(1) 18.50	1.17	6.32	(2) 17.49	0.90	5.15
			(3) 14.81	1.04	7.02	(4) 17.76	0.73	4.11
5696	26.59	10	(1) 17.26	0.49	2.84			
8544	17.20	12	(1) 24.48	0.99	4.04	(2) 25.35	0.99	3.91
11392	13.01	9	(1) 29.14	1.11	3.81	(2) 30.73	0.91	2.96
14346	10.39	9	(1) 34.00	1.34	3.94	(2) 34.76	1.65	4.75
			(3) 17.68	0.86	4.86	(4) 35.10	1.27	3.62
14346	12.68	10	32.63	1.81	5.55			

* Numbers represent the order in which determinations were made.

TABLE II
YOUNG'S MODULUS DATA FOR SERIES B

Compaction Pressure kg/cm ²	Porosity %	No. of Samples	E $\times 10^{-4}$ kg/cm ² (d-dried state)			E $\times 10^{-4}$ kg/cm ² (rewetted and returned to 42% RH)		
			$\bar{x}$	σ	$(\sigma/\bar{x}) \times 100$	$\bar{x}$	σ	$(\sigma/\bar{x}) \times 100$
284.8	58.94	12	(1)* 1.26	0.20	15.87	(2) 3.10	0.28	9.03
569.6	51.92	12	(1) 3.33	0.25	7.51	(2) 5.78	0.47	8.13
1139.2	43.90	12	(1) 6.36	0.38	5.97	(2) 8.61	0.56	6.50
2848	30.70	12	(1) 13.30	0.64	4.81	(2) 16.04	0.59	3.68
4272	24.22	12	(1) 17.55	0.87	4.96	(2) 20.73	1.05	5.07
5696	19.70	12	(1) 19.43	0.57	2.93	(2) 25.69	1.30	5.06
5696	21.80	10	(1) 20.15	1.27	6.30			
8544	12.12	12	(1) 17.65	0.69	3.90	(2) 33.60	1.34	3.99
11392	8.92	9	(1) 19.82	0.83	4.19	(2) 37.29	1.24	3.33
14346	7.12	9	(1) 19.92	0.71	3.54	(2) 39.35	1.22	3.10

* Numbers represent the order in which determinations were made.

After about 15 hours the flow rate for the samples compacted at 5700 and 14,350 kg/cm² is of the same order as the leak rate of the apparatus.

Fig. 4 shows a direct comparison between the helium flow rate of two samples compacted at 5700 kg/cm², one from Series A and one from Series B; densities are 2.374 and 2.327 gm/cc, respectively. The flow rate for Series B exceeds by far that for Series A.

FIG. 3
Helium Inflow for Series A

FIG. 4
Helium Inflow-Comparison
Between Series A and B

FIG. 5
Young's Modulus vs Porosity
for Series A

Young's Modulus

Series A

For the d-dried compacted cement specimens the Young's modulus-

porosity relation is represented by the upper curve on Fig. 5, a semi-log plot. It is linear from 10 to 35% porosity and is similar to the curves reported by Soroka and Sereda (5). The curve extrapolates to 59×10^4 kg/cm² at zero porosity; this is again similar to the value taken from the results of Soroka and Sereda at about 10% porosity. (It has been shown that porosity measurement on a d-dried sample, if done by water, includes inter-layer space as porosity. Helium, on the other hand, can exclude these spaces if the appropriate procedures are used (9).)

Each point on Fig. 5 includes $\bar{x} \pm 2 \sigma$ levels (approximately 95% confidence limits) and $\bar{x}$ (the arithmetic mean) is usually the average of twelve samples. The number of samples tested, the mean, the standard deviation, and the coefficient of variation are included in Table I. Up to 46% porosity the coefficient of variation does not exceed 6.5%, but it is about 12% for the last two points, at 55 and 59% porosity, respectively. The points for the samples that were rewetted and dried back to 42% RH fall almost exactly on the first curve but the coefficient of variation is about 12% and higher for 28% porosity and higher.

Four sets of compacts were d-dried, giving a major reduction of Young's modulus at lower porosities. At 10.39%, reduction was from 34.76×10^4 to 17.68×10^4 kg/cm², and at 23.14%, from 17.5 to 14.8×10^4 kg/cm². The spread demonstrates a clear separation of curves. The other two points, at approximately 35 and 46% porosity, do not show significant deviation from the previous curve, but that at 46 per cent porosity shows a very large coefficient of variation. The samples at 10.39 and 23.14% porosity were rewetted and dried back to 42% RH. The Young's modulus returned to coincide with the points on the upper curve, with a low coefficient of variation. For example, the Young's modulus of the sample at 10.39% porosity changed from 17.68×10^4 kg/cm² in the d-dry condition to 35.10×10^4 kg/cm². The initial d-dry condition was 34.0×10^4 kg/cm² and the initial rewetted condition 34.76×10^4 kg/cm².

