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Response of composite resins to preheating and the resulting strengthening of luted feldspar ceramic

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ABSTRACT

Objective. This study evaluated the influence of preheating different composite resins on their viscosity and strengthening yielded to ceramic.

Methods. Modulus of elasticity, Poisson's ratio, and degree of C=C conversion were measured for three restorative composite resins (Z100–microhybrid; Empress Direct–nanohybrid; Estelite Omega–supranano) and one photoactivated resin cement (RelyX Veneer). Viscosity was measured during a heating-cooling curve (25 °C–69 °C–25 °C) and also using isothermal analyses at 25 °C and 69 °C. Feldspar ceramic disks simulating veneers were bonded with the luting materials. Biaxial flexural strength, characteristic strength, and Weibull modulus were calculated at axial positions ($z = 0$ and $z = -t_2$) of the bilayers. Film thickness was measured and morphology at the bonded interfaces was observed. Data were statistically analyzed ($\alpha = 0.05$).

Results. A gradual decrease in viscosity was noticed as the rheometer temperature gradually increased. Viscosity differences between the composite resins were large at the beginning of the analysis, but minor at 69 °C. At 25 °C, the composites were up to 38 times more viscous than the resin cement; at 69 °C the difference was 5-fold. C=C conversion was similar between all resin-based agents. The resin cement yielded lower film thickness than the composites. All resin-based agents were able to infiltrate the ceramic porosities at the interface and strengthen the ceramic. However, the magnitude of the strengthening effect was higher for the preheated composite resins, particularly at $z = -t_2$.

Significance. Selection of composite resin impacts its response to preheating and the resulting viscosity, film thickness, and magnitude of ceramic strengthening.

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1. Introduction

Feldspar ceramics are widely used for laminate veneers in anterior teeth due to their excellent esthetic properties, although the presence of high content of vitreous phase makes them more fragile than most ceramic systems. In order to strengthen the ceramic and enhance its clinical performance, resin-based agents are used to lute the restorations to dental structures [1,2]. The strengthening effect by adhesive luting has been attributed to infiltration of ceramic porosities and sealing of superficial cracks, leading to a better dissipation of mechanical stresses and lower stress concentration at the ceramic structure [3,4]. The use of resin-based agents with higher modulus of elasticity (E) has been reported as having a positive role on the strengthening effect [4–6].

Resin cements are traditionally used for luting all types of glass ceramics and indirect composites [2]. The use of restorative composite resin as luting agent was first cited by Friedman [7] and nowadays is increasingly popular among dentists. Clinical cases and studies [8–10] as well as laboratory investigations [11–15] have assessed the use of preheated restorative composite as luting agent. Composite resins are less expensive than resin cements and maximize the range of shades available. In addition, composite resins have lower polymerization shrinkage/stress and improved mechanical strength due to increased filler loading. Thus, the exposed cement layer at the bonded interface could be more resistant to intraoral degradation, which is positive since marginal failures are reported as one of the main reasons for failure of ceramic veneers [16,17]. Chipping and minor fractures are also reasons for failure of veneers [16,17], and bonding with preheated composite resins could enhance the ceramic strengthening effect. A recent in vitro study showed that lithium disilicate laminate veneers luted with preheated composite resin had less chipping and higher fracture resistance than luted with resin cement [15]. Another study reported higher bond strength to ceramic bonded with a photoactivated restorative composite compared with a dual-cured resin cement [18].

The main shortcoming of using preheated composite resin is the resulting increased film thickness of the luting agent layer [12,13,19]. Selection of composite resin based on its ability to gain flowability upon preheating needs to be appropriate to minimize issues regarding thick cement layers. Maintaining the gained flowability during luting is another challenge because heat dissipation occurs fast after preheating is ceased and the luting agent is applied to the tooth structure [20]. There is a gap in the literature regarding the effect of preheating distinct composite resin formulations, their gains in flowability, and the resulting effect on ceramic strengthening. Thus, this study evaluated the viscosity behavior of different composite resins upon preheating and the strengthening effect yielded to feldspar ceramic disks simulating veneer restorations. The hypothesis tested was that the choice of preheated composite resin affects the mechanical strength of the bonded ceramic.

2. Materials and methods

2.1. *Study design and materials tested*

This in vitro study investigated the effect of four resin-based agents (three preheated restorative composite resins, one resin cement) on the performance of bonded feldspar ceramic disks simulating veneers. The restorative composite resins tested were one microhybrid (Filtek Z100; 3M ESPE, St. Paul, MN, USA), one nanohybrid (IPS Empress Direct; Ivoclar Vivadent, Schaan, Liechtenstein) and one supranano (Estelite Omega; Tokuyama Dental, Tokyo, Japan). Composites from different manufacturers were selected because they have distinct formulations and could react differently to preheating. The commercial photoactivated resin cement RelyX Veneer (shade A1, 3M ESPE) was tested as a reference. Table 1 presents formulation details of the resin-based agents tested. All composite resins were A1 enamel shades. The feldspar ceramic disks were obtained from Vitablocs Mark II A1C for CAD-CAM (Vita Zahnfabrik, Bad Säckingen, Germany). The response-variables related to characterization of the luting agents were viscosity (n = 5), *E* (n = 3), Poisson's ratio (ν, n = 3), and degree of C=C conversion (n = 3). The response-variables for the bonded ceramic were biaxial flexural strength (σ_{bf}, n = 30) and its characteristic strength (σ_0, n = 30), Weibull modulus (*m*, n = 30), and film thickness (n = 30). Scanning electron microscopy (SEM) was used for investigating the morphology of the ceramic-luting agent interfaces (n = 3).

