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## Review Article

# Composition, Properties, and Standards for Cementitious Ceramic Tile Adhesive: A Review

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## ABSTRACT

The simplicity and widespread use of adhesive mortars for affixing ceramic tiles as ornamental finishes for indoor and outdoor applications has flourished the market of adhesive dry mortars. In tiling technology, attaching ceramic tiles to different types of surfaces requires a mortar with the requisite workability, flexibility, and adherence. With the evolution of building industry, mortars are now manufactured as ready-to-use products in which mineral binders and aggregates are properly combined. In addition to the fundamental components (cement, lime, sand), additional additives and admixtures are frequently added to the mortar formulations to improve their specifications and achieve varied technical features. Depending on their application purposes, incorporation of these additives and admixtures should be examined in depth to obtain the most significant contributions. In this regard, this article examines the already conducted studies and research works concerning Cementitious Tile Adhesives (CTAs) and explores the adhesion mechanisms, CTA constituents, classification, and standards, and performance evaluation of the cementitious ceramic adhesive. The results from extensive research indicate that a number of distinct parameters affect the adhesion properties of the CTAs. However, further studies are still required to enhance their engineering attributes.



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## 1. INTRODUCTION

Adhesive is a substance applied to one or more surfaces of two different components, which permanently binds them together and prevents their separation [1]. Based on the adhesion process, adhesives are often categorized into two types of reactive and non-reactive.

Likewise, they can be divided based on their ingredients, chemical composition, mechanical properties, or physical phase. According to the building application, adhesive materials are categorized as

follows:

- Cementitious adhesives
- Paste or ready-to-use adhesives
- Organic adhesives

## 2. ADHESION MECHANISMS

Several interdependent mechanical, physical, and chemical forces are involved to bond an adhesive to a substrate. The five adhesion mechanisms are mechanical,

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electrostatic, physical absorption, chemical absorption, and diffusion, and it is impossible to isolate these forces from one another [2,3].

### 2.1. Physical Adhesion

This type of adhesion is the result of molecular contact between two materials that are bonded by van der Waals forces. Figure 1 shows a schematic of this adhesion and the force that causes it. Van der Waals force is the weakest force in the adhesive connection but it is reasonably sufficient enough to make strong connections [4].

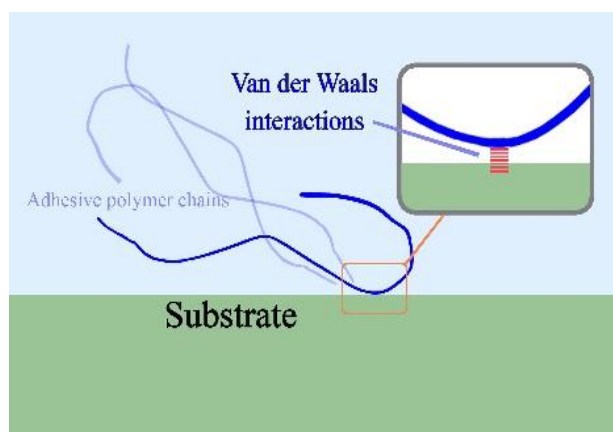


Figure 1. A schematic of surface forces in physical adhesion

### 2.2. Chemical Adhesion

Chemical adhesion is referred to as the formation of ionic, covalent, or hydrogen bonds at the interface of two components. Two substances are connected by either exchanging electrons (ionic bond) or absorbing hydrogen, oxygen or fluorine atoms to a hydrogen atom (hydrogen bond). Chemical bonds are strong and significantly contribute to the adhesion of two components. Figure 2 shows the schematic of this type of connection.

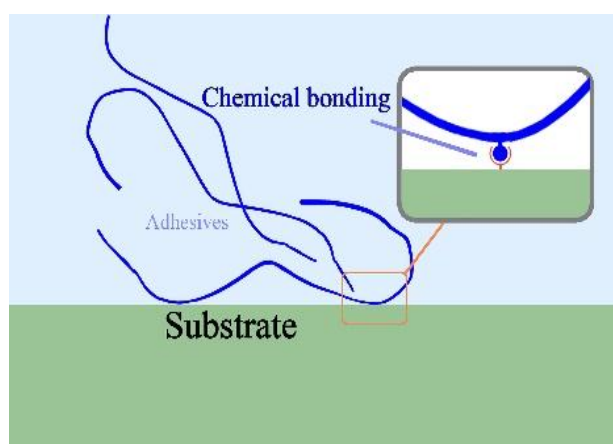


Figure 2. Schematic of the chemical connection between the adhesive and substrate

### 2.3. Diffusion Adhesion

Adhesion of polymer materials results from the interpenetration of chains at the interface. This type of adhesion is created when both the adhesive and substrate are polymers and when they can dissolve in one another. The interface between an adhesive and a substrate before and after diffusion bonding is depicted in Figure 3. Atoms diffuse from one component to another when a polymer adhesive and substrate are contacted with one another and heated, which results in adhesion. Temperature, contact time, polymer molecular weight, and physical state (liquid or solid) all affect the diffusion adhesion [4].

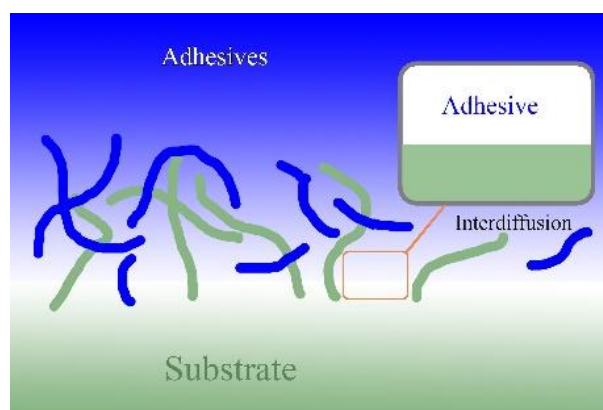


Figure 3. Schematic of diffusion adhesion between the adhesive and the substrate

### 2.4. Electrostatic Adhesion

The theory of electrostatic adhesion refers to the difference in electric charge at the interface between two materials where the electrons are transferred from one material to another. This phenomenon creates an attractive force between these materials which facilitates adhesion. An electrical double layer is depicted in Figure 4 as a result of an adhesive adhering to a substrate. This theory will not be applicable if one or both of the connected materials are insulators [4].

