



UNIVERSIDADE FEDERAL DE UBERLÂNDIA

FACULDADE DE ODONTOLOGIA



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**DESEMPENHO BIOMECÂNICO DE CIMENTOS
DUAL/CORE - VALIDAÇÃO DE PRODUTO
NACIONAL**

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Trabalho de conclusão de curso apresentado a Faculdade de Odontologia da UFU, como requisito parcial para obtenção do título de Graduado em Odontologia.

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DESEMPENHO BIOMECÂNICO DE CIMENTOS DUAL/CORE - VALIDAÇÃO DE PRODUTO NACIONAL

RESUMO

Os cimentos resinosos e a fotoativação podem influenciar as propriedades mecânicas da cimentação de pinos e reconstrução do núcleo em dentes tratados endodonticamente. Este estudo analisou como as propriedades mecânicas são afetadas pela fotoativação imediata ou após 5 minutos em diferentes materiais resinosos utilizados na cimentação de pinos de fibra de vidro (cimentos duais ou cimentos core) e na reconstrução do núcleo (resinas bulk-fill ou cimentos core). Foram testados nove materiais resinosos, incluindo 4 cimentos core: Allcem Core, FGM; LuxaCore Z, DMG; Rebilda DC, VOCOC; Clearfil DC Core Plus, Kuraray; 3 cimentos dual: RelyX Universal, 3M Oral Care; RelyX U200, 3M Oral Care; Allcem Dual, FGM; e 2 resinas compostas bulk-fill: Opus Bulk Fill APS, FGM; e Filtek One Bulk Fill, 3M Oral Care. Os cimentos duais e as resinas compostas core foram fotoativados imediatamente e após 5 minutos. As propriedades medidas incluíram a contração pós-gel (%), resistência flexural (MPa), módulo de elasticidade (MPa), resistência à tração diametral (MPa), resistência à compressão (MPa), dureza Knoop (N/mm²), grau de conversão (%), profundidade de cura (mm), análise termogravimétrica (% de conteúdo de carga) e radiodensidade. Foram usadas microscopia eletrônica de varredura e espectroscopia por energia dispersiva para examinar a morfologia e o conteúdo de carga. O resíduo dos cimentos duais e cimentos core que permaneceu na ponta automistura foi pesado. Os dados de contração pós-gel, análise termogravimétrica, resistência flexural, módulo de elasticidade, resistência à tração diametral, resistência à compressão, profundidade de cura e radiopacidade foram analisados usando ANOVA de 2 vias e ANOVA de medidas repetidas de 2 vias para dureza Knoop e grau de conversão, seguidos do teste de Tukey para identificar diferenças entre os grupos ($\alpha = 0,05$). A fotoativação realizada 5 minutos após a aplicação resultou em uma redução significativa da contração pós-gel em todos os cimentos duais e cimentos core ($p < 0,001$). O aumento na profundidade de cura causou uma diminuição significativa nos valores de dureza Knoop para todos os materiais testados ($p < 0,001$). As resinas bulk-fill e as resinas compostas core apresentaram valores de dureza Knoop superiores aos dos cimentos

duais ($p < 0,001$). A fotoativação após 5 minutos reduziu significativamente os valores de contração pós-gel sem ter um efeito negativo ou de melhoria nas propriedades mecânicas testadas. Os cimentos duais e cimentos core mostraram maior profundidade de cura do que as resinas compostas bulk-fill. Os cimentos resinosos core apresentaram valores de dureza Knoop mais altos do que os cimentos duais e propriedades mecânicas semelhantes às resinas compostas bulk-fill.

PALAVRAS-CHAVE: propriedades mecânicas, propriedades físicas, resinas compostas core, cimento resinoso dual, resinas compostas bulk-fill, contração pós-gel, radiopacidade, fotoativação.

