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# Application of petrography for determining the quality of concrete cured in tropical environment

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**Abstract**

This paper presents the results of a study on the application of petrographic and chemical techniques for determining the quality of concrete cured in tropical environment. Concretes with water-to-cement ratios (w/c) of 0.30 to 0.70 with an increment of 0.10 were made and cured at 35 and 20 °C, and exposed to different durations of moist curing. The results suggest that most of the petrographic and chemical methods can be used for estimation of w/c and cement content for hardened concrete cured in tropical environment with acceptable accuracy.


*Keywords:* Concrete; Curing; Hydration; Petrography; Temperature; w/c ratio

## 1. Introduction

Petrography has been used as a tool for research and forensic investigation of concrete in Europe and North America for many years [1–6]. As the weather conditions in Southeast Asian countries are quite different from those in Europe and North America, the objectives of this research were (1) to investigate the effect of curing on the degree of cement hydration and capillary porosity of concrete using local materials, (2) to determine if the petrographic and chemical methods available can be used to estimate the water-to-cement ratio (w/c) and cement content of hardened concrete cured in tropical environment, and (3) to compare the accuracy of the various methods.

This paper presents the results on the determination of water-to-cement ratio (w/c) and cement content of hardened concrete by petrographic and chemical methods for concrete used in tropical environment. Curing effects on the compressive strength, degree of cement hydration and capillary porosity were investigated and reported elsewhere [8].

For the determination of w/c of concrete, three semi-quantitative petrographic methods were developed some years ago [1–3,5–7]. In the first method, the w/c was estimated based on the capillary porosity of concrete determined by the fluorescence microscopy and the degree of cement hydration. In the second method, the w/c was estimated based on the quantity of unhydrated cement clinker particles determined by point counting on thin section samples. In the third method, the w/c was estimated based on the density and volume of the cement paste, aggregates and concrete determined by point counting on polished sections.

Because the capillary porosity, the degree of cement hydration, and the quantity of unhydrated cement clinker are affected by many factors, such as raw materials, mix proportion of concrete, and curing conditions, accurately determining the w/c becomes a challenging task. In the first two methods mentioned above, it is required to compare the unknown thin section of concrete to be examined with the standard thin sections that have a

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similar degree of cement hydration as the unknown concrete sample. It is therefore important to study the effect of curing conditions on the degree of cement hydration, and make sure that the standard thin sections prepared in the laboratory and cured at standard conditions can be used for the estimation of the w/c of concrete cured at job sites.

For the determination of cement content, two petrographic methods were developed [3,4]. In the first method, the cement content in concrete was calculated from the estimated volume of cement paste from the point-counting results and the estimated w/c. In the second method, the cement content of concrete was calculated from the estimated volume of aggregates from the point-counting results, the measured density of dry concrete, and the estimated degree of cement hydration.

A previous study [8] showed that the degree of cement hydration increased with time and temperature, but the increase beyond 28 days was not significant. The concrete cured at 20 °C water for 28 days had a higher degree of cement hydration and a lower capillary porosity than the concrete cured in water of the same temperature for 7 days followed by exposure to outdoor air for 21 days, but had an opposite trend compared to the concrete cured in 35 °C water for 7 days followed by exposure to outdoor air. However, the differences on the degree of cement hydration and capillary porosity for the concrete cured in these different conditions were not significant. This suggests that the reference thin sections of concrete cured at 20 °C in moist condition for 28 days may be used to estimate the w/c of concrete cured in tropical environment for at least 28 days including 7 days moist curing without the need to determine the degree of cement hydration of concrete under investigation.

## 2. Experimental

### 2.1. Materials

Ordinary Portland cement (ASTM Type I), natural sand, and crushed granite with maximum size of 20 mm were used for all concrete mixes. Chemical admixtures were used for workability purpose, and the information is given together with the concrete mix proportions (Table 1).

