Influence of inorganic filler content of resin luting agents and use of adhesive on the performance of bonded ceramic

Fabíola Jardim Barbon, DDS, MSc,[a] Rafael R. Moraes, DDS, MSc, PhD,[b] Cristina Pereira Isolan, DDS, MSc, PhD,[c] Aloísio Oro Spazzin, DDS, MSc, PhD,[d] and Noéli Boscato, DDS, MSc, PhD[e]

Feldspathic ceramics are commonly used for laminate veneer restorations because of the excellent optical properties[1] associated with their high vitreous phase content.[2-4] The clinical success, longevity, and strengthening of thin feldspathic ceramic restorations are linked to the luting procedures[5-8] and the achievement of an adequate bonded interface between the ceramic and resin-based luting agent (RBLA).[6]

The quality of the bonded interface and the mechanical strength of feldspathic ceramic have been associated with the modulus of elasticity (*E*)[7-9] and the particle size of filler content in RBLAs.[8] However, evidence on the effect that the filler content of RBLAs may have on the performance of bonded veneers is lacking. Inorganic filler content affects the viscosity (η) of RBLAs[10,11] and thus may interfere with

ABSTRACT

Statement of problem. The inorganic filler of resin-based luting agents and the use of an adhesive layer could influence the bonding ability, mechanical performance, and interface morphology of bonded feldspathic ceramic.

Purpose. The purpose of this in vitro study was to investigate the influence of resin-based luting agents loaded with different inorganic filler content, with or without an adhesive, on microtensile bond strength, biaxial flexural strength, and the adhesive interface morphology of bonded ceramic specimens.

Material and methods. Experimental resin-based luting agents with low (55wt%), intermediate (65wt%), and high (75wt%) filler contents were bonded to ceramic specimens, with or without a layer of adhesive. The resin-based luting agents were characterized by measuring viscosity, elastic modulus, Poisson ratio, and degree of conversion (n=3 for each test). The response variables for the bonded ceramic specimens were ceramic-resin microtensile bond strength (n=30), biaxial flexural strength (n=30) and characteristic strength, and Weibull modulus. Scanning electron microscopy was used for fractographic and interface analyses of the specimens fractured in the microtensile test (n=3). Data were subjected to ANOVA with the post hoc Tukey test (α=.05). Weibull moduli were also calculated.

Results. Increased inorganic filler content yielded significantly higher viscosity, stiffness, and film thickness. However, the Poisson ratio and degree of conversion were not affected. The lowest bond strength values were observed for the resin-based luting agents with high inorganic filler content when no adhesive was used and for the resin-based luting agent with low filler content when the adhesive was used. The increase in filler content of the resin-based luting agent appeared to be associated with a higher frequency of mixed failures. Increased filler content resulted in higher biaxial flexural and characteristic strength and decreased structural reliability. The adhesive helped fill irregularities on ceramic and slightly increased film thickness but had only a minor effect on mechanical strength.

Conclusions. Experimental resin-based luting agent loaded with high inorganic filler content strengthened the bonded feldspathic ceramic and yielded significantly higher viscosity and film thickness. In contrast, the bond strength was lower if no adhesive was used. (J Prosthet Dent 2019;122:566.e1-e11)

Clinical implications

An adhesive is indicated to help fill the ceramic irregularities, yielding better bond strength and mechanical performance in thinner ceramic restorations luted with more viscous resin-based luting agents.

their ability to penetrate the micromechanical retention created on ceramic by acid etching,[12,13] in turn influencing the strength of the ceramic.[14,15] Unfilled grooves at the adhesive interface may result in areas of stress concentrations upon loading.[16-18]

The use of adhesive resin on the silanated ceramic surface before application of the RBLA may aid in filling surface irregularities.[13,19] However, the impact of that adhesive on the performance of ceramic veneers has not received much attention. While it is likely that the adhesive layer is too thin to influence the overall strength of the bonded assembly, increased ceramic strengthening has been associated with RBLAs of higher elastic moduli.[9,20] As RBLAs with increased filler content may show differences in elastic moduli and varied ability to penetrate the etched ceramic, their combination with adhesive resin in bonding to ceramic warrants investigation.

In the present study, experimental RBLAs with different inorganic filler content were formulated and characterized. The experimental RBLAs were bonded to a feldspathic ceramic, with or without a layer of adhesive, and the mechanical performance of the bonded interface was also investigated. The null hypotheses tested were that the inorganic filler content of the RBLAs and the use of an adhesive layer would not influence the performance of the bonded feldspathic ceramic interface.

MATERIAL AND METHODS

The experimental design of this in vitro study and the list of materials and equipment used are shown in Figure 1 and Table 1. The experimental RBLAs were formulated with a 1:1 mass ratio of the monomers urethane dimethacrylate and triethyleneglycol dimethacrylate. Camphorquinone (0.4wt%) was used as a photosensitizer and ethyl 4-dimethylamino benzoate (0.8wt%) as a co-initiator. Barium borosilicate glass particles (2 μm average size) coated with 1wt% silane coupling agent were used as fillers. Three different RBLAs were created with different inorganic filler contents: low filler content (55wt %), intermediate filler content (65wt%), and high filler content (75wt%). The materials were mechanically mixed by using a centrifugal mixer (SpeedMixer DAC150) at 1500 rpm for 20 seconds. The commercially available photoactivated RBLA (RelyX Veneer), shade translucent,

was used as a control. This RBLA contains 66wt% filler content, which is similar to the amount in the intermediate experimental RBLA tested.

For η characterization, a parallel plate rheometer (R/S-CPS+) with a temperature controller was used to measure the viscoelastic behavior of the materials (n=3). A 0.5-mL volume of each material was dispensed, and η (Pa.s) was measured for 30 seconds by using 30 counts, 100 seconds^{-1} constant shear rate, and 23 °C temperature. In the E and ν characterization, rectangular specimens (60×10×4 mm) were tested (n=3) by an impulse excitation technique (Sonelastic). E and ν were calculated from the sound emitted by the specimen based on the acoustic response impulse yielded by its natural vibration frequencies.[21-23]

The degree of C=C conversion of the RBLAs (n=3) was evaluated by using a Fourier-transform infrared spectrophotometer (Prestige 21) equipped with an attenuated total reflectance diamond device. A preliminary reading for the unpolymerized material (monomer) was made in the absorbance mode with 32 coadded scans and 4 cm^{-1} resolutions. The material was light-polymerized by using a light-emitting diode unit (Radii; SDI) for 30 seconds at 1200 mW/cm^2 irradiance, and then another spectrum was acquired.[24]

Feldspathic ceramic blocks (VITABLOCS Mark II A1C) were obtained from the manufacturer with dimensions of 12×14×7 mm. For μTBS and failure analysis, the surface of each block was sequentially polished with 600- and 1200-grit SiC abrasive papers (Norton SA) under running water. The polished surfaces were treated with 10% hydrofluoric acid (Condac Porcelain 10%) for 60 seconds, washed for 60 seconds, and dried with water and oil-free compressed air for 30 seconds. The surfaces were additionally etched with 37% phosphoric acid (Condac 37) for 30 seconds for cleaning and then washed and dried. Two layers of a silane coupling agent (RelyX Ceramic Primer) were applied on the surfaces, and after 60 seconds, the silane layer was dried with compressed air for 30 seconds.[25] In 4 blocks, a thin layer of an adhesive (Adper Single Bond 2) was applied at the acid-etched, silanated surface, followed by solvent air-drying. The adhesive was not light-polymerized at this time.