ABSTRACT

The resin-based cements and light activation protocol can influence the mechanical properties of post cementation and core reconstruction in endodontically treated teeth. This study analyzed how the mechanical properties are affected by immediate or 5-minute photoactivation of different resin materials used in cementation of glass fiber posts (dual-cure resin cements or dual-cure resin-core) and to build the core (bulk-fill resins or dual-cure resin-core). Nine resin-based materials were tested, including 4 core cements: Allcem Core, FGM; LuxaCore Z, DMG; Rebilda DC, VOCO; Clearfil DC Core Plus, Kuraray; 3 dual cements: RelyX Universal, 3M Oral Care; RelyX U200, 3M Oral Care; Allcem Dual, FGM; and 2 bulk-fill composite resins: Opus Bulk Fill APS, FGM; and Filtek One Bulk Fill, 3M Oral Care. The dual cements and core composites were photocured immediately and after 5 minutes. The measured properties included post-gel shrinkage (%), flexural strength (MPa), elastic modulus (MPa), diametrical tensile strength (MPa), compressive strength (MPa), Knoop hardness (N/mm^2), degree of conversion (%), depth of cure (mm), thermogravimetric analysis (% filler content), and radiodensity. Scanning electron microscopy and energy-dispersive X-ray spectroscopy were used to examine the morphology and filler content. The residue of the dual cements and core cements that remained in the self-mixing tip was weighed. Post-gel shrinkage, thermogravimetric analysis, flexural strength, elastic modulus, diametrical tensile strength, compressive

strength, depth of cure, and radiopacity data were analyzed using 2-way ANOVA and 2-way repeated-measures ANOVA for Knoop hardness and degree of conversion, followed by Tukey's test to identify differences between groups ($\alpha = 0.05$). Light activation performed 5 minutes after application resulted in a significant reduction in post-gel shrinkage in all dual cements and core cements ($p < 0.001$). Increasing the depth of cure caused a significant decrease in Knoop hardness values for all materials tested ($p < 0.001$). Bulk-fill resins and core composite resins presented higher Knoop hardness values than dual cements ($p < 0.001$). The light activation after 5 minutes significantly reduced post-gel shrinkage values without having a negative or improving effect on the mechanical properties tested. Dual-cure resin-based materials showed greater depth of cure than bulk-fill resins. Resin-core materials showed higher Knoop hardness values than dual cements and similar mechanical properties to bulk-fill resins.

KEYWORDS: mechanical properties, physic properties, resin-core materials, dual-cure resin cement, bulk-fill resin-based composite, post-gel shrinkage, radiopacity, light activation.

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ARTIGO 1

***Artigo enviado para Journal of Prosthodontics**

Mechanical properties of dual-polymerize resin-core, dual-polymerize resin cement and bulk-fill resin composite used for restoring endodontically treated teeth

ABSTRACT

Statement of problem. The resin-based material and light-activation protocol can influence the mechanical properties of the materials used for fiberglass post-cementation and core reconstruction of endodontically treated teeth.

Purpose. To evaluate the influence of immediate or five minute delayed light-activation on the mechanical properties of dual-polymerize resin cements, dual-polymerize resin-core materials compared with bulk-fill composite resins (CRs).

Material and methods. Nine resin based materials were tested: 4 dual-polymerize resin-core materials, (Allcem Core; FGM), (LuxaCore Z; DMG), (Rebilda DC; VOCO), and (Clearfil DC Core Plus; KURARAY), 3 dual-polymerize resin cement, (RelyX Universal; 3M ESPE), (RelyX U200; 3M ESPE), and (Allcem Dual; FGM), 2 bulk-fill CRs, (Opus Bulk Fill APS; FGM), and (Filtek One Bulk Fill; 3M ESPE). The dual-polymerize materials were light-activated using both protocols. The post-gel shrinkage (Shr), flexural strength (FS), elastic modulus (E), Knoop hardness (KH), degree conversion (DC), and depth of polymerization (DoP) were measured (n=10). Data of Shr, FS, E, and DoP were analyzed using two way ANOVA, and for KH and DC using two way repeated measurement ANOVA and Tukey test ($\alpha=.05$).