2.2. *Characterization of the luting agents: viscosity, E, and ν*

Viscosity analyses were carried out using a dynamic oscillation rheometer (R/S-CPS+; Brookfield, Middleboro, MA, USA) at 25 °C (average room temperature) and at 69 °C (preheating temperature). The temperature was set to be the same of the device used for preheating the composite resins in the subsequent tests (Hotset; Technolife, Joinville, SC, Brazil). The rheometer itself heated the specimen. Three separate analyses were performed (n = 5 per group), the first analysis measured the viscosity of the composite resins with temperatures rising from 25 °C to 69 °C, then cooling down to 25 °C. The other two analyses were at isothermal conditions with the aim to isolate the effects of temperature: one kept the temperature at 25 °C and another at 69 °C. In all analyses, a 0.5 mL volume of material was dispensed on the lower plate of the rheometer and positioned with a 0.05 mm gap between the plates. Viscosity (MPa s) was measured for 90 s at a constant shear rate of 2 s^{-1}.

For the analyses of *E* and ν, three rectangular specimens (60 × 10 × 4 mm) were prepared for each material. The composite resins were preheated for 5 min after reaching 69 °C (Hotset; Technolife) before being placed in the rectangular molds. The specimens were photoactivated on both sides for 20 s in each window where the LED curing unit (Radii; SDI, Bayswater, Victoria, Australia, irradiance 1200 mW/cm^2) was positioned to cover the entire mold. The specimens were tested 24 h after storage in water at 37 °C using an

Table 1 – Formulation of the resin-based agents tested in the study.

Resin-based agent	Type	Batch	Monomers and fillers[a]	Filler content[a]
RelyX Veneer (3M ESPE)	Photoactivated resin cement	N901407	Bis-GMA, TEGDMA, silica and ceramic particles	66 wt%
Filtek Z100 (3M ESPE)	Microhybrid composite resin	1734800172	Bis-GMA, TEGDMA, silica/zirconia particles	80 wt%/66 vol%
IPS Empress Direct (Ivoclar Vivadent)	Nanohybrid composite resin	V36475	UDMA, Bis-GMA, TCDMA, ytterbium trifluoride, prepolymer, mixed oxide and glass particles	75–79 wt%/52–59 vol%
Estelite Omega (Tokuyama)	Supranano composite resin	101EZ7	Bis-GMA, TEGDMA, silica/zirconia spherical particles	82 wt%/71 vol%

Bis-GMA: bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; TCDMA: tricyclodocane dimethanol dimethacrylate.

[a] Information provided by the manufacturers.

impulse excitation technique (Sonelastic; ATCP Engenharia Física, Ribeirão Preto, SP, Brazil). E and ν were calculated from the sound emitted by the specimen after it suffered a small stroke. The acoustic response is composed by natural vibration frequencies of the specimen, proportional to the E, and its amplitude declines according to the material damping. Spectra with frequency and amplitude by flexural-torsional vibration were registered by a software (ATCP Sonelastic 2.0), ν was calculated considering isotropy [21].

2.3. *Preparation of specimens for biaxial flexure test, film thickness and C═C conversion*

The feldspar ceramic blocks were milled under water-cooling to obtain a cylindrical shape (12 mm diameter). The resulting cylinders were sectioned using a diamond saw under water cooling generating ceramic disks (0.8 ± 0.1 mm thickness) simulating monolithic veneer restorations. All disks had both sides wet-polished using 600- and 1200-grit SiC papers. The final dimensions of each specimen were measured using a digital caliper with 0.001 mm accuracy (Mitutoyo, Tokyo, Japan). A total of 150 ceramic disks was obtained and randomly allocated into four groups (n = 30), according to the luting agent tested. An additional group (control) was tested in which the ceramic specimens were not acid-etched, silanated, nor coated with any luting agent or adhesive.

The intaglio surface of the ceramic specimens simulating veneers was treated with 10% hydrofluoric acid for 60 s (Dentsply Sirona, York, PA, USA), rinsed for 60 s, and dried with water- and oil-free compressed air for 30 s. Two thin layers of a silane coupling agent (Dentsply Sirona) were applied on the surface, and after 60 s, the silane layer was dried with compressed air for 30 s. A thin layer of unfilled adhesive (Scotchbond Multi-Purpose; 3 M ESPE) was applied over the silanated surface and air thinned. The adhesive was not light-cured at this time. The composite resins were heated at 69 °C for 5 min before luting; the ceramic disks were also preheated in the same device to reduce heat dissipation effects. A standard volume of the luting agent was applied to the center of the disk and a Mylar strip was lightly pressed. The disk was centrally oriented on a loading platform and its top ceramic surface was loaded with a 5 N load for 60 s [5]. Excess luting agent was removed using a microbrush, the adhesive and luting agent were photoactivated through the ceramic disk for 40 s. The thickness of the bonded assembly was measured using the digital caliper. The film thickness (n = 30) was calculated as the difference from the bonded specimen and the ceramic disk alone. The specimens were stored at 37 °C in the dark, for 24 h.

