standards

Review

Standards and Assessment of Construction Products: Case Study of Ceramic Tile Adhesives

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Abstract: This work attempts to draw attention to the importance of a multidimensional approach when creating standard requirements in the assessment of construction products with the example of ceramic tile adhesives (CTAs). CTAs are an essential group of building materials today, the continuous development of which has been noted since the 1960s. However, until 2001, i.e., the year when EN 12004 was published, there were no precise requirements for CTAs at the European level, which often made it difficult or, in extreme cases, even impossible to assess the product objectively. Under the provisions of EN 12004, for twenty years, the basis for the assessment and verification of constancy of performance (AVCP) of CTAs has been adhesion determined by tensile strength. The paper discusses the test methods, paying attention to their imperfections, including the impact of the materials used in measurements, i.e., concrete slab, ceramic tile, and water quality. The results of the multi-annual interlaboratory tests indicate that an essential factor that must be considered in the process of AVCP is test measurement uncertainty. Additionally, it should be remembered that uncertainty also occurs at other assessment levels. It also seems that the simple acceptance rule that does not consider the variability resulting from the measurement uncertainty is inadequate when assessing CTAs.

Keywords: ceramic tile adhesive; measurements uncertainty; assessment of construction products; tensile adhesion strength; market surveillance

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1. Introduction

The principles of the construction products market in the European Union are regulated by the Construction Products Regulation (CPR) [1]. CPR ensures the free movement of construction products within the EU, taking into account the interests and needs of the Member States. This document lays down harmonized rules for the CE marking of construction products and defines the method of declaring their performance with regard to the essential characteristics of construction products. The requirements set out in the CPR are necessary to ensure that reliable information is available to professionals, authorities, and consumers and to allow for the construction products to be compared between manufacturers in the different Member States. A fundamental part of the functioning of the EU market of products is standardization which facilitates the development and maintenance of common technical terminology. Standardization is a link between the requirements of the Member States and the Declaration of Performance (DoP) issued following CPR. The manufacturer prepares DoP for the user of the product. The European system of technical regulations created by CEN, CENELEC, and EOTA is a factor that stimulates competition and innovation while contributing to improving consumer safety and reducing the number of accidents, making European standards a global benchmark [2].

The foundations of standardization are consistency, transparency, openness, consensus, independence from special interests, and efficiency [3]. In principle, the European standards are driven by business and made through a balanced process involving relevant stakeholders. In the EU, standards support market-based competition and help to reduce costs [4]. European standardization is an integral part of supporting global competitiveness, and therefore, the aspect of cooperation with international standardization bodies, namely ISO, IEC, and ITU, is also essential.

The economic landscape of the EU and its international trading partners is still changing, taking on a rather dynamic character in some areas, and therefore it is crucial to adopt standardization processes to these changes. It is essential to consider the verified achievements of science and technology. In Strategy 2030, CEN and CENELEC intend to strengthen their position as independent facilitators between industry, regulators, consumers, and other stakeholders and to ensure that the European standards contribute to the competitiveness and sustainability of the European economy [5]. Recently conducted surveys have shown that the internal market and its functioning is a topic of interest, with stakeholders asking for better coordination from the EU and harmonization across the EU on building standards and overall demanding better regulation of the construction sector [6].

Construction is a branch of the economy and a scientific discipline with specific interrelationships and conditions between science and practice [7]. In construction, a vast number of various building materials are used, which impact the fulfillment of the basic requirements specified in the CPR. Every construction product must be assessed before it is placed on the market [1]. In the case of CE marking, the manufacturer is obliged to assess and verify the construction product's constancy of performance (AVCP) [1]. Due to the variety of construction materials and their intended use, the assessment is carried out at very different levels in countless laboratories worldwide. In theory, as a rule, measurement processes must be unambiguous so that metrologically consistent results are achieved when reproduced anywhere in the world. However, the reality is much more complicated, to mention the situation where the performance values of the product determined in the tests are close to the evaluation criterion. In such a situation, the manufacturer must be aware of the risk associated with the fact that in the control procedures of construction products placed on the market or during periodic inspections, his product may be considered as non-conforming [8]. For some construction products, assessment is more complex due to the products' nature which requires appropriate preparation before use, e.g., mixing with water. For this reason, assessment by market supervision authorities is also multifaceted, i.e., the formal assessment of the DoP and testing of samples of products taken from the market. For this reason, it is crucial to answer the following question: To what extent is the assessment of a construction product based on tests reliable [9]. Another dimension of product evaluation is the relationship between the results of laboratory tests and the actual behavior of the product during its subsequent use, especially in the case of construction materials for which destructive testing methods are used. In the case of using semi-destructive methods to assess construction products and especially non-destructive testing methods, the situation is less complex. For this reason, there has been a significant development of non-destructive testing in recent years for the evaluation of materials in civil engineering. [10]. All the accounts mentioned above present quite a scientific challenge. It is impossible in the EU system with CE marking to carry out the product assessment process without knowing the requirements specified in the harmonized standard or the European Technical Assessment [1].