### 2.2. Concrete mix proportions and curing conditions

Concrete with w/c ratios of 0.30 to 0.70 with an increment of 0.10 was prepared, and cured at 35 and 20 °C, respectively. Cubes of 100 mm and prisms of 100× 100×500 mm were cast from each concrete mix. The mix proportions and properties of the fresh concretes are presented in Table 1. After 24 h curing in moulds covered with plastic sheet, the specimens were demoulded and cured under the following conditions:

Series 1: 20 °C, 6 additional days in water followed by 21 days in air outside the laboratory (sheltered),
Series 2: 20 °C, 27 additional days in water, and
Series 3: 35 °C, 6 additional days in water followed by 21 days in air outside the laboratory (sheltered).

It should be noted that the outdoor temperatures were not exactly 20 or 35 °C (higher than 20 °C but lower than 35 °C).

### 2.3. Experimental methods

Compressive strength, degrees of cement hydration, and capillary porosity of concrete, and pore structure of the

Table 1
Mixture proportion, properties of fresh concrete, and compressive strength

| Mix ID | Design w/c | Temp. of initial water curing (°C) | Materials ($kg/m^3$) | | | | | | Slump (mm) | Air content (%) | Compressive strength, 28 days (MPa) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Cement | Fine aggregate | Coarse aggregate | Water | Admixture 1 Dosage ($kg/m^3$) | Admixture 2 Dosage ($kg/m^3$) | | | |
| 1 | 0.70 | 20 | 275 | 911 | 911 | 192 | 2.72[a] | – | 150 | 5.0 | 25.8 |
| | | 35 | | | | | 2.72[a] | – | 120 | 3.9 | 31.4 |
| 3 | 0.60 | 20 | 325 | 823 | 967 | 195 | 1.50[b] | – | 150 | 1.3 | 34.6 |
| | | 35 | | | | | 1.95[b] | – | 120 | 2.7 | 43.6 |
| 5 | 0.50 | 20 | 375 | 747 | 1031 | 187 | 1.35[b] | – | 145 | 2.2 | 50.2 |
| | | 35 | | | | | 2.70[b] | – | 110 | 1.8 | 58.0 |
| 7 | 0.40 | 20 | 425 | 678 | 1107 | 170 | 4.48[b] | – | 100 | 3.2 | 63.1 |
| | | 35 | | | | | 5.10[b] | – | 110 | 1.7 | 67.5 |
| 9 | 0.30 | 20 | 475 | 613 | 1190 | 142 | 6.28[c] | 1.05[d] | 150 | 2.9 | 89.8 |
| | | 35 | | | | | 6.98[c] | 1.11[d] | 110 | 1.7 | 95.1 |

[a] A lignosulfonate-based water-reducing admixture.
[b] A sodium naphthalene sulfonates/lignosulfonate-based retarding high-range water-reducing admixture.
[c] A polycarboxylate-based superplasticizer.
[d] A hydroxycarboxylic-acid-salts-based retarder.

mortar in concrete were determined at various ages and the results were presented in Ref. [8]. Fluorescent thin sections and polished sections of the concrete cured under different regimes and to different ages were prepared to determine the w/c and cement content of the concretes.

*2.3.1. Determination of water-to-cement ratio of concrete*

*2.3.1.1. Method by optical fluorescence microscopy.* In this method, thin sections of the concrete with the w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured at all the three different conditions, were prepared and evaluated according to Nordtest Method, NT Build 361 [9] except for green tone (GT) intensity. The green tone (GT) intensity of the thin sections was determined by using a JVC 3-CCD Colour Video Camera and image analysis software instead of visual judgement. The green tone values were determined randomly at 12 different paste areas through the thin section and the mean GT value and standard deviation were calculated. A relationship between the mean GT value and the designed w/c of the concrete was established for the concrete cured in water at 20 °C for 28 days by regression analysis. The w/c for all the thin sections of concrete cured under the different conditions was then estimated based on this relationship.

*2.3.1.2. Method by unhydrated cement clinker.* In this method, the w/c of concrete was estimated by counting the number of residual unhydrated clinker particles per millimeter traverse of paste on thin sections [3]. It is believed that at a given curing temperature, the amount of residual clinker particles is inversely proportional to the w/c. The higher the w/c, the lower the amount of residual clinker particles. By establishing a reference curve between the amount of residual clinker particles and the w/c in standard curing condition, the unknown w/c of concrete can be estimated based on this relationship. In this study, the thin sections of the concrete with w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured at 20 °C in water for 7 days followed by air curing outside the laboratory for 21 days, were selected for analysis. The counting of the number of unhydrated clinker particles in the paste was performed under an optical microscope with a magnification of 500× and a semiautomatic counting machine. The counting of 100-mm traverse of the paste was done for each thin section.

