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# Influence of Concrete Strength Class on the Long-Term Static and Dynamic Elastic Moduli of Concrete

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**Abstract:** Construction materials, among which concrete is by far the most used, have followed a trend of continuously increasing demand in real estate. A relatively small number of research works have been published on the long-term material properties of concrete in comparison to studies reporting their findings at standard curing ages of 28 days. This is due, in part, to the length of time one must wait until the intended age of concrete is reached. The present paper contributes to filling this gap of information in terms of the strength and dynamic elastic properties of concrete. The dynamic modulus of elasticity may be used to assess the static modulus of elasticity (Young's modulus), a key property used during the design stage of a structure, in a non-destructive manner. This paper presents the results obtained from laboratory tests on the long-term (6 years) characterization of concrete from the point of view of dynamic shear and longitudinal moduli of elasticity, dynamic Poisson's ratio, static modulus of elasticity, compressive and tensile splitting strengths, and their change depending on the concrete strength class.

**Keywords:** long-term mechanical properties; dynamic modulus of elasticity; dynamic shear modulus; concrete strength class



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## 1. Introduction

The increasing demand in real estate recorded during the past three decades has resulted in a continuous rise in the consumption of raw materials to meet the needs of modern life. Construction materials, among which concrete is by far the most used, have followed this rising trend. Despite economic and sanitary crises, construction markets have continued to expand at a faster or slower pace but are nevertheless growing. According to a recent study, concrete was identified as the second most used commodity in modern society after water [1]. Consequently, it is one of the most intensively studied construction materials. New cements have been developed to create stronger and more durable concretes that are able to meet the highest standards [2].

Although it is one of the biggest consumers of natural resources, the concrete industry has the advantage that it can incorporate large amounts of industrial waste, ranging from recycled materials for aggregates to supplementary cementitious materials, to reduce the consumption of cement in concrete. With every new development, standardized tests are employed to assess whether or not the resulting concrete is suitable for structural applications. This means that the strength and elastic properties of concrete are determined at a fairly early stage, compared to the lifespan of the building it becomes a part of.

A relatively small number of research works have been published on the long-term material properties of concrete in comparison to studies reporting their findings at standard curing ages of 28 days. This is due, in part, to the long time one must wait until the intended age of concrete is reached. Investigations on the durability properties of concrete may break this trend, since these employ accelerated tests in order to simulate years of service life under severe environmental conditions [3,4]. However, given that some of the building stock is fast approaching its designed lifetime, coupled with a growing awareness of the need to understand what happens to concrete in the long run, an increased number of research works dedicated to investigating the long-term properties of concrete and concrete structural elements have been published.

Recent studies have revealed the effect of metakaolin and silica fume on the long-term strength and durability properties of concrete [5]. Both supplementary cementitious materials have an influence on either the compressive strength or internal structure of concrete, but their synergistic long-term effect does not produce significantly improved mechanical properties. However, using metakaolin in combination with polymer admixture has resulted in improving the long-term strength and durability of concrete [6]. Using wastewater as mixing water in concrete was found to have adverse effects on the mechanical properties and durability of concrete in the long run, but the use of industrial treated wastewater had minor negative effects on the abovementioned properties [1]. This represents a significant step forward in reducing the amount of fresh water used in the construction industry.

The effect of curing conditions has also been investigated from the point of view of the long-term properties of concrete. Steam curing is a technique used in the production of prefabricated/precast elements to ensure a higher early-age performance. Several curing procedures have been investigated: steam curing, step-curing and variable-rate curing, each of which was found to have a direct influence on the heat damage effect in concrete, with consequences for its long-term properties [7].

Material properties have a direct effect on the behavior of structural elements in buildings. It was reported that long-term concrete shrinkage results in a decreased magnitude of the load, causing the occurrence of the first crack in reinforced concrete elements and reducing the tension stiffening mechanism for lower longitudinal reinforcement ratios [8].

Some of the concrete poles supporting power lines are made using spun-concrete technology. They are subjected to repeated freeze–thaw cycles during their lifetime in conjunction with various other environmental factors and chemical attacks. The influence of different admixtures on the long-term durability and strength characteristics were investigated and reported in a previous study [9].

There is still a lot of missing information regarding the long-term variation in the mechanical and elastic properties of concrete, with time being the single most important obstacle to gathering the knowledge at a faster pace. The present paper contributes to filling this information gap in terms of the strength and dynamic elastic properties of concrete. The latter characterization offers insightful information concerning the vibration damping characteristics of a material. The dynamic modulus of elasticity may be used to assess the material damping ratio and, by means of empirical conversion equations, to determine in a non-destructive manner the static modulus of elasticity (Young's modulus), a key property used during the design stage of a structure. This paper presents the results obtained from laboratory tests on the long-term (6 years) characterization of concrete from the point of view of the dynamic shear and longitudinal moduli of elasticity, dynamic Poisson's ratio, static modulus of elasticity, and compressive and tensile splitting strengths. The main parameters of the research were the concrete age, which was either 28 days [10] or 6 years, and the concrete strength class (three different strength classes were considered).