Results. Five minute delay to light-activate significantly reduced Shr for all materials ($P<.001$). Increasing the depth significantly reduced the KH for all materials ($P<.001$). Bulk-fill CRs and dual-polymerize resin-core had higher KH values than dual-polymerize resin cements ($P<.001$.)

Conclusions. Delayed five minute light-activation reduced post-gel shrinkage and had no negative effect on mechanical properties. Dual-polymerize resin-core materials exhibited higher KH values than dual-polymerize resin cement, and mechanical properties similar to those of bulk-fill CRs.

CLINICAL IMPLICATIONS

Dual-polymerize resin-core materials had similar or superior physical and mechanical properties than dual-polymerize resin cements and bulk-fill CRs, demonstrating to be an alternative to cement fiberglass post, and to build the core. The five minute delay to light activate the dual-polymerize resin-core materials is recommended to reduce the post-gel shrinkage. Rehabilitation of endodontically treated teeth is a challenge because of the difficulty involved and the variability of materials. Teeth with severe structural damage lack a coronal structure for the retention of the final restoration.^{1,2} Restorative protocols are based on the use of materials with similar mechanical properties associated with the adhesive strategy aimed at mimicking the tooth structure of intact teeth.^{3,4} The use of adhesive procedures with or without fiberglass posts and composite resin (CR) can provide retention for direct or indirect restoration.⁵⁻⁸ The association of dual-polymerized resin cement for luting the fiberglass post and the use of CRs for coronal reconstruction has been the first option for restoring endodontically treated teeth.^{9,10} However; dual-core materials simplify procedures by reducing materials and interfaces, allowing both core build-up and cementation in one step.^{11,2}

Sustained efforts have been made to improve the mechanical performance and retention of the final restoration.¹²⁻¹⁴ In the conventional method, a dual-polymerize resin cement secures the post, while bulk-fill CR rebuild the crown for indirect restorations.^{15,16} However, the mechanical properties of these dual-polymerize resin-core materials need to cover all the requirements expected for CRs used for coronal reconstruction and present proper polymerization during fiberglass post-cementation.¹⁷⁻²⁰

The dual-polymerize resin cement provides control of working time and warrants the restoration to achieve a high degree conversion in areas with limited light penetration.²¹⁻

²⁴ Usually, the resin cements are light-activated immediately after the insertion.^{25,26} However, a five minute delay between the mixing and light-activation.²⁷ The light-activation delay may also improve Knoop Hardness, elastic modulus, and bond strength of the dual-polymerize resin cement, and reduce shrinkage stress.²⁸⁻³¹ However, the effectiveness of the five minute delay light-activation for dual-polymerize resin-core material is not established, and the influence of the composition on the physical and mechanical properties after immediate or five-minute delay light-activation remains uncertain.³²

Therefore, the present study evaluated the immediate and delayed light-activation (five-minutes) impact on mechanical properties of resin-based materials used in restoring endodontically treated teeth, including dual-polymerize resin cement, bulk-fill composite resins and resin core materials. The null hypotheses were that light activation of the dual-polymerize resin cement and dual-polymerize resin-core materials immediately or after five minutes protocols would not modify the mechanical properties, and the physical and mechanical properties of dual-polymerize resin-core materials would be similar to that of the dual-polymerize resin cement and bulk-fill CRs used for restoring endodontic treated teeth.

MATERIAL AND METHODS

Nine resin-based materials used for restoring endodontically treated teeth were tested: 4 dual-polymerize resin-core materials, (Allcem Core; FGM, Clearfil DC Core Plus; KURARAY, LuxaCore Z-Dual; DMG, and Rebilda DC; VOCO), 3 automix dual-polymerize resin cements, (RelyX Universal; 3M ESPE, RelyX U200; 3M ESPE, and Allcem Dual; FGM), and 2 bulk-fill CRs, (Opus Bulk Fill APS; FGM, and Filtek One Bulk Fill; 3M ESPE). The composition and light-polymerize manufacturing recommendations are listed in Table 1. Two light-activation protocols using a multiwave light-curing unit (LCU) (VALO Grand; Ultradent) were used for dual-polymerize resin-based materials: immediately and five minute delay after mixing. The LCU was characterized using a 6 inch integrating sphere

(Labsphere; Ocean Optics) connected to a fiber-optic spectrometer (USB 4000; Ocean Insight).