*2.3.1.3. Method by cement paste density.* According to the mix proportions, relationship between the cement paste density and w/c can be established. In this method, the w/c of concrete was determined based on the estimated density of cement paste which was obtained from the density of concrete and aggregate as well as the volume of cement paste, aggregate, and concrete [3]. The density of the concrete was measured after the concrete was vacuum saturated in water. The density of the aggregate components was determined by direct measurement. The volume of the cement paste and aggregate was determined by point counting on the polished sections, and the density of the cement paste was then calculated and estimated. The volume percentage of the aggregate can also be determined by subtracting the volume of cement paste and air from that of concrete. Based on the relationship between the cement paste density and w/c, the w/c of unknown concrete can be determined with the estimated cement paste density. In this study, the concretes with w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured at all the three conditions, were selected for analysis.

*2.3.2. Determination of cement content in concrete*

As cement content in concrete is another important factor that governs the property of concrete in addition to the w/c, various petrographic methods that could be used to determine the cement content were also studied and compared.

*2.3.2.1. Method by ASTM C 1084: 1997.* The cement content was determined in accordance with the maleic acid extraction procedure of the ASTM C 1084 [10] by subtracting insoluble residual and chemically combined water from the total mass of the concrete sample. For this test, concrete sample was dried at 105±1 °C until constant weight. The sample was then crushed and ground until all the material passes through a 1.18-mm sieve. To determine the chemically combined water, 10 g of the sample was ignited in a furnace at 520±5 °C for 3 h. The percentage of the chemically combined water was then calculated. To determine the insoluble residue, 20 g of the ground sample was digested by maleic acid solution. The solution was then filtered and the residue was dried in an oven. The percentage of the insoluble residue was calculated. The concrete with the w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured in water at 20 °C for 28 days, were selected for this part of study.

*2.3.2.2. Method by paste volume and water-to-cement ratio.* In this method, the cement content was determined from the volume of cement paste in concrete by point counting [5]. This is based on the assumption that the volume of the hardened cement paste is equal to the original volume of the unhydrated cement and water. If the w/c is known, the cement paste content can be used to determine the cement content in concrete.

$$M_{\mathrm{cem}} = V_{\mathrm{p}}/(\mathrm{w/c} + 1/\rho_{\mathrm{cem}}) \quad (1)$$

Where $M_{\mathrm{cem}}$—cement content in kg/m$^3$, $V_{\mathrm{p}}$—volume of cement paste, and $\rho_{\mathrm{cem}}$—density of the cement.

The concretes with w/c ratios of 0.30, 0.40, 0.50, 0.60 and 0.70, cured under all the three conditions, were selected for this part of the analysis.

*2.3.2.3. Method by concrete density, aggregate volume, and degree of cement hydration.* In this method, the cement content of concrete was determined based on the density of the dry concrete sample in combination with the total aggregate content obtained from point counting [4]. The cement content can be calculated from the dry concrete density, the total aggregate content, and degree of cement hydration based on the following equation:

$$M_{\mathrm{cem}} = \left(\rho_{\mathrm{c,\,dry}} - M_{\mathrm{agg}}\right)/(1 + 0.24\alpha) \tag{2}$$

Where $M_{\mathrm{cem}}$—cement content, kg/m$^3$, $\rho_{\mathrm{c,\,dry}}$—dry concrete density, kg/m$^3$, $M_{\mathrm{agg}}$—total aggregates contents by mass, kg/m$^3$, and $\alpha$—degree of hydration which can be chosen on the basis of experience and data from the literature [11,12].

In this study, the concretes with w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured under all the three conditions, were selected for the analysis.

## 3. Results and discussion

### *3.1. Determination of water-to-cement ratio*

#### *3.1.1. Determination of water-to-cement ratio by optical fluorescence microscopy*

The mean green tone (GT) value and standard deviation of all the thin sections are given in Table 2. The results showed a good linear relationship between the mean GT values and w/c for concrete cured at a standard condition of 20 °C for 28 days, and the following equation was obtained after regression analysis with a correlation coefficient of 0.99.

$$\mathrm{w/c} = 0.005\mathrm{GT} + 0.102 \tag{3}$$

The w/c ratios of the concrete cured under the other two conditions are estimated using this equation and are also given in Table 2. The results indicated that the w/c can be estimated with reasonable accuracy of ±0.05.