## 2. Materials and Methods

### 2.1. Materials

The three chosen concrete strength classes were C16/20, C20/25, and C25/30, as classified by Eurocode 2 [11]. A CEM II A-LL 42.5R rapid hardening cement, complying to standard specification [12] and readily available on the market, was selected. This was a composite cement consisting of 65–79% cement clinker and 21–35% a mixture of ground-granulated blast furnace slag (GGBS) and lime.

River aggregates with rounded edges were purchased from a local supplier. The particle size distribution of all types of aggregates is presented in [10]. A constant water/cement ratio of 0.5 was considered for all three mix proportions shown in Table 1. The super-plasticizer represented 0.5% of the cement mass for each mix proportion, and was decided upon after successive laboratory trials. The fresh properties of each concrete mix presented in Table 1 were previously reported in [10].

**Table 1.** Mix proportions used in the research.

| Mix Designation | Cement (C) | Water (W) | W/C | Aggregates | | | Super-Plasticizer |
|---|---|---|---|---|---|---|---|
| | | | | Sand | Sort 4–8 mm | Sort 8–16 mm | |
| | [kg/m$^3$] | [kg/m$^3$] | - | [kg/m$^3$] | [kg/m$^3$] | [kg/m$^3$] | [kg/m$^3$] |
| Mix1 | 320 | 160 | 0.5 | 799 | 309 | 796 | 1.6 |
| Mix 2 | 340 | 170 | | 756 | 302 | 810 | 1.7 |
| Mix 3 | 360 | 180 | | 739 | 294 | 788 | 1.8 |

Mix 1 refers to the C16/20 concrete strength class, whereas Mix 2 and Mix 3 refer to the C20/25 and C25/30 concrete strength classes, respectively.

*2.2. Methods*

A total of 45 cylinders (ϕ100 × 200 mm) were cast for each mix proportion shown in Table 1. Of the initial 45 cylinders, only 15 remained to be tested at the age of 6 years. The remaining 30 specimens were tested at the standard age of 28 days. The specimens were demolded at 24 h after casting and cured in water for 28 days. After the age of 28 days, the samples were kept in laboratory conditions (23 ± 2 °C and relative air humidity of 40–50%) until the day of testing, 6 years later.

The static longitudinal modulus of elasticity was assessed in accordance with the specifications of SR EN 12390:13 [13]. Cyclic loading was applied within the limits mentioned in the code and three individual values were obtained for each specimen. One cylinder was loaded in compression until failure to correctly set the upper and lower limits of the loading cycles. Therefore, the static longitudinal modulus of elasticity was assessed for 14 specimens only, with 3 distinct measurements for each specimen.

The compressive and splitting tensile strengths were determined in accordance with SR EN 12390:3 [14] and SR EN 12390:6 [15], respectively. The loading rate for determining the compressive strength was 0.6 MPa/s (4.71 kN/s), whereas for the tensile splitting strength a loading rate of 0.05 MPa/s (0.4 kN/s) was adopted.

The dynamic longitudinal modulus of elasticity, $E_d$, was assessed following the guidelines of ASTM C215:14 [16] and was based on the first resonant frequency (FRF) obtained from the Impact Echo Method. The dynamic modulus of elasticity for the cylindrical specimens was computed as shown in Equation (1):

$$E_d = D \cdot m \cdot f_{ln}^2 \quad (1)$$

where m is the mass of the sample (kg), $f_{ln}$ is the fundamental frequency of vibration (Hz), and D is a coefficient that depends on both the diameter and the length of the cylinder (Equation (2)):

$$D = 5.093 \cdot \frac{L}{d^2} \quad (2)$$

where L is the length of the cylinder (m) and d is the diameter (m).

The dynamic shear modulus, $G_d$, was assessed following the guidelines of ASTM C215:14 [16] and was based on the fundamental torsional frequency obtained from the Impact Echo Method. Equation (3) was used to calculate the values of the dynamic shear modulus:

$$G_d = B \cdot m \cdot f_t^2 \quad (3)$$

where m is the mass of the sample (kg), $f_t$ is the fundamental torsional frequency (Hz), and B is a coefficient that depends on both the diameter and the length of the cylinder (Equation (4):

$$B = \frac{4LR}{A} \tag{4}$$

where L is the length of the cylinder (m), R is a shape factor equal to 1 for cylindrical specimens [16], and A is the cross-sectional area of the cylinder ($m^2$).

The two dynamic moduli were used to compute the dynamic Poisson's ratio:

$$\mu_d = \frac{E_d}{2G_d} - 1 \tag{5}$$

The schematic representation of the loading procedure used for assessing the dynamic elastic properties of concrete cylinders is shown in Figure 1.