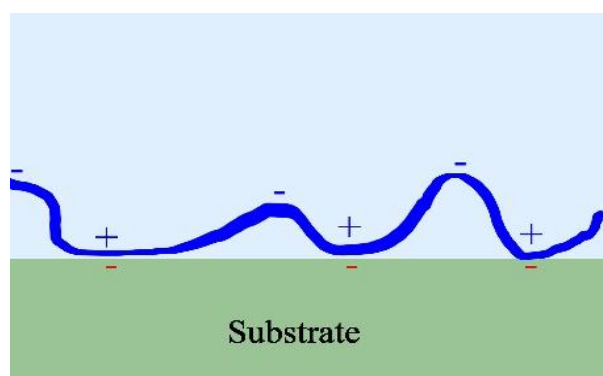


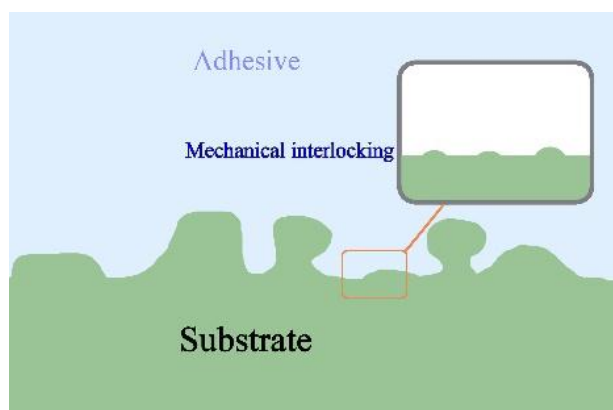
Figure 4. Negative and positive electric charges in the connection of materials

## 2.5. Mechanical Adhesion

According to the principle of mechanical bonding of the adhesives, a proper adhesion can occur only when an adhesive interlocks holes pores, cracks, and other surface roughness of the substrate, and it is mechanically locked to the substrate. Figure 5 illustrates how surfaces have always some degrees of roughness, hence never fully smooth.

To be specific, an adhesive should move the trapped air at the interface, interlock the surface pores, and provide a mechanical lock with the interface. It also implies that the adhesive must possess the necessary rheological characteristics to fill the gaps in a timely manner in addition to wetting the surface [4].

The surface roughness helps the adhesion force to create a larger contact surface. The total surface contact energy rises as a result of this issue, thus increasing the resistance to separation. Of note, the adhesive must wet the substrate to form a strong bond.



**Figure 5.** Mechanical locking between the adhesive and the substrate

## 3. THE HISTORY AND APPLICATION OF DRY MIX MORTARS

The usage of mortars has a long history, dating back to the time when the mineral mortars were widely utilized in building and architectural constructions. Roman construction refers to a state where the cement paste, i.e., a mortar (lime and sand or pozzolan) mixture, was first created and used. Until the 1950s, mortars were made and utilized in the workplace. Mineral adhesives, typically cement and aggregates (mainly quartz sand), are transported to the job site separately and then manually combined in the appropriate ratio. The wet mortar should be mixed with water before usage. The demand for new building materials and technology increased during the 1950s and 1960s in Western Europe, particularly in Germany and the United States of America [5]. Despite the fact that the first official patent for manufacturing and using dry mix mortars was published in Europe in 1893,

the technology based on mixing mortars on-site was gradually supplanted over the years by factory-mixed dry mortars. As a result, it is the only form on the market nowadays. The mortar formula can be controlled in the lab, and the product quality can be greatly enhanced by adding unique additives that in turn change its behavior and performance. Consequently, the product's added value will be raised as well. The ability to create many types of dry mix mortars, each with unique and appropriate properties, for the demands of a particular application, is the fundamental benefit of the ready-to-use mortars. Additionally, by mixing the materials prior to use, this technology enables consumers to prevent mistakes that traditionally occurred when the mortar was created on-site.

## 4. CEMENT-BASED TILE ADHESIVE

In both modern and historic building, ceramics and tiles are frequently used for internal and exterior decoration. Ceramic floor and wall tiles are highly resistant to abrasion, chemical agents, and water, and they are hygienic and washable as well. Rapid development of the supplementary materials to cling to these coatings is justified by their widespread applications. The commercialization of dry mix mortars with various properties is the result of much study in this area. Cement and sand are the conventional ingredients in tile adhesives. The finished product has important properties thanks to the inclusion of re-dispersible polymer particles in the adhesive mix. The type of the used tiles (for instance, pay attention to large-format floor tiles) and substrate to which they are applied affects the performance, efficiency, and durability of the CTAs. Just before usage, cementitious adhesives should be combined with water and put as a thin layer beneath ceramic or tile. Flexibility, deformability, ease of use, reliability, and appropriate adherence of all types of tiles to all types of substrates are the key characteristics to seek for in an adhesive. Most nations throughout the world have standardized the specifications for cementitious CTAs [6].