Composite resin blocks (Llis, shade A2D) with the same dimensions as the ceramic blocks were bonded to the ceramics with 1 of the RBLAs tested, with or without an adhesive. A cementation load of 5 N was applied for 2 minutes and light-polymerized for 40 seconds. The RBLA with a high filler content was heated to 60 °C for 30 minutes before luting.[26,27] Each bonded block was sectioned into 30 beam-shaped composite resin-ceramic specimens with a 0.8-mm^2 bonded area. The beam-shaped specimens were submitted for a μTBS test on a mechanical testing machine (DL500) at a crosshead
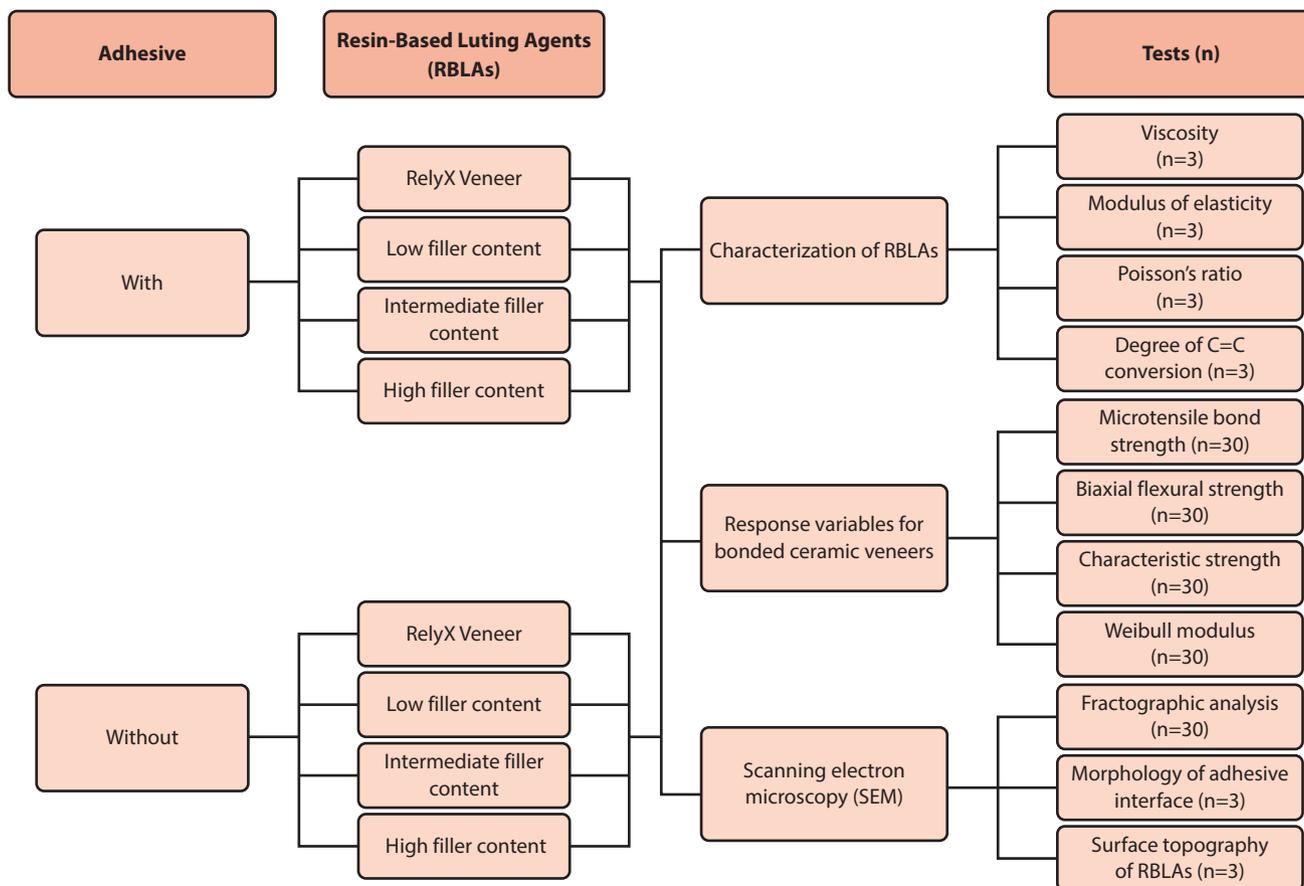
Figure 1. Experimental design.

Table 1. Materials and equipment used

Materials	Manufacturer
Urethane dimethacrylate (UDMA)	Esstech Inc
Triethyleneglycol dimethacrylate (TEGDMA)	Esstech Inc
Camphorquinone	Sigma-Aldrich
Ethyl 4-dimethylamino benzoate (EDAB)	Sigma-Aldrich
Barium-borosilicate glass particles (2 μm)	Esstech Inc
RelyX Veneer shade translucency	3M ESPE
I14 A1 VITABLOCS Mark II for CEREC	VITA Zahnfabrik
SiC abrasive papers	Norton SA
Condac Porcelain 10%	FGM
Condac 37	FGM
RelyX Ceramic Primer	3M ESPE
Adper Single Bond 2	3M ESPE
Llis shade A2D	FGM
Equipment	**Manufacturer**
Asymmetric Centrifugal SpeedMixer DAC150	FlackTek
R/S CPS Rheometer	Brookfield Brasil
Advanced Impulse Excitation Technique Sonelastic	ATCP
Infrared Spectrometer Prestige 21	Shimadzu
Isomet1000 precision cutter	Buhler
EMIC DL500 mechanical testing machine	EMIC Instron
JSM-6610LV Scanning Electron Microscope	JEOL Ltd
Digital Calipers 0-150 mm/0.01 mm	Mitutoyo

Table 2. Means (standard deviations) for Young modulus of elasticity (E), Poisson ratio (v), degree of C=C conversion, and viscosity (η) of resin-based luting agents tested (n=3)

| Resin-Based Luting Agents	η, Pa.s	E, GPa	v	C=C Conversion, %
RelyX Veneer (control)	179 514 (68 834)[b]	10.7 (1.6)[a]	.36 (0.24)[a]	41.3 (2.4)[b]
Low filler content (55wt%)	1936 (476)[d]	6.6 (1.3)[c]	.43 (0.18)[a]	52.7 (1.2)[a]
Intermediate filler content (65wt%)	30 879 (14 345)[c]	7.8 (0.03)[bc]	.44 (0.20)[a]	52.6 (1.2)[a]
High filler content (75wt%)	1 011 288 (779 242)[a]	10.4 (0.8)[ab]	.61 (0.04)[a]	51.7 (4.5)[a]

Different letters in each column indicate significant differences between resin-based luting agents (*P*<.05).

speed of 0.5 mm/min until failure. The fractured specimens were examined under ×40 optical magnification, and the failure modes were classified as premature debonding (specimens debonded spontaneously during sectioning or testing), interfacial failure (adhesive), mixed failure (failure involving more than one substrate, such as the composite resin, RBLA, and/or the ceramic surface), or cohesive failure (failures within one substrate). Premature and cohesive failures were not considered when