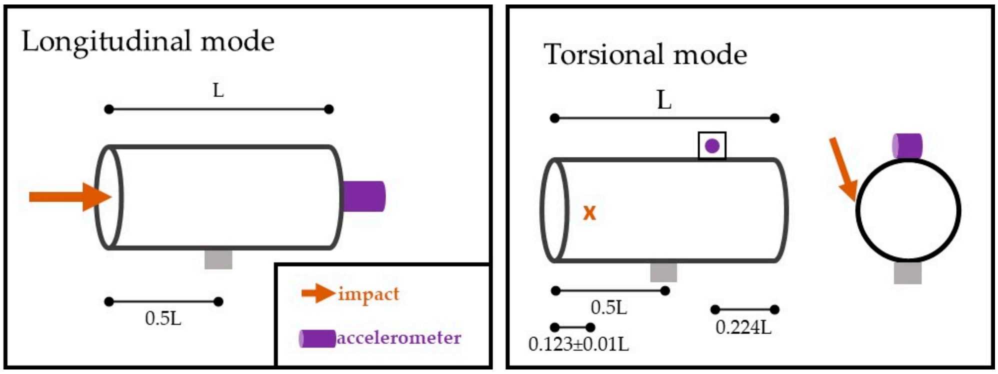



**Figure 1.** Experimental setup for the assessment of dynamic longitudinal and torsional frequencies of vibration.

## 3. Results and Discussions

### *3.1. Static Modulus of Elasticity*

As previously mentioned, the static modulus of elasticity in compression was determined in accordance with currently available standards [13]. The results are presented in Table 2 as the average values of 14 determinations each of these individual values were, in turn, obtained as the average of three measurements [17].

**Table 2.** Static modulus of elasticity.

| Mix Designation | Static Modulus of Elasticity | Standard Deviation | Coefficient of Variation |
|---|---|---|---|
| | [MPa] | [MPa] | [%] |
| Mix 1 | 25,214 | 2461.7 | 9.76 |
| Mix 2 | 31,369 | 1563.1 | 4.98 |
| Mix 3 | 33,147 | 1838.3 | 5.55 |

Similar results were reported on the static modulus of the elasticity of concrete at various ages [18]. There is a rising tendency in the values of the modulus of elasticity with increasing the curing/storage time but it also depends on the curing/storage conditions. Storing the specimens in air, with a low relative humidity, often leads to lower values of the modulus of elasticity compared to those obtained for specimens stored in water for the same time interval. However, it is rather difficult to conduct a direct comparison between the data presented in the scientific literature because of the large variability in the considered concrete mixes, compressive strengths, curing/storing conditions, etc.

It can be observed that the values of the static modulus of elasticity increased with the increasing of the concrete strength class. The values of the standard deviations and coefficients of variation (COV) show a relatively narrow spreading of the results around the median, with higher accuracies for Mix 2 and Mix 3. Similar values for the two statistical terms were reported in the scientific literature for the hardened properties of concrete and their variation with age [19].

### *3.2. Dynamic Longitudinal Modulus of Elasticity*

The determination of the dynamic modulus of elasticity was based on the first resonant frequency of the cylindrical specimen, which was assessed by means of the Impact Echo Method. The free vibration response of all specimens, 15 for each of the mix proportions presented in Table 1, was recorded as shown in Figure 2. Fast Fourier Transform (FFT) was applied to the recorded signal so as to obtain the Fourier spectrum of the specimens from which the fundamental frequency of vibration was identified. For each cylindrical specimen at least four measurements were considered from which the fundamental frequency of vibration was calculated.

