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Effect of pre-cure temperature on the bonding potential of self-etch and self-adhesive resin cements

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ABSTRACT

Objectives. To assess whether the pre-cure temperature of resin cements significantly influenced the bonding potential to dentin.

Methods. Forty extracted molars were randomly divided into 8 groups ($n=5$): Groups (1–4) RelyX Unicem (RU, 3 M ESPE) and Groups (5–8) Panavia F 2.0 (PF, Kuraray Co.), at pre-cure temperatures of 4, 24, 37, and 60 °C, respectively. Cements were used in dual-cure mode for luting composite overlays (Paradigm MZ100, 3 M ESPE) to dentin. Microtensile bond strength testing and scanning electron microscope (SEM) observations of cement–dentin interfaces were performed.

Results. Group 4 had to be eliminated as RU at 60 °C underwent such an accelerated curing that was already set at the time of dispensing. The bond strengths (MPa) measured at refrigerator, room, and intraoral temperature were, respectively: RU 5.4 ± 1.7, 11.4 ± 6.1, 10.6 ± 4.2; PF 7.4 ± 3.7, 13.9 ± 6.2, 12 ± 5.2. The statistical analysis revealed that both luting agents developed a significantly weaker adhesion when used at refrigerator temperature ($p < 0.05$). No statistically significant differences in bond strength were recorded when either cement was used at 24 or 37 °C ($p > 0.05$). Pre-heating of PF to 60 °C resulted in a significant increase in bond strength (20.7 ± 9.4 MPa; $p < 0.05$). SEM observations disclosed an enhanced potential of PF to form a hybrid layer as the temperature increased over 4 °C. RU exhibited a less porous and more homogeneous layer at intraoral than at refrigerated temperature.

Significance. It is advisable to let refrigerator-stored resin cements warm up to at least room temperature prior to clinical use. Pre-heating to 60 °C enhances the bonding potential of PF.

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1. Introduction

Among the materials available for luting indirect restorations and endocanalar posts, a growing interest has been directed toward the use of self-etch and self-adhesive resin cements. The simplification in handling attained with these new agents is expected to make the luting procedure less technique- and operator-sensitive than when using three-step systems [1–5]. Reduced post-operative sensitivity [6], along with moderate pulpal reactions [7], and the ability to adequately bond to different restorative substrates [8] are other clinically relevant properties recognized to self-etch and self-adhesive resin cements. However, limitations have emerged in the bonding potential of these materials to enamel and dentin

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Table 1 – Chemical composition and batch numbers of the tested materials

	ED Primer 2.0	Panavia F 2.0
Panavia F 2.0	Primer A: HEMA, MDP, 5-NMSA, water, accelerator (batch #00186B)	Base paste: hydrophobic aromatic and aliphatic dimethacrylate, sodium aromatic sulphinate, N,N-diethanol-*p*-toluidine, functionalized sodium fluoride, silanized barium glass (batch #00047A)
	Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate (batch #00067B)	Catalyst paste: MDP, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate, silanized silica, photoinitiator, dibenzoyl peroxide (batch #00006A) (filler load 70.8%, particle size 2 μm)

	Powder	Liquid
RelyX Unicem (batch # 70-201115642)	Glass fillers, silica, calcium hydroxide, self-cure initiators, pigments, light-cure initiators (filler load 72% wt, particle size < 9.5 μm)	Methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-cure initiators, light-cure initiators
Paradigm MZ100 (batch #70-2010-3073-4)	bis-GMA, TEGDMA, zirconia silica filler (85% wt)	

HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, *N*-methacryloyl 5-aminosalicylic acid; bis-GMA, bis-phenol A glycidyldimethacrylate; TEGDMA, triethylenglycol dimethacrylate.

[1–5,9–12]. The permeability of the acidic ED Primer (Kuraray Co., Tokyo, Japan) exposed the cement–dentin interface to water-induced changes that negatively affected bond strength of Panavia F (Kuraray Co., Tokyo, Japan) [9]. A limited etching potential and the ability to only superficially interact with dentin were observed for RelyX Unicem (3 M ESPE, Seefeld, Germany) [1–3,5,11,12], and related to the high viscosity of the cement that would hinder deeper resin penetration [3]. In a recent investigation [4], the adhesion to dentin of Panavia F 2.0 and RelyX Unicem benefited from the application of an onlay seating force heavier than finger pressure. Reduction in cement viscosity and porosity, hindrance of water diffusion from dentin, and promotion of chemical and physical interactions possibly accounted for the enhanced coupling.