This review overviews the changes in the assessment and verification of the performance of ceramic tile adhesives (CTAs) in the EU over the past twenty years. Due to the dominant market share of cementitious CTAs, these adhesives constitute the subject of a detailed analysis of the three types of CTAs. Following the requirements of the EN 12004 standard established in 2001, the basis for the assessment and classification of cementitious CTAs is adhesion, which is determined by the tensile strength and the open time determined by adhesion strength after a given time after which the ceramic tile is embedded. The paper presents the measurement methods in detail and discusses their imperfections, including the impact of the materials used in measurements, i.e., concrete slab, ceramic tile, and water quality. The results of many years of interlaboratory comparisons (ILCs) and conclusions from laboratories and producers of CTAs are discussed. It has been pointed out that the knowledge of uncertainty, particularly on the uncertainty resulting from tensile adhesion strength measurement uncertainty (MU), is fundamental when assessing product compliance. The activities of construction supervision authorities in the field of CTAs collected from the market and the conclusions drawn from them for a possible amendment of the requirements and test methods specified in EN 12004, taking into account the achievements of science and technology in the past twenty years, are discussed.

2. Ceramic Tile Adhesives and Their Importance in Construction Today

When discussing CTAs, it should be noted at the outset that there are three types of products, namely cementitious, dispersion-based, and reaction resin CTAs. This division follows the chemical nature of CTA's binders. The division into three types of CTAs is also related to their application properties and final performance. Of the three types of CTAs, cementitious CTAs account for the dominant market share, and the paper is limited to the study of these three CTAs. Currently, CTAs are produced in modern dry-mix mortar plants, in which individual components (raw materials) are stored in silos. Then, they are weighed and transferred to a high-speed blending mixer, packed, and transported to points of sale or directly to the construction site [11]. Before use, the addition of only water and mixing according to the manufacturer's instructions is required. Since the CTA production process is well defined, automatized, and computer-controlled, the resulting construction product is of a well-defined quality and has specific performance characteristics [11,12].

A breakthrough in the development of cementitious CTAs was the receipt of redispersible polymer powder in the 1950s, which represents significant changes in the economy of Western Europe in the 1970s related to the fuel crisis and a substantial increase in labor costs, and the economic transformation of Central and Eastern European countries in the last years of the 1990s. Although it is debated, the CTA market size was valued in 2018 at USD 15.08 billion [13] or USD 14.38 billion in 2020 [14]. The global CTA market is expected to reach USD 20.17 billion by the end of 2026 [14] and in very optimistic forecasts, which from today's perspective seem unlikely, it is expected to reach 40.73 billion by 2026 [13].

CTAs, although their purpose is always to connect the ceramic tile with the substrate, have different properties depending on many factors. Due to technical criteria, modifying the CTA with a redispersible polymer powder is essential. Redispersible polymer powder is a necessary component of CTAs, which helps them to meet today's expectations and to fulfill higher requirements [11,12,15,16]. In 2020, the redispersible polymer powder market was valued at USD 2.0 billion and projected to grow to 2.8 billion by 2025. The market growth is expected to be primarily driven by the growing economies of the Asia Pacific region (China, India, Japan) [17]. Another criterion that distinguishes CTA is geography, the relationship between the economic level of a given world region, and the technical standards applicable there. It is also visible from the perspective of scientific publications [12,18].

In 2020, the global consumption of ceramic tiles was 16,035 billion square meters. As many as 71.5% of the ceramic tiles were installed in Asia, while in Europe the share was 10.0%, for Central-South America—7.8%, Africa—7.0%, and North America—3.4%. In Europe, 65% of the consumption of ceramic tiles was attributed to European Union countries, which represent 1.035 billion square meters [19]. Assuming the consumption of 4 kg of CTA per square meter of ceramic tile, this means that just over 64 million tons of CTAs was consumed in the world and just over 4 million tons of CTAs was consumed in the EU.