The degree of C═C conversion was evaluated using Fourier-transform infrared spectroscopy (Prestige 21; Shimadzu, Tokyo, Japan) equipped with an attenuated total reflectance diamond device. A preliminary reading for the unpolymerized material (monomer) was taken in the absorbance mode using 32 co-added scans and 4 cm^{-1} resolution. Three bonded ceramic specimens for each group were randomly selected and positioned on the spectrometer with the luting agent layer facing the diamond device and another spectrum (polymer) was acquired. The % C═C conversion was calculated, using a baseline technique, considering the intensity of the stretching vibration of aliphatic C═C at 1637 cm^{-1} and, using as an internal standard, the intensity of the aromatic C═C at 1608 cm^{-1}.

2.4. *Biaxial flexural strength (σ_{bf}) test*

The σ_{bf} was evaluated using a ball-on-ring setup on a mechanical testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil). The specimens (n = 30 per group) were positioned on a 10-mm-diameter knife-edged ring support and centrally loaded using a stainless-steel spherical indenter with 4 mm diameter, at a crosshead speed of 1 mm/min until failure. A thin rubber dam sheet was placed between the disk and support to accommodate possible slight irregularities in specimen geometry. The equations used for calculating the σ_{bf} of the uncoated ceramic disks (control group) and bonded specimens (bilayers) were reported in previous studies [5,6]. The E and ν of the ceramic were 70 GPa and 0.25 [22]. σ_{bf} was calculated at z-axial positions at the center of the bilayer specimens, where the ceramic surface at the bonded interface is located (position $z = 0$) and the resin-based agent surface above the ring is located (position $z = -t_2$).

2.5. *Morphology at the bonded interfaces*

Ceramic-luting agent-ceramic sandwiched specimens were prepared (n = 3 per group) to observe the morphology at the bonded interfaces. The specimens were sectioned cross-sectionally using a diamond saw under water cooling and

Table 2 – Means (standard deviations) for viscosity (Pa s) of the different resin-based luting agents tested at room temperature (25 °C) and after preheating (69 °C), n = 5.

Resin-based agent	Temperature		Average viscosity reduction after preheating
	25 °C	69 °C	
RelyX Veneer	545 (71) ^{A,d}	314 (34) ^{B,c}	42.4%
Filtek Z100	7501 (574) ^{A,c}	1549 (160) ^{B,a}	79.3%
Empress Direct	20754 (1422) ^{A,a}	1259 (193) ^{B,b}	93.9%
Estelite Omega	18967 (890) ^{A,b}	1485 (161) ^{B,a}	92.2%

Distinct uppercase letters in the same line indicate significant differences between temperatures; distinct lowercase letters in the same column indicate differences between resin-based agents ($p < 0.05$).

embedded in epoxy resin for the interfaces to be viewed. The specimens were ultrasonically cleaned with distilled water for 30 min and sequentially wet-polished using 600, 1200, 2000, and 2500-grit SiC abrasive papers followed by polishing using felt disc and diamond suspensions of 3 μm and 1 μm. The specimens were ultrasonically cleaned again, dried at 37 °C for 2 h, sputter-coated with gold and examined using SEM (JSM-6610; JEOL Ltd., Tokyo, Japan).

2.6. Statistical analysis

Data for C═C conversion, film thickness, and σ_{fb} were analyzed using One-Way Analysis of Variance (ANOVA) followed by Tukey's post hoc test ($\alpha = 0.05$). Viscosity data were submitted to a Two-Way ANOVA (material × temperature) and Tukey's test. Weibull analysis for σ_{fb} data was performed using the software Minitab v.14 (Minitab Inc., State College, PA, USA). Weibull modulus (m), characteristic strength (σ_0), and 95% upper and lower confidence interval bounds were calculated using the maximum likelihood method. Groups were considered significantly different when the 95% confidence interval bounds did not overlap.

3. Results

3.1. Viscosity as a function of temperature

Table 2 presents the viscosity means (before and after heating) for the different resin-based luting agents tested at room temperature and after preheating. The statistical analysis revealed that the factors material and temperature, and the interaction material × temperature, were all significant ($p < 0.001$). The viscosity at 25 °C was significantly different between all resin-based agents tested ($p < 0.001$). The resin cement was 13–38 times less viscous than the composite resins at room temperature, Empress Direct was 2.8 times more viscous than Filtek Z100. When heated to 69 °C, the resin cement had an average 42.4% reduction in viscosity, whereas viscosity was reduced up to 93.9% for the composite resins. After heating, Empress Direct was significantly less viscous than the other two restorative composites. The resin cement was still the least viscous material after heating, but comparison with the restorative composites was reduced to 5-fold.

Fig. 1 shows the viscosity of the composite resins as a function of time and heating-cooling curve. An increase in viscosity for the three composites was observed in the first 5 s, when the temperature was still below 30 °C, owing to a reaction to shear stress promoted by the rheometer. A gradual decrease in viscosity occurred thereafter as the temperature gradually increased and the shear stress was constant. Although the viscosity of the composite resins was different at the beginning of the analysis, particularly Filtek Z100 compared with the other two materials, differences were minor at 69 °C. When cooling down started (90 s), the composites showed a gradual return to the viscosity observed right after the initial reaction to shearing stress. In contrast to the other two composites, Filtek Z100 remained less viscous at the end of the test when compared to the same temperature in the beginning of the analysis.

