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Multifactorial experimental analysis of concrete compressive strength as a function of time and water-to-cement ratio

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Abstract

Concrete compressive strength is typically tested for fixed nominal values of curing age. Corresponding measurements, even if performed by multiple specimens testing, cannot figure the dispersion of the result attributed to all the significant effective error parameters. This study is a step that aims at the creation of a function that correlates the expected testing result on the compressive strength of concrete specimens to all parameters that will be finally assessed as significant to incorporate. Based on the use of such a multifactorial function, results retrieved from testing procedures that are more loosely defined could be corrected accordingly, in order to be compatible with the strict definition of the testing procedure. Also, the integration of various similar experiments of such a protocol could lead to the standardization of a semi-empirical model on the relation of concrete compressive strength as a function of a great number of testing parameters and mix materials characteristics. This study specifically aims at the experimental investigation a) of the correlation of compressive strength testing results with the parameters of curing age and water to cement ratio through sensitivity analysis and b) of the significance of the cross-correlation between these two parameters. The experimental results were used into a multifactorial regression analysis procedure leading to a sigmoidal - by time - equation. The basic outcome of this study is a multifactorial regression function incorporating both the parameters of the curing age and the water to cement ratio for given qualitative characteristics of the constituents. Although this function corresponds to these qualitative characteristics of the constituents, the sensitivity analysis of this study is expected to have a more global validity. Such semi-empirical models, especially if completed with all the significant parameters, are expected to be useful, among others, for accredited testing laboratories in order to perform their internal quality control program.

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Keywords: Concrete; compressive strength; sigmoidal model; sensitivity analysis

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1. Introduction

Concrete compressive strength is typically determined for fixed nominal values of curing age. Efforts have been made in assessing the effect on compressive strength of the specimen curing age (Féret (1892); Carino & Lew (1983); Freiesleben Hansen & Pedersen (1985); Metwally (2014); Sotiropoulou et al. (2017)) the geometrical deteriorations of the specimen (Abd & Habeeb (2014); Aslani et al. (2017)), the water to cement ratio (Abrams (1927); Yeh (2006); Gavela et al. (2018)), the curing temperature (Carino & Lew (1983); Kim et al. (1998); Un & Baradan (2011)) and the relative humidity where the specimens are exposed during the curing procedure (Un & Baradan (2011)). None of these attempts did include an experimental assessment for the parameters of curing age and water to cement ratio at the same time in a multifactorial model. Therefore none of these studied has provided a sensitivity analysis combining these two parameters. The result of the sensitivity analysis is necessary for an accredited laboratory in order to perform an effective uncertainty budget, that is the statement of a measurement uncertainty, of the components of that measurement uncertainty, and of their calculation and combination (JCGM-200: 2008).

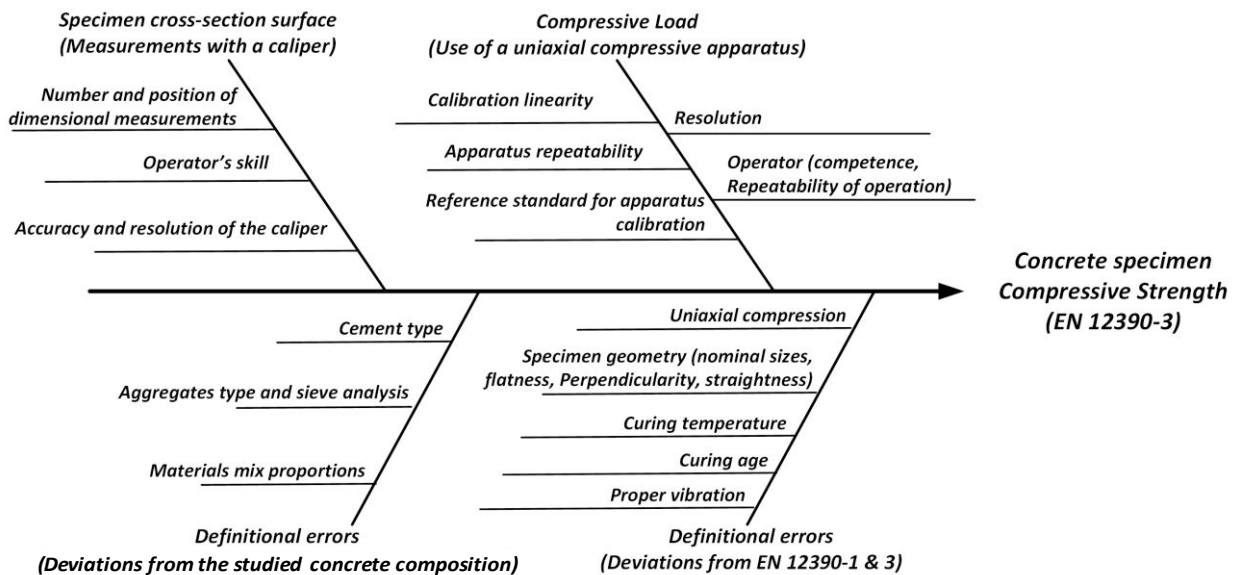


Fig. 1. Ishikawa diagram on the parameters affecting the compressive strength testing according to European Standard EN 12390 series.