Lately, evidence has appeared in the literature that viscosity of resin composites can be decreased and their flowability improved by pre-heating to a temperature of 55–60 °C [13–16]. If it is true that several restorative composite resins have their film thickness significantly decreased following pre-heating [14,15], it seems sensible to verify whether similar changes in the flow properties can be induced also in resin luting agents, possibly favoring their marginal adaptation and interfacial strength.

Beside viscosity, temperature also affects polymerization kinetics of resin composites [17–19]. Significant differences in this regard have emerged between the temperature of storage in refrigerator and intraoral temperature [17,18]. Moreover, resin composite pre-heating to 55–60 °C have been proved to relevantly increase monomer conversion [17–19]. Radical mobility would be promoted directly by thermal effect and indirectly as a result of the lowered system viscosity [17–19].

Enhanced conversion is known to have positive effects on several properties, such as surface hardness [16], flexural strength and modulus, fracture toughness, tensile strength, wear resistance [20] that are clinically relevant also for luting materials.

Additionally, if recent *in vitro* [21] and *in vivo* [22] data seem reassuring regarding the risk of pulp damage with 55–60 °C pre-heating, on the other hand it still remains to be assessed whether the combined curing- and heat-related volumetric changes of the resin [15,23] may balance with the effects of pre-heating on the material mechanical, elastic, and rheological properties.

Based on these premises, it seemed of interest to assess whether temperature changes had an impact on interfacial strength and properties of resin luting agents. Particularly, the following temperature levels were set for testing, as representatives of recurring conditions in the handling of the materials: refrigerator storage (4 °C), room (24 °C), and intraoral (37 °C) temperatures, in addition to 60 °C as the pre-heating temperature delivered by a recently marketed composite heater [15–18]. Two simplified handling and universal use resin cements were selected for the trial.

The tested null hypothesis was that the microtensile bond strength of these luting agents to dentin was not affected by the pre-cure temperature of the cement when used to lute composite onlays. The strength assessment was complemented with microscopic observations of the interfacial morphology.

2. Materials and methods

Forty human third molars, extracted after having obtained informed consent under a protocol reviewed and approved by the institutional review board of the University of Siena, Italy, were selected for being free from caries and previous restorations. Teeth were stored in a 1% chloramine T solution at 4 °C until use in the experiment. Storage in the disinfectant was never longer than 1 month. Twenty-four hours before the bonding procedure, all the teeth were abundantly rinsed with distilled water and placed in distilled water at 37 °C.

The self-adhesive resin cement RelyX Unicem and the self-etch resin cement Panavia F 2.0, whose chemical composition is reported in Table 1, were selected to be used in dual-cure mode for luting composite overlays (Paradigm MZ100, 3 M ESPE, St. Paul, MN).

Paradigm MZ100 blocks were cut with a water-cooled diamond saw (Isomet, Buelher, Lake Bluff, IL) into blocks of 2 mm in thickness and with a surface area sufficient to cover the

bonding surface of the selected teeth. The intaglio surface of each composite block was ground with 180-grit SiC paper, cleaned with ethanol, and air-dried.

Teeth were randomly distributed into eight groups, based on the agent to be used for onlay luting and on the cement pre-cure temperature. The following experimental groups were formed:

Groups (1–4) RelyX Unicem at pre-cure temperatures of 4, 24, 37, and 60 °C, respectively.
Groups (5–8) Panavia F 2.0 at pre-cure temperatures of 4, 24, 37, and 60 °C, respectively.

For testing at the pre-cure temperature of 4 °C, the cements were used right after taking them out of the refrigerator, where they had been stored for at least 1 day.

For testing at pre-cure ambient temperature, the luting agents were left outside the refrigerator, at a room temperature of 24 °C, for at least 1 day.