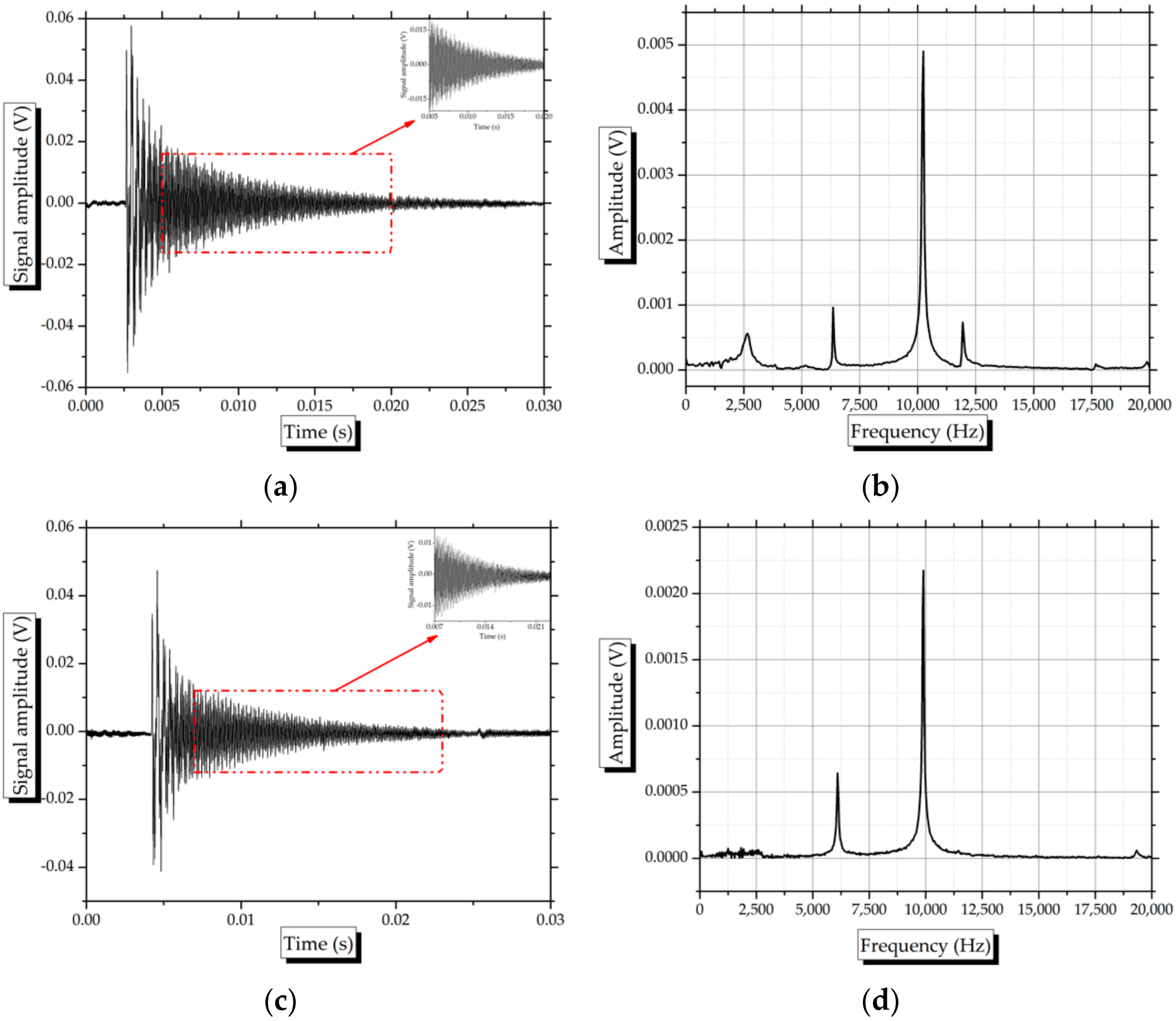
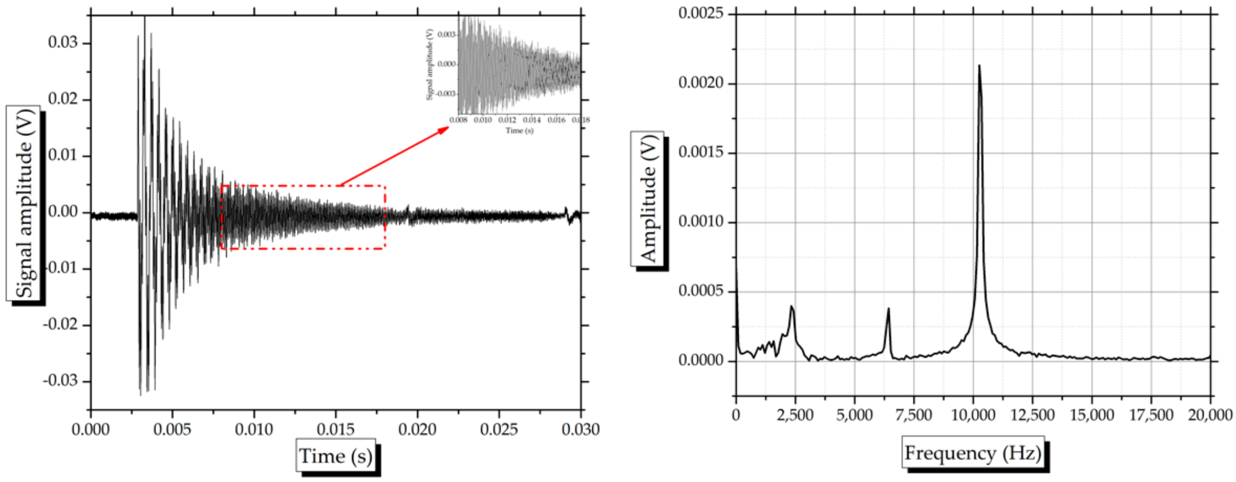



**Figure 2.** *Cont.*




(**e**) (**f**)

**Figure 2.** Measurements for the fundamental frequency of vibration: (**a**) sample of recorded signal for Mix 1; (**b**) FFT for Mix 1; (**c**) sample of recorded signal for Mix 2; (**d**) FFT for Mix 2; (**e**) sample of recorded signal for Mix 3; (**f**) FFT for Mix 3.

The part of the recorded signal considered for the assessment of the fundamental longitudinal frequency of vibration was selected after the initial response of the specimen to the impact load, akin to a transient part (Figure 2a,c,e) of the recorded response signal decay. This would ensure that any possible influence of reflected waves on the boundary of the specimen would be avoided and a cleaner signal would be recorded, similar to a steady-state response [20]. The influence of the initial, transient part on the response spectrum consisted of several peaks of comparable magnitude being identified. On the other hand, the steady state response resulted in a single dominant peak, as can be seen in Figure 2b,d,f.