The analysis of the thin-section images under the optical microscope indicated that at any of the given w/c, there was no significant difference in the intensity of the fluorescent light through the thin sections made from the concrete cured at the different conditions, although their degree of cement hydration and capillary porosities were somewhat different [8]. However, there were clear differences in the GT value between the samples with different w/c ratios.

The results suggest that for concrete cured in tropical environment for at least 28 days including 7 days under moist conditions, the fluorescence microscopy method can be used to estimate the w/c of the concrete without determining the degree of cement hydration.

The results from fluorescence microscopy obtained in this study seem to be reasonable in following two aspects: (1) the variation on the degree of cement hydration, capillary porosity, and type and distribution of hydration products due to temperature change from 20 to 35 °C was small and (2) the ambient relative humidity is relatively high (average of 84%) so that cement can continue to hydrate even after the initial moist curing.

#### *3.1.2. Determination of water-to-cement ratio by unhydrated cement clinker*

The amount of unhydrated cement determined from the point counting conducted on the fluorescent thin sections prepared from the concrete with the w/c ratios from 0.30 to

Table 2
Green tone values for concrete with different w/c ratios

| Curing condition | | | Design w/c | Mean green tone value | Standard deviation | Estimated w/c | Error |
|---|---|---|---|---|---|---|---|
| Temperature of water curing (°C) | Length of water curing (days) | Length of exposure in outdoor air (days) | | | | | |
| 20 | 7 | 21 | 0.70 | 132 | 10 | 0.76 | 0.06 |
| 20 | 28 | – | | 125 | 9 | 0.72 | 0.02 |
| 35 | 7 | 21 | | 126 | 8 | 0.73 | 0.03 |
| 20 | 7 | 21 | 0.60 | 94 | 9 | 0.57 | −0.03 |
| 20 | 28 | – | | 94 | 12 | 0.57 | −0.03 |
| 35 | 7 | 21 | | 97 | 11 | 0.58 | −0.02 |
| 20 | 7 | 21 | 0.50 | 77 | 10 | 0.48 | −0.02 |
| 20 | 28 | – | | 81 | 9 | 0.50 | 0.00 |
| 35 | 7 | 21 | | 80 | 9 | 0.50 | 0.00 |
| 20 | 7 | 21 | 0.40 | 57 | 11 | 0.38 | −0.02 |
| 20 | 28 | – | | 57 | 10 | 0.38 | −0.02 |
| 35 | 7 | 21 | | 61 | 11 | 0.40 | 0.00 |
| 20 | 7 | 21 | 0.30 | 46 | 7 | 0.33 | 0.03 |
| 20 | 28 | – | | 45 | 6 | 0.32 | 0.02 |
| 35 | 7 | 21 | | 43 | 6 | 0.31 | 0.01 |

Table 3
Unhydrated cement clinker vs. w/c

| w/c | Measured unhydrated clinkers in cement paste (%) | Calculated unhydrated clinkers in cement paste (%) |
|---|---|---|
| 0.70 | 1.0 | 19 |
| 0.60 | 6.0 | 29 |
| 0.50 | 6.9 | 35 |
| 0.40 | 15.8 | 43 |
| 0.30 | 12.7 | 46 |

0.70, cured in water at 20 °C for 7 days followed by air curing outside the laboratory for 21 days, is shown in Table 3. In addition, the calculated percentage of unhydrated cement based on the measured degree of cement hydration [8] is also shown in Table 3. The percentage of unhydrated cement was calculated by subtracting the percentage of cement hydrated from the total amount.