For testing at the pre-cure temperatures of 37 and 60 °C, a thermostatically controlled laboratory oven (Precision Thelco, Thermo Fisher Scientific, Walthan, MA) was used to warm up the luting agents. All the materials were allowed to stabilize at the established temperatures for at least 24 h prior to testing [14].

By pre-heating to 60 °C, however, curing of RelyX Unicem was so accelerated that at the time of dispensing, the cement was already set and could not extrude through the capsule tip. Therefore, Group 4 was excluded from the study.

2.1. *Bonding procedure*

On each tooth, a deep coronal dentin substrate was exposed by removing occlusal enamel and superficial dentin with a slow-speed, water-cooled diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL). The exposed dentin surfaces were polished with wet 180-grit SiC paper [24], rinsed copiously with water, and blot-dried with a cotton gauze for 5 s prior to the luting procedure.

The luting cements were handled in strict accordance with the manufacturers' instructions that are reported in Table 2.

By means of a plunger loaded with a box of lead pellets, whose weight was adjusted based on the substrate area [4], a 2 mm thick composite block was placed on the dentin substrate under a pressure of 40 g/mm^2. The standardized pressure was held for the first 5 min of cement autocure reaction. During this period, the luted tooth was kept in the laboratory oven, where environmental conditions simulating the oral cavity (37 °C temperature, 100% relative humidity) were maintained.

Curing was completed by light irradiation from the top of the 2 mm thick composite block for 20 s with a halogen light-curing unit (XL3000, 3 M ESPE, St. Paul, MN) operating at 550 mW/cm^2. Then, on teeth destined to microtensile testing (3 per group), another 2 mm-thick composite block was luted onto the first one using RelyX Unicem in a self-cure mode, in order to ensure an adequate length for specimen gripping.

After a 24-h storage period at 37 °C under 100% relative humidity, each bonded tooth was sectioned occluso-gingivally into serial slabs, using the Isomet saw under water, cooling at very low speed and with no additional pressure. Each slab was then fixed on a glass slide with double-sided gluing tape, and further sectioned into 0.9 mm × 0.9 mm composite–dentin sticks, according to the technique for the "non-trimming" version of the microtensile test. The specimens were stressed to failure under tension using a universal testing machine (Controls, Milano, Italy) at a crosshead speed of 1 mm/min.

2.2. *Statistical analysis of microtensile bond strength data*

Pre-test failures were included in the statistical calculations as zero values and microtensile beams were considered as statistical units. In one first analysis, strengths achieved by Panavia F 2.0 and RelyX Unicem at refrigerator, room, and intraoral pre-cure temperatures were compared using the Kruskall–Wallis analysis of variance on ranks, followed by the Dunn's multiple range test, as the data did not pass the tests of normality and homogeneity of variances. For the same reason, bond strengths of Panavia F 2.0 at 4, 24, 37, and 60 °C pre-cure temperatures had to be compared with a non-parametric analysis (Kruskall–Wallis ANOVA, Dunn's test). In all the analyses, the level of significance was set at $\alpha = 0.05$.

2.3. *Scanning electron microscopy examination*

Two teeth from each experimental group were used for scanning electron microscope observations of the dentin–cement interface. A 2 mm thick slab containing the coupled interface was sectioned from each luted specimen and polished under wet condition with increasingly finer grits of SiC papers (Buelher, #600, #1000, #1200).

The interface was brought into relief by etching with 32% silica-free phosphoric acid gel (Uni-Etch, Bisco, Schaumburg, IL), followed by brief deproteinization with a 2% sodium hypochlorite solution for 60 s. After rinsing with water and air-drying for 10 s, an impression of the interface was taken using a PVS impression material (Affinis, Coltène-Whaledent

Table 2 – Handling of the luting agents

Panavia F 2.0	- Mix equal amounts of ED Primer 2.0 liquids A and B. Apply the mix on the bonding substrate with a brush and leave it undisturbed for 30 s. Dry with a gentle air flow - Mix equal amounts of base and catalyst for 20 s, apply the cement onto the primed substrate	- Let the cement autocure for 5 min at 37 °C and 100% humidity under pressure (for Panavia F 2.0, apply Oxiguard 2.0 along the exposed margins while the specimen is under pressure)
RelyX Unicem	- Activate the capsule for 2 s and mix it for 10 s with Rotomix (3 M ESPE) - Apply the cement onto the substrate	- Light-cure through the composite overlay for 20 s