However, since the Fourier spectra showed a smaller peak around 6000 Hz, for all specimens belonging to each mix proportion additional measurements were taken to confirm the accuracy of the obtained results. Based on the processed data, it was concluded that the smaller peak near 6000 Hz was a combination of the peaks corresponding to the transversal and the torsional frequencies of vibration, as discussed in subsequent sections of this paper.

By applying Equations (1) and (2) the dynamic modulus of elasticity was obtained for all specimens belonging to the considered mix proportions. The data are summarized in Table 3, where the presented values are the averages of all measurements.

**Table 3.** Dynamic longitudinal modulus of elasticity.

| Mix Designation | Dynamic Modulus of Elasticity, $E_d$ | Standard Deviation | Coefficient of Variation |
|---|---|---|---|
| | [MPa] | [MPa] | [%] |
| Mix 1 | 37,874 | 1071.14 | 2.83 |
| Mix 2 | 37,900 | 1221.27 | 3.22 |
| Mix 3 | 39,106 | 685.01 | 1.75 |

The low values of COV imply that the results are very closely spaced with respect to the median value. This means that the elastic properties of the cylinders from each of the three mixes are uniformly distributed throughout the specimens. This is proof that a good homogeneity of the mix was obtained during the mixing process.

### 3.3. Conversion Equations from Dynamic to Static Modulus of Elasticity

The assessment of the dynamic modulus of elasticity has the advantage that it involves a non-destructive method, and can be performed, in general, directly on site. Since, for design and technical assessment purposes, the values of the static modulus of elasticity were considered, conversion equations were proposed.

The considered equations are the ones proposed by Popovics [21], Equation (6), the equation proposed by Lydon and Balendran [22], Equation (7) and the equation available in BS EN 1992-1-1:2004 [23].

$$E_c = \frac{446.09 \times E_d^{1.4}}{\rho_c} \quad (6)$$

$$E_c = 0.83 \times E_d \quad (7)$$

$$E_c = 1.25 \times E_d - 19 \quad (8)$$

where $E_c$ is the static modulus of elasticity (GPa), $E_d$ is the dynamic modulus of elasticity (GPa), and $\rho_c$ is the density of concrete (kg/m$^3$). The obtained results are summarized in Figure 3.

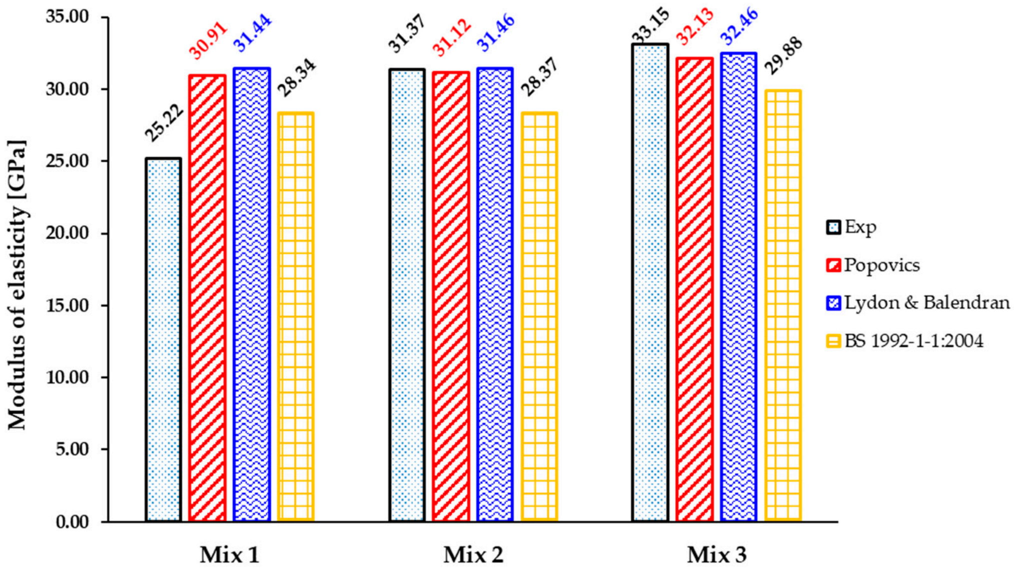



**Figure 3.** Prediction of the static modulus of elasticity.

It can be observed that both Equations (6) and (7) give quite accurate results in terms of predicted value of the static modulus of elasticity for Mixes 2 and 3. All empirical prediction equations overestimated the experimental results of Mix 1 by 12.39–24.66%. Equation (8) underestimates the experimental results for Mixes 2 and 3 by 9.54% and 9.85%, respectively.

Based on the obtained results, it can be concluded that Equations (6) and (7) provide accurate predictions for the values of the static modulus of elasticity in cases of higher concrete strength classes, whereas Equation (8) is better suited for lower concrete strength classes even though it tends to overestimate the experimental results.