3. Assessment and Verification of the Constancy of the Performance of the CTAs

3.1. European Standard EN 12004

Until 2001, CTA requirements for the AVCP did not exist at the level of all EU countries. This situation made it difficult for the investor and contractor to select the appropriate CTA and made it difficult and often impossible to assess the CTA in comparison to other CTAs objectively. The lack of uniform evaluation criteria resulted in different requirements for the same essential characteristics.

A new era in the standardization of CTAs came after EN 12004:2001 [20] was developed in CEN/TC 67. Following the work of CEN/TC 67, and in principle CEN/TC 67/WG 3, the standard requirements for CTAs presented other editions, i.e., EN 12004:2001+A1:2002/AC:2002, EN 12004:2007, EN 12004:2007+A1:2012 and EN 12004-1:2017. The last standard EN 12004-1:2017 has not yet been published in the list of European harmonized standards [21]. For this reason, the basis for AVCP is EN 12004:2007+A1:2012 [22], which is the last of the versions mentioned above of the standards, specified in the list of harmonized European standards published in the Official Journal of the EU [23]. Although the CPR rules have been in force for the last eight years [1], EN 12004:2007+A1:2012 is a standard from the old legal order, i.e., from the period of Directive 89/106/EEC [24].

The EN 12004 standard, in addition to cementitious adhesives, also applies to dispersion adhesives and reactive resin CTAs. It is worth mentioning that the EN 12004 standard divided cementitious CTAs into the following two main classes: with basic properties, marked as C1, and with enhanced parameters, marked as C2. Table 1 shows the requirements for cementitious CTAs under EN 12004:2007+A1:2012.

Table 1. Requirements for cementitious CTAs according to EN 12004:2007+A1:2012 [22].

Fundamental Characteristics		
Characteristics	**Requirement**	**Test Method**
Normal setting adhesives (C1)		
Initial tensile adhesion strength	≥0.5 N/mm^2	8.2 of EN 1348
Tensile adhesion strength after water immersion	≥0.5 N/mm^2	8.3 of EN 1348
Tensile adhesion strength after heat aging	≥0.5 N/mm^2	8.4 of EN 1348
Tensile adhesion strength after freeze–thaw cycles	≥0.5 N/mm^2	8.5 of EN 1348
Open time: tensile adhesion strength	≥0.5 N/mm^2	EN 1346
Fast setting adhesives (C1F)		
Early tensile adhesion strength	≥0.5 N/mm^2	8.2 of EN 1348
Open time: tensile adhesion strength	≥0.5 N/mm^2	EN 1346
All other requirements as in Table 1a		
Optional characteristics		
Special characteristics		
Slip	≤0.5 mm	EN 1308
Extended open time: tensile adhesion strength	≥0.5 N/mm^2	EN 1346
Deformable adhesive: transverse deformation	≥2.5 mm and <5 mm	EN 12002
Highly deformable adhesive: transverse deformation	≥5 mm	EN 12002
Additional characteristics (C2)		
High initial tensile adhesion strength	≥1.0 N/mm^2	8.2 of EN 1348
High initial adhesion strength after water immersion	≥1.0 N/mm^2	8.3 of EN 1348
High tensile adhesion strength after heat aging	≥1.0 N/mm^2	8.4 of EN 1348
High tensile adhesion strength after freeze–thaw cycles	≥1 N/mm^2	8.5 of EN 1348

It is also important to note that there are requirements for the transverse deformability of CTAs. According to the data presented in Table 1 (special characteristics), it is possible to distinguish two types of cementitious CTAs—S1 (deformable) and S2 (highly deformable).

The last version of EN 12004-1:2017 additionally specified requirements for fast setting cementitious CTAs with increased parameters (C2F). These requirements are presented in Table 2. A separate specification of requirements for fast setting cementitious CTAs, for which requirements were already in the earlier version of the standard (EN

12004:2007 + A1:2012), resulted from the need to clarify misunderstandings in the interpretation of the provisions of the previous version (EN 12004:2007+A1:2012).