## 5. CLASSIFICATION AND STANDARDS OF CEMENTITIOUS TILE ADHESIVE

The specifications for the CTAs were defined in 2001 with the establishment of the European Standard EN 12004:2001. EN 12004:2001/A1:2002/AC:2002, EN 12004:2007, EN 12004:2007+A1:2012, and EN 12004-1:2017 are the most recent revisions to the standard. The most recent version of the standard with the requirements for CTAs is EN 12004:2007+A1:2012, which is included in the list of European harmonized standards. The official list of harmonized standards published in the Official

Journal of the European Union has not yet included the EN 12004-1:2017 standard, meaning that it cannot serve as the foundation for both evaluation and verification of the consistency of the performance [7].

Additionally, there are a number of ISO 13007 standards for CTAs, which have nearly identical specifications as EN 12004. The terms pertaining to the components, processes, and application characteristics of the CTAs are defined in ISO 13007-1:2010. It specifies the values of performance requirements for all CTAs. Furthermore, they are covered by ANSI (American National Standard Institute) Standard 118.

The mechanical specifications are used to categorize cementitious tile adhesives. Based on the tensile adhesion strength of the adhesives, the EN 12004 standard establishes two primary classifications, i.e., C1 and C2 [8]. The C1 class adhesive cannot be used for tiles entirely made of glass or in locations with considerable thermal stress (balconies, roofs, terraces, etc.). On the contrary, C2 adhesive enjoys the benefit of being used with all kinds of substrates and tiles. The requirements for CTA in accordance with EN 12004: 2007+A1: 2012 are listed in Table 1 [9].

**TABLE 1.** Characteristics of CTA according to the requirements EN 12004: : 2007+A1:2012

<b>Fundamental Characteristics</b>		
<b>(C1) Normal setting adhesion</b>		
<b>Characteristics</b>	<b>Requirement</b>	<b>Test Method according to EN120004-1</b>
Initial tensile adhesion strength	$\geq 0.5 \text{ N/mm}^2$	
Tensile adhesion strength after water immersion	$\geq 0.5 \text{ N/mm}^2$	8.3
Tensile adhesion strength after heat aging	$\geq 0.5 \text{ N/mm}^2$	
Tensile adhesion strength after freeze/thaw cycles	$\geq 0.5 \text{ N/mm}^2$	
Open time tensile adhesion strength	$\geq 0.5 \text{ N/mm}^2$	8.1
<b>Fast setting adhesion (C1F)</b>		
Initial tensile adhesion strength	$\geq 0.5 \text{ N/mm}^2$	8.3
Open time: tensile adhesion strength	$\geq 0.5 \text{ N/mm}^2$	8.1
All other requirements for C1		
<b>Optional Characteristics</b>		
<b>Special Characteristics</b>		
Slip (T)	$\leq 0.5 \text{ mm}$	8.2
Extended open time (E): tensile adhesion strength	$\geq 0.5 \text{ N/mm}^2$	8.1
Deformable adhesive (S1): transverse deformation	$\geq 2.5 \text{ mm}$	
	$\leq 5 \text{ mm}$	8.6
Highly deformable adhesive (S2): transverse deformation	$\geq 5 \text{ mm}$	
<b>Additional Characteristics (C2)</b>		
Initial tensile adhesion strength	$\geq 1 \text{ N/mm}^2$	8.3

## 6. INGREDIENTS OF CEMENTITIOUS TILE ADHESIVE

The properties of the cementitious adhesives can be significantly affected by the type and proportion of the components, mixing, implementation, and curing conditions. In this section, the materials that are regularly used in the composition of cementitious adhesives are reviewed.

### 6.1. Cement

The hydraulic component of a cementitious system is cement. Utilized as a building material, cement is combined with either fine aggregates to create mortar or with sand aggregates to create concrete.

Cement comes in a variety of forms with different chemical compositions, mechanical characteristics, strengths, and durability.

Portland Cement (PC), which is primarily utilized as a binder in the production of concrete, is the most

frequently used cement [10]. According to the ASTM C150 standard, Portland cement contains about 55 (wt. %) of alite or tricalcium silicate ( $C_3S$ ), 20 (wt. %) of belite or dicalcium silicate ( $C_2S$ ), 10 (wt. %) of tricalcium aluminate ( $C_3A$ ), 8 (wt. %) of tetra calcium aluminofrite ( $C_4AF$ ), 5 (wt. %) of gypsum, and about 2 (wt. %) of sodium and potassium oxide. It should be noted that gypsum is added in order to adjust the setting time; otherwise, cement will be set too quickly [11]. Cements including Portland cement, slag cement, pozzolanic cement, composite cement, and calcium aluminate cement are examples of hydraulic binders. Based on the weight of the dry mix mortar, the amount of cement in the ceramic tile adhesives is typically set between 35 and 50 (wt. %) [12].

#### 6.1.1. Mechanism of Hydration Reactions

Considering the importance of cement behavior in the mechanical properties of CTA, a review is presented on the mechanism of cement hydration reactions in this section. Cement hydration is a complex chemical

reaction between cement and water that leads to the production of a rigid and mechanically resistant material. The progress of these reactions is evaluated based on the setting and curing. Setting and curing are the terms used to describe the initial gradual decline in the cement workability until the mixture is no longer workable and the progressive rise in the mechanical strength. There is no discontinuity between these two processes. When water is added, the reactions that occur are primarily exothermic.

Figure 6 depicts the heat generated as a function of time by the beginning of cement hydration reactions. In the first stage of cement hydration, the temperature rises quickly.

The second phase is referred to as the sleep phase. In this phase, the rate of temperature increment is noticeably lower than that of other phases. The sleep phase takes about one to three hours. The concrete is in a plastic form during this time, enabling transportation to the workplace. The initial setting starts at the end of this phase. In phases III and IV, the concrete begins to harden, and the temperature increases mainly due to the hydration of tricalcium silicate. After 36 hours, Step V begins. As long as water and unhydrated silicates are present, the gradual production of hydration products happens and continues [13].