### 3.4. Dynamic Shear Modulus of Elasticity

The dynamic shear modulus, $G_d$, was computed based on the first torsional frequency of the cylindrical specimens, following the set-up presented in Figure 1. A sample of the recorded response signal of the cylinders is shown in Figure 4a. The recorded signal looks noisy, not as clean as the recorded response signals presented in Figure 2a,c,e.

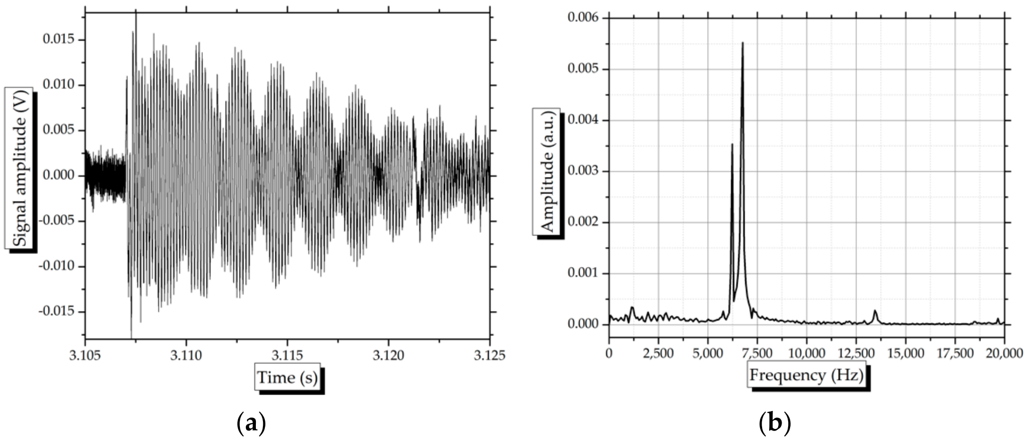



**Figure 4.** Analysis of Mix 1: (**a**) response of cylinder in torsional mode; (**b**) response spectrum.

The FFT response spectrum, Figure 4b, reveals the fact that there are two adjacent peaks that are situated close to each other from the point of view of corresponding frequency. The location of the two peaks matches the location of the smaller peaks present in the Fourier spectra presented in Figure 2b,d,f. The two possible scenarios that were considered were: (1) the two adjacent peaks could be part of a single larger peak and (2) the two peaks could correspond to both torsional and transversal frequencies of vibration.

Consequently, the experimental setup was changed to match the configuration for the determination of the transversal frequency of vibration, in accordance with ASTM C215 [16]. From the Fourier spectrum, the transversal frequency of vibration was identified as being the one corresponding to the lower peak in Figure 4b. This supplementary checking was performed for all specimens to ensure the accuracy and correctness of the obtained results.

The average values of the dynamic shear modulus are summarized in Table 4.

**Table 4.** Dynamic shear modulus.

| Mix Designation | Dynamic Shear Modulus, $G_d$ | Standard Deviation | Coefficient of Variation |
|---|---|---|---|
| | [MPa] | [MPa] | [%] |
| Mix 1 | 14,386 | 450.61 | 3.13 |
| Mix 2 | 14,540 | 581.81 | 4.00 |
| Mix 3 | 15,088 | 384 | 2.55 |

Similar to the values obtained for the statistical terms in cases of static and dynamic moduli of elasticity, very low values for the standard deviation and the COV were obtained in the case of dynamic shear modulus.

Taking into account the shape of Equations (1) and (3), in order to achieve such low values for COV one has to obtain a very low spreading of the values associated to the dimensions of the cylinders, the mass and, most importantly, the corresponding fundamental frequency of vibration. The importance of assessing the frequency with a high accuracy is emphasized by the fact that its squared value is considered in Equations (1) and (3). This means that even a slight increase in the spreading of the results with respect to the median value will be significantly amplified in the final values of the corresponding modulus of elasticity.

### *3.5. Dynamic Poisson's Ratio*

The dynamic Poisson ratio provides a good measurement of the ability of concrete to deform in a direction normal to the line of action of an applied force. The equations provided in ASTM C215 [16] are based on the simplified free-vibration solution of a Timoshenko beam, but exact analytical solutions were also proposed and validated [24].

The results obtained by means of Equation (5) are presented in Table 5. According to previous reports, the dynamic Poisson ratio is larger than the static one [25,26]. Considering the linear relation between Poisson's ratio and the two longitudinal and shear moduli, the accuracy of the results carries over.

**Table 5.** Dynamic Poisson's ratio.

| Mix Designation | Dynamic Poisson's Ratio, $\mu_d$ | Standard Deviation | Coefficient of Variation |
|---|---|---|---|
| | - | - | [%] |
| Mix 1 | 0.303 | 0.0202 | 6.68 |
| Mix 2 | 0.316 | 0.0111 | 3.51 |
| Mix 3 | 0.296 | 0.0147 | 4.95 |

Mixes 1 and 2 seem to be able to dissipate more vibration energy, since they exhibit the largest values for Poisson's ratio, whereas Mix 3 is more rigid. This tendency should be further researched from the point of view of material damping properties, and a further check on the possible correlation between the two parameters should be conducted.