AG, Alstätten, Switzerland). After separation of the impression material from the bonded interface, positive replicas were obtained using a polyether impression material (Permadyne, 3 M ESPE AG, Seefeld, Germany). All replicas were mounted on aluminum stubs, coated with a 15–20 nm thick layer of gold by means of the SC7620 Sputter Coater device (Polaron Range, Quorum Technologies, England), and inspected by a scanning electron microscope (JSM-6060LV, JEOL, Tokyo, Japan).

3. Results

3.1. Microtensile bond strength

Descriptive statistics and statistically significant differences are reported for all the tested groups in Table 3.

Both RelyX Unicem and Panavia F 2.0 developed a significantly weaker adhesion when used at the refrigerator storage temperature ($p < 0.05$). Under this condition, the strengths of the two cements were comparable to each other ($p > 0.05$).

No statistically significant differences in bond strength were recorded when either cement was used at the pre-cure temperature of 24 and 37 °C ($p > 0.05$).

Pre-heating of Panavia F 2.0 to a 60 °C temperature resulted in a significant increase in bond strength as compared with all the other pre-cure temperatures ($p < 0.05$).

3.2. Scanning electron microscopy examination

As the pre-cure temperature was increased over that of refrigerator storage, Panavia F 2.0 exhibited an enhanced ability to form a hybrid layer with resin tags that were longer following pre-heating to 60 °C (Fig. 1). When the luting agent was warmed up to 60 °C, rounded droplets of micron and submicron size appeared diffusely within the adhesive layer (Fig. 1c and d).

When RelyX Unicem was used at the refrigerator temperature, the cement layer appeared more porous and less uniform than when the material was warmed to the intraoral temperature. At either pre-cure temperature, no distinct hybrid layer formation could be seen at the dentin–cement interface (Fig. 2).

4. Discussion

In the light of the microtensile testing results, the null hypothesis that temperature does not affect cement–dentin strength has to be rejected. Particularly, the significantly weaker adhesive potential expressed by either luting agent at the 4 °C pre-cure temperature points out that refrigerator-stored materials should be allowed to reach room temperature prior to placement. Similar indications can be found in recent literature dealing with pre-warming of restorative composite resins [17,18]. Greater system viscosity and poor monomer conversion have been reported for composites cured at refrigerator temperature [16–18]. Similar limitations may have affected the interfacial strengths of resin luting agents in our investigation.

No real need emerged from this study to warm up the luting agent from ambient to intraoral temperature, as similar interfacial strengths were achieved for both materials under either pre-cure temperature.

Conversely, a net gain in bond strength resulted from pre-heating the resin cement system Panavia F 2.0 to 60 °C. The improved marginal adaptation as a result of increased flow [13–16] and the optimized polymerization kinetics [17–20] already demonstrated for restorative composites may explain the enhanced adhesion of the warmed resin cement.

With regard to the microscopic aspects of the cement–dentin interfaces, an improved potential for dentin hybridization seemed to be acquired by Panavia F 2.0 as the pre-cure temperature was increased over that of refrigerator storage. The increased flowability of the pre-heated luting system might have allowed for deeper penetration into dentin.

The positive outcome of Panavia F 2.0 pre-heating in this study encourages further investigation of the potential benefits of warming up resin luting agents prior to placement in the cavity preparation. Particularly, it seems interesting the idea to use filler loads even higher than those of current luting agents formulations for the sake of mechanical properties, curing stress, wear resistance. The highly filled materials could then be made more flowable on application by pre-heating, for the purpose of marginal adaptation and with the additional benefit of optimized polymerization [13–16].