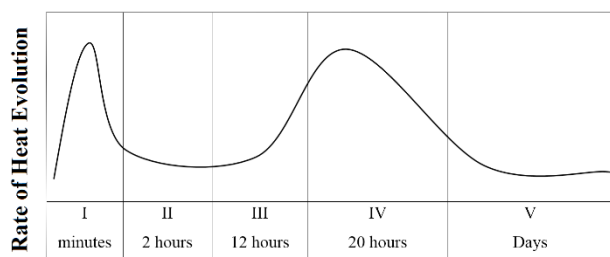


Figure 6. The heat of cement hydration vs. time

A more detailed explanation of hydration reactions is given below. With addition of water, a series of reactions occur. To be specific, ettringite and heat are produced when tricalcium aluminate reacts with gypsum in the presence of water according to the reaction (1).



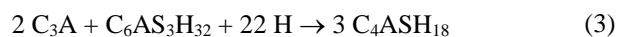
Ettringite consists of needle crystals that are only stable in solution with gypsum. This composition does not incorporate cementitious adhesive strength.

Heat, hydrated CSH, and hydrated lime are produced in reaction (2) when tricalcium silicate (allite) hydrates:



CSH has a fiber structure with a short network, which

greatly contributes to the initial strength of the cement adhesive. Through the consumption of the gypsum (reaction (1)), the ettringite becomes unstable and reacts with residual tricalcium aluminate to form aluminate monosulfate hydrate crystals according to the reaction (3):



Monosulfate crystals are only stable in sulfate-free solutions. In the presence of sulfates, the crystals turn into ettringite, whose crystal size is two and a half times that of monosulfate. An increase in the size causes cracks in the cement exposed to the sulfate attack. Blite (dicalcium silicate) also hydrates to form calcium silicate hydrates and heat according to the reaction (4):



Like the second reaction, calcium silicate hydrates contribute to the cement paste strength. This reaction proceeds more slowly and generates less heat. However, contribution of  $C_2S$  to cement paste strength is initially negligible, and it will be ultimately accountable for durability of the PC concrete.

Gypsum and ferrite go through two sequential reactions:

- In the first reaction, ferrite reacts with gypsum and water and forms ettringite and alumina hydroxides, and hydrated lime.
- Ferrite reacts with newly produced ettringite and creates garnet.

Garnets do little more than take up space; they make no contribution to the cement paste strength. Finally, the hardened Portland cement paste includes the following [11, 14-16]:

- Ettringite: 15-20 %
- Calcium Silicate Hydrate (CSH): 50 to 60 %
- Calcium hydroxide: 20 to 25 %
- Pores: 5 to 6 % (in the form of capillary cavities and trapped air)

## 6.2. Aggregate

The main part in the composition of the tile adhesive is aggregate. For this reason, the aggregate properties have a significant impact on the durability and efficiency of the adhesive mortar.

The properties of aggregates such as mineral and chemical composition, petrographical characteristics, density, hardness, mechanical strength, chemical and physical stability, porosity structure, shape, size, and surface texture are some of the factors that affect the final properties of the adhesive [17]. Cement-based ceramic adhesives contain both coarse and fine aggregates in their composition.



### 6.2.1. Coarse Aggregates

To make the mortar stronger, aggregates serve as the structural support and reinforcement. By providing bending strength and durability and filling in empty gaps, they improve the density of the system [11,18]. Silica sand, dolomite, limestone, light aggregates (such as perlite, polystyrene, hollow glass spheres), etc. can be used as aggregates in ceramic adhesives. The amount of aggregate is usually reported between 50-70 wt. % based on the total weight of the dry mix mortar [12]. The size of the quartz filler (sand), usually used in the CTA, is up to 0.5 mm. In the case of CTAs intended for large format floor tiles, the sand size up to 1.2 mm is used in part (in the combination with 0.5 mm) [12].

### 6.2.2. Fine Aggregate (Filler)

Calcium carbonate is the filler that is most frequently used in cementitious tile adhesive. Fine calcium carbonate enhances consistency and workability of the fresh mix. Of note, it should be noted that due to its nature, limestone powder tends to absorb water, thus making the hardened tile adhesive brittle [12].

### 6.3. Cellulose Ether

Cellulose is a polysaccharide with the chemical formula of  $(C_6H_{10}O_5)_n$  that is an organic substance. It is made up of a linear chain of hundreds to thousands of di-glucose units that are joined together by glycosidic linkages.

Cellulose fibers are made up of bundles of parallel, unbranched chains that are joined together to form fibrils by hydrogen bonds between the hydroxyl groups on nearby chains. Hydrogen bonds cause parallel chains to be arranged in bundles, which gives cellulose fibers their high mechanical strength. Amorphous and crystalline cellulose are both possible [19–20].

One of the essential components of the cement-based tile adhesives is cellulose ether. Cellulose ethers are utilized in hydraulic systems as a water retention agent and have a considerable impact on the adhesive qualities. The water retention agent is essential to achieve the optimum workability of the mortar and to regulate the amount of water needed for the hydration of the cement. The mortar acquires the necessary strength, hence protected from cracks by the adhesive cement being properly hydrated. To get the necessary open time to work with the adhesive, the cellulose ether must retain water. Additionally, cellulose ethers enhance the rheological characteristics of mortar. The consistency of the fresh components is also improved by using polysaccharides such as derivatives of starch [11,21].

Alkyl celluloses, hydroxy-alkyl celluloses, and alkyl-hydroxyl celluloses replaced with two or more alkyl or hydroxy-alkyl groups or a combination of two or more cellulose derivatives can all be found in cellulose ethers. For instance, Hydroxypropyl Methylcellulose (HEMC), Hydroxyethyl Methylcellulose (HPMC),

Carboxymethylcellulose (CMC), Hydroxyethyl Cellulose (HEC), Hydroxypropyl Hydroxyethyl Cellulose (HPHEC), and Ethyl Hydroxyethyl Cellulose (EHEC) are ether celluloses that are used in the composition of cementitious ceramic adhesives [13, 21–22].

