



Fingerprinting of a concrete mix proportion using the acid neutralisation capacity of concrete matrices

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ABSTRACT

Due to the complexity of chemistry in the cement matrices, methods of identification of the concrete mix proportion have been restricted only in either binder type or water content. In this study, a new method for identifying the concrete mix proportion is developed by the acid neutralisation capacity (ANC) of concrete. The suspension of concrete was formed in still water and nitric acid to produce the variation in the pH. The molar concentration of the nitric acid ranged from 0 to 20 mol/kg. As a result, the pH of the suspension for concrete at a given concentration of the nitric acid was dramatically decreased at around 10 in the pH, depending on the aggregate content, of which characteristics was used to determine the aggregate content in a concrete mix. Simultaneously, an increase in the W/C resulted in decreased pH, of which the empirical equation was adopted to determine the W/C. The unique pattern of the ANC curve, including the peak intensity and location, and the fingerprinting spectrum were used to determine binder type in the concrete mix. As binders, OPC, 30% PFA, 60% GGBS and 10% SF were representatively used in this study.

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1. Introduction

It is well known the concrete mix proportion is crucial to the strength and durability of concrete structures in terms of W/C and cement content. Moreover, binder type in a concrete mix strongly influences the durability against a severe environment: pozzolanic materials such as pulverised fuel ash, silica fume and ground granulated blast furnace slag are representative binders for enhancing the resistance to chemical deteriorations of a concrete structure. Once a concrete structure, however, is damaged, the concrete mix proportion is required to judge the optimised repair/rehabilitation technique depending on the state, exposure condition and history of the structure. Notwithstanding, the absence of the concrete mix design often occurs, leading to a difficulty in predicting the state of structure and in judging the repair method, in particular, in case that the structure was built very long time ago.

To identify the concrete mix proportion, some techniques have been developed up to the present, but there is no promising method developed to identify the concrete mix design encompassing binder type, W/C, cement content and aggregate content. Of them, the inductively coupled plasma method has been often used to determine the binder type, quantifying trace elements such as Ba, Sr, Mn, Ti, K and Mg in cement [1,2]. Alternatively, strontium

isotope (i.e. Sr⁸⁷ to Sr⁸⁶) was investigated to identify cement and rock types and thus to predict the risk of alkali silica reaction [3]. However, these methods are restricted to identifying the binder type, with no information on the concrete mix proportion. Moreover, as the trace element exists at a extremely level in cement, the accuracy of detection is not fully convinced. On the other hand, an accousto-ultrasonic technique was applied to determine the water content or/and W/C in a concrete mix, analysing the wave pattern including the intensity and frequency passed through a concrete body [4], although no information on binder and cement type was provided.

Glass and Buenfeld [5] currently suggested that the acid neutralisation capacity (ANC) of cement hydration products was used to determine an unknown concrete mix design including the cement content and cement type. However, the ANC pattern obtained by differentiation of the pH–acid concentration curve is not applicable to determine the W/C and aggregate content, as only cement paste was used to determine the ANC, assuming that aggregate has no impact on the chemistry regarding the ANC. The ANC technique has been used mainly to investigate the leaching resistance of solid cement-based materials [6]. In this method, the titration curve (pH against molar equivalent of acid) shows several plateaus, representing the resistance of different cement hydration products to a pH reduction [7]. Hence, it seems that the pH dependent dissolution of cement hydration products may characterise the concrete mix proportion.

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In this study, a new technique of analysing the ANC data was presented to identify a concrete mix design. The binder type, aggregate portion and W/C were quantified by adopting the ANC curve obtained from the suspension of dust sample containing cement paste, mortar or concrete. The empirical equations indicating the relation between the ANC and concrete mix proportion were derived. Then, these equations were used to identify a concrete mix design, of which the applicability was confirmed by an example study.

2. Experiments

2.1. Specimen fabrication

Specimens were manufactured by cement paste, mortar and concrete at the variation in a free water cement ratio (W/C), aggregate content and binder type. The weight ratio of cement, water and aggregate for the mix proportion of specimens is summarised in Table 1. Ordinary Portland cement (OPC), and mixtures of OPC with 30% pulverised fuel ash (PFA), 60% ground granulated blast furnace slag (GGBS) and 10% silica fume (SF) were used for binders. Their oxide composition is given in Table 2. The density of coarse and fine aggregate accounted for 2.68 and 2.63 respectively, and their absorption was below 1.0%. The maximum size of coarse aggregate was 10 mm. The ratio of fine aggregate to total sustained 0.4 for all concrete mixes. The aggregate used in this study was assumed inert in the chemistry of hydration process to avoid. Paste, mortar and concrete were cast in a cylindrical mould (100 mm in the diameter and 200 mm in the height) and demoulded after 24 h. The specimens were wrapped in a polythene film to avoid a leaching out of chemical components from the specimens, which may often occur in the wet curing. Then, the specimens were cured in a chamber at 20 ± 2 °C for 200 days.