Although it seems that there is a relationship between the amount of unhydrated clinker and w/c, the coefficient of correlation between them was relatively low. Therefore, it may not have enough accuracy for estimating the unknown w/c by just counting the unhydrated clinker particles for the curing conditions used in this study. In addition to that, curing temperature and age also have significant influence on the quantities of unhydrated cement. At temperatures below 20 °C and short curing time, e.g., 3 to 7 days, the relationship between the w/c and unhydrated cement may be good; however, at temperatures below 20 °C but longer curing time, e.g., over 7 days, or at higher temperatures, e.g., over 20 °C, the relationship between w/c and unhydrated cement may be poor.

As the temperature used in French's study [3] was below 20 °C and the concretes examined were cured for only a few days, which are quite different from this study, i.e., higher temperature and longer curing time, the results from these two studies may not be comparable. However, the test accuracy can be improved by measuring the average spacing between the visible unhydrated or partially hydrated cement grains. The spacing increases with the increase in w/c.

### *3.1.3. Determination of water-to-cement ratio by cement paste density*

Cement paste density calculated from the concrete mix proportion is shown in Table 4. The relationships between the paste density and w/c for concrete cured under the three conditions are given as follows with correlation coefficient of ≥0.99.

Concrete cured at 20 °C in water for 28 days:

$$\rho_p = 2712 - 2375\ \text{w/c} \tag{4a}$$

Concrete cured at 20 °C in water for 7 days followed by 21 days in air outside the laboratory

$$\rho_p = 2685 - 2347\ \text{w/c} \tag{4b}$$

Concrete cured at 35 °C in water for 7 days followed by 21 days in air outside the laboratory.

$$\rho_p = 2315 - 1914\ \text{w/c} \tag{4c}$$

The proportions of concrete ingredients determined from the point-counting method are also presented in Table 4. As the maximum aggregate size used was approximately 20 mm, ~2000 points on each specimen was counted based on the recommendation of the ASTM C 457: 1990 [13]. As used by most petrographers, aggregate particles below 2 mm were considered as fine aggregate in the point counting. Because the sieve analysis of the fine aggregate showed that approximately 10% of particles had sizes greater than 2 mm, the fine aggregate volume determined by the point counting was adjusted upwards for 10% and the coarse aggregate volume was then adjusted accordingly. Based on these results and the concrete density, the cement paste density

Table 4
Measured concrete proportion, concrete density, cement paste density, and w/c

| Curing condition | | | Design w/c | Calculated cement paste density (kg/m$^3$) | Measured proportion of concrete (% by volume) | | | | Concrete density (kg/m$^3$) | Measured cement paste density (kg/m$^3$) | Calculated w/c | Error |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Temperature of water curing (°C) | Length of water curing (days) | Length of exposure in outdoor air (days) | | | Coarse aggregate (>2mm) | Fine aggregate (<2mm) | Cement paste | Air | | | | |
| 20 | 7 | 21 | 0.70 | 1095 | 32.3 | 38.5 | 26.6 | 2.6 | 2121 | 1034 | 0.70 | 0.00 |
| 20 | 28 | – | | | 34.7 | 37.3 | 25.2 | 2.8 | 2128 | 990 | 0.72 | 0.02 |
| 35 | 7 | 21 | | | 35.6 | 34.0 | 28.6 | 1.8 | 2144 | 1143 | 0.72 | 0.02 |
| 20 | 7 | 21 | 0.60 | 1280 | 38.0 | 30.5 | 29.8 | 1.7 | 2183 | 1318 | 0.58 | −0.02 |
| 20 | 28 | – | | | 36.9 | 29.6 | 31.9 | 1.6 | 2172 | 1361 | 0.57 | −0.03 |
| 35 | 7 | 21 | | | 37.2 | 28.9 | 32.3 | 1.6 | 2190 | 1431 | 0.57 | −0.03 |
| 20 | 7 | 21 | 0.50 | 1448 | 40.4 | 27.4 | 30.9 | 1.3 | 2231 | 1481 | 0.51 | 0.01 |
| 20 | 28 | – | | | 39.8 | 26.8 | 31.8 | 1.6 | 2225 | 1518 | 0.50 | 0.00 |
| 35 | 7 | 21 | | | 40.4 | 25.8 | 32.6 | 1.2 | 2227 | 1518 | 0.52 | 0.02 |
| 20 | 7 | 21 | 0.40 | 1642 | 41.7 | 23.9 | 32.4 | 2.0 | 2288 | 1761 | 0.39 | −0.01 |
| 20 | 28 | – | | | 41.5 | 24.1 | 31.9 | 2.5 | 2287 | 1786 | 0.39 | −0.01 |
| 35 | 7 | 21 | | | 42.2 | 23.0 | 33.5 | 1.3 | 2294 | 1751 | 0.40 | 0.00 |
| 20 | 7 | 21 | 0.30 | 1885 | 43.7 | 21.2 | 33.4 | 1.7 | 2364 | 1986 | 0.30 | 0.00 |
| 20 | 28 | – | | | 44.7 | 20.4 | 33.2 | 1.7 | 2359 | 1966 | 0.31 | 0.01 |
| 35 | 7 | 21 | | | 43.6 | 22.5 | 32.9 | 1.0 | 2376 | 1941 | 0.30 | 0.00 |

