geosciences

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Durability of Two-Component Grout in Tunneling Applications: A Laboratory Test Campaign

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Abstract: Today, two-component grout is the most widely used backfilling technology in shielded mechanized tunneling. Despite its intensive use, however, very scant information pertaining to the durability of this material is available in the scientific literature. In this work, the aging of two-component grout is studied by curing grout samples using three different modalities. Furthermore, the action of air on two-component grout is studied by assessing the dehydration process, which is a phenomenon that occurs when the material is cured without being completely embedded in soil/rock. Uniaxial compression tests and three-point flexural tests have been carried out for mechanical characterization. The results reveal that in a curing environment made of sand, a moisture of 5% is sufficient to guarantee correct curing of the grout and extend the mechanical performance to three years, whereas the action of air is potentially dangerous, since the grout suffers strongly from dehydration. Despite this dehydration process, however, the mechanical performance of the grout also tends to increase for samples cured under the action of air until a very high level of cracking and shrinkage is reached. A discussion of the limitations on the uniaxial compression strength as the main mechanical parameter for the characterization of two-component grout concludes the work.

Keywords: two-component grout; durability; two-component grout aging process

Citation: Todaro, C.; Carigi, A.; Peila, D. Durability of Two-Component Grout in Tunneling Applications: A Laboratory Test Campaign. *Geosciences* **2024**, *14*, 302. https://doi.org/10.3390/geosciences14110302

Academic Editor: Mohamed Shahin

Received: 1 October 2024
Revised: 3 November 2024
Accepted: 8 November 2024
Published: 10 November 2024

1. Introduction

When tunneling with shield machines, the backfilling phase is of paramount importance. Shielding technology is used in soils and weak rocks, since it ensures the stability of the tunnel over the short term, and it also permits the installation of linings that provide stability over the longer term. Since the linings are installed under the protection of the shield, the diameter of the inner shield is greater than the extrados of the lining, meaning that a gap (hereinafter called the annulus) exists. This void must be continuously and synchronously filled with a grouting material [1] in order to avoid displacement of the lining, with consequent surface settlement (in the case of shallow tunnels) and misalignment of the lining (with consequent waterproofing issues). This gap has an order of magnitude of centimeters [2], and a huge amount of material is therefore required to properly perform the backfilling phase [3,4]. Various materials have been studied and tested for this purpose [5,6], but two-component grout is currently the most commonly used around the world. The reason for this is related to its technical and operative advantages [7]: the management of the two fluids is fairly simple; the chemical and physical stability of both components is ensured for at least 72 h; gelation occurs in seconds; and hardening of the final grout starts instantaneously after gelation. For all these reasons, two-component grout technology easily satisfies all the requirements for a good backfilling phase, i.e., locking the lining in the designed position, bearing the back-up load, reducing surface settlement, and spreading the eventual punctual load over a larger surface of the lining [8]. After gelation, the mechanical and elastic properties start to improve within a short time [9,10]. Càmara [11] and Pelizza et al. [12] have also underlined that two-component grout exhibits anti-wash-out properties; however, the only available information on innovative cement-based materials for underground works that were expressly designed with anti-wash-out properties has been provided by [13,14], and these materials have not yet been applied in backfilling applications.

Despite many applications worldwide [15], current knowledge of the durability of the grout obtained through the use of this technology is very limited. The authors of [16] reported on a series of durability tests that involved curing samples of two-component grout while stressing them under a sodium sulfate solution, which is an approach that was specifically planned to simulate the Lyon–Turin jobsite. Tests performed during one year of curing showed a decreasing trend in the mechanical performance in terms of uniaxial compression strength. No information pertaining to the durability of the grout under standard modalities was provided in this work.

In fact, the changes in the mechanical properties of two-component grout over long curing times are unknown for certain curing conditions. It should be noted that each construction site is unique, with different geologies, different moisture contents, different groundwater chemism and different curing temperatures being only a few of the parameters that could strongly affect the performance of the grout over long curing periods.

In this research, two different aspects were considered, and their effects on the durability of two-component grout were investigated: the effects of aging and of air exposure (dehydration). A double campaign of laboratory tests was performed, which involved casting several samples of the same two-component grout and curing them for different times under different conditions. The first set of tests concerned samples cured for up to three years under a controlled environment, while the second involved samples cured with no embedding in rock or soil, i.e., under the direct action of air. For a better understanding of the research, two different stress actions can be recognized for the first and the second set of tests, respectively: the time and the air. This last set of tests has been planned considering the heaviest stress, in terms of dehydration, that can be applied on the grout in a reasonable time-span.

The outcomes suggest that if a minimum water content of the embedding soil is ensured, the strength of the two-component grout increases over time. On the other side, it has been experimentally proved that the mechanical characterization alone is not enough in order to completely understand the “state of health” of the grout, since increasing *UCS* could be misleading, hiding potential volume reduction (shrinkage).

Although the results were obtained at the laboratory scale, they can be considered very important for a better understanding of the nature of this backfilling technology in relation to both the durability and the curing condition of the grout.

2. Two-Component Grout and the Issue of Durability

Two-component grout technology is based on two fluids (components A and B) that are pumped from a batching station to the machine tanks typically without the use of intermediate pumps. Both components remain as fluids until the mixing phase, which takes place a few centimeters before the nozzles [7,12]. Immediately following turbulent contact between the components, the gelation process starts and the material progressively loses its fluidity. The ‘gel time’ is defined as the period that elapses between the first contact of the components and the loss of fluidity of the obtained grout. If the backfilling project has been correctly set up, the mixed grout is ejected from the nozzles in a shorter time than the gel time, and the pattern of nozzles is geometrically arranged in order to completely fill the annulus. Component A is a cement mortar made up of water, bentonite and chemicals to ensure low viscosity and long workability. Youn et al. [17], Schulte-Schrepping and Breitenbücher [18] and Song et al. [19] have suggested the use of innovative ingredients for backfilling grout with the aim of increasing the mechanical performance, including slag, blast furnace and alkali-activated industrial solid waste. Component B is commonly a silicate solution [20], which is provided “ready to use” from chemical suppliers. The core of two-component grout technology is the mixing design, which is obviously strongly dependent on the requirements and location of the construction site (raw ingredients are commonly obtained from the surroundings). According to Todaro et al. [15], the following ranges can be assumed as typical with data drawn from real construction sites: cement 230–480 kg/m^3, water 730–872 kg/m^3, bentonite 25–60 kg/m^3, retarding/fluidifying agent 1–7 L/m^3, accelerator 50–100 L/m^3.