The post-gel shrinkage was calculated using the strain gauge test performed in a dark room with temperature control (22 ± 1 °C). The resin-based material was placed (n=5) in hemispheres on top of a biaxial strain-gauge (CEA-06-032WT-120; Excel Sensores) that measured the shrinkage stresses in both parallel and perpendicular directions. A strain conditioner (ADS05000IP; Lynx) converted the electrical resistance variations in the strain gauge into voltage variations and Shr values using a quarter-bridge with an internal reference resistor (120Ω). The maximum value at stabilization of the curves was used as the post-gel linear shrinkage and converted to a volumetric percentage by multiplying by 3 and 100%.

A three point bending test was used to calculate the flexural strength (MPa) and elastic modulus (MPa) according to ISO 4049 (2019).³³ Specimens of all resin-based materials (n=10) were prepared using a 2 mm × 2 mm × 25 mm rubber matrix in a dark room. To minimize the presence of bubbles and smooth the surface, the matrix was placed on a glass plate with Mylar strip separator (KDent; Quimidrol). The matrix was filled, sealed with another Mylar strip and light-activated at three points using two different protocols.. Specimens were stored for 24 h in a dry oven at 37 °C. Three point bending test was conducted using a universal testing machine (Instron ElectroPuls E3000; Instron) at a crosshead speed of 0.5 mm/minute. The FS was calculated using the following formula: $\alpha = 3FL / (2wt^2)$, where F was the maximum force applied (N), L the distance between the support beams (mm), w the width of the specimen (mm), and t the thickness of the specimen (mm). The deflection (mm) until failure was determined using a deflectometer (W-E401-J, E-Series Deflectometer; Instron) positioned at the center of the sample base during loading. The elastic modulus (MPa) was determined using the formula $E = FL^3 / 4BH^3 d$, where F is the maximum load (N), L is the length of the specimen (mm), B is the width of the specimen (mm), H is the height of the specimen (mm), and d is the deflection (mm) at the maximum load.³⁴

Each resin-based material was manipulated and inserted into a bipartite cylindrical matrix with a diameter of 4 mm and a thickness of 10 mm. The LCU tip was positioned near the

polyester strips and stabilized with metal support before light activation using 2 protocols..²² The specimens were stored in dark envelopes, dry location at 37 °C for 24 h. The Knoop hardness (N/mm²) along the length of each specimen was measured at 50 g for 15 s using a microhardness tester (FM700; Future-Tech). Three measurements were performed at 5 locations: 0, 2.5, 5.0, 7.5, and 10 mm.

The specimens for DC were inserted into a bipartite cylindrical matrix with a diameter of 4 mm and a thickness of 10 mm. The DC was measured in 3 points: one measurement made on the top, other in the middle of the specimen, and the last on the bottom, with a medium-infrared FTIR spectrometer (Vertex 70; Bruker Optik) using a software program (OPUS v.7.8; Bruker Optik). The spectra were obtained between internal standard aromatic C=C bonds (1608 cm⁻¹) and aliphatic C=C bonds (1638 cm⁻¹). The baseline of each unpolymerized cement resin was recorded by scanning 32 times over a range of 400–4000 cm⁻¹ at a resolution of 4 cm⁻¹. Data were collected at 25 ±1 °C and 60 ±5% humidity conditions in a dark room with yellow light. Due to the lack of internal reference aromatic peaks at (1608 cm⁻¹) in resin cement Rely X Universal, the (1710 cm⁻¹) peak height was used.²³ The formula used to calculate the degree conversion was $DC (\%) = (1 - C/U) \times 100$. Following DoP measurement, specimens were removed from the matrix, submerged in 100% acetone (NEON; Persoalab) for an hour, then dried for another hour at room temperature (22 ±1 °C). The specimens were photographed using an EOS camera (D7200; Nikon) coupled with a 105 mm lens, and measured with a digital caliper (Pinça Vernier Digimatic Digital Absoluta; Mitutoyo), then the values were divided by 2 following the ISO 4049 recommendations.³⁵