This study aims at the experimental investigation of the correlation of compressive strength testing results with the parameters of curing age and water to cement ratio at the same time, through sensitivity analysis. A sigmoidal curve was used to fit the experimental results, something that has already been proposed by Carino (1983) and Freiesleben Hansen & Pedersen (1985), but was not followed up experimentally in the past years.

All of the parameters presented in Fig.1 affect the compressive strength testing according to European Standard EN 12390 series. Some essential questions are: to what extent do all these parameters correlate to each other and to the result of the test procedure? Are the results of the testing procedure valid if these parameters fail to be accurately determined? For example, should a testing result be put aside if the curing age of the specimen deviates by a few days from the typical nominal 28-days value?

The population of parameters affecting the result of a compressive strength test is big so unless performing sensitivity analysis for any subset of these parameters via a specialized experiment it is almost impossible to assess the impact of this subset of parameters. Studying the effect of all the above parameters in one single experiment for various levels of those parameters would lead to an enormous specimens' population. For this reason in the frame of an extended study aiming at the creation of a function that correlates the testing result on the compressive strength of concrete specimens to all the significant of the above parameters, only the experimental investigation of the correlation of compressive strength testing results with the parameters of curing age and water to cement ratio was examined. The in-

tegration of various similar experiments of such a protocol by various laboratories and for various parameters of the test procedure could speed the achievement of a standardized semi-empirical model on the relation of concrete compressive strength as a function of a great number of testing parameters and mix materials characteristics (Gavela et al. (2017)).

Nomenclature

$CS(t)$	concrete specimen Compressive Strength at time (curing age) t [MPa]
CS_{inf}	concrete specimen Compressive Strength @ infinite time (curing age) and a specific level of WtC
WtC	concrete specimen Water-to-Cement ratio [-]
t	curing age of the concrete specimen [days]
$P(t)$	proportion of the final value, CS_{inf} , at curing age t
τ	curing age reference value [days]
n	regression parameter related to the shape of the sigmoidal curve [-]
c_0	regression parameter related to the estimation of the final value CS_{inf} [MPa]
c_1	regression parameter related to the estimation of the final value CS_{inf} [MPa]

2. Experimental design and method

The testing procedure followed for the realization of the experiment is exactly the one described in the European Standard EN 12390-3:2009.

2.1. Experimental design

The produced specimens were compatible with the requirements of the European Standard EN 12390-1, and especially with the definition of cubic specimens with dimension 15 cm. Cement type CEM II 42.5 was used. Crushed fine and coarse limestone aggregates were used. The concrete mix compositions are shown in Table 1.

For each of the concrete compositions 13 specimens were prepared. Slump test for each composition was performed according to EN 12350-2 and Slump test results are shown in Table 1. Many different metal moulds were used for the preparation of these specimens. The use of many different moulds leads to an expected and reasonable dispersion of the essential geometrical characteristics of the specimens (flatness and perpendicularity of the specimen surfaces, accuracy of the specimens' dimensions compared to the nominal dimensions according to EN 12390-1). Consequently, the parameter of the specimen's geometry is expected to have contributed into the results of this study.

Table 1. Mix compositions.

Composition	Cement [kg/m ³]	WtC [-]	Sand [kg/m ³]	coarse aggregates	coarse aggregates	Superplasticizer [kg/m ³]	Slump (mm)
				4-16mm [kg/m ³]	16-31.5mm [kg/m ³]		
A	280	0.46	1112.9	372.0	679.0	6.16	30
B	280	0.48	1104.7	369.2	674.0	6.16	70
C	280	0.50	1090.7	364.5	665.5	6.16	100
D	280	0.52	1088.2	363.7	664.0	6.16	140
E	280	0.54	1081.3	361.4	659.7	5.32	180

The curing procedure followed the requirements of EN 12390-2. After demolding the specimens were immersed in water. The curing temperature was about 20 °C. Three specimens from each composition were tested at 3 days of curing age. Also, two specimens from each composition were tested at curing ages equal to 7, 14, 28, 60 and 90 days.