Table 2. Requirements for cementitious CTAs according to EN 12004-1:2017 [21].

Optional Characteristics		
Characteristics	**Requirement**	**Test Method**
	Fast setting adhesives (C2F)	
Early tensile adhesion strength	$\geq$1.0 N/mm^2	8.3 of EN 12004-2:2017
Open time: tensile adhesion strength	$\geq$1.0 N/mm^2	8.1 of EN 12004-2:2017

All other requirements as in Table 1d of EN 12004-1:2017.

ISO/TC 189 adapted the requirements of EN 12004:2001 and EN 12002:2002 [25], and a series of four ISO 13007 standards specifying requirements and test methods for CTAs and grouts was developed and published in 2004. Since ISO was and still is a global standardization organization, the terminology and technical requirements for CTAs initially published in EN 12004:2001 due to ISO 13007-1:2004 [26] have become commonplace worldwide. ISO last reviewed and confirmed the provisions of the ISO 13007-1:2014 standard in 2019 [27].

3.2. Testing of CTAs

The adoption of tensile adhesion strength and open time (maximum time after CTA application when ceramic tiles can be embedded in the adhesive layer to obtain the tensile adhesion strength value equal to 0.5 N/mm^2 (C1) or 1.0 N/mm^2 (C2)) as the fundamental characteristics of CTAs was criticized [15,28]. It was noted that the shear stresses in the substrate—CTA—ceramic tile system (parallel shear force) would better characterize the system compared to the vertically acting force when determining the tensile adhesion strength. However, the difficulties of shear strength determination are incomparably more significant than the relatively simple task of tensile adhesion strength measurements. For this reason, the shear strength is not normally determined for cementitious tile adhesives.

CTA adhesion measurement is performed after the adhesive has been stored in four different conditions, which simulate the real-life conditions in which the cementitious CTAs are applied. Nevertheless, it is essential to underline that conditioning according to EN 12004 does not always provide information about the long-term service life performance of CTAs. For this reason, there are postulates and proposed methods of assessing the long-term performance of CTAs [29,30]. However, the subject of this review is the assessment of EN 12004. At this stage of the review, it is also important to mention that the determination of the tensile adhesion strength, similar to other measurement methods, is related to the dispersion of the obtained results, which will be discussed in more detail later in this paper [31,32]. Therefore, it is crucial to determine the influence of various factors related to the test method.

An experiment consisting of determining the initial adhesion strength of seven different CTAs in ten laboratories using two other concrete slabs was described by Felixberger [15]. The study organizer provided one of the concretes slabs, and each participating laboratory provided the second. Both concrete slabs were compliant with EN 1323:2007 [33]. As a result of the tests, it was found that the concrete slab affected the value of the determined CTA adhesion. It was also found that for cementitious CTAs characterized by lower tensile adhesion strength values, the differences between individual measurements were more significant than in the case of CTAs with higher adhesion.

The properties of ceramic tiles used for tensile adhesion strength measurements are specified in EN 1348:2007 [34]. Niziurska determined the tensile adhesion strength of CTA using ten different ceramic tiles [35]. All of the tested ceramic tiles met the requirements of EN 1348:2007, and all other materials used in the study were the same. Significant differences in tensile adhesion strength were observed, which did not correlate with the results of the phase composition test, the study of the water absorption of the ceramic tiles, and the observations of the microstructure of the surface of the ceramic tiles. Two years later, in the same laboratory, the influence of the water used for seasoning the samples on the tensile adhesion strength of six different CTAs was tested [36]. Samples of CTAs were stored in three types of water, including distilled water (pH = 7.09, specific conductivity = 0.040 mS/cm), tap water (pH = 8.25, specific conductivity = 0.805 mS /cm) and softened water (pH = 8.63, specific conductivity = 1.228 mS /cm). It was found that the type of water used for seasoning samples has a great influence on the adhesion of CTAs (differences between the average tensile adhesion strength values ranging from 16% to 66% in the case of one of the CTAs were observed). Samples stored in distilled water were characterized by higher adhesion than those stored in tap water or softened tap water.