Results of the viscosity analyses at isothermal conditions are presented in Fig. 2. These analyses were carried out in order to isolate the effects of temperature, keeping it constant to evaluate the effect of the shearing stress imposed by the rheometer. At 25 °C, it is possible to note that the shear stress promotes an increase in viscosity in the first 10–15 s. A decrease in viscosity follows until reaching more or less constant viscosity values after 20 s. The differences in viscosity were clear between materials, especially Filtek Z100 and RelyX Veneer compared to the other two composite resins. The isothermal analysis at 69 °C showed an increase in viscosity at the first few seconds of the test, even for the resin cement. A decrease in viscosity follows and viscosity stabilization occurs faster compared with the same analysis at 25 °C. The viscosity was similar between the three restorative composites after 12–15 s.

3.2. E, υ, C═C conversion, film thickness, and morphology at the bonded interfaces

Table 3 shows the results for E, υ, degree of C═C conversion, and film thickness. E and υ were not statistically compared, the values were used for calculating σ_{bf}. The C═C conversion was similar between the resin-based agents tested ($p = 0.161$). The film thickness of Empress Direct and Estelite Omega were significantly greater compared with Filtek Z100. The resin cement had significantly lower film thickness than the three restorative composite resins. SEM images of the ceramic bonded interfaces are shown in Fig. 3. It is possible to notice that the acid etched ceramic surface is irregular but all resin-based agents were able to completely infiltrate the surface porosities. The adhesive layer applied to ceramic before the luting material can also be noticed.

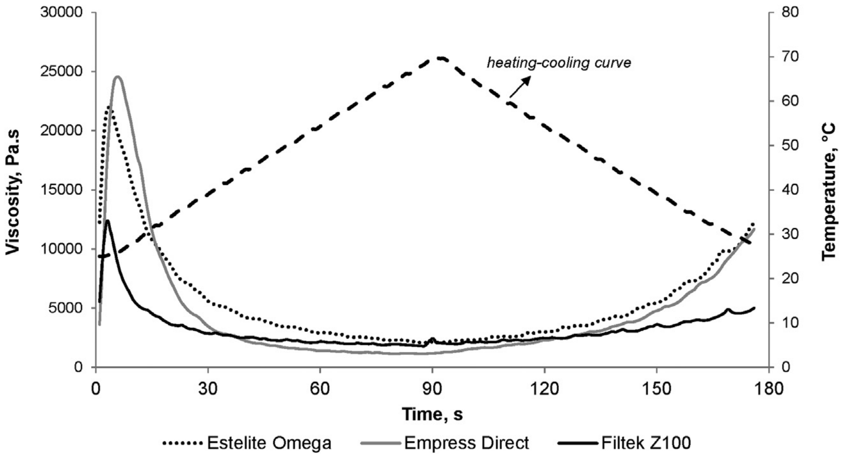

Fig. 1 – Viscosity of the restorative composite resins during the heating-cooling curve. The temperature was increased from 25 °C to 69 °C during 90 s, then reduced back to 25 °C for another 90 s. An increase in viscosity was observed in the first 5 s, when the temperature was still below 30 °C, owing to a reaction to shear stress promoted by the rheometer. A gradual decrease in viscosity occurs thereafter. Differences in viscosity are minor at 69 °C. Filtek Z100 remains less viscous at the end of the test when compared to the same temperature in the beginning of the analysis.

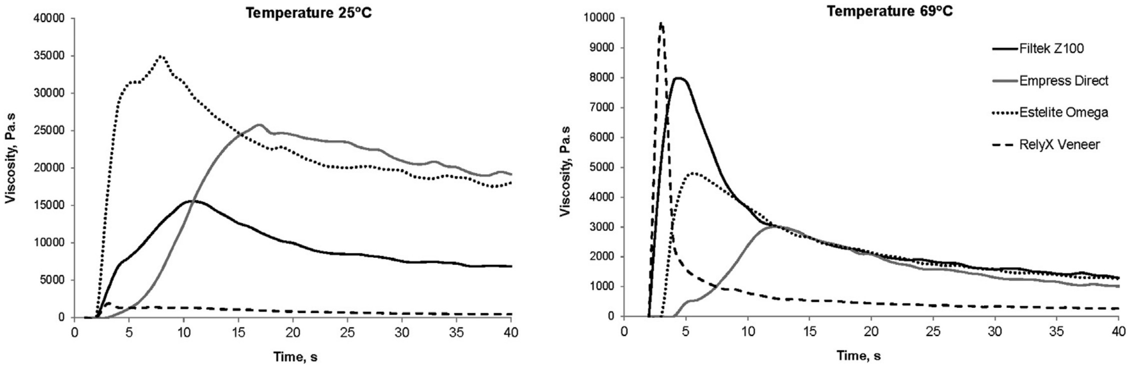

Fig. 2 – Viscosity of all resin-based agents at isothermal conditions, i.e. at constant temperatures of 25 °C or 69 °C. Note that the Y-axes have different range of values in the graphs. At 25 °C, the shear stress promotes an increase in viscosity in the first 10–15 s, then a decrease in viscosity follows. The isothermal analysis at 69 °C showed an increase in viscosity at the first few seconds of the test, even for the resin cement, with a faster decrease in viscosity and stabilization compared with the same analysis at 25 °C.

