

Assessment of Cement Content in Cured Concrete by Chemical Study



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Research Article

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Abstract

An effort was made in this investigation to determine the cement content in cured concrete. A test procedure used to determine calcium oxide which further leads to the assessment of cement in concrete that has been hardened was applied to the laboratory test. The content of cement of three different concrete samples that have been hardened of known proportions was precisely determined by applying the simplified California Test 403 procedure. The California Test 403 method originally used in order to determine the calcium oxide concentration in hardened concrete has been simplified by modifying several steps. The test results are verified by parameter determination of the composition in concrete by X-ray diffraction (XRD) using the JCP (Joint Committee of powder diffraction standards file) database. Good agreement between the results obtained by the titration and analytical methods of the instrument and the actual results is observed.

1. Introduction

Concrete is a heterogeneous composite construction product obtained by mixing and hardening components such as cement, aggregates and water. The performance of concrete depends on the quality of the components, their proportions, their location and the contact conditions. Cement plays an important role in binding the ingredients together and creating concrete that is hard, strong and cohesive with age. There are several types of cement, in which Portland cement is commonly used in general civil construction [10]. Portland cement is manufactured from the same rocks, ores, and minerals that make up concrete aggregates. Furthermore, no Portland cement component is sufficiently consistent from one cement maker to the next. Therefore, testing concrete is the current practice that includes other mineral admixtures such as fly ash, silica fume, etc. in a rather complex mixture. It is difficult to determine the percentage of cement in hardened concrete due to the lack of a perfect procedure for determining the proportions of concrete. In addition, the use of organic admixtures in concrete as admixtures in concrete has made it more difficult to determine the actual proportions of materials in the concrete in the solid state after bridge construction/collapse. However, the difference in composition (element) between cement and aggregate is one of the correct choices for determining cement in hardened concrete. As a result, chemical examination of hardened concrete can reveal a plethora of information regarding the mix's composition and potential degradation reasons. Documentation on the chemical analysis of hardened concrete is rarely available. California Test 403 is a chemical analytical procedure commonly used to determine the proportions of components in concrete. The ASTM approach involves chemically detecting the amount of dissolved silica and calcium oxide in a sample and then indirectly computing the percentage of cement by assuming or establishing from the original cement's analysis. The initial amounts of silica and calcium oxide in cement were determined. This approach produces reliable findings, especially if initial cement and aggregate samples are available, but it is time intensive and cannot be used on concrete with aggregates that develop considerable amounts of silica and calcium oxide under test circumstances. Hime briefly described several chemical analytical procedures commonly used to evaluate the cement content in hardened concrete [1]. Basically, all chemical analysis procedures

are based on the chemical composition of cement. The calcium oxide content, the soluble silica content, the sulfate content, and the chloride content are the less enumerable determinations of cement content. Covault and Poovey employed neutron activation studies to determine cement content. By adding the radioactivity and determining the cement content from the cement content VB count rate curve, the volume of cement in a radioactive concrete trial was determined. This technique, however, necessitates the use of costly irradiation and counting equipment. Furthermore, it cannot be used with calcium-containing aggregates. Iddings and his colleagues studied the feasibility of several analysts for enabling nuclear techniques, stable tracer analysis, measurement of natural radioactivity, and copper dilution. taste in the determination of cement in concrete. Neutron activation analysis was shown to be quick once again, albeit it was impeded by inaccuracies caused by variables typical in concrete structures. It also necessitates an expensive installation. Other techniques are either uneconomical or only practical under ideal conditions. It should also be mentioned that when employing radioisotopes, personnel safety must be assured against any radiation dangers [9]. The analysis will incur an additional cost as a result of this requirement. Kossivas devised a method for calculating the cement content of a concrete sample using its sulphate concentration. This method necessitates the knowledge of the cement's sulphate content as well as the fact that all sulphates are obtained from the cement. The usage of aggregates containing excessive levels of sulphates can lead to serious problems. In addition, the overall characterization of the aggregates in terms of their suitability for use in final construction was determined by petrographic analysis as well as by X-ray diffraction (XRD) [2]. The petrographic analysis confirms the composition of the original aggregate which helps to fill in the gaps to infer the test results. Therefore, petrographic analysis can still be one of the useful methods for the assessment and characterization of concrete. In this study, an attempt was made to find out how much cement is in hardened concrete samples of known proportions, in order to compare the results and find the accuracy. The XRD technique is a flexible, non-destructive method for revealing detailed information on the chemical composition and crystal structure of natural and artificial materials. The chemical test procedure suggested in this article is a simplified version of the California 03 test. Here, the procedure was developed as a result of various practical problems raised during the process. tested according to California 03 test. Concrete with iron content exceeding traceable limits can be easily analyzed with this simplified procedure.