Table 3 – Descriptive statistics of the microtensile bond strengths measured for the two cements at the experimental temperatures

Cement	Temperature	N	Mean (MPa)	Standard deviation	Significance ($p < 0.05$)
RelyX Unicem	Group 1: 4 °C	40	5.4	1.7	A
	Group 2: 24 °C	42	11.4	6.1	B
	Group 3: 37 °C	38	10.6	4.2	B
	Group 4: 60 °C	Not tested			
Panavia F 2.0	Group 5: 4 °C	41	7.4	3.7	A a
	Group 6: 24 °C	38	13.9	6.2	B b
	Group 7: 37 °C	43	12	5.2	B b
	Group 8: 60 °C	40	20.7	9.4	c

Different upper case letters indicate statistically significant differences in the strengths measured at 4, 24, and 37 °C for the two cements. Different lower case letters indicate significant differences in strengths measured for Panavia F 2.0 at the four tested temperatures.

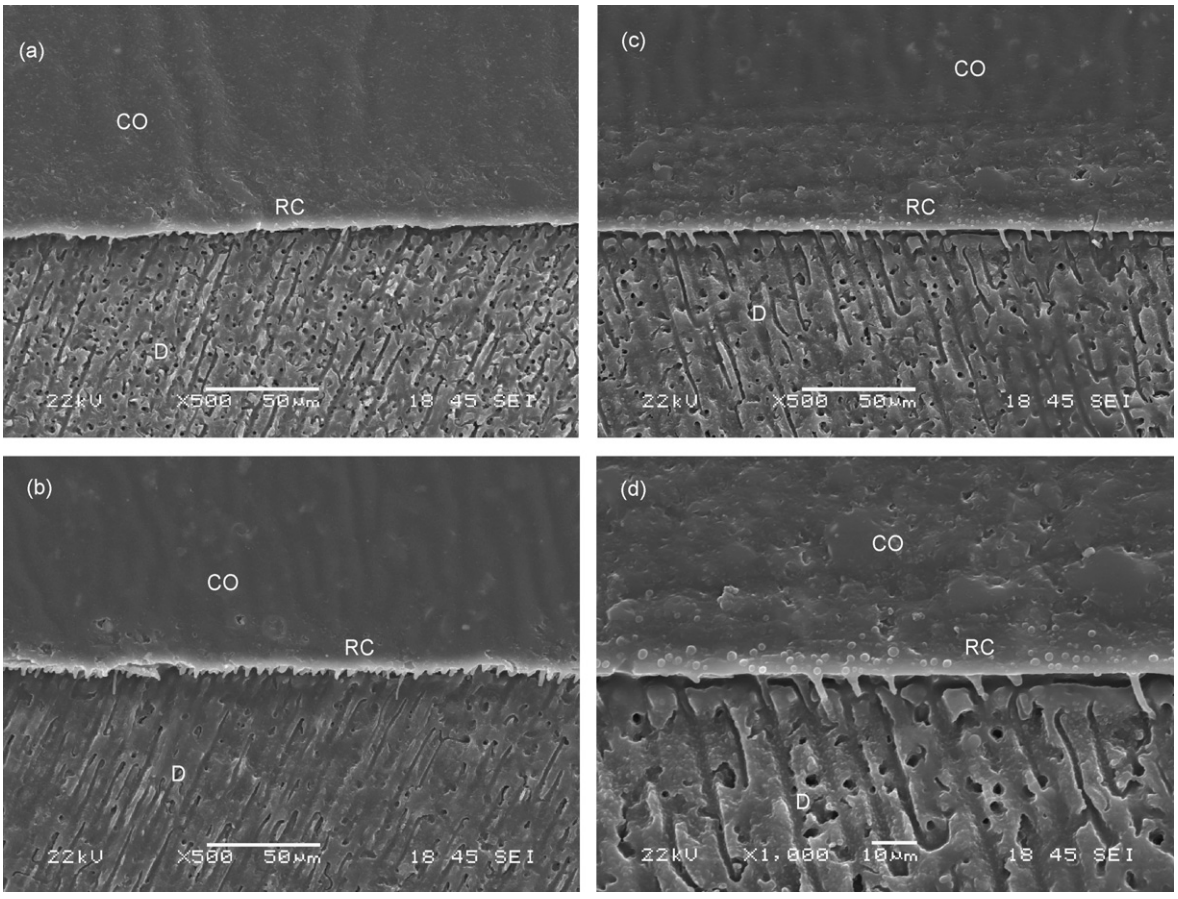

Fig. 1 – SEM micrographs of specimens luted with Panavia F 2.0. (a) The interface developed when the cement was used at the refrigerator storage temperature of 4 °C. A thin hybridized smear layer with few resin tags was visible. (b) The hybridized smear layer became thicker and resin tags appeared more diffuse when the material was warmed up to the intraoral temperature. (c) When the material was pre-heated to 60 °C, longer resin tags were formed and the cement layer appeared uniform (X500, bar = 50 μm. CO = composite overlay, RC = resin cement, D = dentin). (d) Higher magnification of the adhesive interface when the luting agent was warmed up to 60 °C. Rounded droplets of micron and submicron size were diffusely present within the adhesive layer (X1000, bar = 10 μm. CO = composite overlay, RC = resin cement, D = dentin).

Also, it appeared from this study that the increase in resin shrinkage ensuing Panavia F 2.0 heating to 60 °C was of no concern, and was purportedly well balanced by the favorable effects on the material mechanical properties, as both interfacial strength and microscopic aspects were suggestive of a valid cement–dentin coupling.