Table 3. Means (95% confidence intervals) for microtensile bond strength (μTBS), characteristic strength (μ$_0$), and Weibull modulus (m), n=30

Resin-Based Luting Agents	μTBS, MPa		μ$_0$, MPa		m	
	Without Adhesive	With Adhesive	Without Adhesive	With Adhesive	Without Adhesive	With Adhesive
RelyX Veneer	26.0 (23.3-28.6)^{B,ab}	33.9 (30.2-37.6)^{A,a}	28.1 (25.1-31.4)^{B,a}	37.6 (33.8-41.7)^{A,a}	3.5 (2.6-4.6)^{A,a}	3.7 (2.8-4.8)^{A,a}
Low	25.1 (22.6-27.6)^{A,ab}	23.4 (21.6-25.2)^{A,c}	27.6 (25.2-30.3)^{A,a}	25.3 (23.5-27.3)^{A,b}	4.2 (3.1-5.6)^{A,a}	5.1 (3.9-6.7)^{A,a}
Intermediate	28.1 (25.8-30.4)^{A,a}	28.9 (26.6-31.2)^{A,ab}	30.4 (28.0-33.6)^{A,a}	31.5 (29.2-34.0)^{A,a}	4.4 (3.4-5.7)^{A,a}	5.2 (3.9-7.0)^{A,a}
High	21.4 (19.9-22.9)^{B,b}	25.6 (23.2-28.0)^{A,bc}	23.1 (21.5-24.6)^{B,b}	29.8 (26.1-34.0)^{A,ab}	6.2 (4.5-8.6)^{A,a}	3.0 (2.3-3.9)^{B,a}

Low (55wt%), intermediate (65wt%), and high (75wt%) refer to filler content of experimental resin-based luting agents. m Indicates reliability of bond strength, and μ$_0$ indicates value at which 63.21% of specimens tested.[37,38] Uppercase letters in same row indicate significant differences, with or without layer of adhesive; lowercase letters in same column indicate differences between luting agents (P<.05).
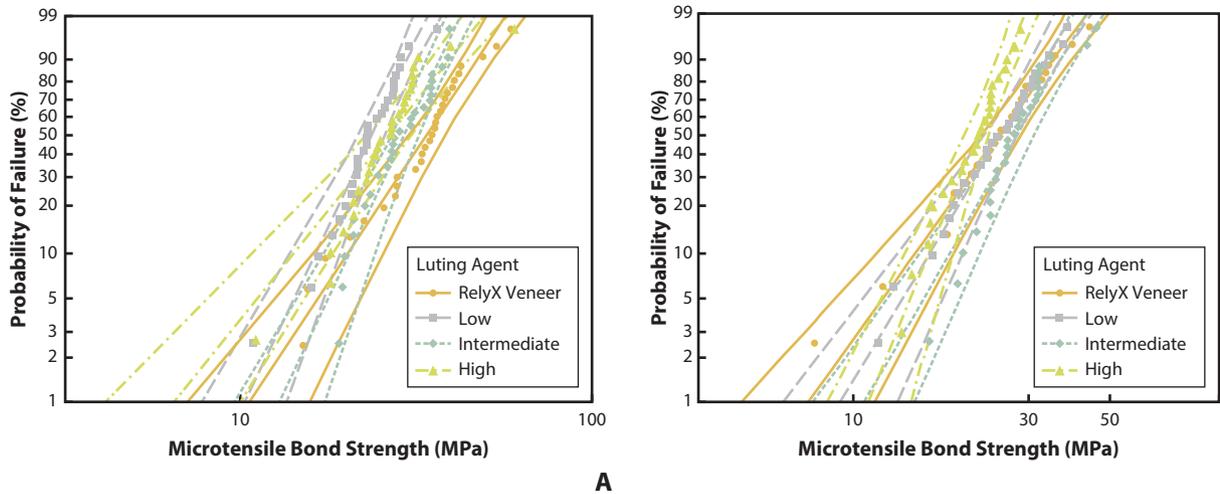
Figure 2. Weibull plots showing probability of failure (%) versus microtensile bond strength (MPa) for all resin-based luting agents. A, With adhesive. B, Without adhesive. No significant differences in Weibull moduli observed across groups except higher m observed when adhesive used for luting agent with high filler content.

calculating the μTBS. The specimens of each group were examined with SEM (JSM-6610) to determine the fracture origin based on fractographical principles.[28]

For σ$_{bf}$ ceramic disks (0.8 ±0.1 mm in thickness and 12 mm in diameter) were obtained and wet-polished, simulating monolithic restorations (n=30). A monolayer ceramic group was tested, in which the specimens were acid-etched and silanated but not coated with any RBLA or adhesive. In the 8 bonded groups, one side of each ceramic disk was prepared with the same procedures described in μTBS. The top ceramic surface was loaded with a 5-N load for 60 seconds.[9] The RBLA was light-polymerized through the ceramic disk for 40 seconds. The thickness of the RBLA layer was subsequently measured.

The σ$_{bf}$ of the specimens was evaluated with a ball-on-ring arrangement on a mechanical testing machine (DL500) at a crosshead speed of 1 mm/min until failure. The σ$_{bf}$ of the uncoated ceramic disks (monolayer ceramic group) was calculated.[9,12,29,30] The σ$_{bf}$ of the resin-coated specimens was calculated according to the analytical solutions described in previous studies.[6,9,31,32] The 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 RBLA surface above the ring of the ball-on-ring setup is located (position z=-t$_2$).

For the statistical analysis, data from the characterization of the RBLAs (E, ν, C=C conversion, and η) were analyzed with 1-way ANOVA. Nonparametric data were transformed on ranks. Then, σ$_{bf}$ and μTBS data were analyzed with 2-way ANOVA (filler content of the RBLA×use of adhesive resin). Post hoc comparisons were carried out with the Tukey test (α=.05). Weibull moduli were also calculated for σ$_{bf}$ and μTBS data by using a software program (Minitab, version 14; Minitab Inc). The parameters m, σ$_0$, μ$_0$, and 95% upper and lower confidence bounds were calculated with the maximum likelihood method. RBLAs were considered significantly different when the 95% confidence interval bounds did not overlap. Descriptive data were used to report the failure modes.