The compressive strength measurements were carried out on an Avery 7112 CCG hydraulic uniaxial testing machine. The uniaxial compressive equipment that was used for performing the tests had recently been calibrated and

the calibration was verified by an accredited laboratory according to the requirements of the International Standard 7500-1. It could be assumed that the calibration of the uniaxial compressive equipment contributes to the combined uncertainty of the regression result only by the effect of the corresponding systematic errors. These systematic errors could be assumed statistically equal to the uncertainty of the reference values provided by the reference standard used during the calibration procedure.

The essential contribution of this study lies in the experimental design for the combination of curing ages and the parameter of water to cement mass ratio. Specimen curing age values at the time of compressive strength testing were selected to include the typical value of 28-days. Water to cement ratio values were selected to be separated by equivalent intervals of 0.02. At the same time superplasticizer's mix proportions were kept the same except from the mixture with the more water to cement ratio. In this mixture a small decrease of superplasticizer's mix proportion was needed in order the mixture is not becoming segregated. The cement content expressed in kg/m^3 was selected to be kept constant. So, inevitably, it was impossible to change the water-to-cement ratio and keep the content of aggregates unchanged as expressed in kg/m^3 . Otherwise the base for the calculation of constituents' content would not be in all cases equal to 1 m^3 .

2.2. Method of analysis

The above mentioned experimental results were used into a multifactorial regression analysis procedure leading to a sigmoidal - by time equation:

$$CS(t) = CS_{inf} \cdot e^{[-(\tau/t)^n]} = (c_0 + c_1 \cdot WtC) \cdot e^{[-(\tau/t)^n]} \quad (1)$$

In the above Eq.(1), $CS_{inf} = c_0 + c_1 \cdot WtC$ provides the value of compressive strength estimated for infinite curing age, which could be called the final compressive strength. The other part of the equation, the exponential, provides the proportion of the final compressive strength reached at curing age t :

$$P(t) = e^{[-(\tau/t)^n]} \quad (2)$$

Sensitivity analysis and application of the law of propagation of uncertainty is easily performed according to the ISO GUM procedure when such a multifactorial function is used. Specifically, the sensitivity coefficients C_{WtC} and C_t can be estimated as the corresponding derivatives of the function in Eq.(1). These coefficients provide an assessment for the uncertainty of the result of concrete specimen compressive strength measurement which is attributed to the uncertainty in estimating the values for water-to-cement ratio and curing age, respectively. These two sensitivity coefficients are provided by the following equations:

$$C_{WtC} = c_1 \cdot e^{[-(\tau/t)^n]} = c_1 \cdot P(t) \quad (3)$$

$$C_t = \frac{CS(t) \cdot n \cdot \tau^n}{t^{n+1}} \quad (4)$$

A laboratory performing testing in well-known concrete syntheses could use Eq.(1) in the frame of quality control. That is, for concrete specimens that are similar in synthesis as those used for establishing Eq.(1), the result of any future compressive strength testing should not deviated significantly from the reference value provided by Eq.(1). For significantly different syntheses a laboratory should repeat the herein presented experimental procedure in order to fit Eq.(1) to the results of the corresponding compressive strength tests.

3. Results and discussion

The regression procedure provided a statistically significant multifactorial function (see Fig.2a for the relation of the multifactorial model as a function of WtC and Fig.2b for the relation of the model as a function of curing age, t ,

with parameter values: $c_0=143\pm 24$ MPa, $c_f=-136\pm 38$ MPa, $\tau=0.45\pm 0.18$ days and $n=1.0\pm 0.2$, at a confidence level of 95%. The fitting quality is very satisfying ($R^2 = 0.92$). One of the laboratory test results was omitted as an outlier, so 64 results were used in the regression procedure, instead of 65.

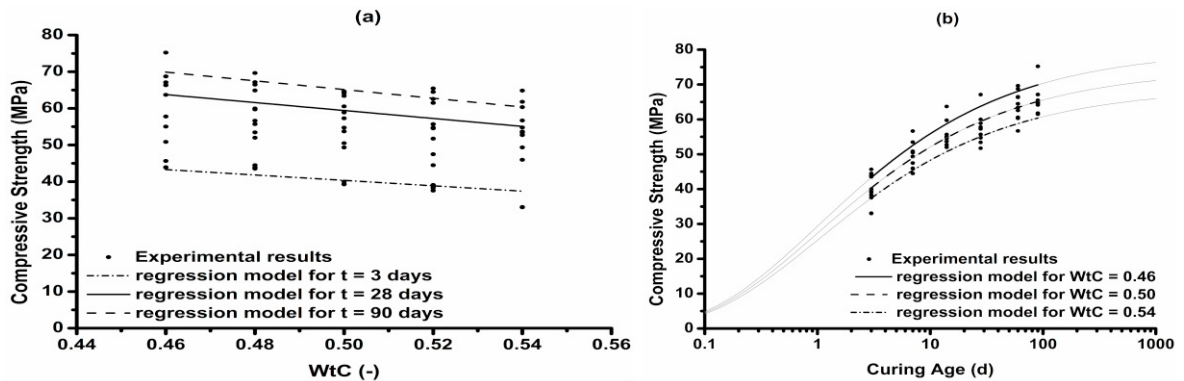


Fig. 2. Multifactorial regression model as a function of (a) Water-to-Cement ratio (WtC) and (b) curing age t .

