Available online at www.sciencedirect.com

SCIENCE DIRECT®

Cement and Concrete Research 35 (2005) 1855–1858

Cement and Concrete Research

Analytic methods and theory of quantitative stereology for the determination of concrete proportioning in structural components

Xiong Zhang[a,*], Jihong Han[b]

[a]Building Material Institute, Tongji University, 1239 Siping Road, Shanghai 200092, China
[b]Shanghai Research Institute of Building Sciences, 75 Wanping South road, Shanghai 200032, China

Received 23 January 2004; accepted 10 October 2004

Abstract

The analytic methods and the theory of the quantitative stereology combining with micro-analysis for concrete proportion on structural components were systemically described in this paper, specifically, the effective mix proportion of structural concrete was identified through three levels macroscopic (coarse aggregate), mesoscopic (fine aggregate) and microscopic (cement paste). Moreover, the porosity and hydration degree of structural concrete could be understood based on the compositional analysis of aggregate and cement stone. The results of the experimental analysis indicated that the mix proportion of concrete was similar to the effective mix proportion, the systemic error of the method could be amended, and the relative errors among configurations of materials were less than 4%, which validated the accuracy and the applicability of the methods.

Keywords: Quantitative stereology; Mixture proportioning; Concrete

1. Introduction

In recent years, the building industry has been developing rapidly, but problems due to the low quality of construction are taking place frequently, amongst which there are quite a number related to the concrete quality. This may be as a result of abnormal setting time, lower strength than designed, more structural cracks of concrete, etc., all of which are nearly related to the constituent materials and mix proportioning of concrete. Losing control of the dosage of gel-forming materials, water and aggregate would certainly result in the low strength of concrete and therefore problems due to low quality.

Once quality problems have occurred, it is very important to find out their causes, by analysing what differences there are between the actual mix proportion of the structural concrete and the designed mix proportion.

Many efforts have been made by experts to find the effective methods for quantitatively identifying the mix proportion of structural concrete, but still only qualitative analysis for separating coarse aggregate is available: this is far from what is required. In this paper, new analytical methods and the theory of the quantitative stereology combined with micro-analysis for concrete proportion on structural components were systemically described. By this means the effective mix proportion of structural concrete could be quantified based on three levels: macroscopic (coarse aggregate), mesoscopic (fine aggregate) and microscopic (cement paste). Moreover, the porosity and degree of hydration of structural concrete could be understood based on the compositional analysis

* Corresponding author.
E-mail address: tclsys@mail.tongji.edu.cn (X. Zhang).

0008-8846/$ - see front matter © 2005 Published by Elsevier Ltd.
doi:10.1016/j.cemconres.2004.10.039

of aggregate and cement paste. All of these measurements could be the basis, and provide assistance, for construction problem analysis.

2. Experimental method

2.1. Sample preparation

The following raw materials were used:

- Cement. 42.5 Ordinary Portland cement.
- Gravels. Three types of gravels identified as A, B and C were used in this study. A and B are detritus with dark and light color. For details see Table 1.
- Sands. Apparent density is 2.67×10^3 kg/m^3. Water ratio is 0.6% (this part is included in the W_0). Mud ratio is 0.3% (a Chinese standard, show the quality of sands). Modulus of fineness is 2.43.

All mix proportions of concrete samples in this experiment are shown in Table 2.

The preparation for analytic samples with quantitative stereology method was as follows:

- Coarse aggregate samples: for each group, three $10\times10\times40$ cm^3 samples were cut into sections and used for image analysis of coarse aggregate. According to the symbolic statistical analysis, 5 sections are chosen in each sample and therefore 15 sections are chosen in each group so as to represent the samples adequately [3].
- Fine aggregate samples: samples were made from the sections used for image analysis of coarse aggregate. In this study, the sections of concrete are chosen from Group E (mix proportion is $C_0/S_0/G_0/W_0$=1:1.90:3.52:0.52). Sample making occurs in three steps: firstly break up the concrete sections, then make sulfur slides using the pieces only with sands and cement stone. Finally use the slides for image analysis of fine aggregate after rough, gentle and fine grinding and polishing. Four slides were used in each section of this experiment, 9 sections per group, and 36 slides in all.

Samples from structural concrete were obtained as concrete cores (100 mm diameter and 100 mm length) were drilled from structural components on site. More than 8 cores were used for mix proportion analysis are and each core was sliced into more than 8 sections samples, according to the symbolic statistical means [3].