It should however be pointed out that the configuration factor of this study's setting (onlay luting on a flat dental substrate) is highly favorable to stress dissipation. It would then be of interest to verify whether pre-heating brings similar beneficial effects on cement interfacial strength and adaptation under less favorable geometric configurations, such as luting of crowns, inlays, and endocanalar posts.

Additionally, it should be investigated whether pre-heating of the resin luting agent may be advantageous also for bonding to enamel, such as in veneer luting. In this regard, one recent study reported that the penetration of a self-etch adhesive in ground and unground enamel was greater at room than at refrigerated temperature [25].

Moreover, the possibility to reduce light exposure time still achieving similar monomer conversion, so far investigated only for restorative resin composites [17–19], should be evaluated also for resin luting agents.

It also remains to be ascertained whether pre-heating the luting agent up to 60 °C may involve any risk of pulp damage, especially in consideration of the close pulp proximity existing when indirect restorations are luted to vital prepared teeth. Data lately appeared in literature [21,22] are relieving in this regard, also in consideration of the rapid cool down, the material undergoes between removal from the heater and application on the dental substrate [13]. Temperature drop is expected to be even more relevant in the handling of luting agents that involves a mixing step.

Rapid cooling during mixing and dispensing may also explain why no significant differences in cements strengths and interfacial properties emerged in this study between 37 °C pre-heating and use at room temperature.

Recent work has pointed out that unused restorative composites can be re-heated with no concern about monomer conversion, which was also unaffected by prolonged resin heating. Yet, these conditions need to be verified for resin cements [13].