2. Analytical Procedure

Accurate concrete analysis requires good procedure and a qualified analyst with good knowledge of the composition and properties of cement and concrete. Sometimes avoidance or tolerance is not possible and then the analyst must then disclose not just the findings of his investigation, but also the direction and size of the mistake connected with that outcome. Hardened concrete is mainly a mixture of cement and aggregates. Here mainly discussed the procedure for determining the ratio of cement in concrete [8]. The cement content of a hardened concrete sample is commonly calculated by assuming that all of the calcium or acid-soluble silica in the concrete sample comes from port-land cement components [7]. Although the percentages of calcium oxide and silicon dioxide in Portland cement vary by grade and by cement maker, the cement is expected to have between 63.5 and 21% dissolved silica (SiO_2). In most

cases, an error of less than 5% in the computation of portland cement will result. If the aggregate contains limestone or dolomite, the calcium content of Portland cement will be incorrectly high when tested. Because most aggregates' silicon components are insoluble, soluble silica is commonly used as a substitute. Because most aggregate silicon components are insoluble, soluble silica is often regarded as a reliable approach for determining cement concentration [6].

In general, aggregates without limestone components are preferred for concrete use. The California 03 test procedure used to analyse hardened concrete for calcium oxide content is discussed here.

3. California Test 403 To Determine The Calcium Oxide Content

Step by step procedures for California Test 403 method are shown in Fig. 1.

4. Properties Of Test Specimen

The chemical composition of the cement sample used for the concrete is shown in Table 1. Three different hardened concrete samples of known proportions were collected. The cement used to prepare the concrete samples for this study contained 43.50% CaO as mentioned in the manufacturer's report. Descriptions of concrete core samples, namely A, B, C, which are approximately four times larger in diameter than the maximum aggregate size used in the preparation of concrete, are presented in Table 2 [3].

Table 1
Oxide compositions of Portland cement

Compositions	Contents in %
CaO	43.48
SiO ₂	16.95
IR	15.52
LOI	1.61
MgO	1.65
SO ₃	2.72
Fly Ash	17.98

Table 2
Ingredient of the hardened concrete samples

Sample	Cement (%)	Sand (%)	Aggregate (%)	Water (%)
A	18.00	22.32	51.41	8.27
B	17.25	22.81	51.62	8.30
C	18.80	21.77	51.14	8.27

5. Test Preparation

Air-dried concrete cores are weighed and stored in water for 24 hrs., then dried at 110°C for another 24 hrs. The sample is ignited at 550°C to remove absorbed moisture. The concrete sample is then ground into dust. However, not all particles are completely crushed when sprayed. The milled sample is therefore sieved through 4.75 mm aperture and the percentage pass is determined as shown in Table 3. Then, approximately 100 grams of finer particles are sieved through 40 or 420 micrometre sieves. 2 grams of sample passed through a 420-micron sieve was then used for the determination of calcium oxide. The procedure to be applied to find out how much calcium oxide there is. The process for determining calcium oxide in concrete is outlined in the next section.

Table 3
Particle size analysis of concrete samples

Particulars	A	B	C
Amount of passing through 4.75mm sieve (kg)	2.852	3.413	2.396
Amount of retained in 4.75mm sieve (kg)	4.658	5.824	3.892
Percentage of passing through 4.75mm sieve (%)	37.47	36.96	38.10

6. Test Procedure Adopted

The different steps applied to perform the analytical procedure are shown in the following diagram [Figure 2). Several steps of the California Test 403 procedure for the determination of calcium oxide in concrete have been modified and applied to the analysis of concrete samples.

