

# Strength Parameters of Composite Cements and Storage Temperature

Subjects: Dentistry, Oral Surgery & Medicine

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Fixed restorations are now among the most common restorations in modern dental prosthodontics. The view in prosthodontics of maximum preparation economy is causing an increased interest in the mechanical properties of cements. Among the most important properties of materials used for indirect cementation are mechanical properties, i.e., hardness and compressive strength. These properties can change as a result of changes in physical factors. Some studies indicated that mechanical properties, such as flexural strength, polymerization shrinkage, and conversion factor, did not change after heating the composite material. According to some researchers, preheating the composite material increased its conversion degree, which consequently led to an increase in hardness and fracture toughness, an increase in flexural strength and an increase in elastic modulus, and an increase in abrasion resistance.

Keywords: heated composite cement ; cooled composite cement ; dual composite cement ; mechanical properties

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## 1. Introduction

Fixed prosthetic restorations are currently one of the most commonly performed restorations in dental prosthodontics. The durability of these types of restorations is dependent among others on one of the most important forces called retention. Retention, as the force that holds the restoration on the prosthetic pillar, is determined by the geometric configuration of the pillar tooth walls created during preparation, which directly translates into the size of the bonding area, between the tooth's own tissues and the prosthetic restoration. The most important factor influencing the retention of a fixed prosthetic restoration is the strength of cement adhesion to the tooth surface and prosthetic restoration. The mechanical properties of the cement, such as hardness and compressive strength, are the factors that determine proper retention of the fixed prosthetic restoration. Among the cements currently and most widely used in prosthodontics, composite cements seems to be the most popular. Composite cements can be divided according to the method of tooth surface preparation and according to the method of initiation of the polymerization process. The preferably clinical classification of composite cements is based on the method of surface preparation. According to this classification, cements are divided into resin, self-etch, and self-adhesive. Currently, self-adhesive cements are the most commonly used cements by clinicians, and their popularity is due to the simplified application procedure, which significantly saves time spent on the cementing procedure <sup>[1][2]</sup>.

Self-adhesive cements are modified resin cements that bond to hard tissue in two stages. In the first step, the superficial layers of hard tissue are demineralized by the action of acidic monomer groups, which leads to the formation of a bond between the methacrylates and the enamel and dentin. Later, metal ions from the fillers are released and further bonding to enamel and dentin occurs <sup>[1][2]</sup>. These cements do not require acid conditioning of the tooth surface or application of a bonding agent prior to cementation <sup>[3]</sup>. According to the manufacturers, the functional monomers are able to chemically bond calcium to hydroxyapatite, which in the case of this group of cements is one of the bonding mechanisms responsible for the retention of the restoration <sup>[4][5][6]</sup>. Of the many analyses and conclusions regarding composite cements in the literature, the most noteworthy are those that translate significantly to clinical work. It has been concluded that if the grinding limit is in the enamel and dentin, conventional composite cements should be the materials of choice because of their strong bond to the enamel. It was also noted that the use of selective enamel etching prior to the use of resin and highly recommended for self-etch cements. It has also been shown that the strength of self-etch cements with dentin, compared to conventional composite cements, is higher. Additionally, it is known that despite the lack of clear recommendations for bonding systems, in the case of self-adhesive cements, the additional use of phosphoric acid is necessary to develop the enamel surface. Studies show that selective etching of the enamel significantly improves the bonding of the cement to this tissue <sup>[7][8]</sup>.

## 2. Changes in Strength Parameters of Composite Cements as Affected by Storage Temperature

There are reports indicating that storage conditions of composite cements can significantly affect their bond strength. Ozer F. et al. <sup>[9]</sup> conducted a comparison of the bonding performance of three different self-adhesive resin cements to human dentin after storage under two different temperature conditions. The test cements used in both study groups were: Clearfil SA (CSA), G-Cem (GC), and Bis-Cem (BC). The cements of the first group were stored in a refrigerator at  $6 \pm 2^\circ\text{C}$  for 3 months and the cements of the second group were stored at a constant room temperature of  $19 \pm 2^\circ\text{C}$  for an additional 3 months. Each group consisted of 6 teeth and 24 dentin scrapings, and cementation was performed in both storage groups according to the manufacturer's recommendations. After determining the shear bond strength (SBS), bond forces were calculated and translated into values expressed in MPa. The bond strength values in the two storage groups were significantly different from each other.

The compressive strength of composite cements was measured by other investigators in water at  $37^\circ\text{C}$  and at 0.5, 1, 5, and 60 min after removal from the water bath. All of the cements tested showed a very rapid decrease in temperature after removal from the water bath, and a marked increase in strength of all cements was shown within the first minutes after removal from the water bath <sup>[10]</sup>.