was calculated and the w/c of concrete was determined (Table 4). The results showed acceptable and satisfactory accuracy. There is no obvious difference between the analytical accuracy for concrete cured under the three different conditions.

### 3.1.4. Comparison of the methods for estimating water-to-cement ratio

A comparison of the three test methods indicated that both the fluorescence method and the cement paste density method have acceptable analytical accuracy for the w/c, i.e., around $\pm 0.05$. The unhydrated cement clinker method is only applicable to certain conditions, and does not seem to be applicable to concrete cured in tropical environment.

The selection of method for estimating w/c should be based on the actual situation, as each method may have its own advantages and disadvantages under different situations. For example, if a quick and rough estimation is needed and the condition is applicable, the unhydrated cement clinker method may be adopted. If an accurate estimation is needed, both the fluorescence method and the cement paste density method should be applied.

## 3.2. Determination of cement content in concrete

### 3.2.1. Determination of cement content by ASTM C 1084: 1997

Cement content for concrete with the w/c ratios of 0.30, 0.40, 0.50, 0.60, and 0.70, cured in water at 20 °C for 28 days determined in accordance with the maleic acid extraction procedure of the ASTM C 1084 [10], is shown in Table 5. The cement contents determined by this method were lower than the calculated values from the concrete mix design except for the sample with w/c of 0.70. The lower cement content determined by the ASTM method could be because (a) incomplete digestion of cement due to partial carbonation of concrete during curing and sample preparation, and (b) the unhydrated iron phases of the portland cement may not be soluble in the maleic acid and, this may bias the cement content to a low value. The latter may be significant for concrete at early age but less significant for concrete at later age. The lower value obtained for the concrete with w/c of 0.70 could be related to segregations that might have occurred for the concrete samples used. The error of the estimation by this method is within 6% (22 kg/m$^3$) in this study, which is lower than the standard accuracy of 40 kg/m$^3$ stated in ASTM C 1084.

### 3.2.2. Determination of cement content by paste volume and water-to-cement ratio

Based on the estimated w/c and the paste content determined by point counting, the cement content was calculated and presented in Table 6. The cement content obtained by this method was generally higher than the calculated one except for the concrete with the w/c of 0.70. The reason for the higher values could be due to the very fine material present in the aggregate which might have been counted as part of the cement paste during the point-counting process. Assuming 1% of aggregate (by mass) was counted as part of the cement paste, this would be equivalent to 25 kg of cement per cubic meter of concrete for the concrete with a w/c of 0.50. For the concrete with w/c of 0.70, the cement content obtained by this method showed a relatively lower value compared to the calculated one. In this case, segregation might have occurred for the concrete beam used for the petrographic analyses. For this method, the accuracy of w/c determination also affects the accuracy of the cement content. According to the results, the error for the estimation of cement content was ≤12%. There is no obvious difference between the test errors for the concretes cured under different conditions.