7. Analysis Of Concrete Samples

The three samples mentioned in Table 2 were originally tested to analyze the application of the California Test 403 procedure and some steps were changed as necessary. Step 3 of the California 403 trial was found to be ineffective with all samples included in their starting formulation. However, the addition of iron crystals in the samples resulted in a slightly reddish color, ensuring that the iron composition was traceable in the sample to aid the test step. Therefore, the modified step 4, as shown in Fig. 1 was considered for all samples to perform further experiments. The analytical results obtained by the adopted

procedure are shown in Fig. 2 shown in Table 4. The calcium oxide content of each sample was initially determined for powder passing 4.75 mm and extrapolated. for subsequent full concrete samples.

Table 4
Chemical analysis of concrete samples

Particulars	A	B	C
KmnO ₄ required for titration (ml)	71	72	74
Percentage of CaO (%) (sample passes through 4.75 mm sieve)	20.15	20.00	20.71
For whole concrete CaO (%)	7.56	7.34	7.90
Cement (%)	17.36	16.88	18.14

8. Xrd Inspection

X-ray diffraction is a widely accepted instrumental procedure used to offer extensive information on natural and artificial materials' chemical makeup and crystal structure. There are many cases where researchers have explored XRD methods for qualitative and quantitative analysis of cement systems and have proven to be popular and reliable (Chatterjee A. K.,2006 and Jacek Hulimka and Marta Kaluza, 2020). Concrete powder samples were prepared for XRD analysis and spectroscopic analysis was performed by X-ray diffraction [Model: D8 Advance; Brand: Bruker, Germany] using the JCP 2:00 database and the chemical compositions were evaluated as shown in Table 5. The XRD model describes the chemical compositions and one of them is shown in Fig. 3.

Table 5
Chemical analysis of overall concrete samples

Sample	Calcium oxide content (%)			Error (%)	
	True	Evaluated	XRD	True Vs Evaluated	True Vs XRD
A	7.82	7.56	8.55	-3.59	+ 9.08
B	7.50	7.35	8.17	-2.20	+ 8.67
C	8.16	7.90	8.93	-3.44	+ 9.19

9. Results And Discussions

The assessed calcium oxide content in each concrete sample was compared with the actual percentage of calcium oxide in the respective concrete samples and was found to be in good agreement. In addition, the calcium oxide content in each concrete sample observed by XRD analysis is also close to the estimated estimate. Therefore, the adopted simplified California 403 test can be established as one of the most accurate methods for evaluating cement in concrete samples.

10 Conclusions

The overall quality of hardened concrete depends mainly on the quality of its components. Calcium in cement has the main task of binding concrete components together and forming heterogeneous concrete slabs. It is clear that the presence of different elements of the individual components leads to the heterogeneity of the hardened concrete and increases the complexity of the mixture. Therefore, it is quite difficult to separate the individual components/components from the hardened concrete. In addition, it is not possible to determine the percentage of cement in concrete that has been hardened by conventional procedures. The chemical test procedure is quite precisely applied to concrete manufactured with calcium-free aggregates. The analyses and experiments in this paper lead to the following findings.

- 1) The California 403 test is one of the most accurate procedures for determining calcium oxide in hardened concrete with traceable amounts of elemental iron and calcium-free aggregates.
- 2) In normal concrete, mortar (cement and fine aggregate) accounts for about 40% of the total weight of concrete. So, in the case of concrete with calcium-free aggregates, aggregates larger than 4.75mm can be filtered and grout can be used for chemical analysis.
- 3) Step 3 of the California 403 Test does not give positive results if less than trace iron oxide is present. Instead, steps 4 and 5 of the adopted chemical analysis procedures can be useful for determining the exact calcium oxide and cement percentage in concrete that has been hardened
- 4) The method applied provides an exact amount of calcium oxide as well as the cement percentage in the concrete with a variation of 10%.