Data of Shr, , FS, E, KH, DC and DoP were first analyzed for normal distribution (Shapiro-Wilk test) and homoscedasticity (Levene test). The twoway analysis of variance was performed for the Shr, , FS, E, and DoP data. The two-way ANOVA with repeated measures was used for KH and DC. All tests used an ($\alpha=0.05$), and all analyses were performed using a software program (Sigma Plot, v13.1; Systat Software Inc).

RESULTS

The Shr mean (%) and standard deviation values of the light-activated resin-based materials obtained using both protocols are shown in Table 2. The two way ANOVA showed a significant difference between the resin-based materials ($P<.001$), and between the light-activation protocols ($P<.001$). The Tukey test showed that five minute delay light curing protocol, significantly reduced Shr for all dual-polymerize materials. When light-activated after five minutes, all dual-polymerize materials had lower Shr values than the bulk-fill CRs. The dual-polymerize resin cements, in general, had a lower Shr than the dual-polymerize resin-core materials when the light was activated immediately. However, this difference disappeared when the light was activated after five minutes.

The FS mean (MPa) and standard deviation values of all tested resin-based materials are shown in Table 3. The two way ANOVA showed a significant difference between resin-based materials ($P<.001$), but no significant difference for light-activation protocols ($P=.302$). Rebuilda DC had the highest FS value, followed by bulk-fill CRs ($P<.001$).

The mean E (MPa) and standard deviation values of all the resin-based materials are shown in Table 3. The twoway ANOVA showed a significant difference between resin-based materials ($P<.001$), but no significant difference between light-activation protocols ($P=.101$). OPUS Bulk-fill APS had the highest E value, followed by Clearfil DC Core Plus, and RelyX Universal had the lowest E values ($P<.001$).

The KH mean (N/mm²) values and standard deviations of all the resin-based materials using different light-activation protocols are shown in Figure 1. There was a strong correlation between depth increase and reduction in KH values for all tested materials ($P<.001$). The two way ANOVA showed a significant influence of the resin-based materials ($P<.001$), light-activation protocol ($P<.001$), and the interaction between both study factors ($P<.001$). The Tukey test showed that bulk-fill CRs and dual-polymerize resin-core materials generally had higher KH values than dual-polymerize resin cements. The KH values were always significantly higher at the top than at a depth of 5 mm or deeper. A five minute light activation delay increased the KH values in deep locations for

RelyX Universal, Luxacore Z, Allcem Core, and RelyX U200 at 0 to 5 mm. No influence of the light-curing protocol was observed for Clearfil DC Core Plus or Allcem Dual.

The DC mean (%) values and standard deviations of all the resin-based materials using different light-activation protocols are shown in Figure 2. There was a strong correlation between the depth increase and reduction in the DC values for all materials ($P<.001$). The twoway ANOVA showed a significant influence of the resin-based materials ($P<.001$), light-activation protocol ($P<.001$), and the interaction between study factors ($P<.001$). Tukey test showed that bulk-fill CRs and dual-polymerize resin-core materials had higher DC values than dual-cure resin cements. The DC values were significantly higher at the top than at 10 mm depth. A five minute light activation delay had no significant influence on the DC values, irrespective of the resin-based material tested.

The DoP mean (mm) values and standard deviations of all the resin-based materials using different light-activation protocols are shown in Figure 1. The two way ANOVA showed that there was a significant influence of the resin-based materials ($P<.001$) but not of the light-curing protocol ($P=.477$). Tukey test showed that the bulk-fill CRs had a significantly lower polymerize depth than the dual-polymerize resin-based materials. A five minute light activation delay did not influence the DoP values.