There are also reports on the use of heated composite material for cementing intermediate works. The idea of using restorative materials after they have been preheated has been used for almost 40 years, and the material that was used first after preheating was a regular composite resin <sup>[11]</sup>. In light of current research, it is even believed that this preheated composite material can compete with dual-cure composite cements when cementing indirect restorations. In fact, studies have shown that preheating the composite material used for cavity filling can find application in the adhesive cementation procedure. The heated composite material has higher fluidity, elastic modulus, and microhardness. The higher fluidity can reduce the viscosity of the material, which can directly affect the application method and improve marginal tightness <sup>[12][13]</sup> <sup>[14]</sup>.

Skapska A et al. <sup>[15]</sup> conducted a pilot study to compare selected mechanical properties of a heated composite material and a self-adhesive composite cement. The aim of their work was to compare selected mechanical properties, compressive strength, and modulus of elasticity of the heated composite material Enamel Plus Hri (Micerium) and dual composite cement RelyX U200 (3M). For the test, specimens of each material type were prepared in the shape of a  $5 \times 3$  mm cylinder, using silicone molds. The material temperature ( $50^\circ\text{C}$ ) was obtained using an Ena-Heat heating device (Micerium). The test was performed using an Instron 8501 hydraulic pulse oximeter, which is a universal testing machine. The compressive strength test was used, and the modulus of elasticity was calculated. Analysis of the results showed that the average compressive strength the heated composite material was 530 or 327 MPa, depending of the material. The study also showed that the heated composite material has a higher modulus of elasticity ( $7.9 \pm 1.48$  GPa) and thus is more rigid compared to the self-adhesive composite cement ( $5.9 \pm 0.35$  GPa).

In another study, Skapska et al. conducted a study to compare composite cements with preheated composite materials. Enamel Plus Hri (Micerium) composite material and RelyX U200 Automix (3M) dual composite cement were tested for microhardness, compressive strength, flexural strength, diametric compressive strength, and modulus of elasticity. The composite materials were heated to  $50^\circ\text{C}$  before polymerization. Higher values of microhardness (by 67.36%), compressive strength (by 41.84%), elastic modulus (by 17.75%), flexural strength (by 36.03%), and diametral tensile strength (by 45.52%) were obtained using heated Enamel Plus Hri composite material compared to RelyX U200 adhesive cement <sup>[16]</sup>.

Additionally, it has also been shown that heating can increase the fluidity of composites characterized by a regular consistency, which can translate into improved adaptation of the material to the walls of the prepared cavity. In the study of Sanjukta Deb et al. <sup>[17]</sup>, the preheating of the resin composites exhibited a significant decrease in film thickness after preheating, so it can be crucial from the clinical point of view <sup>[17]</sup>. The authors suggested that enhancing flow is resulted from thermal energy that translates to higher molecular motion <sup>[17]</sup>. The heated material showed less viscosity, which consequently translated into a better fit to the edges of the cavity. This resulted in better adhesion at the border of the material with the tooth's own tissues and reduced marginal microleakage. Minimizing marginal microleakage resulted in a lower tendency for secondary cavity caries and resulted in clinical success in terms of long-term bonding of the material to the tooth's own tissues <sup>[18][19][20]</sup>. Studies on the modulus of elasticity have shown that similar values of the modulus of elasticity of dentin and cement result in proper functioning of the prosthetic restoration in the stomatognathic system, and in this context, it should be mentioned that the average value of the modulus of elasticity for dentin is considered to be 19 GPa <sup>[21]</sup>. Based on the assumption that, since the dual composite cement contains filler in its composition, such as the

composite material, only in a smaller amount, because the filler constitutes 30%–75% of the volume in the case of the adhesive cement and the composite material contains 60%–80% of this component, it could be hypothesized that even minimal heating of the composite cement alone could improve its mechanical properties.

Morais et al. [22] performed a study to evaluate the effect of preheated, dual-cured resin cements on the bond strength of indirect restorations to dentin. The research hypothesis was that preheating the dual composite cement provided greater bond strength to dentin, compared to using the cement at room temperature, regardless of the activation mode. The study was conducted on forty freshly extracted and erupted human molars. Composite discs mimicking indirect composite restorations and three dual composite cements Variolink II (Esthetic DC), Calibra (Dentsply Sirona), and Excite (Ivoclar) were used. Previously prepared teeth were assigned to eight groups depending on the cement temperature (25 or 50 °C), the type of cement among the three selected for the study, and the activation mode (dual bond or self-curing mode). The specimens were cut to obtain multiple bonded beams with a cross-sectional area of 1 mm<sup>2</sup> for tensile strength testing and then tested using a universal testing machine. In conclusion, it was found that elevated temperature before polymerization may promote higher tensile strength over dentin in indirect restorations. The increase in microtensile bond strength (μTBS) values at 50 °C (from 33.38 to 47.12 MPa) was due to the presence of the self-curing component of benzoyl peroxide (BPO) in Variolink II [22]. This component is activated by heat and decomposes faster into free radicals at higher temperatures compared to room temperature. As a consequence of this, it may contribute to an increase in μTBS values. However, the effectiveness of heating the composite dual cement on μTBS was product dependent, as the greatest effect in terms of increased μTBS was observed for cements with low self-curing components. For Variolink II cement, significant differences were found for temperature and activation mode factors in μTBS results. For Calibra cement, no significant difference was found in μTBS values as a function of temperature and activation mode [22].