To observe the surface topography of the RBLAs tested and the morphology of the bonded interfaces, disks (6×2 mm) of each RBLA (n=3) and ceramic-RBLA-ceramic sandwiched specimens were obtained (n=3).[33] The specimens were sputter-coated with gold and examined by SEM. These specimens were also
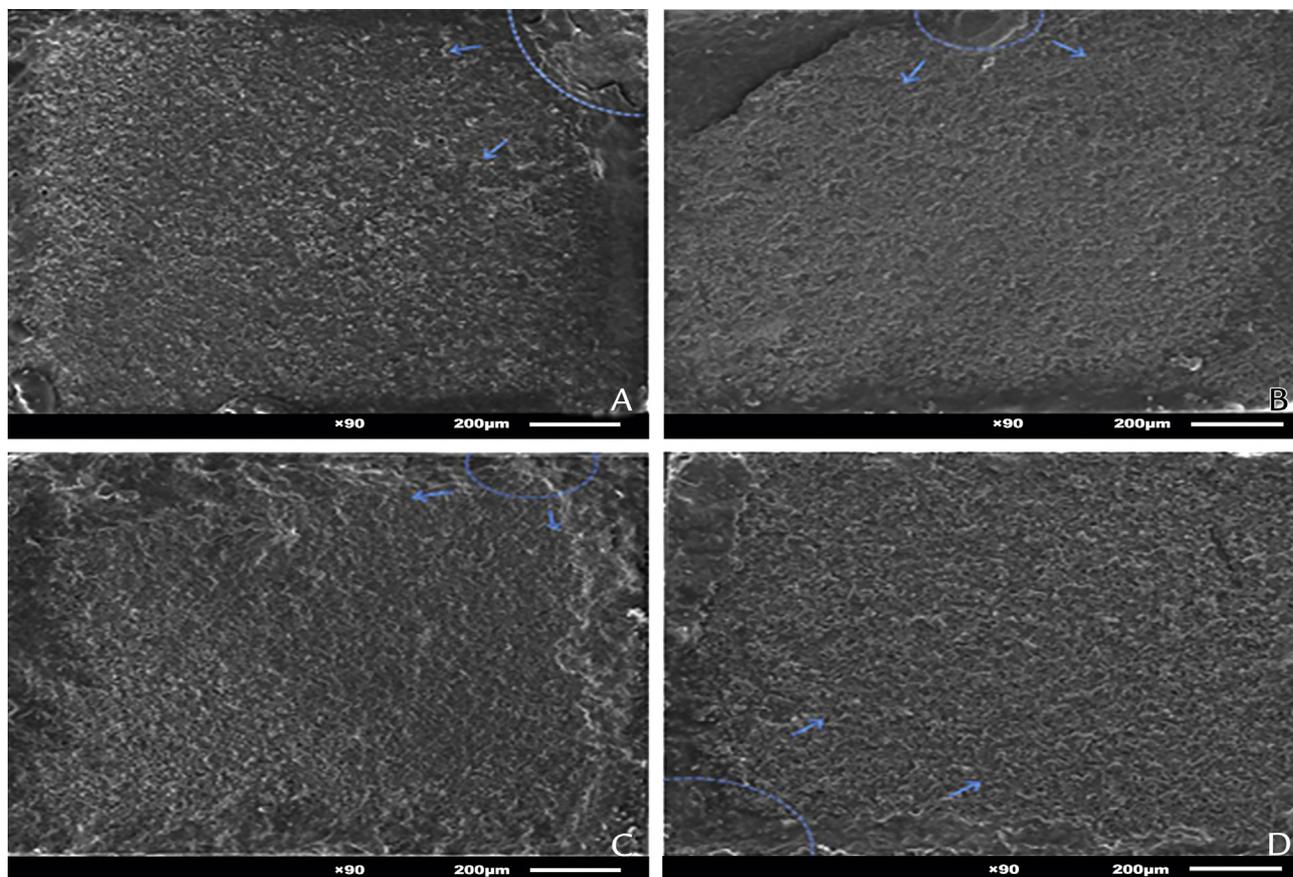
Figure 3. Scanning electron microscope images of surfaces from fractured specimens in microtensile bond strength test. Original magnification ×90. Dotted lines indicate critical flaws and arrows indicate hackle marks. Images show resin-based luting agents without adhesive: A, control; C, low; E: intermediate; G, high. With adhesive B, control; D, low; F, intermediate; H, high. C, D, G, Interfacial failures. B, E, F, H, Mixed failures. Crack origins generally observed in border of specimens. When adhesive used, fracture events starting in adhesive layer and reaching luting agent at bonded interface commonly observed.

used for measuring the film thickness in the different groups.

RESULTS

As shown in Table 2, the increase in inorganic filler content of the experimental RBLAs affected η significantly. The control material had an η varying from that of experimental RBLAs with high to intermediate filler contents. The inorganic filler content also affected the stiffness of the RBLAs. The control material had a similar E to the experimental RBLA with a high filler content. However, the v or C=C conversion was not affected, but the control material had lower C=C conversion than for all RBLAs.

The μTBS data are summarized in Table 3. The Weibull plot for the μTBS of all RBLAs is shown in Figure 2. The factors filler content of the RBLA (P<.001; f=9.0) and the use of an adhesive layer (P=.004; f=8.7) were both significant, as was their interaction (P=.002; f=5.1). When an adhesive was not used, the experimental RBLA with a high filler content yielded lower μTBS to the feldspathic ceramic than the RBLA with an intermediate filler content. When the adhesive was used, the RBLA with a low filler content yielded lower μTBS than the intermediate and the control luting agents. Weibull moduli had higher μ_0 when the adhesive was used than if adhesive was not used for the more viscous luting agents. No significant differences in Weibull moduli were observed across RBLAs, except the higher m observed when adhesive was not used for the RBLA with a high filler content.

The increase in filler content of the RBLA appeared to be associated with a higher frequency of mixed failures. The distribution of interfacial-mixed failures was 50% to 43% (low), 40% to 53% (intermediate), 23% to 67% (high), and 40% to 57% (control) when the adhesive was used. The frequency of premature and cohesive failures was not high for almost all RBLAs, varying between 3% and 9% of the total failures. Figure 3 shows representative SEM pictures of interfacial and mixed failure modes along with identification of fracture features.
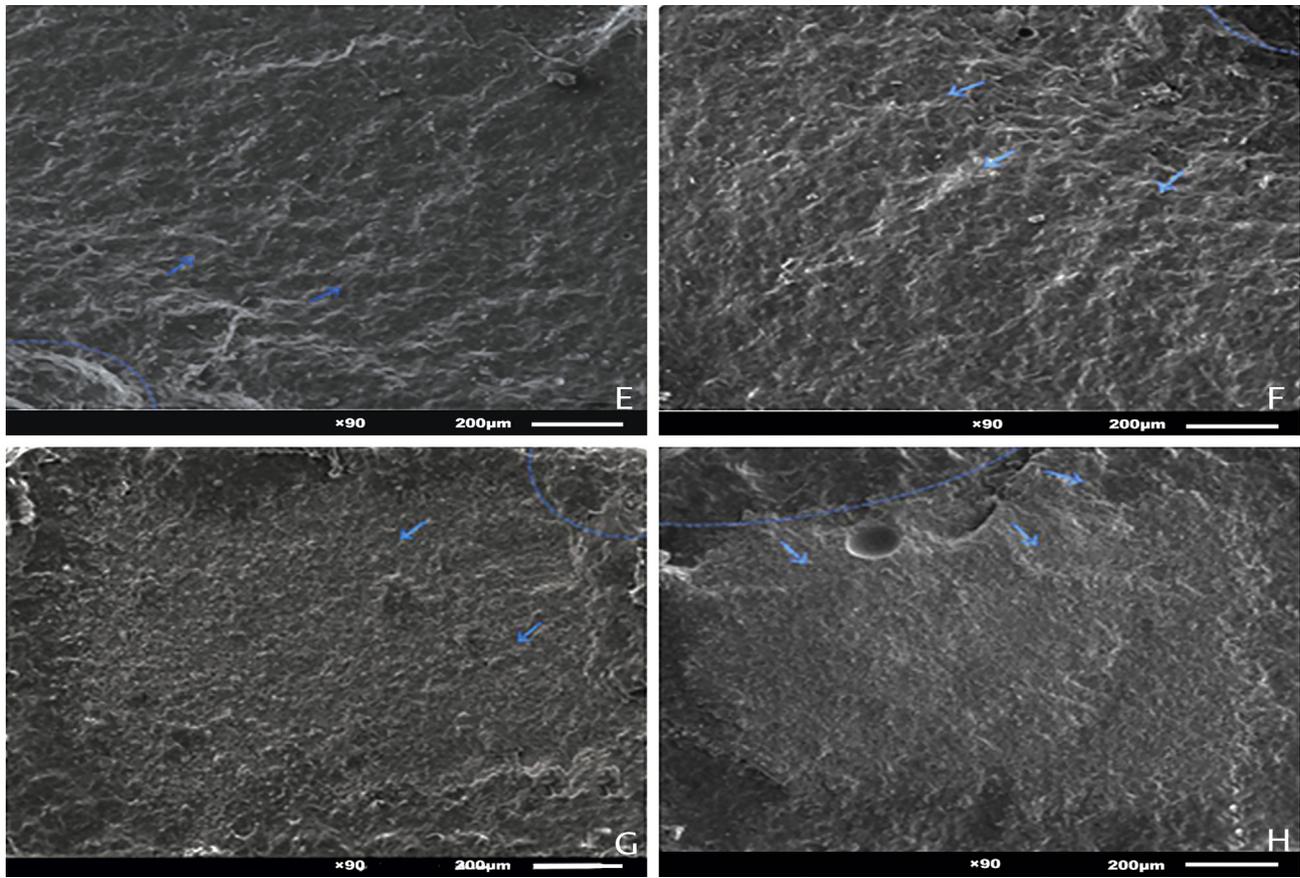
Figure 3. (*continued*).