Table 1
Three types of gravels in this experiment

Type	Apparent density ($\times10^3$ kg/m^3)	Water ratio (%)	Mud ratio (%)
A	2.72	0.1	0.2
B	2.67	0.2	0.8
C	2.72	0.2	0.3

Table 2
All mix proportions of concrete samples in this experiment

No.	Strength grade	Particle size of gravel (mm)	W/C	Sand ratio (%)	Mix proportion $C_0/S_0/G_0/W_0$
A	C25	5–40 (A)	0.58	35	1:2.16:4.00:0.58
B	C25	5–40 (B)	0.54	31	1:1.16:3.83:0.54
C	C35	5–40 (A)	0.48	32	1:1.57:3.35:0.48
D	C35	5–40 (B)	0.44	27	1:1.18:3.19:0.44
E	C30	5–40 (A)	0.52	35	1:1.90:3.52:0.52
F	C30	5–40 (A)	0.48	29	1:1.41:3.45:0.48
H	C30	5–40 (A)	0.48	30	1:1.30:3.03:0.48
I	C30	5–31.5 (C)	0.40	30	1:1.17:2.74:0.40
J	C30	16–31.5 (C)	0.46	30	1:1.14:3.26:0.46
K	C30	5–16 (C)	0.48	38	1:1.67:2.73:0.48
L	C30	16–31.5 (C)	0.52	41	1:1.41:3.45:0.52

2.2. Measurements

2.2.1. Analysis of quantitative stereology for coarse and fine aggregates

With the development of image analysis instruments, quantitative stereology works as a good method for estimating 3D structures, quantifying them through the measurement and calculation of diagnostic parameters of 2D structures [1]. A LEICA QUANTIMET 600 was used as image analysis instrument in this study [2].

(1) Image acquisition

Coarse aggregate: To clean the sections of concrete samples, we wet equally the surface with wet dishcloth, and then these sections were captured with a video camera to acquire grey images. According to the symbolic statistical means, the numbers of samples should be 8 [3].

Fine aggregate: An optical microscope with the magnification of 50× was used to analyse the volume content of fine aggregates in concrete. In order to assure analysis reliability and gain enough data points, at least 4 parts of mortar in one concrete sample slice must be selected and 10 contiguous images in each part of mortar must be captured.

(2) Parameters measurement:

The following parameters were measured automatically in the binary images: the numbers and areas of gravels and sand and their respective particle size distribution.

According to the fundamentals of quantitative stereology, the particle volume of gravels and sand in concrete can be both gained by the statistical analysis of these parameters [4,5].

2.2.2. Apparent density of concrete

The apparent density of concrete can be calculated on the basis of density definition formulas after measuring the size of concrete sample, calculating its volume, and weighting it.

2.2.3. Chemically bound water of concrete (W_b)

Measuring the bound water weights of paste by drying the paste sample out of concrete at the temperature of 60 °C and firing it at the temperature of 900 °C till constant weight to measure the ignition loss, and then according to the weight ratio of paste in concrete, the bound water weight of concrete can be calculated.

3. Method and theory for determining the effective mix proportion of structural concrete

The design formula of the mix proportion based on the volume method of concrete is shown as follows [6]:

$$C/\rho_c + G/\rho_{ag} + S/\rho_{as} + W/\rho_w + 10\alpha = 1000L \qquad (1)$$

where C/ρ_c is the volume of cement, $G/\rho_{ag}=V_{ag}$ is the volume of gravel, $S/\rho_{as}=V_{as}$ is the volume of sand, W/ρ_w is the volume of water, 10α is the volume of air, which is close to 10 without air-entrained admixture.

The effective mix proportion of structural concrete can be calculated according to the following method and theory by using the above analytic data.

3.1. Concrete aggregate content

From the volume percent of concrete gravels V_{ag} and the volume percent of sands V_{as} determined by the quantitative stereology method, the concrete gravel content G (kg/m^3) and sand content S (kg/m^3) as well as sand ratio can be calculated.

Table 3
The original volume content of gravels for all concrete samples (V_{Vog})

No.	Weight ratio of gravels (%)	Apparent density of gravels ($\times 10^3$ kg/m^3)	Apparent density of concrete ($\times 10^3$ kg/m^3)	V_{Vog} (%)
A	51.71	2.72	2.37	45.1
B	54.21	2.67	2.36	47.9
C	52.29	2.72	2.39	45.9
D	54.90	2.67	2.40	49.3
E	50.75	2.72	2.38	44.4
F	54.44	2.72	2.38	47.6
H	52.16	2.72	2.37	45.4
I	51.55	2.72	2.41	45.7
J	55.58	2.72	2.39	48.8
K	46.40	2.72	2.43	41.5
L	54.06	2.72	2.39	47.5

Table 4
The amended volume content of gravels (V_{ag})

No.	A_{ag}	λ_g	V_{Vog}	V_{ag}
A	5070.21	1.158	45.1	52.2
B	4598.74	1.051	47.9	50.3
C	5168.57	1.181	45.9	54.2
D	4978.71	1.137	49.3	56.1
E	4952.83	1.132	44.4	50.3
F	5116.74	1.169	47.6	55.6
H	4545.74	1.039	45.4	47.2
I	5006.02	1.144	45.7	52.3
J	4889.23	1.117	48.8	54.5
K	4239.21	0.969	41.5	40.2
L	5436.36	1.242	47.5	59.0

3.2. Cement content

The cement content can be calculated as follows:

$$C = D - G - S - W_b \qquad (2)$$

where C (kg/m^3) is cement content, D (kg/m^3) is density of concrete, G (kg/m^3) is the coarse aggregate content, S is fine aggregate content, and W_b (kg/m^3) is bound water.