Some researchers have observed that the use of elevated temperature in glass ionomer cements improves marginal adhesion, reduces working time, and increases surface microhardness up to the 4 mm level [23][24]. Resin-based materials, such as hybrid composite resins, resin cements, and siloranes, have also been subjected to laboratory studies to evaluate the effects on their physical and photoactivation properties. It was found that the average heating temperature of these materials found in the literature is 54–68 °C and considered safe for pulp in the study conducted [25][26][27]. According to a study by Knezevic et al., the limiting safety temperature for pulp in the context of heating composite materials is 68 °C [28]. Some researchers believe that the harmful threshold for living dental pulp tissue is a 5.5 °C increase in dental pulp temperature [29]. There are also reports confirming that heating composite cement to 60 °C prior to polymerization reduced viscosity and allowed for increased free radical mobility [17][30]. As a consequence, these materials achieved higher monomer conversion at elevated temperatures than when used at room temperature [31][32][33][34][35]. When free monomers are present (lower degree of conversion), it is important to note that they can cause severe allergic reactions, and their occurrence is a highly undesirable phenomenon. The incompletely reacted monomers may also leach into the oral environment, into saliva, and consequently reduce the mechanical strength of the restorations, alter their color, and create the possibility of the development of unfavorable bacterial flora in the oral environment [17].

When monomer conversion is closely related to the mechanical properties of the contained polymer, improved bond strength of indirect restorations to dentin can be expected, even if the polymerization of the dual-bond composite cement is based solely on self-curing components in a chemical reaction [36][37][38].

Studies of microtensile bond strength and resin-dentin adhesive bonding have shown that preheating composite resin for cementing procedures may not improve microtensile bond strength, although it can be used to reduce material viscosity and improve adhesive bonding strength. Ten experimental groups were set up using human molars with three different composite materials. These included Rely X ARC resin cement and Venus and Z250 XT composite resins. The composite resins were tested both at room temperature and when heated to 64 °C. The filling depth was tested using intermediate composite restorations with a height of 2 or 4 mm, previously fabricated on cylindrical molds. Adhesive and luting procedures were performed under simulated pulp pressure. After cementation, teeth were cut into beams with a cross-sectional area of 1 mm<sup>2</sup> at the adhesive interface and subjected to tensile testing at 0.5 mm/min. The characteristics of the adhesive interfaces were evaluated under a scanning electron microscope (SEM). When cementing 2 mm restorations, Z250 XT composite resin preheated or at room temperature achieved significantly higher microtensile bond strength than RelyX ARC cement. At this depth, Venus composite resin did not differ from the resin cement, and for 4 mm restorations only, preheated Venus exhibited significantly higher microtensile bond strengths than RelyX ARC. Preheating the composite resin resulted in a thinner luting bond, with a more intimate interaction between the luting agent and the adhesive layer [39].

In addition to the temperature factor of preheating itself, the time required to achieve the desired fluidity and thus improve the properties of the filler material is equally important. From the available studies, it appears that there is a very wide

variation in heating times. The most common time used to anneal the material was 15 min, and the time span for annealing composite materials, among the available studies, ranged from 40 s to 24 h. In the oral cavity, the action of a constant temperature stimulus affects the maturity of dental materials and improves their properties. High temperatures, reached above the glass transition temperature, can cause irreversible deformation and changes in properties, even over a short period of time <sup>[40][41][42][43]</sup>.

An important consideration regarding the storage conditions of composite cements is the recommendation for storage at or below room temperature. Some composite materials require manufacturer-recommended refrigerated storage, but researchers recommend removing them from the refrigerator just before use. This is attributed to the disruption of some of the properties of the composite resin under reduced temperature conditions, related to the significant dynamics of the composite temperature drop after removal from the heater. A 50% drop in temperature was recorded within 2 min. The best monomer conversion results were achieved when the heated composite was dispensed and used as quickly as possible. Neither repeated nor prolonged heating of the composite significantly affected monomer conversion <sup>[33]</sup>.

From the collected studies, composite resins when heated show significant improvement in physical and strength properties <sup>[44][45][46][47][48][49][50][51][52][53][54]</sup>, such as an increase in conversion rate and a decrease in polymerization shrinkage <sup>[52][53][54][55][56][57][58]</sup>.

Significant differences in the mechanical properties of dual composite cements have been found with chemical activation alone than with light curing alone <sup>[59]</sup>. It has been noted that dual cements also exhibit the greatest fault tolerance during earning compared to resin matrix cements, which are among the most demanding and complicated cements in terms of cementing procedure <sup>[60][61]</sup>.

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