Crack origins were generally observed at the border of the specimens. When the adhesive was used, fracture events starting at the adhesive interface, or adhesive layer, and reaching the luting agent layer were noticed.

The σ_{bf} data are summarized in Table 4. The Weibull plots are presented in Figure 4. At z=0, the filler content of the RBLA ($P<.001$; f=129.7) and that of the adhesive layer ($P=.001$; f=10.4) were both significant factors, as was their interaction ($P<.001$; f=5.7). The increase in filler content of the experimental RBLAs was associated with increased σ_{bf} and σ_0; that is, they strengthened the bonded feldspathic ceramic. In contrast, the layer of adhesive reduced the σ_{bf} for the intermediate and the control luting agents. The luting agent with a high filler content showed the lower m across RBLAs. At z=-t_2, the filler content of the RBLA ($P<.001$; f=195.9) was a significant factor, but the factor with or without a layer of adhesive was not ($P=.104$; f=2.7). The interaction between the factors was also significant ($P=.026$; f=3.2). The increase in filler content of the experimental RBLAs again strengthened the bonded ceramic, but the layer of adhesive had a minor effect.

SEM images of the RBLA surfaces are shown in Figure 5. The experimental luting agents showed irregular filler particles of slightly more varied particle size distributed at the surfaces compared with the control RBLA. Figure 6 shows SEM images of the ceramic-RBLA bonded interfaces, with or without a layer of adhesive and film thickness.

DISCUSSION

The results of this study provide evidence that both the content of the inorganic filler of RBLAs and an adhesive layer had significant influence on bonding ability, mechanical performance, and morphology at the adhesive interfaces of bonded feldspathic ceramic specimens. Therefore, the null hypotheses tested were rejected. The increase in filler content generated more viscous and stiffer RBLAs, although not in a linear fashion (Table 2). These characteristics, in turn, affected the bonding ability and the reinforcing effect of the ceramic by adhesive cementation. Previous studies have reported a positive correlation between E and filler fraction in particulate composite resins.[22,23,33] The E is improved because the rigid glass particles have a much higher stiffness than the polymer matrix. The increase in η is a consequence because more fillers mean lower interparticle distances and incremental particle-resin and particle-particle interactions, affecting flowability of the unpolymerized paste.

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

Resin-Based Luting Agents	σ_{bf}, MPa		σ_0, MPa		m	
	Without Adhesive	With Adhesive	Without Adhesive	With Adhesive	Without Adhesive	With Adhesive
Axial Position z=0						
Ceramic	111 (108-115)[d]	-	116 (112-120)[d]	-	10.3 (8.0-13.4)[a]	-
RelyX Veneer	138 (132-144)[A,c]	121 (115-127)[B,c]	146 (138-153)[A,c]	128 (122-135)[B,c]	7.5 (5.9-9.6)[A,ab]	7.5 (5.8-9.6)[A,a]
Low	131 (124-138)[A,c]	122 (117-127)[A,c]	139 (130-149)[A,c]	128 (124-133)[A,c]	5.6 (4.4-7.0)[B,bc]	10.1 (7.7-13.4)[A,a]
Intermediate	161 (148-174)[A,b]	150 (136-164)[B,b]	175 (160-191)[A,b]	164 (148-182)[A,b]	4.3 (3.4-5.5)[A,cd]	3.7 (2.9-4.7)[A,b]
High	239 (206-272)[A,a]	258 (233-283)[A,a]	269 (235-308)[A,a]	285 (257-315)[A,a]	2.8 (2.2-3.6)[A,d]	3.7 (3.0-4.8)[A,b]
Axial position z=-t₂						
RelyX Veneer	26.5 (24.3-28.7)[A,b]	27.2 (24.9-29.5)[A,b]	29.0 (26.3-31.9)[A,b]	29.8 (27.3-32.5)[A,b]	3.9 (3.1-4.9)[A,a]	4.4 (3.4-5.7)[A,ab]
Low	17.6 (15.7-19.5)[A,c]	16.3 (15.3-17.3)[A,d]	19.5 (17.4-22.0)[A,c]	17.5 (16.4-18.6)[A,c]	3.3 (2.6-4.2)[B,ab]	5.9 (4.6-7.6)[A,a]
Intermediate	26.8 (23.7-29.9)[A,b]	23.1 (20.6-25.6)[B,c]	29.8 (26.3-33.7)[A,b]	25.6 (22.8-28.7)[A,b]	3.0 (2.4-3.9)[A,ab]	3.3 (2.6-4.2)[A,b]
High	69.1 (56.7-81.5)[A,a]	67.7 (59.0-76.4)[A,a]	78.3 (65.8-93.3)[A,a]	75.8 (66.2-86.7)[A,a]	2.2 (1.7-2.8)[A,b]	2.8 (2.2-3.6)[A,b]