Irrespective of the ingredients and the mix design used, information linked to the durability is very scarce in the scientific literature. As is well known, durability is defined as the ability of a material to preserve its properties throughout the designed lifetime without requiring excessive maintenance operations or repairs. The durability of two-component grout has never been investigated in depth, and very limited information is available. Pelizza et al. [12] reported that two-component grout works properly over time if the embedding soil can provide a certain moisture content to the backfilling material; however, these authors clearly stated that it was difficult to predict the behavior of two-component grout over the long term. A further step was made in this area of laboratory research by Peila et al. [21], who reported that the moisture content of the soil was not sufficient to ensure the durability of two-component grout and that the permeability of the soil that embeds the two-component grout should also be lower than 10^{-8} m/s. Only with a certain degree of waterproofing can the mechanical properties of the grout can be preserved over time.

In conclusion, no research has yet been conducted to answer legitimate questions such as the following:

- Is the mechanical performance of two-component grout preserved over time (for curing times longer than the standard threshold of 28 days)?
- Can different curing environments affect the mechanical performance after a certain curing time?
- Since other authors have highlighted the role of the embedding soil, what would happen if the two-component grout was left to cure with no form of protection?

Although the last question is a theoretical one, representing a situation that would never occur on a construction site, it is a scenario that might simulate extreme excavation conditions, for example a shallow tunnel in a dry, coarse medium or situations that may locally and temporarily happen in cases such as bypass excavations.

3. Research Path and Sample Production

The basic idea of this research was to assess whether, and to what degree, aging and the action of air can affect the performance of two-component grout. For both analyses, the uniaxial compression strength (UCS) and the indirect tensile strength (T_0) were used as mechanical testing parameters. This choice has been made according to the "history" of the two-component grout. In fact, from the first studies available in the scientific literature [15], the uniaxial compression strength was selected as the main parameter for evaluating if a certain two-component grout satisfies the technical specifications. Nowadays, more complex numerical models require always more realistic parameters, such as the elastic moduli or the shear strength, but for this first work, only the above-mentioned mechanical strengths have been assessed. A study of samples cured under the action of air was also carried out, including monitoring of the weight loss due to dehydration and mechanical characterization.

The relationship between the two test campaigns is the durability (defined as the aptitude of a material to survive without exhibiting damage). The durability can be assessed by selecting a "stressing action". In the aging test campaign, the stressing action was the "time" (with a view to the environment of curing, for simulating three real construction site conditions), while in the air test campaign, the stress action was the air.

3.1. Sample Production

Samples of two-component grout were produced according to the mix design described by Todaro et al. [22]. The dosages of ingredients, expressed in kg/m^3, were as follows: water 853; bentonite 30; cement type I 52.5 R 230; retarding/fluidifying agent 3.5; accelerator 81. Once the correct amount of each ingredient had been weighed, the stirrer was switched on and the production of component A started, following the well-established procedure described by Todaro et al. [22] and summarized in Table 1.

Table 1. Mixing procedure for the production of component A.

Ingredients Added	Phases	Impeller Rotation Speed (rpm)	Duration (min)
Water alone in the mixing tank	Start	800	/
Bentonite	Bentonite activation	2000	7
Cement	Mixing phase	2000	3
Retarding/fluidifying agent	End of mixing phase	2000	2

Casting of the two-component grout was performed according to the procedure described by Todaro et al. [23]. Specimens of dimension 40 × 40 × 160 mm were produced in compliance with CEN [24]. Figure 1 shows some samples after the casting phase.

Figure 1. (**Left**): two-component grout samples cast following Todaro et al. [23]; (**right**): the sample trimming phase.

It should be noted that the procedures used here for the production of component A, the casting phase and the curing phase, are not standardized; many authors have used their own procedures, taking into account the properties of the grout to be assessed [25].

3.2. Curing Conditions for Aging Tests

Samples for the aging study were produced according to the procedure described in Section 3.1. After 24 h following casting, the samples were demolded and split into three different groups, corresponding to three different curing environments (also called "curing modalities"), as summarized in Table 2.

Table 2. Curing modalities.

ID	Curing Modality
1	Samples stored completely under water
2	Samples stored in sand with a moisture content of 5%
3	Samples stored in sand with a moisture content of 10%

At least nine specimens were produced for each testing period, corresponding to at least three specimens for each curing modality. After casting and demolding, which was carried out 24 h later, samples were placed in water (ID 1) or in sand previously humidified with a certain amount of water (IDs 2 and 3). Special care was taken to ensure complete contact between the sand and the samples. After the addition of a layer of sand about 1–2 cm thick at the bottom of the plastic tanks, samples were vertically located. The samples were then gently covered, and contact between the samples and the sand was ensured by hand. After complete filling of the plastic tank (volume of 25–30 L), the plug was applied and the tank was hermetically sealed. Figure 2 shows an example of the burial process.

Figure 2. Burial process for samples of two-component grout under modalities 2 and 3.

The curing temperature of 23 ± 2 °C was controlled and recorded throughout the curing period.

The sand used in this process was a fluvial type (Figure 3). This material was selected in view of its fineness, as it was able to ensure good contact with the sample and consequently good and homogeneous transmission of humidity to the samples (coarser sands were discarded due to a higher void left between particles and the sample surfaces).

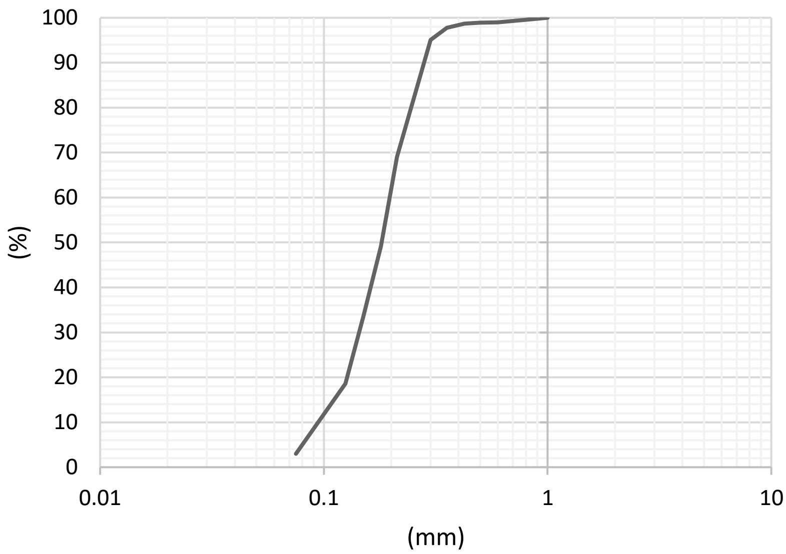

Figure 3. Grain size distribution of the sand used for curing the samples of two-component grout in the aging tests.