Abbreviations

CaO: Calcium oxide; HCL: Hydrochloric acid; H_2O_2 : Hydrogen peroxide; H_2SO_4 : Sulfuric acid;
IR: iridium; JCP: Joint Committee of powder diffraction standards file; LOI: Loss on ignition; $KMnO_4$: Potassium permanganate; MgO: Magnesium oxide; NH_4OH : Ammonium hydroxide; SiO_2 : Silicon dioxide;
XRD: X-ray diffraction

Declarations

Authors' contributions

Author is a post Ph.D. researcher, did the investigations and wrote the manuscript.

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Figures

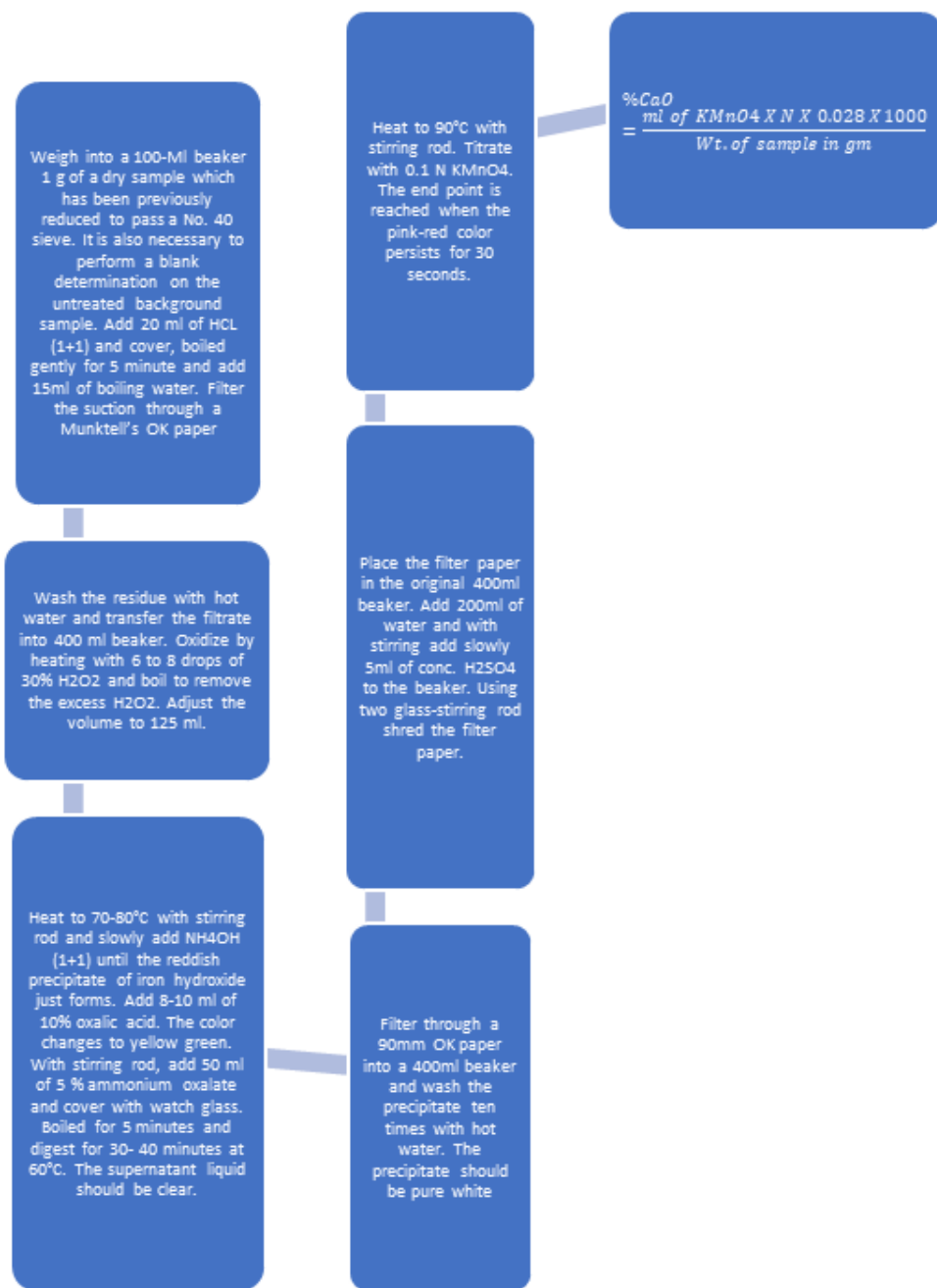


Figure 1

California Test 403 Procedure

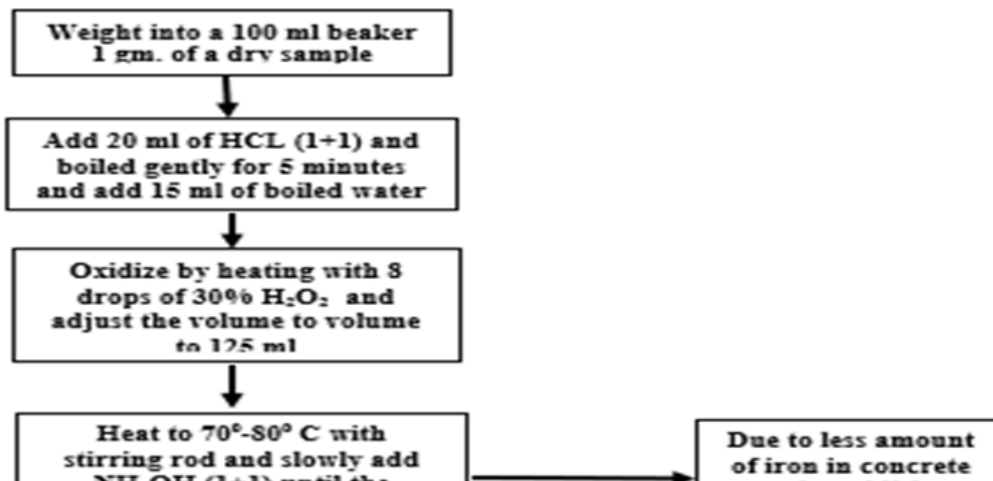


Figure 2

Flow of the adopted chemical procedure

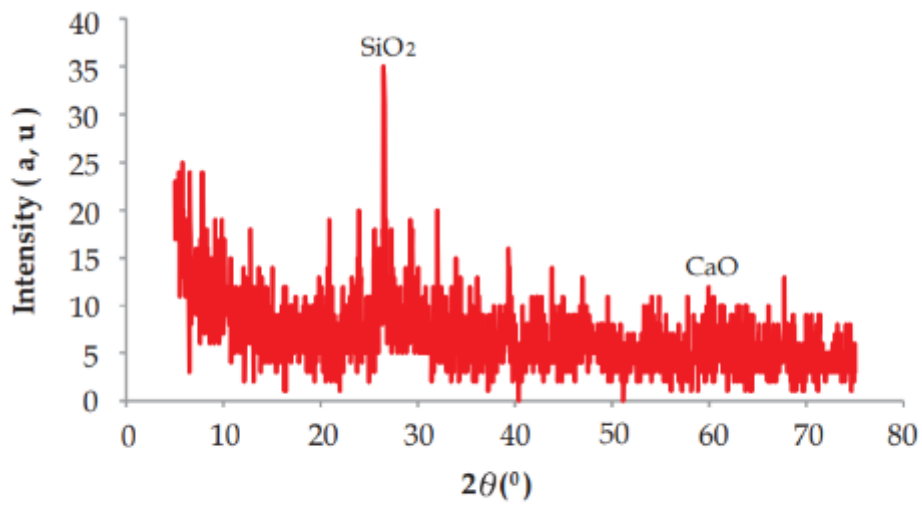


Figure 3

XRD plot of 4.75 mm size passing sample C