Low (55wt%), intermediate (65wt%), and high (75wt%) refer to filler content of experimental resin-based luting agents. m Indicates reliability of biaxial flexural strength, and σ_0 indicates value at which 63.21% of the specimens tested.[37,38] Uppercase letters in same row indicate significant differences for each condition with or without layer of adhesive; lowercase letters in same column indicate differences between luting agents ($P<.05$).
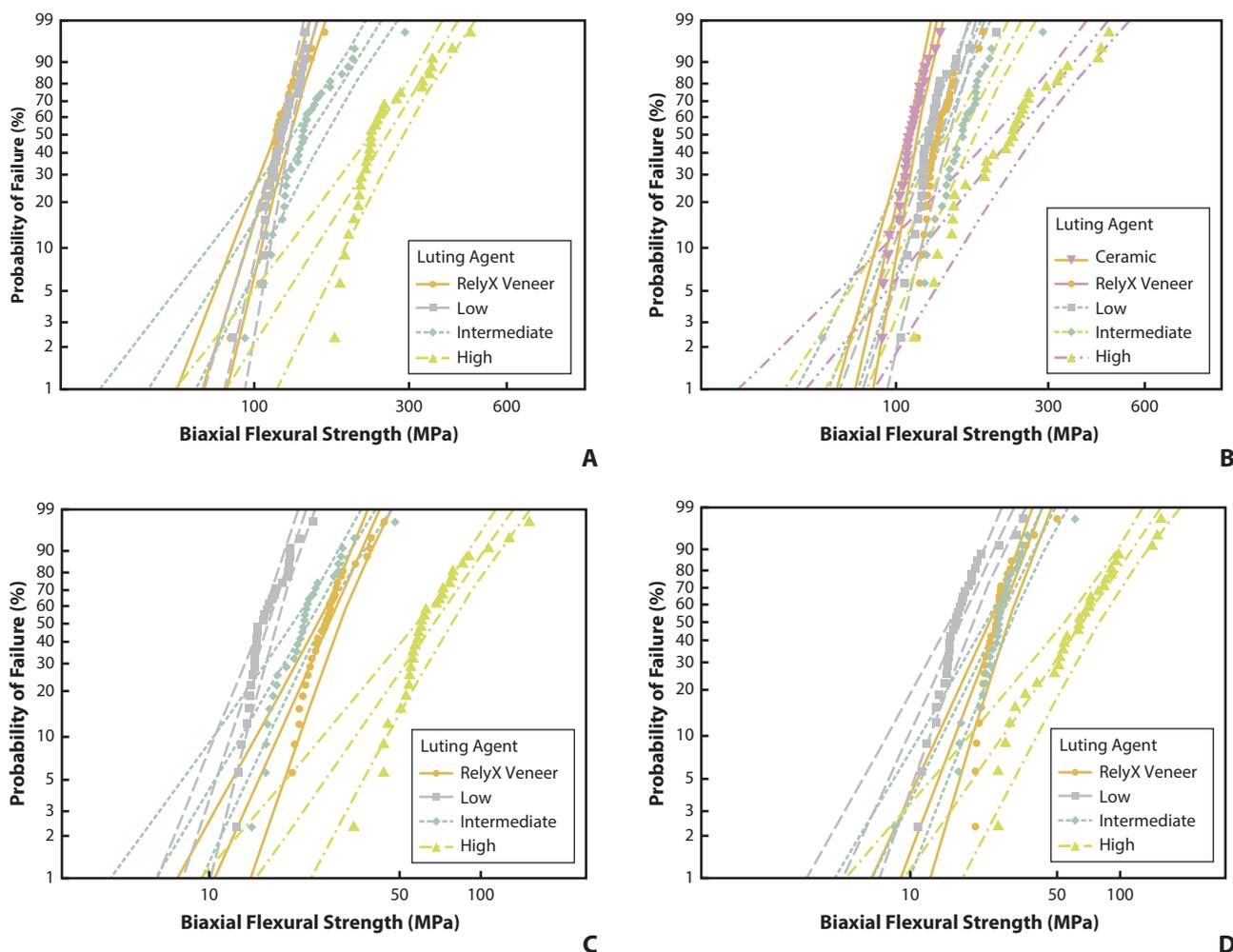
Figure 4. Weibull plots showing probability of failure (%) versus biaxial flexural strength (σ_{bf}, MPa) for all RBLAs at axial positions z=0 and z=-t₂. A, B, z=0. C, D, z=-t₂. At z=0, structural reliability generally negatively affected by increase in inorganic filler content in RBLAs. At z=-t₂, structural reliability higher for RBLA with low filler content than for other experimental luting agents.
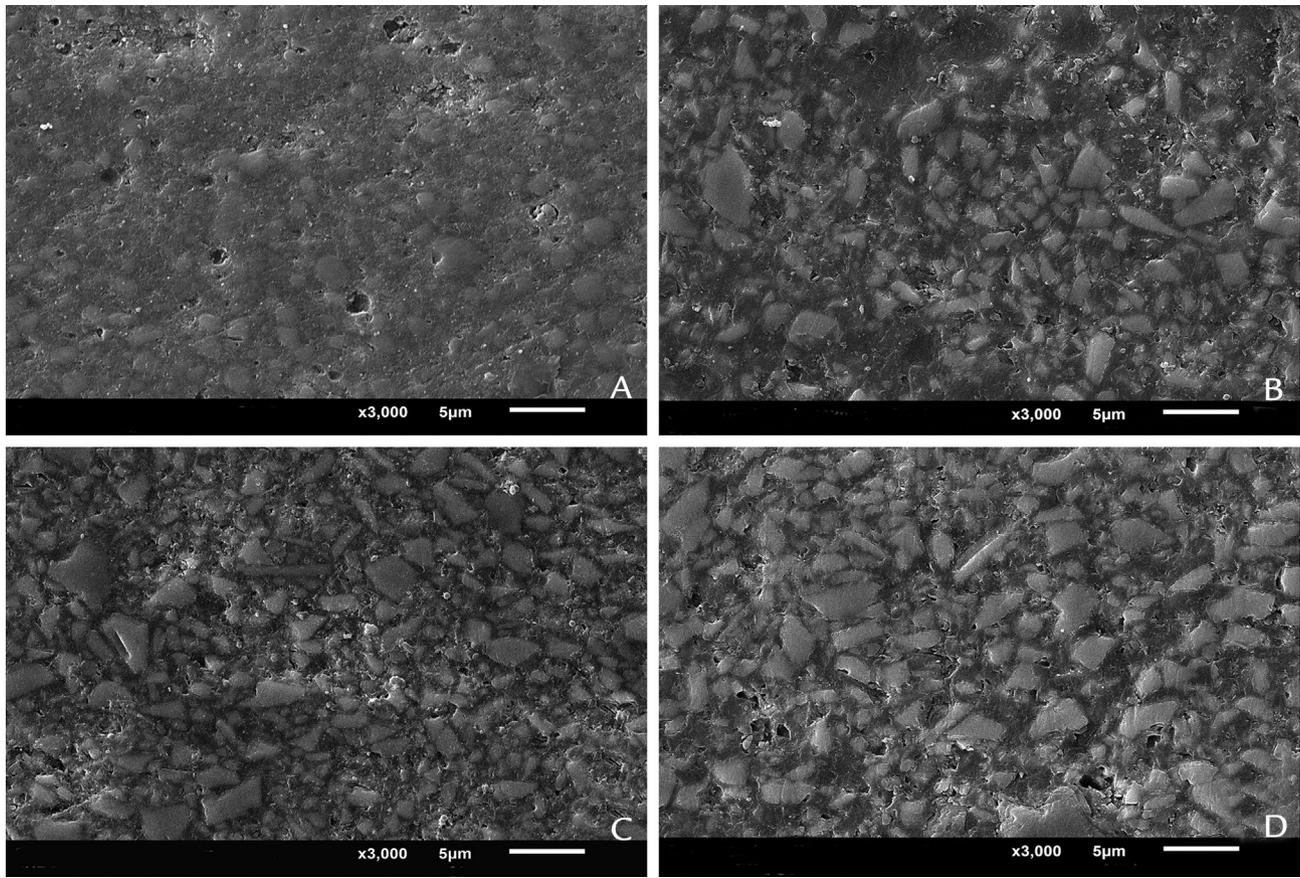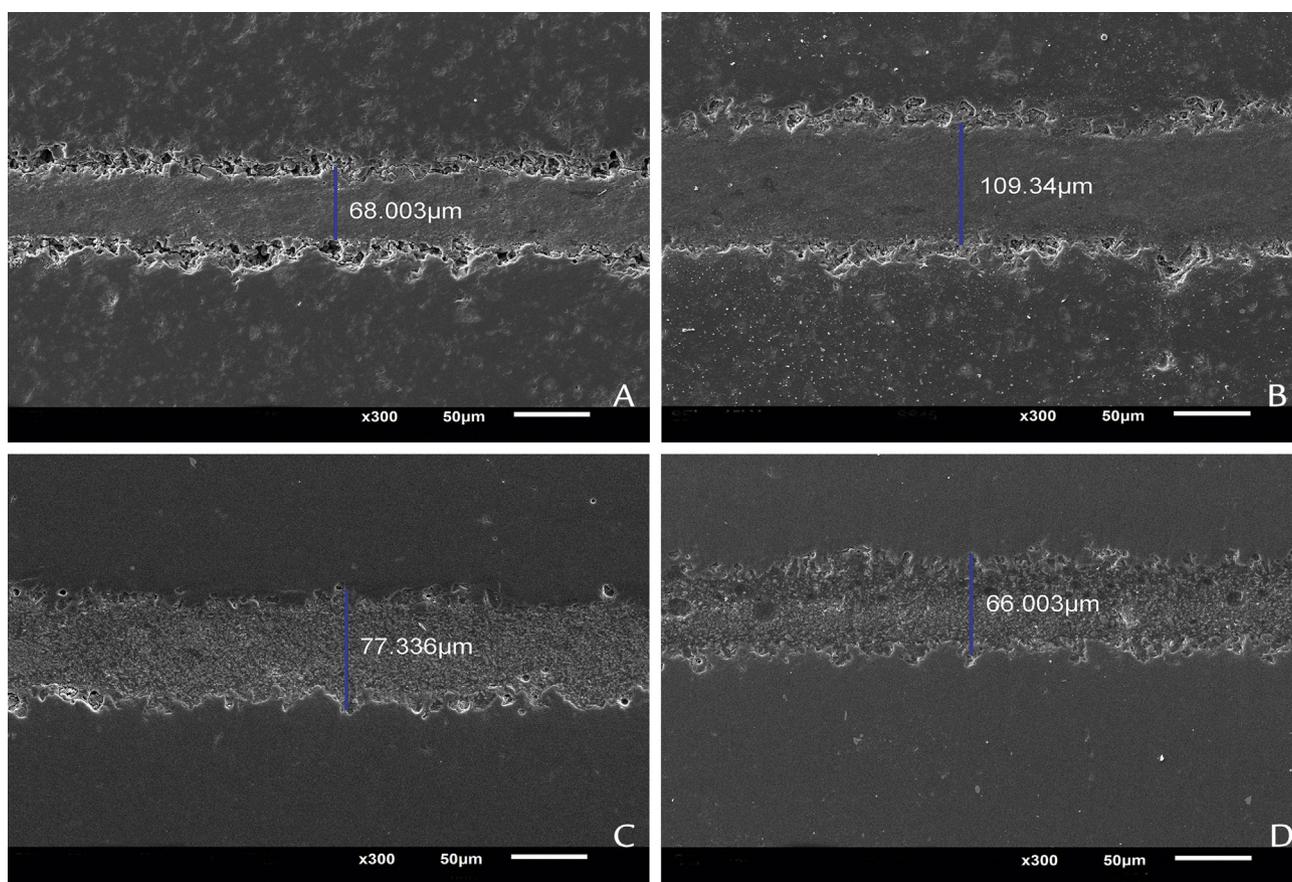
Figure 5. Scanning electron microscope images of surface of luting agents. Original magnification ×3000. A, Control luting agent showed smooth surface with round filler particles without larger variation in particles sizes. Experimental luting agents (B, low; C, intermediate; D, high) showed irregular filler particles with larger variation in particles sizes distributed at surfaces.