### 3.2.3. Determination of cement content by concrete density, aggregate volume, and degree of cement hydration

Based on the estimated degree of cement hydration, dry concrete density, and aggregate content from the point counting, the cement contents were calculated using Eq. (2) and presented in Table 7. The cement content obtained by this method was generally higher than the one calculated from concrete mix design except for the concrete with w/c of 0.70. The higher values could be because the degrees of cement hydration, which was determined experimentally at 520 °C [10] and used in the calculation, was lower than the actual ones. Besides, some fine particles in the aggregates could have been counted as part of the cement paste as explained earlier, and this may result in lower total aggregate content and hence higher calculated cement contents. For the concrete with w/c of 0.70, segregation might have occurred for the concrete specimen used. According to the results, the error of the estimation of

Table 5
Cement content determined by ASTM C 1084

| w/c | Dry sample weight (g) | Dry concrete density (kg/m$^3$) | Insoluble residue (%) | Chemically combined water (%) | Cement content by weight (%) | Cement content (kg/m$^3$) | Design cement content (kg/m$^3$) | Error (kg/m$^3$) | Error (%) |
|---|---|---|---|---|---|---|---|---|---|
| 0.70 | 20 | 2128 | 84.68 | 2.52 | 13.7 | 291 | 275 | 16 | 5.8 |
| 0.60 | 20 | 2172 | 84.20 | 2.50 | 14.1 | 306 | 325 | −19 | −5.8 |
| 0.50 | 20 | 2275 | 81.91 | 2.59 | 16.0 | 364 | 375 | −11 | −2.9 |
| 0.40 | 20 | 2287 | 80.48 | 2.62 | 17.6 | 403 | 425 | −22 | −5.2 |
| 0.30 | 20 | 2359 | 78.40 | 2.60 | 19.5 | 460 | 475 | −15 | −3.2 |

Table 6
Cement content determined by the paste volume and w/c

| Curing condition | | | Design w/c | Measured cement paste volume (l/m$^3$) | Estimated w/c[a] | Cement density (g/cm$^3$) | Calculated cement content (kg/m$^3$) | Designed cement content (kg/m$^3$) | Error (kg/m$^3$) | Error (%) |
|---|---|---|---|---|---|---|---|---|---|---|
| Temperature of water curing (°C) | Length of water curing (days) | Length of exposure in outdoor air (days) | | | | | | | | |
| 20 | 7 | 21 | 0.70 | 266 | 0.76 | 3.12 | 246 | 275 | −29 | −10.5 |
| 20 | 28 | – | | 252 | 0.72 | | 242 | | −33 | −12.0 |
| 35 | 7 | 21 | | 286 | 0.73 | | 272 | | −3 | −1.1 |
| 20 | 7 | 21 | 0.60 | 298 | 0.57 | 3.12 | 335 | 325 | 10 | 3.1 |
| 20 | 28 | – | | 319 | 0.57 | | 358 | | 33 | 10.1 |
| 35 | 7 | 21 | | 323 | 0.58 | | 359 | | 34 | 10.5 |
| 20 | 7 | 21 | 0.50 | 309 | 0.48 | 3.12 | 386 | 375 | 11 | 2.9 |
| 20 | 28 | – | | 318 | 0.50 | | 388 | | 13 | 3.4 |
| 35 | 7 | 21 | | 326 | 0.50 | | 397 | | 22 | 5.9 |
| 20 | 7 | 21 | 0.40 | 324 | 0.38 | 3.12 | 463 | 425 | 38 | 8.9 |
| 20 | 28 | – | | 319 | 0.38 | | 455 | | 30 | 7.0 |
| 35 | 7 | 21 | | 335 | 0.40 | | 465 | | 40 | 9.4 |
| 20 | 7 | 21 | 0.30 | 334 | 0.33 | 3.12 | 513 | 475 | 38 | 8.0 |
| 20 | 28 | – | | 332 | 0.32 | | 518 | | 43 | 9.0 |
| 35 | 7 | 21 | | 329 | 0.31 | | 522 | | 47 | 9.9 |

[a] Determined using florescence microscopy method.

cement content by this method is relatively high (up to almost 25%). There is no obvious difference between the test errors for the concretes cured at different conditions.

#### 3.2.4. Comparison of the methods for estimating cement content

A comparison of the three test methods indicated that, in general, the ASTM C 1084 method underestimated the cement content, whereas the other two methods overestimated the cement content. The accuracy of the estimation was in a descending order as: the ASTM C 1084 method>method by paste volume and w/c>method by concrete density, aggregate volume, and degree of cement hydration. It seems that the accuracy of the ASTM method and the paste volume and w/c method is acceptable in most situations.