3.3. Interlaboratory Tests of CTAs

From the perspective of the manufacturer responsible for the construction product placed on the market, it is crucial to reproduce the results, i.e., test results of the same product obtained using the same method in different laboratories by different operators using other equipment. Each laboratory needs to confirm its competence independently, which enables their participation in proficiency testing (PT). It is one of the three types of interlaboratory comparisons (ILCs) in which a much larger population of commercial (manufacturer) laboratories can take part. The primary purpose of PT is to check the homogeneity of the pool of results obtained for a specific product and determine what part of the results fall [37]. Requirements for the competencies of the organizers and the development and implementation of PT are set out in EN ISO/IEC 17043:2010 [38].

In 2007, the Romanian laboratory Ceprocim (notified laboratory in the scope of EN 12004) started PT of CTAs. Nine laboratories, most of them Romanian, participated in the first edition of the project in 2008–2009 [39]. Twenty-seven laboratories from nine countries (Austria, Bulgaria, Croatia, the Czech Republic, Germany, Poland, Portugal, Romania, and Slovenia) participated in the fifth edition [40]. Twenty-seven laboratories from Austria, Germany, Greece, Italy, Mauritius, Poland, Portugal, Republic of Moldova, Romania, Slovenia, Spain and the United Arab Emirates participated in the last edition, the twelfth, of PT [41]. Participants of all editions of PT represented both accredited and non-credited laboratories according to EN ISO/IEC 17025 [42]. The *z*-score analysis showed that more than 90% of the test results obtained by participating laboratories could be described as "satisfactory" according to EN ISO/IEC 17043. The remaining effects were questionable or unsatisfactory in some editions [39–41]. Twelve editions of the PTs unambiguously demonstrated that constant participation in laboratory PT programs improves the quality of the work of laboratories. In this respect, the organizers of the study achieved the intended goal [39–41]. However, this evaluation was performed from the perspective of the requirements of EN ISO/IEC 17043, i.e., a standard which specifies general requirements for the development and implementation of PT. These requirements are intended for application to all PT programs for different technical requirements in other application areas. Additionally, in this respect, PTs [37,43] are primarily assessed. For PT measurements organized by Ceprocim, among the laboratories classified as "satisfactory", some provided 1.3 N/mm^2 and some obtained the result of 2.4 N/mm^2. From the producer's perspective, these are significant differences. Such significant differences can mean that the product is non-compliant with requirements in many cases. The results of PTs are not satisfactory from the perspective of manufacturers of CTA. Manufacturers of CTAs, who are aware of the "imperfections" of the tensile adhesion strength measurement, have to consider it in their risk analysis. Otherwise, it CTAs may not meet the acceptance criteria in re-assessments of the product conducted by market surveillance authorities [44,45]. Additionally, market surveillance authorities in Poland apply the simple acceptance rule when assessing a construction product, which does not consider the variability resulting from measurement uncertainty [45].

4. Conclusions

The EN 12004 standard was a crucial element in organizing the EU CTAs market. Following the adoption by ISO of the classification and test methods specified in EN 12004, the requirements for CTAs were harmonized worldwide.

The research on the influence of auxiliary materials (concrete slabs and ceramic tiles) used in the tensile adhesion strength measurement showed that they significantly impact the result. In some cases, they may determine whether or not the obtained value meets the acceptance criteria. This situation is unsafe for the manufacturer when the actual properties of the CTA do not differ much from the threshold value, which is the basis for the product evaluation. Each producer that places a product on the market is exposed to the risk that the product assessed by market surveillance authorities will not be positively verified. For this reason, it is essential to be aware of the uncertainty value in the study of a given characteristic. Considering the variability resulting from measurement uncertainty in the risk analysis, the manufacturer can minimize the risk of a negative assessment by market surveillance authorities.

The results of the multi-annual ILCs/PTs of CTAs have shown that, on the one hand, laboratories participating in the PTs obtain primarily satisfactory results. Undoubtedly, continuous participation in PTs increases laboratory competence. On the other hand, acceptable differences from the perspective of the testing laboratory may or not even be sufficient for producers.

In the light of the test results described above, it is reasonable to consider an amendment to the requirements of EN 12004 and an obligatory consideration of the measurement uncertainty in the assessment of CTAs. Differences between test results obtained in different laboratories are a natural phenomenon, and the value of measurement uncertainty indicates the range of differences. However, assessments and verification of the constancy of performance should consider the measurement uncertainty in this assessment due to the specificity and imperfections related to tensile adhesion strength measurements. The new EN 12004 should also consider a procedure for doubtful cases.