3.3. Curing Conditions for the Action of Air

The specimens intended to be tested under the action of air were produced differently, and special molds made of polystyrene were used. The purpose of this disposable solution was to guarantee an easy demolding phase, which could be correctly performed after a few minutes after casting, in order to expose the samples to air since the very first phases of curing. About 10 min after the end of the gelation phase, the two-component grout gained enough strength to be handled without damage to its structure, which would be impossible with the PVC molds used in Section 3.1.

The samples used for the weight loss study were located in specific plastic containers (one sample in each container), and they were raised 2–3 mm from the bottom by means of small cylinders. This arrangement was chosen in order to ensure contact with the air over the total surface of each sample. The reason for using plastic containers was to avoid the loss of part of the sample during the dehydration process (as explained later, two-component grout has a strong tendency to shrink if air is acting on the surface). Samples were stored in a protected area away from direct sunlight and air currents. A decision was taken not to implement climate control, although both the temperature and relative humidity (RH%) of the air were monitored over time. This decision was not an easy one to make, and it was the subject of some discussion, but allowing the above-mentioned parameters to fluctuate permitted us to understand whether these variations could affect the dehydration process.

Samples produced for mechanical assessment were cured under similar conditions: they were placed on a polystyrene layer instead of containers but anyway raised to ensure air contact on all the sides of the samples (Figure 4). For this series of tests, the curing temperature was 22 °C on average, and the relative humidity was within the range 70–80%.

Figure 4. Samples cured under the action of air. The small wooden cylinders allowed air to reach the bottom of the samples. These samples were produced for mechanical assessment.

4. Tests and Procedures

4.1. Mechanical Tests on the Aged Samples

The mechanical tests performed on samples cured via modalities 1, 2 and 3 were performed following CEN [24]. The uniaxial compression strength (*UCS*) and the indirect tensile strength (T_0) were then calculated using the following equations:

$$UCS = \frac{F_{peak}\ (\mathrm{N})}{1600\ (\mathrm{mm}^2)}\ (\mathrm{MPa}) \tag{1}$$

$$T_0 = \frac{1.5 *\ F_{peak}(\mathrm{N}) * L\ (\mathrm{mm})}{b^3\ (\mathrm{mm}^3)}\ (\mathrm{MPa}) \tag{2}$$

where 1600 mm^2 is the active surface over which the standard "compression frame" (or in jargon "comprimitor") applied the compression force; F_{peak} is the maximum value reached during the test (i.e., the force that caused breaking of the sample); and L is the distance between the two lower supports of the three-point flexural test frame (in this case, L = 100 mm). b is the length of the square of the cross-section of the sample, in this case 40 mm.

Assessment of *UCS* and T_0 was carried out after a pre-scheduled curing period. The frequency of testing was higher during the first year and was then carried out annually in years two and three.

4.2. Monitoring of the Dehydration Process via Weight Assessment

The weight lost during the dehydration process of the two-component grout was assessed using dedicated samples cured in special tanks, as described above. Three different samples were tested over time: weight assessments were performed every 5 min during the first hour of curing and then hourly over one day and daily over about 30 days. For longer curing times, weight assessments were performed weekly. This schedule was chosen based on preliminary tests that highlighted the different rates of the phenomenon over time, which was higher in the initial hours/days.

The test campaign ended after about 60 days. During the tests, the weight was assessed without removing the samples from the containers, taking care to consider always the tare weights. This precaution was essential to avoid the potential weight loss for crumbling of the samples.

The weight assessment was performed using a laboratory precision scale (p = 0.01 g).

4.3. Mechanical Tests on Samples Cured Under the Action of Air

Due to the fast deterioration process that characterized the two-component grout cured under the action of air, mechanical characterization was difficult. It should be noted that the equations given in Section 4.1 could not be directly used. T_0 was not assessed, since the cross-section of the sample was deformed and the three points of the three-point frame were not aligned. Furthermore, due to crumbling, it was difficult to move the sample over the three-point frame without damaging the sample (unlike the comprimitor used for the *UCS*, the one used for the indirect tensile tests supported the sample only at two lines of contact, corresponding to the bottom supports of the frame).

For the *UCS* assessment, Equation (1) was also unusable in its standard form. As depicted in Figure 5 (showing a sample after two weeks of curing), samples cured under the action of air tended to change in volume due to the evaporation/dehydration process. In Figure 5, the "real" cross-sections, present under a fragile and weak crust, are highlighted in red: on the left, the red rectangle identifies the longitudinal cross-section, while on the right, the transversal cross-section is shown as a red square.

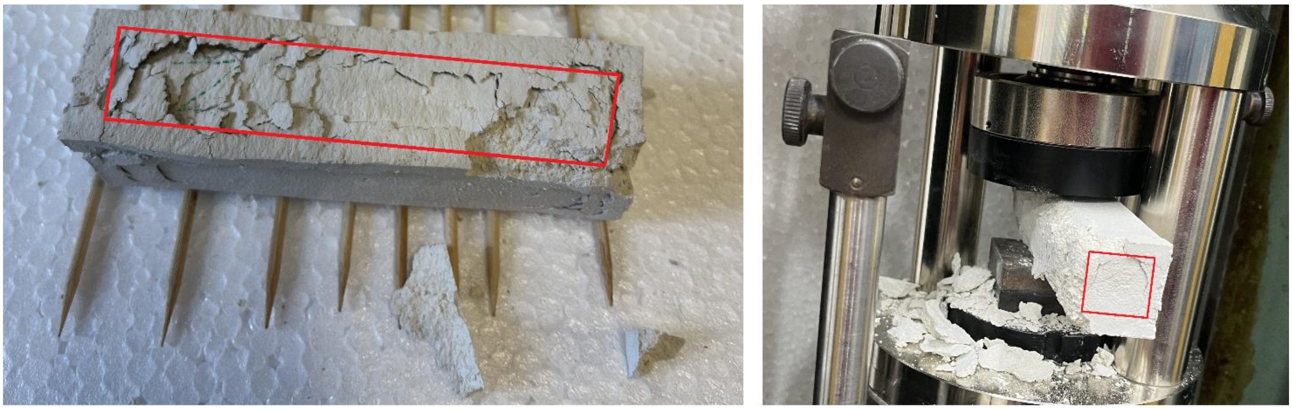

Figure 5. Example of a two-component grout sample cured under the action of air for two weeks. The "real" cross-sections (longitudinal and transversal) are shown by red lines.