Numerous studies have explored how cellulose ethers can affect the properties of cementitious adhesive. The capacity of the cellulose ethers to interact with cement and bind the cement matrix and aggregates to prevent their separation in the paste was highlighted by Hayakawa and Soshiroda in 1986 [23–24]. Tanaka et al. [25] used an additive containing cellulose ether which can boost the compressive strength while simultaneously increase fluidity and workability.

Polysaccharide derivatives were introduced Yamamuro et al. [26] to investigate include ionic and hydrophobic functional groups that could raise the viscosity of cement solution. Additionally, cellulose ether enhances the bond between the cement and substrate. In cementitious adhesives made from cellulose ether, Ghio et al. [27–28] confirmed an increase in the thixotropy and improvement in the workability of the mortar.

In another study, the relationship between the HPMC viscosities and ceramic adhesive characteristics showed that the absence of ether cellulose in the formulation prevented the adhesive from meeting EN 12004 criteria. Additionally, upon increasing the viscosity of the cellulose ether from 15 Pa.s up to 70 Pa.s, the adhesive qualities and adherence were also enhanced. Further analysis and inspection of the hardened mortar microstructure revealed that the amount of C-S-H phase rose as the viscosity of methylcellulose increased. After combining the adhesive powder with water for the first 24 hours, addition of ether cellulose to the composition lowered the hydration rate of the cement [29].

The additional benefit of the celluloses, especially HEMC and HPMC, is that they stabilize the air bubbles during the mixing stage. Workability depends on the stabilization of air bubbles. Air bubbles, however, weaken the mortar's ability to adhere. The relationship between the adhesive strength and resistance to flexibility is an exponential function of density. Creation of an optimal system with appropriate workability and sufficient mechanical strength is therefore required [30].

There is a significant affinity between the water molecules and cellulose ethers. Each cellulose ether molecule is surrounded by a hydrated sphere that makes the fluid phase become immobile and give cement the ability to retain water. To ensure enough water retention and, as a result, enough water for complete hydration of the cement, cellulose ethers are utilized in the composition of tile adhesives. High water retention also guarantees the continued wettability of the adhesive once applied to the substrate (open time). This matter gains even more significance, especially when it is hot or windy outside [26].

The ability of the cementitious adhesives to resist slipping, particularly on non-horizontal surfaces, is an optional feature. This feature comes from the modified cellulose ethers, which give the fresh mortar paste more consistency by thickening and increasing its viscosity. The viscosity of the mortar is further increased by using modified cellulose ether with a certain molecular weight and a thickening such starch or Polyacrylic Amide (PAA). Additionally, cellulose has a thickening effect on the fresh mortar paste [26].

It should be noted that the delay in cement hydration is one of the additional effects of using these macromolecules in mortar composition.

The chemical composition of the cellulose ether molecule is largely responsible for this delay. According to the observations, portlandite is not developed when ether cellulose is adsorbed on the cement clinker phases. Cellulose ether has a significant impact on the hydrated calcium silicate precipitation, which reduces the number of initial hydrated calcium silicate nuclei and delays the development of a continuous hydrated calcium silicate shell surrounding the tricalcium silicate grain. Finally, it prevents the formation of a dense layer of hydrated calcium silicate that is permeable in the system.

The dissolving rate of calcium aluminate gradually declines when cellulose ether is added to a cement matrix, and ettringite, hence precipitation of calcium hydroaluminate. When compared to hydroxypropyl methyl cellulose, this issue occurs more frequently with HEC [10].

The delay in the cement setting is proportional to the number of methoxyl groups. Therefore, it is essential to employ the optimal amounts of cellulose derivatives to enhance the capabilities of the adhesive without jeopardizing those properties by delaying cement hydration [31].

Water immediately evaporates from the adhesive surface in a fresh mortar. A thin layer of cellulose ether was formed after about five minutes that prevented further evaporation. The initial layer of the mortar is partially dissolved as the water moves from the core to the surface before evaporating. The cellulose ether is brought to the surface of the adhesive by this water flow, where it accumulates over time to the point that the film is no longer dissolved. The adhesive qualities at the mortar/tile contact are gradually diminished by this layer, which prevents tile adhesive from getting wet. Migration of cellulose ether to the surface to create a film is inhibited by the adsorption of cellulose ether on the cement grain, which results in a prolonged setting delay.

However, regulation of the film is also vital because a too thin film dissolves and permits surface evaporation. Creation of this film is necessary to prevent drying of the applied mortar surface. However, as a result of the high concentration of cellulose ether in the pore solution, a thick film decreases the wetting between the tile and the adhesive and the adhesive characteristics.

Cement hydration and latex film production both contribute to the system strength during the adhesive hardening process. The water concentration gradient, which is produced by capillary transport to the substrate, mortar evaporation, and cement hydration, is one of the key characteristics during hardening. Early mortar drying can significantly minimize cement hydration. The capacity of the cellulose ether to hold water and the small size of the pores prevent rapid drying. As a result, the adhesive is strengthened by increasing the concentration of redispersible polymer and cellulose ether at the contact between the tile and the substrate. On the contrary, the entrapped air in the paste prevents mixing during tiling by creating a film as a result of the migration of the cellulose ether to the surface. The trapped air persists at the tile-mortar interface where it lowers the contact surface and, consequently, the adhesive properties [31]. As a result, optimizing the film thickness is crucial for the CTA characteristics.