Table 3 – Means (95% confidence intervals) for elastic modulus (E, n = 3), Poisson's ratio (υ, n = 3), degree of C = C conversion (n = 3), and film thickness (n = 30).

Resin-based agent	E, GPa	υ	C—C conversion, %	Film thickness, μm
RelyX Veneer	10.7 (8.9–12.5)	0.36 (0.27–0.45)	76 (64–89)^a	59 (49–69)^c
Filtek Z100	20.2 (20.1–20.3)	0.20 (0.17–0.23)	61 (49–74)^a	106 (91–121)^b
Empress Direct	10.9 (9.5–12.3)	0.64 (0.48–0.79)	52 (39–64)^a	165 (144–186)^a
Estelite Omega	12.8 (10.8–14.8)	0.56 (0.44–0.68)	58 (46–71)^a	196 (169–223)^a

Distinct letters in each column indicate significant differences between the resin-based agents.

3.3. *Ceramic flexural strength and Weibull analysis*

Table 4 presents the results for σ_{bf}, σ_0, and Weibull modulus for the two axial positions of the bonded ceramic specimens. At z = 0, all luting agents showed higher σ_{bf} than the control group (untreated ceramic). Estelite Omega yielded significantly higher σ_{bf} than RelyX Veneer. The σ_0 of the control group was significantly lower than all bonded groups; σ_0 was not different when the resin cement was compared with the restorative composites. Estelite Omega had higher σ_0 than Filtek Z100. The m was higher for the control group compared with RelyX Veneer. At $z = -t_2$, σ_{bf} and σ_0 of Estelite Omega were significantly higher, but m was lower, compared with the resin cement. Weibull plots for both axial positions of the ceramic disks are shown in Fig. 4.

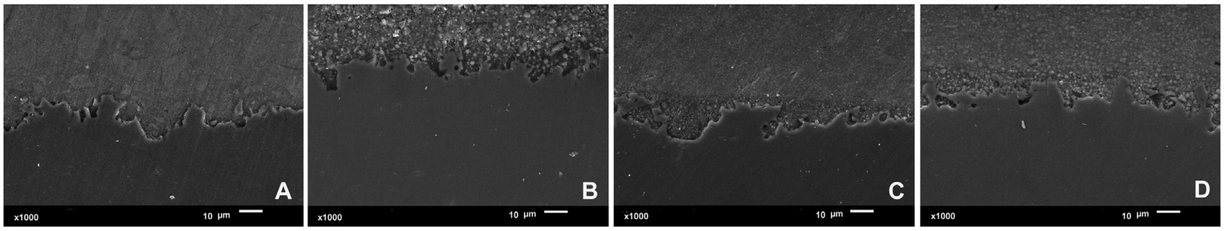

Fig. 3 – SEM images (×1000 magnification) of the ceramic bonded interfaces luted with RelyX Veneer at 25 °C (A), Estelite Omega at 69 °C (B), Empress Direct at 69 °C (C), and Filtek Z100 at 69 °C (D). The acid-etched ceramic surface is irregular but all resin-based agents were able to completely infiltrate the surface porosities. The adhesive layer applied before the luting material can also be noticed.

Table 4 – Estimates (95% confidence intervals) for biaxial flexural strength (σ_{bf}), characteristic strength (σ_0), and Weibull modulus (m), n = 30.

Group	Axial position z = 0			Axial position z = −t$_2$		
	σ_{bf}, MPa	σ_0, MPa	m	σ_{bf}, MPa	σ_0, MPa	m
Control (ceramic)	127 (122–132)^c	131 (128–134)^c	17.1 (13.2–22.1)^a	–	–	–
RelyX Veneer	144 (139–149)^b	151 (145–157)^{ab}	10.0 (7.7–12.9)^b	24.6 (22.0–27.2)^c	25.8 (24.7–26.9)^c	8.9 (6.9–11.4)^a
Filtek Z100	145 (140–150)^{ab}	151 (147–155)^b	13.0 (9.8–17.2)^{ab}	49.2 (46.6–51.8)^b	51.9 (49.7–54.3)^b	8.5 (6.6–11.1)^{ab}
Empress Direct	152 (147–157)^{ab}	159 (153–165)^{ab}	10.5 (8.0–13.8)^{ab}	48.8 (46.2–51.4)^b	51.9 (49.0–54.9)^b	6.7 (6.7–8.6) ^{a}
Estelite Omega	155 (150–160)^a	162 (156–168)^a	10.7 (8.2–13.9)^{ab}	54.9 (52.2–57.5)^a	59.3 (55.0–63.9)^a	5.1 (4.0–6.6)^b

Distinct letters in each column indicate significant differences between groups.