Recognition of the "real" cross-sections was completed simply by testing the cortical parts of the sample that could be easily removed from the core. By the term "real", we mean the effective resistant volume of the sample, without the crumbled volume that was impaired by the action of air, and that had little strength against applied forces. We therefore decided to change Equation (1) in terms of the denominator of the expression. Equation (3) was developed as follows:

$$UCS = \frac{F_{peak}\ (\mathrm{N})}{b^{*}(\mathrm{mm}) * 40\ (\mathrm{mm})}\ (\mathrm{MPa}) \tag{3}$$

where F_{peak} is always the maximum compression force applied during the uniaxial compression test, and the value of b^* is illustrated in Figure 6 and represents the length of the new cross-section of the sample, which is able to identify the real volume that can face the compression strength.

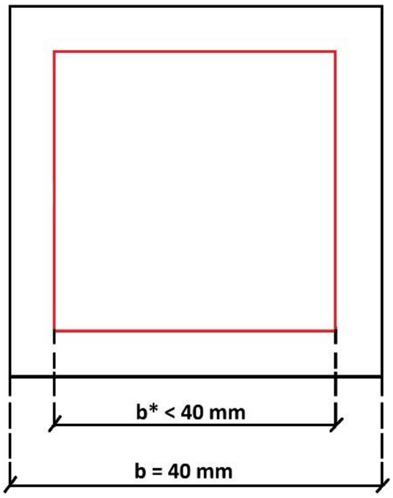

Figure 6. Difference between *b* and *b**. *b** is the length of the cross-section for the two-component grout sample after dehydration due to the action of air. The black line identifies the standard cross-section of the sample, while the red one identifies the cross-section after the shrinkage.

Although the value of *b** was fairly constant over a single analyzed sample, the value used in Equation (3) for a specific sample was an average of at least five measurements performed with a calliper. For comparison, the value of *UCS* for each sample cured under the action of air was also calculated using Equation (1), i.e., neglecting the reduction in the active surface over which the compression force was applied. The results from Equation (3) are referred to in the following as “real” (*UCS*-real), while those obtained with Equation (1) are referred to as “theoretical” (*UCS*-theo).

UCS tests on samples of two-component grout cured under the action of air were performed after 1, 3, 7 and 14 days. Due to the fast deterioration of the samples, it was not possible to continue these tests over longer curing times. To enable a comparison, a parallel campaign was carried out where samples were cured in water, following the curing modality 1 described in Section 3.2. This last set of samples was tested in compliance with CEN [24], i.e., using Equation (1), and the results are referred to in the following as *UCS*-water.

5. Results

5.1. Aging

Results pertaining to the aging test campaign are reported in Figures 7–9. Figure 7 refers to samples cured via modality 1, while Figure 8 shows the results for samples cured with modality 2 and Figure 9 shows those for samples cured with modality 3. In these graphs, each marker “x” represents the results of a particular test, while the continuous lines represent the average results for all tests carried out at a certain curing time (average function).

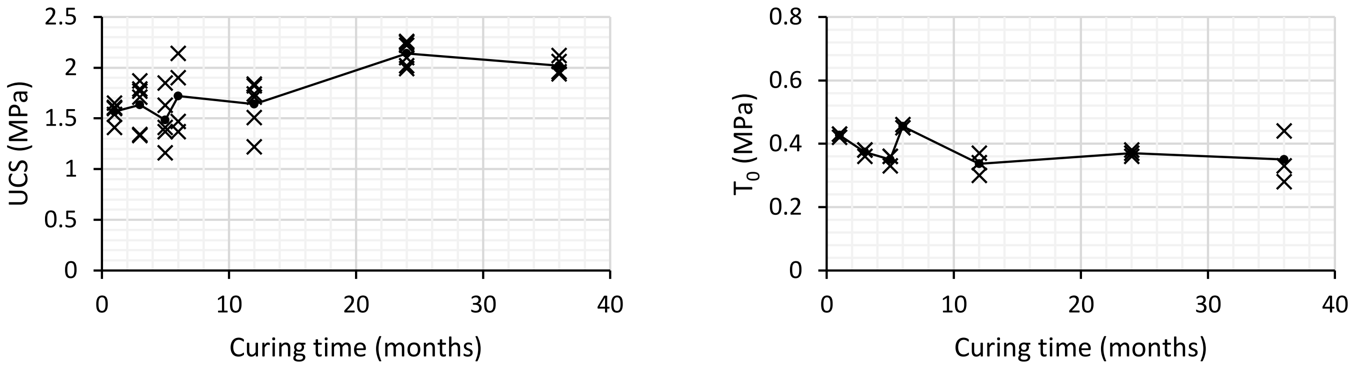

Figure 7. *UCS* (**left**) and T_0 (**right**) for samples cured via modality 1. Each "x" is a tested sample.

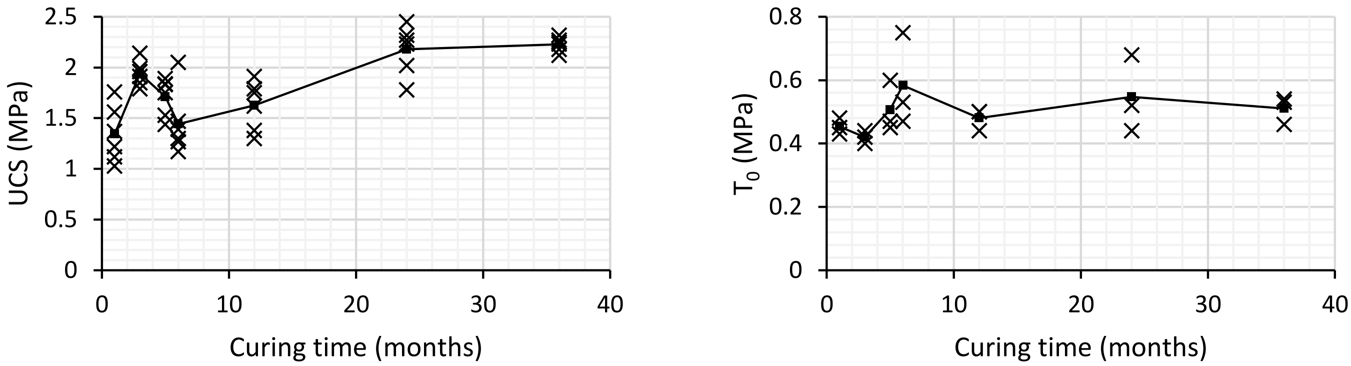

Figure 8. *UCS* (**left**) and T_0 (**right**) for samples cured via modality 2. Each "x" is a tested sample.