In the present study, when the adhesive was not used, the RBLA with a high filler content had lower bonding ability, but higher characteristic bond strength was observed with the adhesive. In contrast, the adhesive negatively affected the bonding ability of the RBLA with a low filler content (Table 3). Then, the low-filler-content RBLA was fluid enough to fill the irregularities created on the ceramic by acid etching, but the more viscous material was not. In the low-filler-content RBLA, the combination of adhesive plus luting agent was likely to be sufficiently fluid to generate adequate micromechanical interlocking with the ceramic. These findings suggest that the decision as to when an adhesive is indicated to be used for bonding ceramic veneers should consider the filler content and η of the luting agent (Table 2). Another finding was the increased frequency of mixed, cohesive, and even premature failures when the filler content in the luting agent was higher. All these observations are consistent with findings from the biaxial flexural test, which indicated that the structural reliability of the bonded veneers was negatively influenced by the increase in filler content of the RBLA.

The lower reliability of the ceramic structure bonded with a viscous luting agent might be explained by an uneven distribution of structural defects at the cement layer or bonded interface, affecting its behavior under loading (Table 4). The strengthening of the ceramic by adhesive cementation relies on the formation of a homogeneous polymer-ceramic interphase. In the present study, the adhesive layer had a minor effect on mechanical strength. However, the SEM analysis showed that the adhesive aided the more viscous luting agents in filling the ceramic irregularities (Fig. 6). This is consistent with a previous study[13] which showed that application of adhesive was able to infiltrate all unfilled voids of a ceramic, even when the acid etching time was prolonged. This reinforces the need for an adhesive layer when glass-ceramics are luted with viscous RBLAs despite the increment in film thickness it might generate (Fig. 6). Differences in η of the luting agent might also affect its wettability on ceramic surfaces, especially when an adhesive has been previously applied.[7,9,12,20,26]

Despite some shortcomings of the use of RBLAs with a high filler content, the mechanical tests provided a clear

Figure 6. Scanning electron microscope images of morphology at bonded interface and film thickness of ceramic-luting agent sandwiched specimens. Original magnification ×300. A, Control; B, Control with adhesive; C, Low; D, Low with adhesive.

picture that the increment in filler content was positively associated with the flexural and characteristic strengths of the bonded specimens (Table 4). Previous studies have shown that the E of the luting agent is positively associated with the strengthening of glass-ceramics.[7,9,12,20] This phenomenon may be explained by the increase in stress concentration at the luting agent layer and reduction of stresses reaching the ceramic when the mismatch in stiffness between the luting agent and ceramic is lower.[9] Increased inorganic particle loading increases the toughness of composite resins, acting on the stress transfer from the polymer to fillers, provided that the particles are homogeneously distributed and effectively coupled to the matrix.[23] The increase of the fracture toughness by adding fillers has been attributed to mechanisms including crack bowing or branching, increase in line energy at the crack front, or polymer plastic deformation around fillers leading to higher fracture energy.[33] Differences in viscoelasticity deriving from different filler contents in the RBLAs may occur, although it is likely that all the agents tested in this study may accommodate some elastic and plastic

deformation, contributing to the ceramic reinforcing effect.