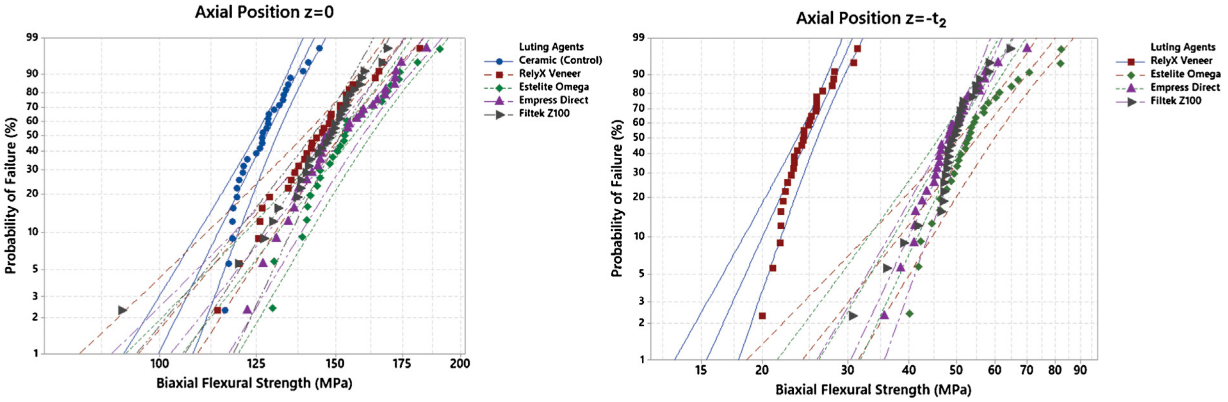

Fig. 4 – Weibull plots for axial positions z = 0 and z = −t$_2$.

4. Discussion

Results of the present study provide evidence that distinct composite resins react differently to preheating and the resulting viscosity, flowability, and film thickness might be affected. In addition, it was observed that selecting a preheated composite resin to lute feldspar ceramics may affect the mechanical performance of the bonded assembly. Therefore, the hypothesis tested cannot be rejected. To the best of authors' knowledge, this is the first study to show such a result, since previous studies have concentrated in comparing preheated composites with room temperature composites or with resin cements, or usually did not test different composites to lute ceramic structures [10–12,14,15,18–20]. The clinical implications of the present findings are following addressed.

The composite resins tested herein differ in resin phase components and particularly in type and fraction of filler particles. These differences led to varied responses to preheating. When analyzed in the rheometer, both the increased temperature and shearing stress reduced the viscosity of the composite resins, and even of the resin cement. These findings are in line with those of previous studies showing that dental composite resins exhibit non-Newtonian nature and pseudoplasticity shear thinning [23,24]. Lower viscosity upon preheating is explained by the expected reduction in fluid viscosity and increased molecular motion by thermal energy, whereas shear thinning occurs due to the disturbance of the interactions between the matrix and fillers, increasing flowability [25,26]. This is why the composite resin should be properly selected to preheat and lute ceramics. A previous study [23] tested 15 different composite resins and showed that changes in viscosity after heating at 37 °C ranged between

41% and 92%, although a higher temperature is needed for luting procedures. Another rheological study highlighted that the influence of preheating on viscosity depends on composite type and even its brand [24]. When comparing different materials, the viscosity generally increases as the filler loading is increased, which explains the higher viscosity observed for the composite resins compared with the resin cement. In addition, particle size and shape, interlocking between filler particles, particle-particle and particle-matrix interfacial interactions may vary among materials and influence the viscoelastic properties. The type and quantity of crosslinking and diluent monomers may also have implications in the response to preheating. Filtek Z100, for instance, was the least viscous material at 25 °C likely due to a high content of TEGDMA. However, this composite had the lowest average reduction in viscosity after preheating.

The isothermal analyses indicated that the shear stress imposed by the rheometer reduced viscosity and increased flowability, but that composite resins may react differently to shear thinning. One good example is the behavior observed for preheated Filtek Z100, as the composite did not return to its initial viscosity when cooled back to room temperature. This is likely related to the hydrodynamic force, strain and heat leading to an alteration in the microstructure of the non-polymerized composite paste [27]. In corroboration, it has been reported that depending on the composite resin, cooling down after preheating increased its shear storage modulus and that a non-linear, unstable behavior during the cooling step occurred [24]. Another study reported improved rheological properties for preheated composite resins even after cooling [27]. For Filtek Z100, this behavior might be considered positive since it could improve its flowability when a cementation load is applied and the material is pressed against the tooth structure and ceramic restoration. This may be the reason why this composite was the most viscous after preheating but still yielded the lowest film thickness among the restorative composite resins tested. Clinically, pseudoplasticity and increased flowability when preheated composites are subjected to external strain may occur in other instances, e.g. when extruding the material from a compule or syringe. Application of ultrasound energy [28] when luting ceramics with preheated composite resins may additionally aid in improving flowability and reducing film thickness, and should be evaluated in a further study. Also, if the laminate veneer is not too thin and the ceramic structure is not too fragile, increased seating pressure by the dentist could also aid in reducing film thickness.

Improved rheological properties even after cooling are important when preheated composite resins are used as luting agents. A short interval in between preheating the composite and actually applying it to the ceramic or tooth surface is crucial, especially for reducing film thickness, since it has been estimated that the temperature decreases 50% after 2 min and 90% 5 min after heating is ceased [20]. In order to minimize these effects, the ceramic disks simulating veneers were also preheated in this study. The same can be accomplished easily in the clinical setup by using a similar heating device employed herein or even a oven, but the tooth structure will not be at the same temperature, thus some reduction is flowability when the luting agent is applied to the tooth should be expected. Therefore, use of ultrasound excitation energy before photoactivation seems to be even more important in the technique for optimizing film thickness. It should be noted that all luting agents had film thicknesses within the range commonly observed clinically for ceramic restorations [29], although the resin cement yielded lower film thickness than all preheated composite resins.