The result on CS_{inf} is shown in Table 2. These results provide the benefit of estimating directly the final compressive strength of the tested concrete synthesis with an uncertainty comparable to that for estimating the 28 day strength as the mean from a specific number of tested specimens.

Table 2. Estimation of CS_{inf} by use of Eq.(1) for three levels of water-to-cement ratio ($k=2$ for expanded uncertainty).

WtC [-]	CS_{inf} [MPa]
0.46	80 ± 10
0.50	74 ± 9
0.54	69 ± 9

Despite the fact that this regression function corresponds to specific qualitative characteristics of the constituents, the sensitivity analysis of this study is expected to have a more global validity. For example, using Eq.(3) the result for C_{WtC} for curing ages of 7, 28 and 90 days was estimated at 89, 109 and 119 MPa. This means that if we assume a maximum error on WtC of about ± 0.02 and a triangular distribution for a type B estimation of WtC standard uncertainty, this would correspond to an effect on the compressive strength of the specimen of 0.7, 0.9 and 1.0 MPa. On the other hand, if we assume a maximum error on the curing age t of about ± 1 day and a triangular distribution for the corresponding type B estimation of t standard uncertainty, Eq.(4) would estimate an effect on the compressive strength of the specimen at 0.6, 0.1 and much less than 0.1 MPa. It is interesting to compare these estimations to the results of previous studies (Gavela et al. (2018)) where the expanded uncertainty ($k=2$) for testing one single specimen according to the EN 12390 series procedure was estimated at about 17% for similar concrete syntheses. It is obvious that water to cement ratio and curing age errors cannot build up the major part of the testing procedure uncertainty. Major uncertainty parameters should be other like the geometry of the specimen which is not easily assessed in an experimental way and the compressive apparatus repeatability.

Application of Eq.(2) provides a value of about 80% of the final compressive strength of each specimen been reached at a curing age of 28 days. It is also evident by Fig.2b that compressive strength still increases significantly after the curing age of 28 days. When testing the specimens only at that curing age, independent for how many are the replicate specimens being used, this figure cannot be accomplished. The result will be always assessed on the basis of an assumed proportion of the final compressive strength been reached at 28 days. A laboratory, or a producer, wishing to estimate the final compressive strength of a series of specimens should apply Eq.(1). An interesting idea coming from this would be not to test 5 or 6 specimens at exactly 28 days, but testing them in consequent time intervals (e.g. 5, 10, 15, 20, 25 and 30 days) and thus producing the sigmoidal curve of Eq.(1). This would provide directly the result on CS_{inf} , with no need for $P(t)$ assumption.

Such semi-empirical models, especially if completed with all the significant parameters, are expected to be useful, among others, for accredited testing laboratories in order to perform their internal quality control program. Specifically, testing results lying outside the prediction bands of Eq.(1) should be considered as outliers.

Finally, one more possibility provided for performing quality control of compressive strength testing is that there is no strict bound for completing the test procedure at the exact nominal 28-days curing age. That means, if a laboratory misses to perform the test at exactly 28 days of curing age, or if a verification testing is to be performed at a significant time interval after the nominal 28-days curing age, it is feasible to reduce the test result by using the Eq.(1) to the corresponding value, at curing age equal to the nominal 28-days.

4. Conclusions

The mean compressive strength produced as a result of testing according to EN 12390-3 can be estimated by a sigmoidal-by-time multifactorial regression function incorporating both the water to cement ratio and curing age parameters. The use of this multifactorial function provides the opportunity to assess whether the compressive strength of the tested synthesis has a significant trend to increase after the nominal curing age of 28-days. The sensitivity coefficient of compressive strength as related to water to cement ratio is a function of curing age, specifically the relation of compressive strength with water to cement ratio is well represented by a line for which the slope changes in a sigmoidal relation with curing age. The sensitivity coefficient of compressive strength as related to curing age is also changing by time. At a curing age of 28 days it is expected to be non significant. So, deviations in the order of a few days from the definition of 28 days do not affect significantly the compressive strength test result.

The sensitivity on the effect of water to cement ratio and curing age uncertainty is minor as compared to the combined uncertainty of the test result at 28 days.

The results of this study are useful for a laboratory seeking accreditation on the method of EN 12390 series.

The study could be further extended by proper experiments on a multifactorial sigmoidal curve incorporating also other significant parameters such as the curing temperature, the aggregates characteristics and the type of cement.

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