2.2. Measurement of the ANC

Prior to testing for the acid neutralisation capacity (ANC), specimens were dried in an oven at 50 ± 2 °C for 3 days to remain only

Table 1
Weight ratio of cement, water and aggregate for the mix proportion against the weight of cement.

System	Water	Cement	Fine aggregate	Coarse aggregate
Paste	0.30	1	–	–
	0.35	1	–	–
	0.40 ^a	1	–	–
	0.45	1	–	–
	0.50	1	–	–
	0.55	1	–	–
	0.60	1	–	–
Mortar	0.30	1	3.12	–
			2.45	–
			1.95	–
	0.40	1	3.17	–
			2.45	–
			1.90	–
	0.50	1	3.21	–
			2.45	–
			1.88	–
Concrete	0.30	1	2.87	4.31
			2.37	3.55
			1.99	2.99
	0.40	1	2.77	4.15
			2.45	3.68
			1.89	2.83
	0.50	1	2.67	4.00
			2.16	3.24
			1.78	2.67

^a Binder includes OPC, 30% PFA, 60% GGBS and 10% SF.

chemical residues in the cement matrices. Then, the specimens were crushed and ground in bulk size to produce powder sample with no segregation, of which 3.0 g was weighed. Twenty-eight levels of acidic solution containing 2 M nitric acid and equivalent distilled water were added to the powder samples to produce the suspension. The details of the acidic concentration at a given volume are given elsewhere [8]. After stirring the suspension for 10 min using a magnetic stirrer, the pH of the suspension was measured by a pH metre, until no change in the pH value was observed. Once the relation between the pH values and the concentration of acid was obtained, the ANC was determined by following equation:

$$\text{ANC} = \frac{\Delta C}{\Delta \text{pH}} \quad (1)$$

where ΔC is the concentration of the acid added to the suspension and ΔpH is a change in the pH values against ΔC . The higher ANC imposes the higher resistance of the suspension against acidification. The replication for each sample was always three and their average value at the variation in the acid was taken in determining the pH dependent dissolution in terms of the ANC.

3. Results

3.1. Aggregate content

A reduction of the pH values of the OPC suspension to nitric acid is depicted in Fig. 1 depending on the W/C and system of specimens (i.e. paste, mortar, concrete). The concentration of nitric acid was expressed as the equivalent acid concentration to the unit weight of powder samples, which was fabricated in paste, mortar and concrete to differentiate the weight of aggregate in mixes. It is seen that the pH of the suspension was sharply decreased at a given concentration of the nitric acid, since the pH value reached 10 in the pH with an increase in the nitric acid concentration. It seems that a significant decrease in the pH of the suspension is attributed to the limited buffering capacity of cement matrices against a pH fall. As cement hydration products have the resistance against a pH reduction up to the value of around 10.0 in the pH, the pH of the suspension significantly decreased after 10 in the pH due to the absence of further chemical resistance of hydration products [9]. It suggests that the more portion of cement paste (i.e. the lower aggregate content) in a concrete mix would produce the higher resistance to a pH reduction up to 10 in the pH: the higher concentration of nitric acid to the suspension is necessary for a sharp pH fall. Thus, the content of aggregate in a concrete mix can be calculated by the resistance to a pH reduction, in terms of the nitric acid concentration required to meet a pH value.

In this study, it was found that an increase in the aggregate content in OPC paste, mortar and concrete resulted in a decrease in the nitric acid concentration required to lower the pH of the suspension up to 10, as seen in Fig. 2. The nitric acid concentration for concrete/mortar mixes was obtained from Fig. 1, while the concentration for cement paste was calculated by extrapolating the pH-acid concentration curve, because the pH of paste sample always indicated the higher values than 10. It is evident that paste sample ranked the highest acid concentration to meet 10 in the pH for the suspension, ranging from 22.9 to 25.5 mol/kg. When the specimens contained aggregates (concretes and mortars), an increase in the aggregate content in the concrete/mortar mixes resulted in a decrease in the nitric acid concentration required to lower the pH of the suspension to 10, irrespective of W/C and specimen system. The relation between the aggregate content and the acid concentration was fitted by the quadratic equation, which could be used in detecting the aggregate content in an unknown concrete mix. For example, once the acid concentration required

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