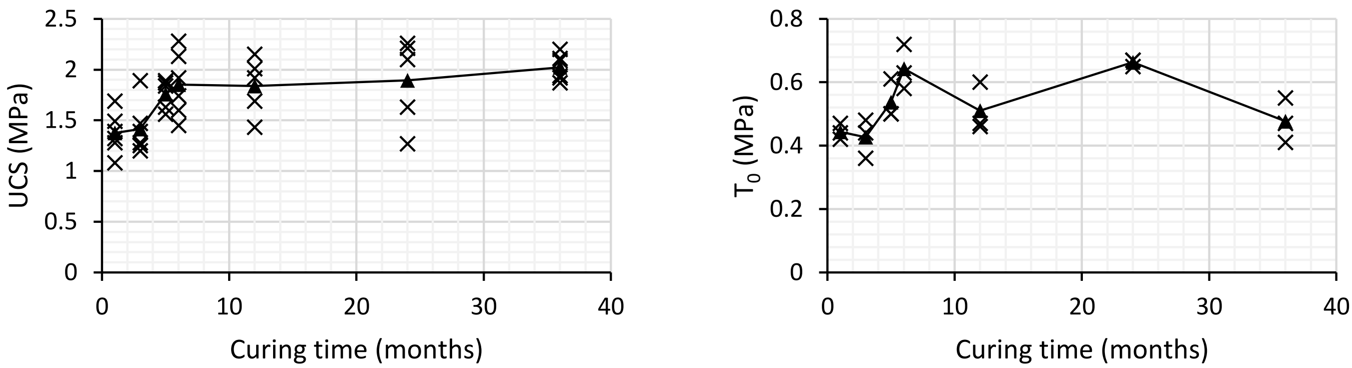

Figure 9. *UCS* (**left**) and T_0 (**right**) for samples cured via modality 3. Each "x" is a tested sample.

Table 3 shows the results of a simple statistical analysis, where "av" means average, "sd" means standard deviation, and CV is the coefficient of variation.

Table 3. Statistical analysis of the results of aging tests.

	Curing Modality	**Curing Time (Months)**													
		1		**3**		**5**		**6**		**12**		**24**		**36**	
		T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*
av (MPa)	1	0.43	1.57	0.37	1.64	0.35	1.48	0.46	1.72	0.34	1.64	0.37	2.14	0.35	2.02
sd (MPa)		0.00	0.08	0.01	0.22	0.01	0.24	0.01	0.31	0.03	0.22	0.01	0.11	0.07	0.07
CV		0.01	0.05	0.03	0.13	0.04	0.16	0.01	0.18	0.09	0.13	0.02	0.05	0.19	0.04

Table 3. *Cont.*

	Curing Modality	Curing Time (Months)													
		1		3		5		6		12		24		36	
		T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*	T_0	*UCS*
av (MPa)	2	0.45	1.34	0.42	1.94	0.51	1.71	0.58	1.44	0.48	1.63	0.55	2.18	0.51	2.23
sd (MPa)		0.02	0.25	0.02	0.11	0.07	0.17	0.12	0.29	0.03	0.22	0.10	0.22	0.04	0.06
CV		0.05	0.19	0.04	0.06	0.13	0.10	0.20	0.20	0.06	0.14	0.18	0.10	0.07	0.03
av (MPa)	3	0.44	1.38	0.43	1.41	0.54	1.76	0.64	1.85	0.51	1.84	0.66	1.89	0.48	2.02
sd (MPa)		0.02	0.19	0.05	0.23	0.05	0.12	0.06	0.29	0.06	0.23	0.01	0.38	0.06	0.12
CV		0.05	0.14	0.12	0.16	0.10	0.07	0.09	0.16	0.13	0.13	0.01	0.20	0.12	0.06

5.2. Dehydration Process

Figure 10 shows the data recorded during the dehydration tests. The average starting weight of the samples was equal to 298.33 g, and the test campaign ended when the samples had a weight close to 100 g, which was about 60 days after casting. The weight assessment was performed hourly for the first 8 h and then daily for 1 week. The acquisition frequency was later reduced since the dehydration process was less fast, and a very high acquisition frequency was considered useless. Figure 10 also shows data from the curing environment in terms of temperature and relative humidity.

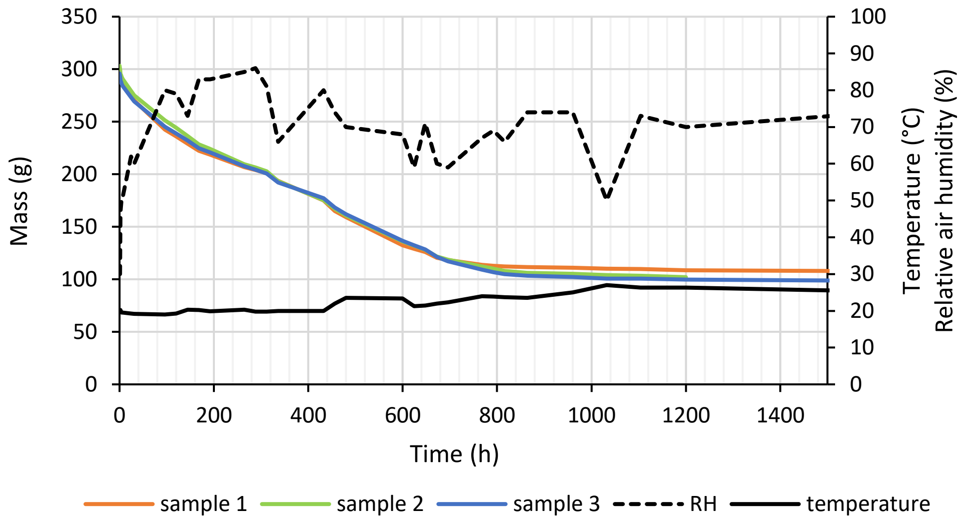

Figure 10. Results of the tests carried out to monitor weight loss.

Figure 11 shows the average trend of the weight for the three studied samples in percentages with respect to the original weight (after casting). This curve is referred to as the "bulk function", and it describes in a synthetic way the weight loss of the studied two-component grout under the action of air. The bulk function was interpolated to find an equation that could fit the experimental data in order to obtain an instrument that could predict the dehydration process. After several different simulations in which we applied both parabolic and exponential equations, it was decided to describe the two parts of the experimental function analytically by splitting it into two parts. Equations (4) and (5) provided the best description of the experimental data:

$$bulkvalue(\%) = 7 * 10^{-5}t^2 - 0.1334t + 100 \, for \; \leq t \leq 800 \text{ h} \quad (4)$$

$$bulkvalue(\%) = -29 * 10^{-4}t + 38.816 \, for \; 800 < t \leq 1512 \text{ h} \quad (5)$$

where *t* is the time in hours, and the *bulk value* (%) expresses the bulk percentage after a certain period of action of air on the two-component grout. The value of 800 h has been selected as the point of interception between the two tangents of the main 2 two parts of the average graph (Figure 11).