The three new series of samples, at approximately 55, 27 and 12.7% porosity, fit closely the upper curve for the first d-dry condition. The coefficient of variation of the modulus of these samples was relatively low,

showing clearly that these characteristics are quite reproducible.

Series B

The Young's modulus-porosity relation for Series B is presented on Fig. 6 and Table II. The plot is again a semi-log and the coefficients of variation are, in general, less than 5% below 55% porosity. The first d-dried condition is represented by the lower curve, which is similar to the curve for the second d-dried condition of Series A. Again, there is little increase in Young's modulus from 25 to 7% porosity. A new series represented by one point at about 22% porosity illustrates further the reproducibility of the curves. After rewetting and drying to 42%, Young's modulus increases over the whole porosity range well beyond the 95% confidence limit over most of the range. The difference in the two curves is large at both ends of the porosity scale but with a lower coefficient of variation at the low porosity. The curve for the rewetted samples is linear up to about 40% porosity and when extrapolated to zero gives a value of $55 \times 10^4 \text{ kg/cm}^2$ for Young's modulus. This is similar to the value of $59 \times 10^4 \text{ kg/cm}^2$ for the first d-dried and rewetted curves of Series A, and indeed the whole curve is similar. The two curves are plotted together on Fig. 7.

FIG. 6

Young's Modulus vs Porosity
for Series B

FIG. 7

Comparison of Young's Modulus -
Porosity Curves for Series A and B

Discussion

These results show very clearly that the inclusion of interlayer water molecules in the structure of the silicates increases the Young's modulus, in some cases by over 100% (see Fig. 6). This has been shown before (3), but the coefficient of variation in the present work is much lower than was previously observed. In addition, by compacting the material after it has been d-dried it is possible to attain the same high values for Young's modulus as were obtained with interlayer water present. This occurs at the higher pressures of compaction. Fig. 7 shows these curves on the same diagram. The curve for Series A follows the curve for Series B without interlayer water present at low pressures as well as the curve for Series B with interlayer water present at high pressures. The transition takes place beyond 4200 kg/cm^2 . The density values on Fig. 2 show that there is no further increase in density for Series B after 5600 kg/cm^2 , and that the values for Series A continue to increase. Helium flow results show that as the pressure of compaction is increased it is more difficult for helium to penetrate the spaces. It seems clear that when compaction occurs at high pressures with interlayer water removed the layers are forced closer together than they are when interlayer water has not been removed. The high Young's modulus indicates that either some new solid bonds have been established or the surfaces have been forced so close together that solid-to-solid attractive forces contribute to the higher modulus.

That interlayer water behaves as a solid bridge between the layers is now apparent, and it is clear that this space cannot be included as porosity, especially since the interlayer water is only removed at very low humidities. The equivalent role of the water is re-emphasized when Series A is rewetted and Young's modulus remains constant, but when the samples are dried again the moduli drop to values similar to those for Series B, which has also been dried. This means that, on rewetting, water can re-enter the interlayer positions in spite of the fact that the layers are closer together than normal; the water seems to compensate exactly for any decrease in Young's modulus when the layers move apart. If interlayer

water is again removed, the layers revert to a position similar to that in Series B where drying took place after compaction. This latter position appears to be a more stable state. A second rewetting of Series A again returned the Young's modulus to the original high value. There are thus three configurations for the layers:

- 1) Layers separated, with H_2O molecules occupying the interlayer positions. These molecules play a reinforcing role, giving a high value for Young's modulus.
- 2) Water removed from between the layers, resulting only in partial collapse. There is a reduction in Young's modulus of over 50% in some cases.
- 3) Layers pressed closer together than in configuration (2), resulting in a Young's modulus identical to that of configuration (1).

Increase of Young's modulus owing to compaction of d-dried cement appears to take place in such a manner that up to pressures of 4200 kg/cm^2 or 5600 kg/cm^2 the main mechanism is a reduction of porosity because particles are pushed together. Beyond 5600 kg/cm^2 Young's modulus increases very little by this process (see Fig. 5, lower curve.) If the interlayer positions are vacant, compaction of the interlayer system occurs at high pressures and Young's modulus continues to increase. This is confirmed by density and helium flow measurements.

Conclusions

The role of interlayer H_2O molecules in increasing Young's modulus is confirmed. If d-dried hydrated cement is compacted, Young's modulus values may be obtained identical to those obtained when interlayer water is present.

Three configurations exist for the layers:

- 1) H_2O molecules between layers;
- 2) layers partially collapsed and all interlayer water removed;
- 3) layers forced closer to each other by compaction when interlayer water is not present.

Interlayer water acts, then, as in configuration (3) and must be regarded as part of the solid structure of hydrated portland cement.

Acknowledgements

The author wishes to acknowledge the valuable assistance of S. Dods in performing experiments and gathering data.

This paper is a contribution from the Division of Building Research, National Research Council of Canada, and is published with the approval of the Director of the Division.

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