*3.6. Compressive and Tensile Splitting Strengths*

The compressive strength was measured on 10 cylinders in accordance with SR EN 12390-3 [14]. The load was applied at a constant rate of 0.6 MPa/s (4.71 kN/s). The obtained values are shown in Figure 5.

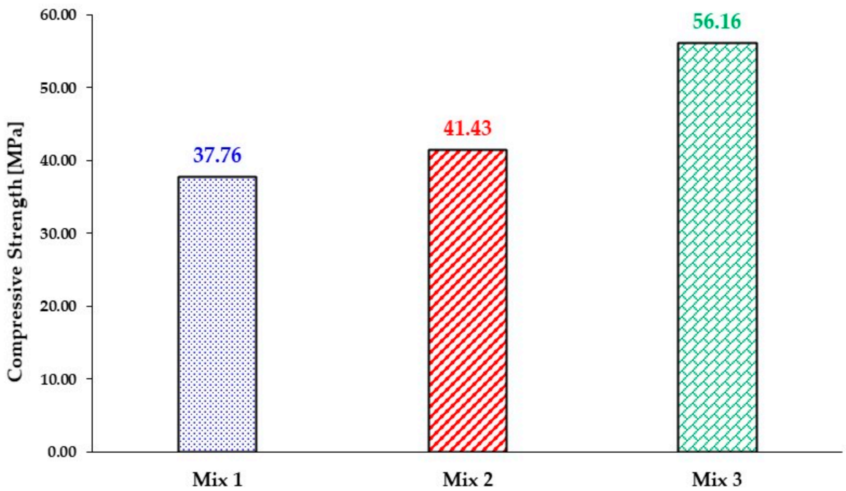



**Figure 5.** Compressive strength at 6 years.

Compared to the data presented in [10], when similar tests were conducted on cylinders made from the same mix proportions, the value of the compressive strength increased by 5.81%, 9.23%, and 47.37% for Mixes 1, 2, and 3, respectively. The possible explanation for such a significant increase in the compressive strength value of Mix 3 compared to the other mixes, all other parameters being similar (water/cement ratio, curing conditions, storage conditions), may be related to the fact that Mix 3 contains the highest amount of cement. The statistical analysis confirms the accuracy of the obtained results with COV values of 4.86%, 0.92%, and 1.61% for Mix 1, Mix 2 and Mix 3, respectively.

A 8.06% increase in the value of the compressive strength at the age of 365 days (1 year) was reported in [1], whereas a 15.07% increase, at the age of 600 days (1.6 years) was reported in [5]. Neither of the studies had a target compressive strength pertaining to a certain concrete strength class and the water to cement ratio was 0.4. Additionally, neither of the studies mention the storage conditions of the concrete specimens, after the standard curing age of 28 days, until the tests were conducted. Similar trends were reported in [27] for the control mix, although the authors did not provide any numerical data.

The conversion equations from compressive strength to static modulus of elasticity were checked for their accuracy and suitability for being applied to rubberized concrete, although they were not specifically developed and proposed for this purpose. Hence, the equation given by Eurocode 2 [11], Equation (9), and the equation given in ACI 318-14 [28], Equation (10), were investigated. The obtained results are summarized in Table 6.

$$E_{cm}(t) = \left(\frac{f_{cm}(t)}{f_{cm}}\right)^{0.3} \times E_{cm},\ \text{where}\ f_{cm} = f_{ck} + 8[\text{MPa}] \quad (9)$$

$$E_c = 4.7\sqrt{f_c{}'} \quad (10)$$

where $f_c{}'$ and $f_{ck}$ are the compressive strength of concrete obtained from laboratory investigations (MPa), $E_c$ and $E_{cm}$ are the static moduli of elasticity (MPa), and $f_{cm}(t)$ and $E_{cm}(t)$ are the compressive strength of the modulus of elasticity at the age of 6 years, respectively.

**Table 6.** Predicted values for the static modulus of elasticity as a function of the compressive strength.

| Mix Designation | $E_c$ (Eurocode 2, Equation (9)) | Exp/$E_c$, Equation (9) | $E_c$ (ACI 318-14, Equation (10)) | Exp/$E_c$, Equation (10) |
|---|---|---|---|---|
| | [MPa] | - | [MPa] | - |
| Mix 1 | 31,840 | 0.79 | 28,883 | 0.87 |
| Mix 2 | 33,122 | 0.95 | 30,252 | 1.04 |
| Mix 3 | 36,107 | 0.92 | 35,223 | 0.94 |

It can be observed that both Equations (9) and (10) give quite accurate predictions for the static modulus of elasticity of the three mixes. Equation (9) tends to underestimate the results, yielding conservative values, whereas Equation (10) slightly overestimates the results in the case of Mix 2.

The tensile splitting strength was determined in five samples in accordance with SR EN 12390:6 [15]. The specimens were loaded at a loading rate of 0.05 MPa/s (0.4 kN/s). The obtained results are shown in Figure 6. The statistical analysis confirms the accuracy of the obtained results with values for COV of 10.71%, 9.1%, and 3.52% for Mix 1, Mix 2, and Mix 3, respectively.