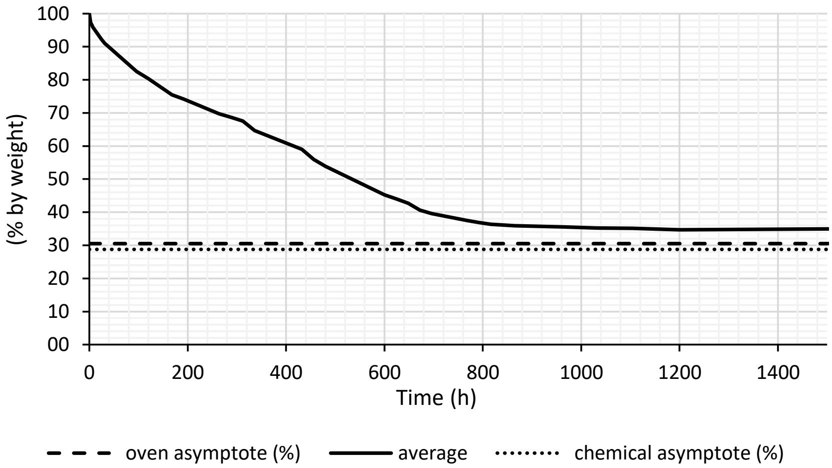

Figure 11. Bulk function for the two-component grout, describing the dehydration process.

It is important also to note that in Figure 11, to have a reference of the expected value of the final weight, two different asymptotes are shown. The first is the "oven asymptote", which is equal to 30.5% and was computed by drying a freshly cast two-component grout sample in an oven at a temperature of 80 °C for a sufficient time to guarantee the stability of the residual weight. The second is the "chemical asymptote", which was computed based on the mix design of the two-component grout and computing the weight of the water added. The chemical asymptote is slightly lower than the oven one with a value of 28.8% (meaning that a sample with the mix under study is made up of about 72.1% of water by weight). The reason that the chemical asymptote is lower is because a certain amount of water (about 2% by weight) is chemically and physically bonded to the grout, and the energy provided by the oven is not sufficient to break these bonds.

The strong sensibility of the two-component grout to the action of air described analytically in the graphs in Figures 10 and 11 can be also clearly seen from the pictures in Figures 12 and 13, which show the tested samples after one and two months of curing, respectively, under the action of air.

Figure 12. Tested samples of two-component grout after one month of curing.

Figure 13. Tested samples of two-component grout after about two months of curing.

5.3. Action of Air

The results for the mechanical characterization of samples cured under the action of air are shown in Figure 14. The images shown on the chart (from left to right) correspond to samples tested after 1, 7 and 14 days, respectively. In order to avoid overcrowding of the graph, only average values are reported. For the curing time of 1 day, values of *UCS*-theo and *UCS*-real coincide.

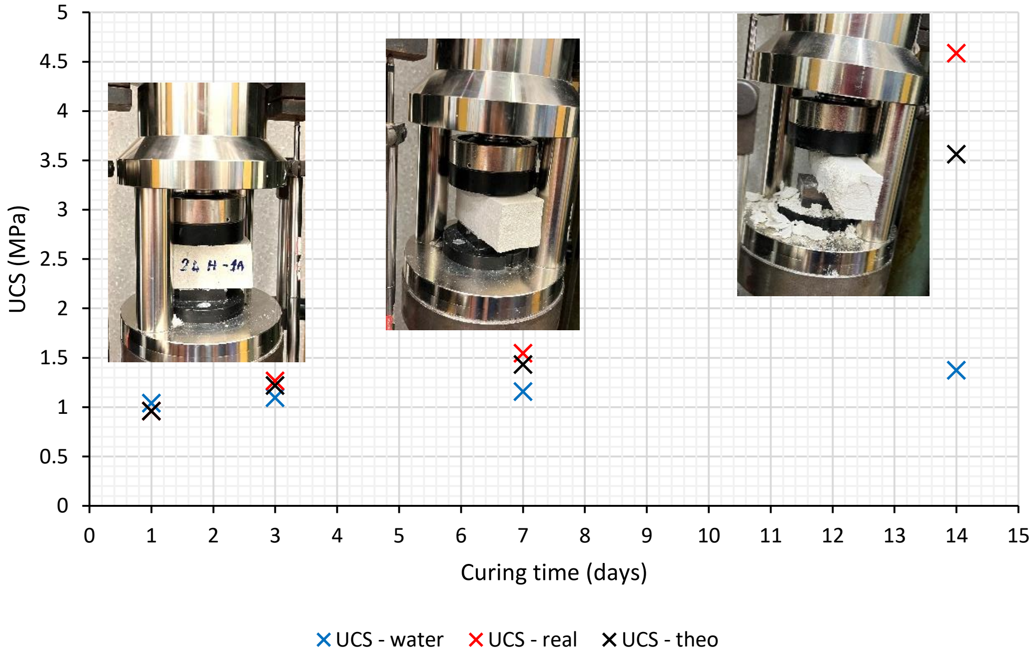

Figure 14. *UCS* results for samples cured under the action of air.

Table 4 shows the results of a simple statistical analysis, where "av" means average, "sd" means standard deviation, and CV means the coefficient of variation.

Table 4. Statistical analysis of the results of tests carried out on samples cured under the action of air.

	Curing Time (Days)											
	1			**3**			**7**			**14**		
	***UCS*—Water**	***UCS*—Real**	***UCS*—Theo**	***UCS*—Water**	***UCS*—Real**	***UCS*—Theo**	***UCS*—Water**	***UCS*—Real**	***UCS*—Theo**	***UCS*—Water**	***UCS*—Real**	***UCS*—Theo**
av (MPa)	1.04	0.96	0.96	1.09	1.27	1.22	1.16	1.54	1.43	1.37	4.58	3.56
sd (MPa)	0.02	0.10	0.10	0.08	0.20	0.19	0.15	0.21	0.17	0.06	1.76	1.25
CV	0.01	0.10	0.10	0.07	0.16	0.16	0.13	0.14	0.12	0.04	0.38	0.35

6. Discussion

Before proceeding with a discussion of the results, it is important to provide some details of the statistical analysis performed here. It should be noted that in the available scientific literature on the mechanical characterization of two-component grout, information from statistical analyses is rarely reported. The reason for this is associated with the sensitivity of the material, as it is very susceptible to the procedures of casting and curing [26]. Consequently, a simple analysis based on the standard deviation for the two-component grout is not sufficient. In fact, for this material, the standard deviation can be averagely higher than that one related to other cement-based materials, giving the wrong perception of a not accurate set of outcomes. Furthermore, a simple analysis based on the standard deviation is unsuitable for comparing the results presented by different authors. For these reasons, the main parameter chosen for the statistical analysis in this work was the CV, i.e., the ratio between the standard deviation and the average value.