#### 6.4. Polymers

The so-called polymer-modified flexible tile adhesives, the second generation of thin-bed tile adhesives, were released on the market in the beginning of the 1980s. This important development in the adhesive technology was made possible by Wacker Chemie's invention of Re-dispersible Polymer Powder (RPP) in 1953. Improved efficiency, flexibility, and adhesion are the key benefits of RPP in the formulation of ceramic adhesives [11]. To improve the stickiness and shear resistance, polymers are added to the adhesive formulations [32]. The tensile strength, plasticity, wear resistance, and flexural strength of the mortar are all enhanced by redistributable polymers, which also produce a suitable degree of flexibility. These polymers also increase the water retention capacity of the mortar and prevent its water evaporation by forming a film. The polymer film closes the pores and cracks in the dried grout, thus preventing water evaporation. Additionally, by forming an additional bond, the polymer strengthens the mechanical properties of the mortar [33]. In the polymer dispersion process during the cement hydration in the adhesive mortar, two hypotheses are proposed. The first theory states that there is no chemical reaction between the polymer and cement components. Upon using polymers, the water amount in the system decreases and leads to the integration of polymer particles, thus causing the gradual formation of a three-dimensional polymer network and increasing the strength and toughness of the adhesive mortar. The second theory postulates that in addition to this occurrence, polymer particles and cement hydration products interact chemically that results in the formation of complexes that either delay or hasten the hydration of the cement system [11,34].

Elastomeric and thermoplastic powders are the two categories into which the re-dispersible polymers on the

market can be divided. Styrene Butadiene Rubber (SBR) is an elastomeric powder while poly (ethylene-vinyl acetate), poly (vinyl acetate-vinyl verstate), poly (styrene-acrylic ester), and polyacrylic ester are thermoplastic powders (PAE). Examples of polymers that are frequently used in the formulation of ceramic adhesives include vinyl acetate, copolymers of vinyl acetate with ethylene, copolymers of vinyl acetate with ethylene and vinyl ester, copolymers of vinyl acetate with ethylene and acrylic ester, copolymers of vinyl acetate with ethylene and vinyl chloride, copolymers of styrene acrylic ester, and copolymers of styrene-1. The flexible butadiene and rigid styrene chain in the SBR structure increases adhesion, durability, and mechanical qualities in the concrete mortars [35–38]. EVA, or poly (ethylene-vinyl acetate), is another type of re-dispersible polymers that is frequently used due to its satisfactory working with the cement base system [39–41]. VA/VeoVa, often known as poly (vinyl acetate-vinyl versatate), is another polymer that has drawn academic attention. The polymer, with its remarkable resistance in alkaline environments, gains three side chains of the alpha alkyl molecule due to the presence of the versatate group [42–45]. The compressive and tensile strengths as well as the water retention effect improve with a rise in the poly (styrene-acrylic ester) or SAE to cement ratio [46–47]. Additionally, polyacrylic ester, or PAE, proved to have adequate workability and improved mechanical characteristics [48–49].

The mechanical properties of polymers change drastically at the glass transition temperature ( $T_g$ ) due to the molecular motion. Below  $T_g$ , polymers are relatively rigid, inflexible, and brittle with no translational or rotational movement of the atoms in the chain while above it, they are soft and flexible. The main advantages of RPP in thin-bed tile adhesive are [31]:

- Improved adhesion between the substrate and adhesive;
- Greater flexibility facilitates use of adhesive for big tiles and lowers the shear stress on the substrate, tile adhesive, and tiles.
- Improvement in the rheological characteristics facilitates easier mixing, workability, and adhesive wetting of tiles. Copolymers of Ethylene and Vinyl Acetate (EVA) or styrene and butyl acrylate are the typical RPPs used for tile adhesive. Between 0 and 5 % of CTA contains redistributable polymer powder [13,21].

In their study, Jenni and et al. confirmed that the RPP could boost the adhesive strength of the mortar owing to the impact of polymer on the microstructure and adhesive strength of the cement tile adhesive. Microstructural analyses demonstrate that the polymer can improve the adhesive capabilities when entirely and uniformly dispersed in the cement-polymer matrix. On the sample surface, containing cellulose ether, polymer films with

the width of 10-100  $\mu\text{m}$  were detected. Therefore, they came to the conclusion that the formation of this film in the entire matrix was the reason for enhancing the adhesion strength of the adhesive. The type of re-dispersible polymer utilized (SA, EVA, or VC) affects the tendency to create cellulose ether-polymer composite films and their morphology [32]. In a cement (CEM 1 32.5R) and sand combination, Schulze [50] examined the effects of utilizing SAE (polystyrene-acrylic ester) and EVA (poly (ethylene-vinyl acetate)) in both internal and external weather conditions. After 28 days in the open, the adhesion strength of the EVA-modified mortar was higher than that of the SAE-modified mortar and higher than that of the conventional cement-sand mixture. All mortars showed a steady rise in their adhesion strength over the course of 10 years, and all samples attained their maximum strength. However, compared to the initial amount of the EVA-modified mortar, the tensile adhesion strength of the control sample (standard cement and sand mixture) was considerably low. Over 10 years, no increase in the strength of the control or un-modified sample was reported in indoor condition. Due to the mortar's ability to keep a suitable amount of water for the cement hydration, the adhesives that contained EVA and SAE in their composition exhibited similar performance, and their adhesion strength increased during 10 years. The aggregates are kept together and adhere better as a result of the application of polymer powders. Similar to the previous study, it was found that the polymer could distribute water throughout the system and prevent the polymer from separating water from the adhesive paste. It could also retain water in the adhesive composition and reduce the amount of water required to prepare the adhesive paste [43].