As previously mentioned in the assumptions, standardization works use proven achievements of science and technology, are carried out in openness, take into account the public interest, which is very important and participation is voluntary. The consensus is the basis of the standardization process. The final product of this work, which is a standard, is perceived differently by the standardization bodies themselves, authorities, primarily market surveillance, science, customers, and industry. The situation in testing the adhesion of ceramic tiles adhesives described in this review perfectly illustrates how difficult it is to create a standard that satisfies all stakeholders.

To illustrate the complexity of the situation described in the review in the scope of measuring the adhesion of CTAs according to EN 12004, Figure 1 presents in a new approach the test results described in the recently published results of the study comparing two editions of ILCs [46]. The bottom part of Figure 1 shows the tensile adhesion strength results of the same CTA, a class C2 adhesive, determined after immersion in water, which was obtained by nineteen testing laboratories participating in the study and distinguishing the dominant failure pattern (two different colors). Ten laboratories indicated cohesive failure pattern (CF-A) as dominant, and seven as the dominant failure pattern indicated adhesive failure between the adhesive and tile (AF-T). Two out of nineteen laboratories showed a mixed failure mode model, i.e., 5% CF-A and 95% AF-T in one case and 20% CF-A and 80% AF-T in the second one. The middle part of Figure 1 shows the results of the *z*-score analysis, which is used to determine the competencies of laboratories participating in the ILCs. In the case of eighteen laboratories, *z*-scores were obtained and classified according to ISO 13528 [47] as "satisfactory", i.e., meeting the condition $|z| < 2$, marked in green in Figure 1. Only in the case of one laboratory (marked as number 15) was the value of the z-score above 2.0, which, following the requirements of ISO 13528, means that the obtained result was classified as "questionable". In the upper part of Figure 1, the result of the construction supervision authority assessment is presented using a simple acceptance rule that does not consider the variability resulting from the measurement uncertainty.

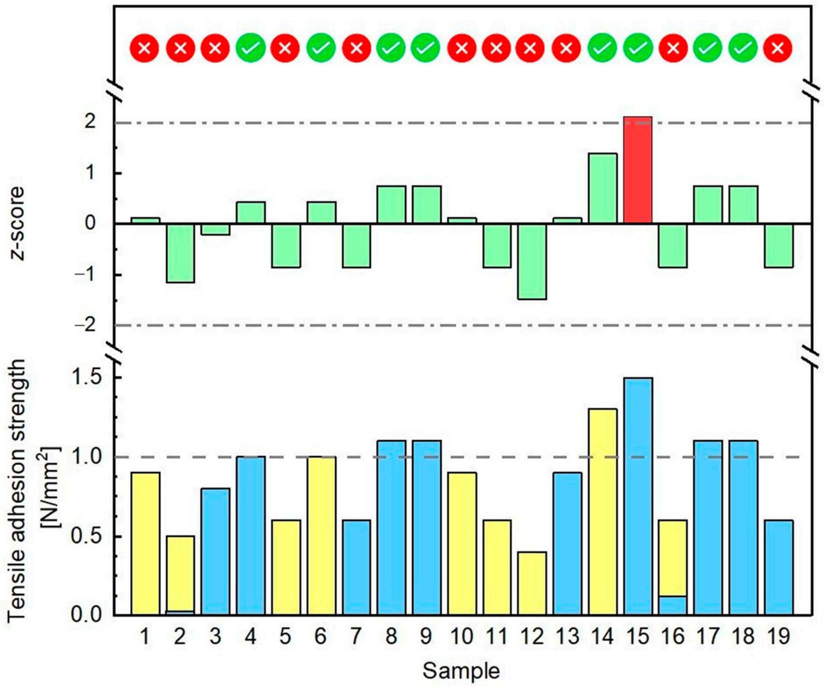

Figure 1. The results of the tensile adhesion strength of class C2 cementitious tile adhesive (CTA) after immersion in water obtained by nineteen laboratories participating in the ILC [46]. Legend: (■) adhesive failure between adhesive and tile (AF-T); (■) cohesive failure within adhesive (CF-A); (■) |z| < 2 (satisfactory); (■) 2 < |z| < 3 (questionable); (✓) samples that were assessed by the construction supervision as meeting the requirements for CTA class C2 (>1.0 N/mm^2 following EN 12004); (✗) samples that the construction supervision assessed as not meeting the requirements for CTA class C2.

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