The particles used in the experimental RBLAs had an irregular shape, 2 μm in average size, and a moderate narrow size distribution (Fig. 5). Perhaps different results would have been observed if the particles had been smaller or round. This assumption is reinforced by the different results usually observed for the control luting agent tested as a reference compared with the experimental materials. Composite resins containing round particles may have higher flexural strength, flexural modulus, and hardness than composite resins with irregular-shaped particles.[33] Round particles have been reported to enhance the fracture strength because stresses tend to develop at sharp edges of fillers, although, in the same study, the E of resin-based composite resins was independent of filler shape, particularly when the size range of fillers was small.[23] In addition, the combination of relatively small and varied-size fillers, especially round in shape, could allow denser packing. However, more fillers would mean even higher η. The RBLA with a higher filler content was so viscous that it
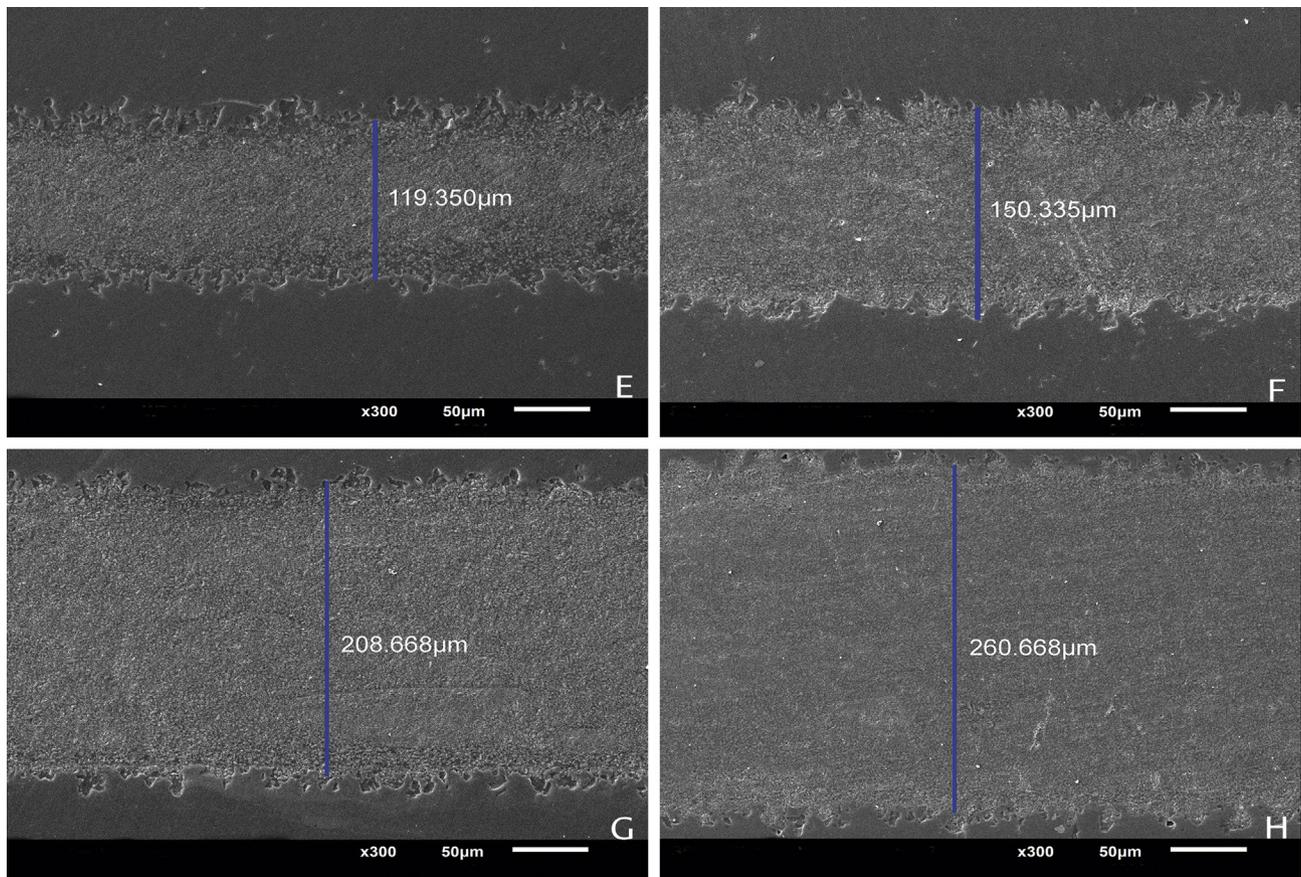
Figure 6. (*continued*). E, Intermediate; F, Intermediate with adhesive; G, High; H, High with adhesive. Layer of adhesive helped to fill with resin irregularities in grooves created by acid etching. Increased filler loading of luting agent associated with increased film thickness. Layer of adhesive increased film thickness except for luting agent with low filler content.

had to be preheated at 60 °C before luting; otherwise, it would have led to even poorer penetration of the ceramic roughness. The use of preheated composites for luting ceramic restorations has gained attention. Different devices can be used for preheating (such as HotSet [Calset], water bath, hot air oven),[26,34-36] as well as different temperatures up to 69 °C.[26,27] In the present study, the RBLA with a high filler content was preheated to 60 °C in a hot air oven.[27] One of the shortcomings of this technique is the increased film thickness,[26] which was also observed in the present study. However, the technique is reported to have advantages including increased degree of C=C conversion of the composite resin[27] and generating higher fracture strength and less chipping than luting with resin cements.[28] The results of this study corroborate the higher strengthening of the ceramic with preheated composite resin, but the C=C conversion did not differ from that of the other RBLAs.

As selecting an appropriate RBLA and the use of adhesive can increase the structural reliability of luted feldspathic ceramic restorations, further studies taking into account aging effects are warranted to further investigate the influence of the variation of filler content and other characteristics of the luting agents on the bond strength to ceramic and the magnitude and stability of ceramic strengthening. Additionally, studies should evaluate the effects on bonding of these materials to dentin and enamel.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Increased inorganic filler content of the experimental resin-based luting agents strengthened the bonded feldspathic ceramic.
2. The luting agent with a high filler content yielded significantly higher viscosity and film thickness. In contrast, the bond strength was lower and the structural reliability decreased if an adhesive was not used.
3. In the clinical situation, when more viscous luting agents are used, such as preheated composite resin, an adhesive is indicated to help improve the performance of bonded ceramic.

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Corresponding author:
Dr Noéli Boscato
Graduate Program in Dentistry
Federal University of Pelotas
Rua Gonçalves Chaves 457, Room 508 96015-560
Pelotas, RS
BRAZIL
Email: noeliboscato@gmail.com