The most viscous composite resin at room temperature was the most fluid material at 69 °C, reinforcing the fact that the formulation plays a significant role on the response of the composite resin to preheating. The resin cement was also preheated in the isothermal analyses, showing a reduction in viscosity below 43%. Despite the mentioned differences observed between the resin-based materials tested, they were all able to fill up the irregularities on the acid-etched ceramic surface. A continuity of the interlocking at the interface is an important aspect related to the strengthening effect of resin-based agents yielded to ceramic structures, and it is likely that the use of a layer of unfilled adhesive aided in filling up the ceramic surface porosities [30]. All resin-based agents tested, including the resin cement, were able to strengthen the ceramic structure as compared with the control group. In addition, the degree of C=C conversion was not different among the materials, likely because all composite resins were preheated to the same temperature, leading to increased monomer mobility and polymer crosslinking.

The mechanical strength of the bonded specimens was calculated at two axial positions of the bilayers. In the clinical scenario, the cement layer will be additionally attached to the supporting tooth structure and improve stress dissipation. At $z = 0$, the differences in mechanical strength yielded by the resin cement compared to the restorative composites were not large (up to 7.3%). However, the structural reliability was lower for the ceramic luted with the resin cement as compared with control ceramic. A $z = -t_2$, differences between the luting materials were large, with gains in strengthening ranging between 98% and 129% when preheated restorative materials were compared with the resin cement. In contrast to the findings at $z = 0$, the differences observed at the cement layer might be of clinical significance because the mismatch in mechanical properties between the luting agent layer, tooth structure, and ceramic might be associated with higher stresses reaching the restoration and lower magnitude of ceramic strengthening [6,31]. Differences in mechanical properties of the luting agent layer and the resulting changes in stress dissipation to supporting tooth structures might explain the results reported by Gresnigt et al. [15]. The authors luted lithium disilicate veneers to teeth and subjected the restorations to cyclic loading. As a result, chipping and fractures were more frequently observed, and the number of endured mechanical cycles and fracture loads were significantly lower, when the veneers were luted with resin cement compared with preheated composite resin.

In previous studies, the *E* of resin-based agents was reported as having a significant role on ceramic strengthening [4–6]. However, in the present study, the stiffest preheated composite resin did not generate the highest strengthening. It seems that the thickness of the luting agent layer had a major role on the reinforcing effect. In biaxial flexure testing, the failure stresses are sensitive to variations in thicknesses of layers because they may modify the neutral axis and the magnitude of shear stresses at the bonded interface [32]. Therefore, this study raises a point that a thicker layer of resin-based luting agent layer perhaps might be positive for the reinforcement of thin feldspar ceramic veneers. It has to be further considered that, unlike other studies [15,33], results of the present investigation did not take into account the effects that mechanical aging might have on degradation at the cement layer and ceramic-cement interface with time. The differences between materials found here could be even more appreciable if aging conditions were present, thus further studies are warranted.

5. Conclusions

Distinct composite resins respond differently to preheating, leading to differences in viscosity and flowability. As a result, film thickness and the magnitude of ceramic strengthening depend on the selection of preheated composite resin used as luting agent.

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REFERENCES

[1] Perroni AP, Bergoli CD, Dos Santos MB, Moraes RR, Boscato N. Spectrophotometric analysis of clinical factors related to the color of ceramic restorations: a pilot study. J Prosthet Dent 2017;118:611–6.

[2] Perroni AP, Kaizer MR, Della Bona A, Moraes RR, Boscato N. Influence of light-cured luting agents and associated factors on the color of ceramic laminate veneers: a systematic review of in vitro studies. Dent Mater 2018;34:1610–24.

[3] Fleming GJ, Maguire FR, Bhamra G, Burke FM, Marquis PM. The strengthening mechanism of resin cements on porcelain surfaces. J Dent Res 2006;85:271–6.

[4] Spazzin AO, Bacchi A, Alessandretti R, Santos MB, Basso GR, Griggs J, et al. Ceramic strengthening by tuning the elastic moduli of resin-based luting agent. Dent Mater 2017;33:358–66.

[5] Addison O, Sodhi A, Fleming GJ. Seating load parameters impact on dental ceramic reinforcement conferred by cementation with resin-cements. Dent Mater 2010;26:915–21.

[6] Spazzin AO, Guarda GB, Oliveira-Ogliari A, Leal FB, Correr-Sobrinho L, Moraes RR. Strengthening of porcelain provided by resin cements and flowable composites. Oper Dent 2016;41:179–88.

[7] Friedman M. Multiple potential of etched porcelain laminate veneers. J Am Dent Assoc 1987. Spec No:83E-87E.

[8] Gresnigt MM, Cune MS, Schuitemaker J, van der Made SA, Meisberger EW, Magne P, et al. Performance of ceramic laminate veneers with immediate dentine sealing: an 11 year prospective clinical trial. Dent Mater 2019;35:1042–52.