For curing modality 1 (Figure 7), the *UCS* showed an overall increase, reaching 2 MPa after about 24 months of curing. At curing times of 5 and 12 months, an inversion of the average function can be observed. The results for 6 months of curing were more scattered (CV = 0.18), but all outcomes showed CV values lower than 0.2. With regard to T_0, we note that the average function does not show an increasing trend. The values of this function are in the range 0.34 to 0.46 MPa, and the global trend can be assumed to be constant. Pertaining to the precision, if the results for a curing time of 36 months are excluded (CV = 0.19), the CV is lower than 0.1, showing a higher precision than in the *UCS* test campaign. The higher dispersion obtained at 36 months of curing could be correlated to samples not perfectly casted. The inversion of the trend at a curing time of five months (both for *UCS* and T_0) may be related again to some casting or curing problem related to this set of samples. It is important to note that the ratio UCS/T_0 also shows an increasing trend from about 3.7 after one month to about 5.8 at 36 months.

For curing modality 2 (Figure 8), the *UCS* average function shows an increasing trend, although a fluctuation can be seen after 3 months of curing. However, the corresponding CV for this curing time is 0.06, and it can therefore be stated that the results are close to the average value. The results for the accuracy at 36 months are higher (CV = 0.03), while those at 6 months are less accurate (CV = 0.2). On average, the values of CV for this *UCS* test campaign were equal to or lower than 0.2. For this curing modality, a value of 2 MPa for the *UCS* was reached after about 24 months of curing. For T_0, the average function was obtained from rather scattered values compared to those for curing modality 1, although the maximum value of CV was close to 0.2. However, also in this case, an increasing trend was not seen: the function domain ranged between 0.42 and 0.58 MPa, which is slightly higher than for curing modality 1. UCS/T_0 also showed an increase with a value of 3.0 after 1 month and 4.4 after 36 months.

For curing modality 3 (Figure 9), in the same way as for the other two cases, the average function showed an increasing trend, and there are no outliers in the chart that should be mentioned. However, the data were more scattered. Except at 5 and 36 months (CV = 0.07), all of the values of CV ranged between 0.13 and 0.20. The lowest quality of data was seen for 24 months of curing, with CV = 0.2, equal to 20% of the corresponding average *UCS*. Unlike the other curing modalities, a value of 2 MPa was reached only after 36 months. For T_0, the average function ranged between 0.43 and 0.66 MPa without a clear increase over time. The maximum value of CV was 0.13. The ratio UCS/T_0 started at about 3.1 after 1 month and reached more than 4.0 after 36 months.

Concluding, with reference to the whole aging test campaign, the curing condition that provides better results cannot be recognized univocally. Considering in fact the *UCS*, samples cured with modality 1 provide the highest values for the curing time for 2 years (close to the value of samples cured with modality 2), while the lowest values are obtained for curing times of 6 and 12 months. Conversely, as concerning T_0, curing modality 1 provides the worst result. This analysis pointed out once again the difference between the standard concrete and the two-component grout since (for standard concrete, the curing modality 1 is likely the one that maximizes the final achievable strength).

With regard to the dehydration process, it can be seen from Figure 10 that the three trends corresponding to the three tested samples are very similar. From the charts in Figure 10, it can be observed that in the complete absence of an embedding medium for the two-component grout, the material suffers strong dehydration and is completely cracked and almost completely reduced to powder. More specifically, the material begins to lose water immediately after casting, which continues up to about 1000 h. The curing environment experienced fluctuations in terms of temperature and relative humidity (19 < T (°C) < 27 and 30 < RH (%) < 86), but these variations did not seem to affect the dehydration curves. An analysis of the bulk functions and asymptotes in Figure 11 provides us with further interesting information pertaining to the dehydration process. The bulk function tends toward the oven asymptote without reaching it; in fact, the experimental curve becomes quite flat after about 1200 h of curing at a bulk value of about 35% (5% higher than the oven asymptote). It can be speculated that at $t \rightarrow \infty$, the bulk function will not reach the oven asymptote, because part of the water is chemically bonded to the cement and other ingredients and part of the water is adsorbed by the material. In conclusion, the real asymptote of the bulk function can be predicted as between 30.5% (the oven asymptote) and 35%. With regard to the chemical asymptote, Figure 11 shows once again that water is the main element of the two-component grout (more than 70% by weight). However, as a last remark, it should be noted that the real chemical asymptote is lower than the one reported on the chart. The value of 28.8% should be further reduced considering the water in the accelerator. Component B also contains water, but this amount was not considered in this computation, since the composition of component B is a trade secret.

On the basis of the results obtained here, it can be concluded that the two-component grout is very sensitive to the dehydration process. Indeed, if there is no embedding medium and air can act on the grout, a few days are sufficient to cause destruction of the material. This result supports the theory proposed by Pelizza et al. [27] and Peila et al. [21]. Furthermore, the obtained bulk function is an important instrument that can be used to estimate the quantity of water lost as a function of time. It should be noted, however, that Equations (4) and (5) are valid only for the studied mix design and specific environmental conditions, although the trends could be considered valid for all standard two-component grout material.

For the mechanical characterization tests performed on samples cured under the action of air, the quality of the results was higher than for the aging test campaign with the exception of a curing time of 14 days. The values of CV were lower than 0.16 in each case, except for a curing time of 14 days, where the results were more scattered (CV close to 0.35). The reason for this higher dispersion is due to the poor condition of the samples, which are strongly fractured as a consequence of the dehydration process. The results for 1 day of curing for *UCS*-real and *UCS*-theo have the same value because of a very low dehydration process (about 7% according to Equation (4)). The functions in Figure 14 show an increasing trend, but it is notable that samples cured without water exhibit higher values of *UCS* than the ones of samples cured in water irrespective of whether Equations (1) or (3) are used. If, after 24 h, values of *UCS* are all similar and close to 1 MPa, at 3 days of curing, the *UCS*-real is higher than the *UCS*-water of 16%, and this percentage is set to increase, reaching values of 34% and 234% after 7 and 14 days, respectively. Due to these unexpected results, the parameter *UCS*-theo was introduced (the aim was to remove the potential influence of mistakes in the assessment of b*), but the values for *UCS*-theo were 11%, 24% and 159% higher than those for *UCS*-water for 3, 7 and 14 days of curing, respectively. For completeness, it should be noted that the value for *UCS*-water after 28 days of curing was found to be close to 1.6 MPa (the same order of magnitude as samples cured for only seven days under the action of air).