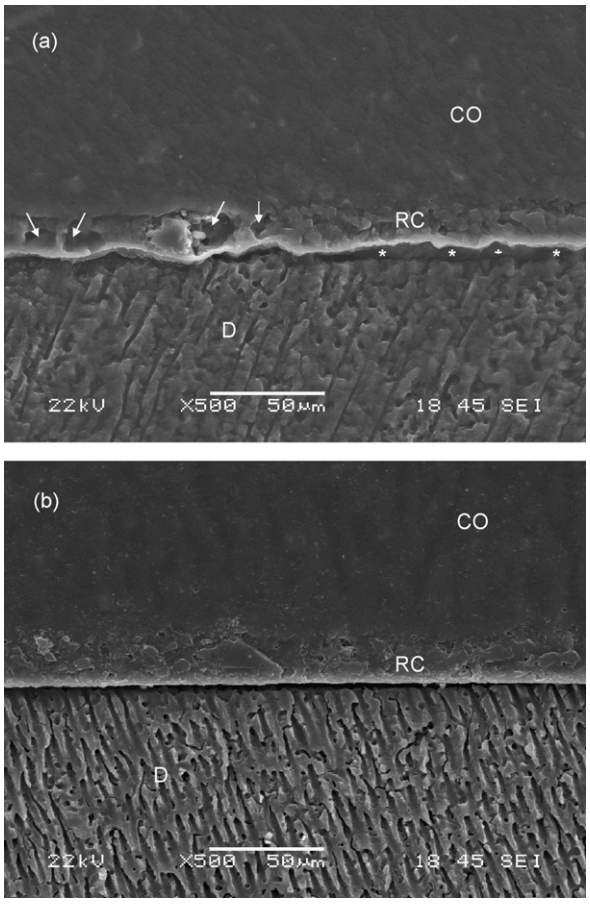

Fig. 2 – SEM micrographs of specimens luted with RelyX Unicem (X500, bar = 50 μm. CO = composite overlay, RC = resin cement, D = dentin). (a) When the cement was utilized at the refrigerator temperature, no definite hybrid layer could be seen. Discontinuities were present along the interface with dentin (asterisks), and the luting agent exhibited porosities (arrows). (b) A more homogeneous cement layer was formed following pre-heating to intraoral temperature. Still no signs of hybrid layer formation were detectable.

Particularly, it would be important to establish whether repeated heating may have an effect on the shelf life of self-etch adhesives. In this regard, the finding of diffuse droplets within the adhesive layer of Panavia F 2.0 following pre-warming at 60 °C (Fig. 1d) deserves attention, as it may represent the microscopic manifestation of phase separation phenomena occurring in the self-etch adhesive as a result of heating. Such phenomena have raised the concern of researchers, as they may expose the adhesive interface to degradation over time, thus affecting the durability of the bond [26]. The long-term stability of bonds established by pre-heated resin luting agents should therefore be assessed in future research.

With regard to RelyX Unicem, on commencing our study, pre-warming to 60 °C seemed justified by the intention to reduce viscosity and improve adaptation to the substrate, thus promoting the contribution of chemical reactions and physical interactions to the overall adhesive mechanism. In addition to viscosity [3], also a low degree of conversion even after light-curing [27] has been indicated as a possible limitation to the strength of RelyX Unicem. Therefore, the enhancement in polymerization kinetics ensuing pre-heating was seen as potentially beneficial.

Nevertheless, 60 °C pre-heating of RelyX Unicem revealed of no use, since setting was so accelerated that at the time of dispensing, the cement could not be extruded from the capsule. Conceivably, heat was effective at catalyzing the chemical reactions that add up to radical polymerization in the setting process of RelyX Unicem [1]. A recent study reported that the setting of glass ionomer cements was hastened under the application of an external heat source, most likely as a result of the activation of polyalkenoic acid chains cross-linking by calcium ions [28]. With regard to RelyX Unicem, it can similarly be speculated that at a certain temperature level above 37 °C the reactions of phosphorylated monomers with ions leached from the basic filler particles were catalyzed, remarkably accelerating the course of setting.

Still, it appears advisable to use RelyX Unicem at room or intraoral temperature, as a more homogeneous cement layer with greater interfacial strength is obtained under such conditions (Table 3; Fig. 2).

In the awareness of the existence of a composite heating device with documented reliability [15–18], the decision was made in this study to use a laboratory oven for pre-warming, as RelyX Unicem capsules would not fit into the commercial device.

Finally, it is worth mentioning that a standardized onlay cementation pressure of 40 g/mm^2 was applied in this experiment in consideration of the outcome of a previous investigation, showing enhanced dentin adhesion when Panavia F 2.0 and RelyX Unicem were maintained, during the autocure period, under a seating force greater than finger pressure [4].

5. Conclusion

In conclusion, pre-cure temperature has a significant influence on adhesion to dentin of a self-etch and a self-adhesive resin cement.

It is advisable to let the refrigerator-stored materials warm up to at least room temperature prior to clinical use, as significantly weaker adhesion is developed at a low temperature.

The bonding potential expressed by either cement following 37 °C pre-heating is similar to when using the materials at room temperature, in plausible relation to the rapid cool down occurring during mixing and dispensing.

Warming up to 60 °C, the working temperature of a commercial composite heater, is of no use with RelyX Unicem, as curing is so accelerated that the cement is already set at the time of dispensing.

Conversely, 60 °C pre-heating effects a significant improvement in interfacial strength and adaptation of Panavia F 2.0, supposedly in relation to the occurrence of favorable changes in flowability, mechanical properties, and polymerization kinetics.

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