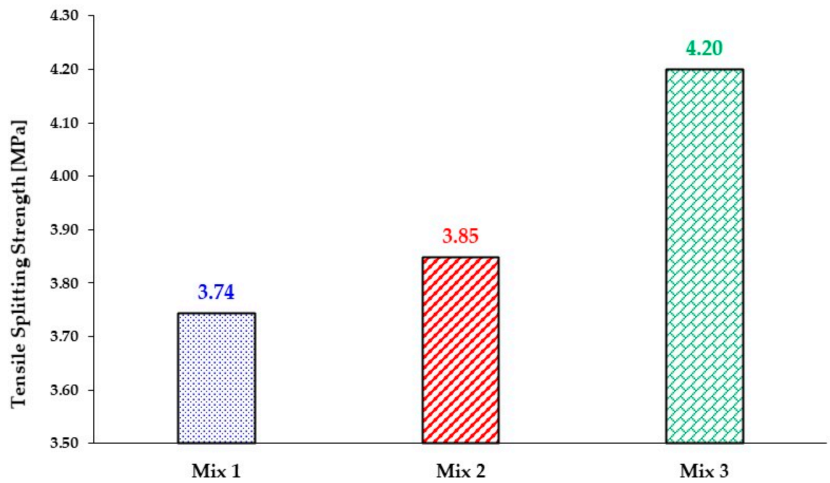
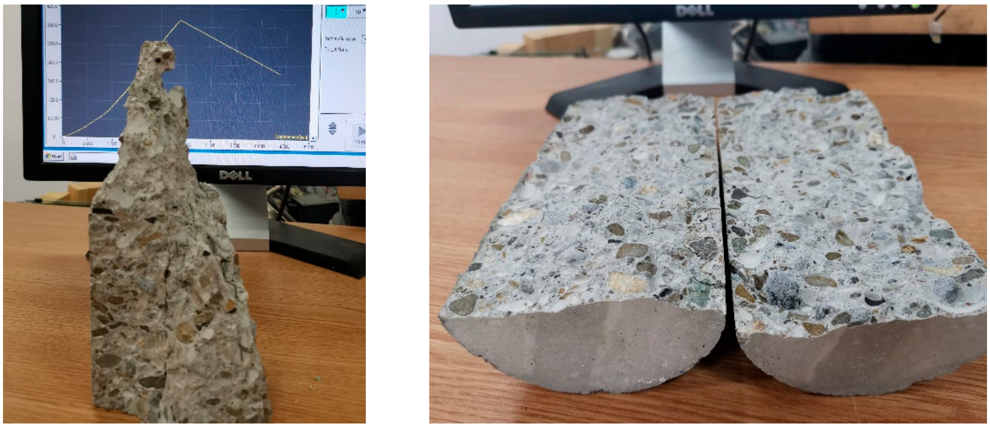



**Figure 6.** Tensile splitting strength of the Ref and RuC mixes.

Samples of a failed specimen in both compression and tensile splitting are presented in Figure 7a,b, respectively. The formation of the hourglass shape for the specimen that failed in compression as well two resulting halves of the cylinder subjected to compression along the generatrix are proof of the consistency of the results presented in this paper.




(**a**) (**b**)

**Figure 7.** Samples of failed specimens: (**a**) in compression; (**b**) tensile splitting test.

## 4. Conclusions

The paper presents an experimental program aimed at assessing the long-term material properties of Portland cement concrete. Based on the obtained results, the following conclusions can be drawn:

There is a clear tendency of the values of the mechanical properties increasing with the age of the concrete and with the concrete strength class. The compressive strength increases by 5.81% to 47.37% compared to the values obtained at the age of 28 days. The increasing trend is not as steep as other results reported in the scientific literature because the specimens were stored in a relatively dry environment. The storage conditions, most importantly the relative air humidity, are known to have a direct influence on the evolution of the material properties in time.

The values of the dynamic modulus of elasticity are between 18~50% higher than the static modulus of elasticity, with the lower percentage corresponding to the higher concrete strength class.

The dynamic Poisson's ratio is lower for the higher concrete strength class owing to the higher rigidity of the material. This may have a direct influence on the material damping properties of concrete and the authors believe it warrants further investigation.

The empirical conversion equations, from the dynamic modulus of elasticity to the static one, provide a good agreement between the analytical values and the experimental ones, with some of the equations being on the conservative side. A similar accuracy was observed when determining the static modulus of elasticity in terms of the compressive strength at the age of 6 years. The available equations from the design code overestimate the experimental results by as much as 8%. This implies that they can be applied with a fair degree of confidence when in situ non-destructive experimental data are available.

The accuracy and repeatability of the presented data were checked using two statistical parameters that provide valuable information on the spread of data with respect to the median value. For each investigated parameter reported in the paper, the standard deviation and the coefficient of variation showed consistently low values. It can be concluded that the values of the material properties were uniformly distributed over the 15 samples for each mix, the resulting concretes were homogeneous, and the experimental investigations were thoroughly and systematically conducted.

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