On the basis of other works on two-component grout, values for the CV between 0.2 and 0.4 are commonly accepted [8,26]. In this research, a value of 0.2 was never exceeded, showing a good quality of the data. In regard to the aging tests, we note that irrespective of the curing modality, two-component grout cannot be considered fully cured after 28 days, since the *UCS* continues to increase over time up to 36 months. The samples cured according to modality 2 (i.e., with a lower water content of the sand) did not suffer from a lack of water as expected. This is a very important result, since it has been experimentally proved that a low water content (5%) of the soil that embeds the two-component grout is sufficient for correct curing of the material and good mechanical performance, according to the mix design. Lower moisture content could, however, be sufficient to ensure a good curing, but a specific test campaign should be developed. The other curing modalities (1 and 3) also exhibited an increasing trend in *UCS* over time. The order of magnitude of the values of *UCS* reached was similar irrespective of the curing modality. For the tensile strength, we note that the average functions show a trend which does not show an increase. Two hypotheses can be proposed: (i) T_0 is independent of the curing time and depends only on the mix design, or (ii) the three-point-flexural test is not suitable for assessing the trend in the tensile strength of the two-component grout.

Finally, with regard to the UCS/T_0 ratio, we observed that irrespective of the curing time and modality, the values were always smaller than 10, representing another significant difference from standard concrete. All the considerations discussed above are valid only if curing takes place in a soil with a water content equal to or higher than 5%; if the two-component grout is cured under the action of air instead, a dehydration process occurs. The magnitude of this phenomenon is significant, and it cannot be absolutely compared with that of the standard mortar or concrete. Stockholders in mechanized tunneling need not be concerned about the results reported here for the dehydration process, since on a construction site, the possibility of direct contact between air and the backfilling grout is remote. It should be in fact remarked that the choice of testing the curing under the air action was taken in order to maximize the stress on the grout in a reasonable time-span. However, the evidence presented here should be considered a "red flag" in terms of durability, since on a construction site where a tunnel could be built in soil with a water content of close to zero, or in cases where the two-component grout is exposed to air due to niches or bypasses excavation, special care should be taken with regard to the issue of durability. In this case, the possibility of cracking and a reduction in the volume of the injected two-component grout should be taken into account when the mix design is set up. However, despite the reduction in volume, the *UCS* is positively affected by curing for up to 14 days under the action of air. The reason for this strong increase in *UCS* can be speculated in the variation in the water/cement ratio (w/c) of the grout, as summarized in Table 5 and Figure 15.

Starting from a value of 3.71 (from the original mix design), a reduction of about 50% is seen after 14 days of curing, with the decreasing trend shown in Figure 15 (function of Equation (4), i.e., the water lost). It can be assumed that although dehydration under the action of air has the disadvantage of cracking and crumbling of the material, it has also a positive effect in terms of the reduction in w/c, which causes an increase in *UCS*. A last comment pertaining to the shrinkage phenomenon and the *UCS* is due. It is important to report that *UCS* alone is not sufficient to ensure the good quality of a two-component grout but also the shrinkage should be taken into account to characterize the backfilling material. A good *UCS*, that satisfies the technical specification of a construction site, is not enough to tell that a two-component grout is suitable: it could lose water and increase its shrinkage but at the same time increase *UCS* in time. So, the *UCS* assessment should be always correlated with a shrinkage assessment especially in non-standard curing environment.

Table 5. Water–cement reduction as a function of the curing time under the action of air.

Curing Time (Days)	w/c	Decrease in w/c (%)
0	3.71	/
1	3.31	10.74
3	3.09	16.54
7	2.43	34.41
14	1.87	49.53

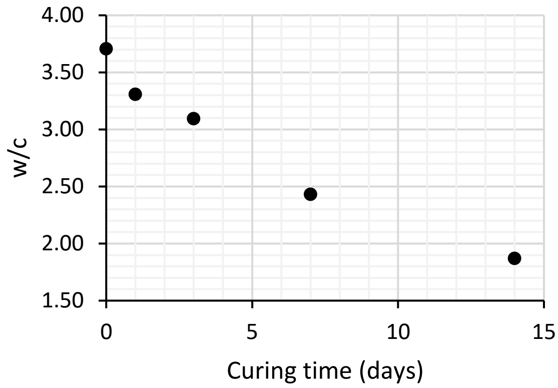

Figure 15. Water–cement ratio as a function of curing time under the action of air.

7. Conclusions

Two-component grout is currently the most commonly used material for backfilling, but no studies of its durability have ever been carried out. In this research, concerning the aging and the air action, we found the following:

- A minimum value of 5% guarantees correct curing of the grout with a slow but continuous increase in *UCS* over time; it should be also reported that regarding the state of the art, it is not possible to exclude that also curing modalities with lower water content could ensure a suitable curing;
- The increase in *UCS* despite the progressive destruction of the samples highlights the importance of assessing the shrinkage of the material; the evidence presented in this work should encourage stockholders involved in mechanized tunneling to reconsider the common acceptance tests for two-component grout.

In conclusion, particular care should be taken on construction sites where tunnels are built in dry geological formations; the performance of the grout could be affected by the dry environment, and a durability test campaign is strongly suggested with optimization of the mix design in order to handle the difficult curing conditions. It should also be reported that the elastic properties of the grout and the shear strength could be also affected by the curing condition as well as the *UCS*. These parameters have not been considered in this first work on durability but the research is still ongoing and is also focused on them.

Author Contributions: Conceptualization, C.T.; methodology, C.T. and A.C.; validation, C.T., D.P. and A.C.; formal analysis, C.T.; investigation, C.T. and A.C.; data curation, C.T. and A.C.; writing—original draft preparation, C.T.; writing—review and editing, C.T. and A.C.; supervision, D.P.; funding acquisition, D.P. All authors have read and agreed to the published version of the manuscript.

Funding: This work was developed at the Department of Environment, Land and Infrastructure Engineering (DIATI) in the frame of the "Department of Excellence" on Climate Transition (2023–2027).

Data Availability Statement: Research data are available on request.

Acknowledgments: The authors want to thank Utt Mapei for having supplied the chemical ingredients and for the given technical support. They also thank Buzzi Unicem for the cement supplied.

Conflicts of Interest: The authors declare no conflict of interest.

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