The selection of method for estimating cement content should also be based on the actual situation, as each method may have its own advantages and disadvantages under different situations. For example, if the w/c of concrete to be tested is known or accurate determination of the w/c is possible, the method by paste volume and w/c should be used. As some method tends to underestimate whereas others tend to overestimate the cement content, a combination of these methods may be useful.

Table 7
Cement content determined by concrete density, aggregate volume, and degree of cement hydration

| Curing condition | | | Design w/c | Concrete density (kg/m$^3$) | Coarse aggregate (>2 mm) mass[a] (kg/m$^3$) | Fine aggregate (<2 mm) mass[a] (kg/m$^3$) | Degree of cement hydration[b] | Calculated cement content (kg/m$^3$) | Designed cement content (kg/m$^3$) | Error (kg/m$^3$) | Error (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Temperature of water curing (°C) | Length of water curing (days) | Length of exposure in outdoor air (days) | | | | | | | | | |
| 20 | 7 | 21 | 0.70 | 2121 | 853 | 993 | 0.81 | 230 | 275 | −45 | −16.4 |
| 20 | 28 | – | | 2128 | 916 | 962 | 0.83 | 208 | | −67 | −24.4 |
| 35 | 7 | 21 | | 2144 | 940 | 877 | 0.85 | 272 | | −3 | −1.1 |
| 20 | 7 | 21 | 0.60 | 2183 | 1003 | 787 | 0.71 | 336 | 325 | 11 | 3.4 |
| 20 | 28 | – | | 2172 | 974 | 764 | 0.71 | 371 | | 46 | 14.2 |
| 35 | 7 | 21 | | 2196 | 982 | 746 | 0.76 | 391 | | 66 | 20.3 |
| 20 | 7 | 21 | 0.50 | 2231 | 1067 | 707 | 0.65 | 395 | 375 | 20 | 5.3 |
| 20 | 28 | – | | 2225 | 1051 | 691 | 0.67 | 416 | | 41 | 10.9 |
| 35 | 7 | 21 | | 2227 | 1067 | 666 | 0.70 | 423 | | 48 | 12.8 |
| 20 | 7 | 21 | 0.40 | 2288 | 1101 | 617 | 0.57 | 501 | 425 | 76 | 17.9 |
| 20 | 28 | – | | 2287 | 1096 | 622 | 0.60 | 497 | | 74 | 17.4 |
| 35 | 7 | 21 | | 2294 | 1114 | 593 | 0.62 | 511 | | 86 | 20.2 |
| 20 | 7 | 21 | 0.30 | 2364 | 1154 | 547 | 0.54 | 587 | 475 | 112 | 23.6 |
| 20 | 28 | – | | 2359 | 1180 | 526 | 0.55 | 577 | | 102 | 21.5 |
| 35 | 7 | 21 | | 2376 | 1151 | 581 | 0.58 | 560 | | 85 | 17.9 |

[a] Aggregate mass, calculated by using aggregate volumes and their densities (i.e., fine aggregate: 2.58, coarse aggregate: 2.64).
[b] Degree of cement hydration, taken from the test results in Ref. [8].

## 4. Conclusions and recommendations

Based on the limited results available, the following conclusions may be drawn.

### 4.1. Determination of w/c

It appears that there was no significant difference in the intensity of the fluorescent light through the thin sections of concrete cured at the different conditions, although their capillary porosities were somewhat different. The w/c of concrete can be estimated by this method with reasonable accuracy of $\pm 0.05$.

The w/c determined by the paste density method can provide a good estimate of the original w/c so long as the volume of the paste and density of the concrete can be determined accurately.

The method for estimation of w/c from the unhydrated cement clinker does not seem to be applicable to concrete cured in tropical environment.

### 4.2. Determination of cement content

The cement contents estimated by the cement paste volume and the w/c as well as by the chemical method agreed well with the designed values. However, the cement content estimated by the method using concrete density/aggregate content/degree of cement hydration showed relatively high errors compared to the designed values. As some of the test methods underestimate while others overestimate the cement content, a combination of these methods may be useful.

### 4.3. Recommendations

For young concrete, thin sections from concrete with different w/c ratios and degrees of cement hydration should be prepared for estimating the w/c by fluorescent microscopy in order to achieve an accurate analytical result.

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