[9] Signore A, Kaitsas V, Tonoli A, Angiero F, Silvestrini-Biavati A, Benedicenti S. Sectional porcelain veneers for a maxillary midline diastema closure: a case report. Quintessence Int 2013;44:201–6.

[10] D'Arcangelo C, De Angelis F, Vadini M, D'Amario M. Clinical evaluation on porcelain laminate veneers bonded with light-cured composite: results up to 7 years. Clin Oral Invest 2012;16:1071–9.

[11] Almeida JR, Schmitt GU, Kaizer MR, Boscato N, Moraes RR. Resin-based luting agents and color stability of bonded ceramic veneers. J Prosthet Dent 2015;114:272–7.

[12] Mounajjed R, Salinas TJ, Ingr T, Azar B. Effect of different resin luting cements on the marginal fit of lithium disilicate pressed crowns. J Prosthet Dent 2018;119:975–80.

[13] Sampaio CS, Barbosa JM, Caceres E, Rigo LC, Coelho PG, Bonfante EA, et al. Volumetric shrinkage and film thickness of cementation materials for veneers: an in vitro 3D microcomputed tomography analysis. J Prosthet Dent 2017;117:784–91.

[14] Magne P, Razaghy M, Carvalho MA, Soares LM. Luting of inlays, onlays, and overlays with preheated restorative composite resin does not prevent seating accuracy. Int J Esthet Dent 2018;13:318–32.

[15] Gresnigt MM, Ozcan M, Carvalho M, Lazari P, Cune MS, Razavi P, et al. Effect of luting agent on the load to failure and accelerated-fatigue resistance of lithium disilicate laminate veneers. Dent Mater 2017;33:1392–401.

[16] Fradeani M, Redemagni M, Corrado M. Porcelain laminate veneers: 6- to 12-year clinical evaluation—a retrospective study. Int J Periodontics Restor Dent 2005;25:9–17.

[17] Layton DM, Walton TR. The up to 21-year clinical outcome and survival of feldspathic porcelain veneers: accounting for clustering. Int J Prosthodont 2012;25:604–12.

[18] Kameyama A, Bonroy K, Elsen C, Lührs AK, Suyama Y, Peumans M, et al. Luting of CAD/CAM ceramic inlays: direct composite versus dual-cure luting cement. Biomed Mater Eng 2015;25:279–88.

[19] Blalock JS, Holmes RG, Rueggeberg FA. Effect of temperature on unpolymerized composite resin film thickness. J Prosthet Dent 2006;96:424–32.

[20] Daronch M, Rueggeberg FA, Moss L, Goes MF. Clinically relevant issues related to preheating composites. J Esthet Restor Dent 2006;18:340–50.

[21] Schwantz JK, Oliveira-Ogliari A, Meereis CT, Leal FB, Ogliari FA, Moraes RR. Characterization of bis-acryl composite resins for provisional restorations. Braz Dent J 2017;28:354–61.

[22] Zeng K, Oden A, Rowcliffe D. Evaluation of mechanical properties of dental ceramic core materials in combination with porcelains. Int J Prosthodont 1998;11:183–9.

[23] Al-Ahdal K, Silikas N, Watts DC. Rheological properties of resin composites according to variations in composition and temperature. Dent Mater 2014;30:517–24.

[24] Metalwala Z, Khoshroo K, Rasoulianboroujeni M, Tahriri M, Johnson A, Baeten J, et al. Rheological properties of contemporary nanohybrid dental resin composites: the influence of preheating. Polym Test 2018;72: 157–63.

[25] Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. Dent Mater 2006;22:515–26.

[26] Lee I, Chang J, Ferracane J. Slumping resistance and viscoelasticity prior to setting of dental composites. Dent Mater 2008;24:1586–93.

[27] Ahn KH, Lim S, Kum KY, Chang SW. Effect of preheating on the viscoelastic properties of dental composite under different deformation conditions. Dent Mater J 2015;34:702–6.

[28] Schmidlin PR, Zehnder M, Schulup-Mityklo C, Gohring TN. Interface evaluation after manual and ultrasonic insertion of standardized class I inlays using composite resin materials of different viscosity. Acta Odontol Scand 2005;63:205–12.
[29] Akin A, Toksavul S, Toman M. Clinical marginal and internal adaptation of maxillary anterior single all-ceramic crowns and 2-year randomized controlled clinical trial. J Prosthodont 2015;24:345–50.
[30] Naves LZ, Soares CJ, Moraes RR, Gonçalves LS, Sinhoreti MAC, Correr-Sobrinho L. Surface/interface morphology and bond strength to glass ceramic etched for different periods. Oper Dent 2010;35:420–7.
[31] Cao X, Fleming GJ, Addison O. The impact of resin-coating on sub-critical crack extension in a porcelain laminate veneer material. Dent Mater 2017;33:498–504.
[32] Young WC, Budynas RG, Sadegh AM. Roark's formulas for stress and strain. 8th edition New York: The McGraw-Hill Companies; 2011.
[33] Barbon FJ, Moraes RR, Boscato N, Alessandretti R, Spazzin AO. Feldspar ceramic strength and the reinforcing effect by adhesive cementation under accelerated aging. Braz Dent J 2018;29:202–7.