### 6.5. Accelerator

Hydraulic systems typically use the accelerator. Accelerators speed up the hydration process and shorten the setting period of the cement, hence an increase in the tensile strength of the adhesive. There are two types of additives: organic and inorganic. Chlorides, such as calcium, sodium, and potassium chlorides, nitrites, such as sodium nitrate and calcium nitrate, sulfates, such as sodium sulfate and calcium sulfate, thiocyanates, like calcium thiocyanate and sodium thiocyanate, hydroxides, include sodium hydroxide, potassium hydroxide, and calcium hydroxide, carbonates such as calcium carbonate, sodium carbonate, Amines like diethanolamine and triethanolamine, calcium salts, and organic acids like calcium formate, calcium acetate, and maleic anhydride are examples of organic substances. According to reports, the accelerator should be used in the range of 0.1 and 0.3 wt. % [21].

### 6.6. Retarder

Cementitious ceramic adhesives use a retarding



ingredient. The main task of the retarders is to postpone cement hydration so that workers have adequate time to manipulate the paste in an open area [18]. Mineral retarders like lead and zinc oxides, phosphates, magnesium salts, fluorides, and borates are common. Examples of organic retarders are the Na, Ca, and  $\text{NH}_4$  salts of lingsulfonic acids, adipic acid, citric acid, tartaric acid (together with citric acid), gluconic acid, heptonic acid, succinic acid, and polysaccharides. The retarders included in the formulation of cement adhesives are saccharides and their salts such as glucose, sodium gluconate, fructose, galactose, sucrose, xylose, ribose, arabinose, sucrose, mannose, oligosucrose, dextran, lignosulfonates, phosphoric acids and their salts, and boric acid. Followed by creating a compound with monosulfates, citric acid functions as an accelerator in the early stages of hydration before acting as a retarder. According to the reports, commercial lignosulfonates without sugar yielded good results in terms of delaying the hydration of tricalcium aluminate ( $\text{C}_3\text{A}$ ) and tricalcium silicate ( $\text{C}_3\text{S}$ ). Zinc oxide is an inorganic retarder that slows the hydration of  $\text{C}_3\text{S}$  but has no effect on the hydration of  $\text{C}_3\text{A}$  or gypsum [21]. Retarders are typically added to the adhesive compositions in amounts ranging from 0.01 to 0.05 wt. % [13].

### 6.7. Fibers

Fibers are frequently used to strengthen and prevent creep in cementitious materials. The significant effect of the fibers is to bridge at the crack tip to resist crack propagation in the cement matrix. The connection of the fiber to cement paste is an essential factor that affects the performance of fiber-reinforced cement composite. The bonding energy (adhesion) between these materials includes surface interactions (chemical bonding) and mechanical interactions (interlocking). Fibers can be classified into two groups depending on their average length: long fibers with a higher aspect ratio between 200 and 500 are mainly used to strengthen the mortar while short fibers with the ratio of the overall dimensions between 20 and 60 affect the characteristics of the fresh mortar. A typical dose of macro fiber with more than 40 mm in length is 3-8  $\text{kg}/\text{m}^3$ . The microfibers are 6-12 mm long with the typical dosage of 0.6-1.0  $\text{kg}/\text{m}^3$ . Macrofibers are used to increase the strength of the tile

adhesive [51]. Polypropylene fibers are used commonly in cementitious systems.

### 6.8. Other Additives

Thickening agents, secondary water retention agents, wetting agents, defoamers, superplasticizers, dispersants, surface activating agents, calcium chelating agents, calcium complexing agents, and water repellants are the additives that are used in the composition of tile adhesives [52]. Plasticizers, superplasticizers, dispersants or water reducing agents are additives that disperse binder particles and increase the fluidity of the binder (cement or plaster). The superplasticizer improves the fluidity of the mortar and increases the opening time; it also has a positive effect on the mechanical strength of the adhesive [53]. Among the water-reducing agents are the melamine-based agents, lignin-based agents, and poly-carboxylate-based compounds, to name a few [21]. The defoamers often include polyether, silicone, alcohol, mineral oils and non-ionic surfactants. The usual amount of defoaming additives is between 0.2-4 % by weight (based on dry mix mortar). Gelatin, polyethylene glycol, lignin sulfonate, naphthalene-sulfonate, polycarboxylate ether, polystyrene sulfonates, calcium salts from organic acids such as calcium formate, bentonite, montmorillonite, polyamide fibers, metallic aluminum, fibers Propylene, polyvinyl alcohol, vinyl acetate-based polymers, styrene, butadiene, ethylene, and etc. are different types of the additives of cementitious ceramic adhesives [21]. A summary of the most general additives used in the CTAs is given in Table 2.

Cementitious ceramic adhesives are categorized into two classes of C1 and C2 in EN 12004, as previously discussed. The amount of polymer combined with other ingredients in the adhesive composition is the primary difference between C1 and C2 tile adhesive. Table 3 gives some recommendations for the composition of ceramic adhesives based on cement. To enhance the qualities of their formulation, a majority of producers use composition in this range together with other additions.

It has been claimed that the CTA composition occasionally includes as many as 15 different types of components [18].

**TABLE 2.** The most common additives in CTA composition cement-based ceramic adhesives

Additive	Example
Redispersible Polymer	EVA, SBR, SAE, PVA
Water Retention	HPMC, HEMC
Superplastisizer	Polycarboxylate, Melamine, Lignin
Accelerator	Calcium Formate
Retarder	Glucose, Lignosulfonate
Fiber	Polypropylene
Defoamer	Mineral Oils

TABLE 3. The general formulation of the cement-based ceramic adhesives

Ingredient	Example	Amount	
		Class C1	Class C2
Binder	Portland Cement	30-50	30-40
Aggregate	Silica Sand	45-70	45-60
Filler	Calcium Carbonate	5-10	5-10
Redispersible Polymer	EVA, SBR, SAE	0-3	3-6
Water Retention	HPMC	0.2-0.5	0.2-0.5
Additives	Accelerator, Retarder, Defoamer,...	< 2	< 2

## 7. EVALUATION OF THE PERFORMANCE OF THE CEMENTITIOUS CERAMIC ADHESIVE

Assessment and verification of constancy of performance (AVCP) of CTA is carried out considering the EN 12004:2007+A1:2012. The conditions of these tests are summarized below [54].