3.3. Added water in concrete (W)

Added water can be calculated according to a rearranged Eq. (1), as follows:

$$W = \left(1000L - C/\rho_c - G/\rho_{ag} - S/\rho_{as} - 10\alpha\right)\cdot\rho_w \qquad (4)$$

4. Validation for the analytic methods of quantitative stereology

4.1. Quantitative stereology error adjustment

4.1.1. Coarse aggregate adjustment

The original volume content of gravels can be calculated according to the following formula:

$$\frac{G_0}{G_0 + S_0 + C_0 + W_0} = \frac{V_g\rho_g}{V\cdot\rho} = V_{Vog}\cdot\frac{\rho_g}{\rho} \qquad (4)$$

where G, V and ρ represent the weight, volume and the apparent density of concrete, respectively, and V_{Vog} represents the original volume content of gravels; the left part of the formula is namely the weight ratio of gravels.

Table 5
The volume content of sands V_{Vos} and the average value

No.	1	2	3	4	5	6	7	8	Average value
V_{Vos}	0.32	0.28	0.31	0.25	0.31	0.30	0.26	0.37	0.30

Table 6
The comparison between the calculated mix proportion and the effective one of concrete sample

The mix proportion	Cement (kg/m^3)	Water (kg/m^3)	Gravel (kg/m^3)	Sand (kg/m^3)
The calculated one	441	194	1257	513
The effective one	431	200	1223	531
Relative error (%)	2.3	3.0	2.8	3.4

The calculated results of V_{Vog} for all groups in this experiment are shown in Table 3.

Errors between V_{Vog} and V_{ag} come from two parts:

(1) The systemic errors of the image analysis instrument, which result in a systemic error for area data of graphics. Research indicates that they are relatively steady and can be corrected as follows [3]:

$$\lambda_{\mathrm{g}} = A_{\mathrm{ag}}/4377 \tag{5}$$

$$V_{\mathrm{ag}} = V_{\mathrm{Vog}} \times \lambda_{\mathrm{g}} \tag{6}$$

Hence, A_{ag} is the area percent of gravel phase in concrete image, λ_{g} is the amended error. This shows that increased volume content of gravels results in higher amended errors λ_{g}.

So according to formula (6), the amended volume content of gravels V_{ag} can be calculated and are shown in Table 4.

(2) The operating errors, which are caused by image compilation from widening of the lines or underestimation of gravel particles with small area, which make the calculated area appear smaller than original one. These errors should be taken into account by statistical means.

4.1.2. Fine aggregate adjustment

The original volume content of sands V_{Vos} can be calculated according to the similarly formula:

$$\frac{S_0}{G_0 + S_0 + C_0 + W_0} = \frac{V_{\mathrm{S}}}{V} \cdot \frac{\rho_{\mathrm{S}}}{\rho} = V_{\mathrm{Vos}} \cdot \frac{\rho_{\mathrm{S}}}{\rho} \tag{7}$$

Because of the systemic errors and the operating errors, there should be a modulus λ_{s} in the following formula:

$$V_{\mathrm{as}} = V_{\mathrm{Vos}} \times \lambda_{\mathrm{s}} \tag{8}$$

According to the data of the volume content of sands V_{Vos} and their average value for 8 concrete groups shown in Table 5, the modulus λ_{s} can be obtained by statistical methods.

4.2. Validation of quantitative stereology analysis

The comparison between the actual mix proportion and the calculated mix proportion of concrete by quantitative stereology are shown in Table 6. The results indicate that the calculated mix proportion of concrete is similar to the actual mix proportion, and the relative errors of all items are less than 4%, which shows that the analytic method of quantitative stereology for the mix proportion of concrete is comparatively accurate.

5. Conclusions

1. The actual mix proportion of structural concrete can be identified in three levels of macroscopic (coarse aggregate), mesoscopic (fine aggregate) and microscopic (cement paste) using the analytic methods and theory of the quantitative stereology. Moreover, the porosity and hydration degree of structural concrete could be understood based on the compositional analysis of aggregate and cement stone.
2. The results of the experimental analysis indicated that the mix proportion of concrete was similar to the actual mix proportion, the systemic error of the method could be corrected, and the relative errors among configurations of materials were less than 4%.

Acknowledgments

The authors would like to express their appreciation for the financial support of this project provided by the State Fund of Natural Sciences, China (No. 50078041).

References

[1] G.E. Pellissier, S.M. Purdy, Translation by Sun Huilin and Ma Jiyu, Stereology and quantitative auric phase, Publishing company of mechanical industry, Beijing, 1980.
[2] LEICA Q600 User Manual-Appendix A: 15–20.
[3] Han Jihong, PhD dissertation of Tongji university, 2001.
[4] C.F. Mora, A.K.H. Kwan, Sphericity, shape factor and convexity measurement of course aggregate for concrete using digital image processing, Cem. Concr. Res. 30 (3) (2000) 351–358.
[5] H. Gudmundsson, S. Chatterji, A. Damgaard Jensen, The measurement of paste content in hardened concrete using automatic image analyzing technique, Cem. Concr. Res. 9 (5) (1979) 607–612.
[6] Wu Keru, Zhang Xiong, Building Materials, Publishing company of Tongji university, 1998.