### 7.1. Open Time

The open time test is designed to study the behavior of the adhesive after application to the substrate and exposure to air. Applying the adhesive to a large surface requires more time and then, the adhesive will be exposed to air. As mentioned, one of the crucial components of the adhesive is cellulose ether, whose main feature is the retention of water in its molecular structure. This feature is combined with the formation of a thin layer on the surface to prevent water evaporation and increase the wettability of the adhesive.

Storage conditions: 27 days in standard conditions (temperature  $23 \pm 2$  °C and humidity  $50 \pm 5$  %).

### 7.2. Initial Adhesion

The purpose of this test is to evaluate the performance of an adhesive that is not subjected to particular stress during its working life, for instance, inside the building.

Storage conditions: 28 days in standard conditions (temperature  $23 \pm 2$  °C and humidity  $50 \pm 5$  %).

### 7.3. Heat Aging

In hot climates, the temperature on the building facade that is exposed to sunlight can easily reach 70 °C in the summer, which leads to rapid drying, rapid setting, and fracture of the cementitious tile adhesive. These application conditions have a significant effect on the long-term safe adhesion of tiling. The purpose of this test is to identify the vital factors for tiling under such difficult conditions and find a solution to enhance the durability and permanence of tiling.

Storage conditions: 14 days in standard conditions, then 14 days at 70 °C, and then one day in standard conditions. When the temperature of the plates reaches 23 °C, the tiles are pulled from the concrete plate until they are removed.

### 7.4. Immersion in Water

Adhesives that are used for adhering tiles on the exterior of the building or in wet facilities such as swimming pools and bathrooms, necessarily have high resistance to water. In other words, water resistance is defined as the adhesive ability to withstand contact with water without fracture. Of note, this term should not be confused with the term "waterproof".

Storage conditions: 7 days in standard conditions plus 21 days immersion in water. This test is performed immediately after the tiles are removed from the water, while still wet, to best simulate actual conditions.

### 7.5. Freeze/Thaw Cycles

Water that has penetrated the tile during installation may expand in volume as a result of freezing. As the freezing and thawing cycles continue, the mechanical stresses caused by this volumetric shift will grow and become more harmful. The tile body as well as its surface (especially if it is glazed) and the adhesive (which in some cases results in the loss of adhesion) can all be destroyed by stress.

One of the crucial requirements, especially for use in areas with high humidity levels, is the ability to withstand these cycles without suffering significantly from loss in adhesive strength.

Storage requirements: 7 days in standard conditions plus 21 days immersion in water plus 25 cycles of freezing and thawing cycles: 2 hours at -15 °C / 2 hours at 15 °C.

## 8. CHALLENGES AND THE FUTURE PATH OF CERAMIC TILE ADHESIVE

Like any other technology, cement-based ceramic adhesives encounter a number of difficulties. When using tile adhesive, the following factors can hinder the desired outcome:

1. The change in the shape of the mortar consisting of the adhesive, compared to the ceramic tiles on it as a result of shrinkage;
2. Different displacements as a result of heat, humidity, or other effects occur between the ceramic tile, substrate, and adhesive which devastate the system;
3. Deterioration of the cement that supports the

adhesive;

4. Inadequate surface cleaning;
5. Structural movements such vibrations and settling issues;
6. Inappropriate material selection.

Some recommendations to prevent these flaws and obliterations are given in the following:

1. Appropriate material selection based on the size and efficiency;
2. Designing compatible structures, such as sufficient drainage;
3. Appropriate and right installation techniques. According to the reports, wind, rain, humidity, and attack of pollutants on the outside tiling systems of buildings would weaken the adhesive strength of the ceramic adhesive that leads to the eventual destruction of the system [55].

A market analysis states that the value of the ceramic adhesives market was 15.08 billion dollars in 2018 and is predicted to elevate up to 40.73 billion dollars by 2026 [56]. The market size of ceramic adhesives may be difficult to estimate, but it is evident that CTAs will be a major component of the construction industry in the years to come. Additionally, it appears necessary to run evaluation programs, like inter-laboratory comparisons, to guarantee the proper performance of the product and evaluation methods as well as the accuracy of the used equipment in order to advance the market for these products and their sustainable development.

## 9. CONCLUSION

The present study aims to present an overview of the CTAs whose composition has an impact on its adhesion qualities. Re-dispersible Polymer Powder (RPP) and other cementitious constituents interact with cement components to improve their physical and mechanical properties such as increased adhesion strength, reduced shrinkage, and low water absorption. Another component that significantly affects the CTA characteristics is cellulose ether. The RPP/CE decision has a significant impact on the end-use values, i.e., whether they are in the fresh or hardened form. The mortar viscosity, setting time, and film formation were all affected by the CE concentration in the pore solution in the fresh condition. The dry tensile adhesion strength was enhanced by CE film in their hardened state, which also prevented evaporation at the tile-mortar interface. The thickness of the CE/RPP layer also had an impact on the tensile strength after heat aging or immersion in water. Increasing the amounts of polymer and cellulose ether content in the system could often improve adhesion after heat aging. However, in contrast to the situations with the smallest amounts of these contents, such an increase could shorten the open time. The CTA formulation according to the desired properties based on the

EN12004:2007+A1:2012 could be optimized by using the various additives.

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