DISCUSSION

The present study evaluated resin- material type and light-activation protocol on the mechanical properties of materials used in restorations for endodontically treated teeth. Delaying light activation for five minutes after mixing the dual-polymerize resin-based materials significantly reduced Shr and resulted in similar or improved mechanical properties compared to immediate light-activation. This finding led to the rejection of the first null hypothesis.

The Shr values were significantly reduced for all tested dual-polymerize resin materials when a five minute delayed light-activation protocol was used. This occurs because the resin-based material undergoes the change of the monomers to polymers gradually, not

instantaneously, which helps to reduce shrinkage stresses.³⁰ A five minute delay in light activation also increases the material's pH-buffering capacity, which is essential for allowing adequate polymerization to occur.²⁷ Several studies have shown that delay can improve the Knoop hardness, elastic modulus, and bond strength of the dual-polymerize resin cement, while reducing residual shrinkage stress.^{28,31} Due to a higher gel phase, the material can better dissipate the stress caused by shrinkage during volume reduction. However, immediate photoactivation leads to a rapid shrinkage of particles, creating a "shock" effect within the material. This higher gel phase allows for an initial polymerization, and this gradual change facilitates a faster vitrification process in the resin material.³⁴

The verification of mechanical properties of dual-polymerize resin-core materials, dual-polymerize resin cements, and bulk-fill CRs revealed significant differences, leading to the rejection of the second null hypothesis. Due to the material dependence and strong influence of DC on properties, CR performance varies significantly across material types.⁵ However, in the present study, DC, DoP, and KH did not differ between the immediate and delayed light-activation protocols. Interestingly, the five minute delay only increased the KH values for RelyX Universal. Different light-curing protocols significantly impact properties like Shr, KH, E, and FS values, which are not accompanied by variations in the DC.^{16,25} Development of the DC continues for some time even after light activation is completed.^{23,24} High DC values can also be associated with a less stable or strongly connected polymer network, potentially explaining the more pronounced reduction in KH values in deeper locations.

The KH values obtained in the present study can be compared with dentin hardness, which has to overcome cracking in brittle materials.²⁴ As the depth of the material increased, the present study observed a decrease in KH values. This can be attributed to light attenuation. Light attenuation reduces the energy delivered to deep areas, making the chemical reaction alone insufficient to determine the same polymerization through the material depth. This finding highlights the importance of using LCUs with high power and well-distributed light on the LCU tip.^{20,22} The dual-polymerize resin materials had higher KH values than bulk-fill CRs at deeper locations because of the chemical reaction.²³ The higher DC values observed for dual-polymerize resin cement resulted in higher KH

values.¹³ However, the bulk-fill CR depends on the light reaching a depth greater than 4 to 5 mm, confirming that bulk-fill restoration does not constitute a single increment of CR.

Dual-polymerize resin materials are characterized by combining light-activated free-radical polymerization for efficiency with self-cure for deeper areas where light penetration is limited.^{21,26} Uneven light distribution creates stronger mechanics in the surface, while the bottom weaker properties due to light attenuation. . Inadequately polymerized resin-based materials do not achieve the correct DC, leading to microleakage, wear resistance, recurrent caries, discoloration, degradation, and a decrease in mechanical properties.^{1,35}

The FS is an important mechanical property of brittle materials.¹⁷ ISO 4049 prescribes a minimum FS of 50 MPa for luting materials to support masticatory loading.^{33,34} Differences among materials were found due to different filler content, monomer integration, and filler size that are directly related to the mechanical properties.¹⁸ In the present study, different types of monomers influence the FS, such as the incorporation of UDMA.. This monomer is more flexible than bisphenol A-glycidyl methacrylate (BIS-GMA) with a molecular weight (MW) of 512 g/mol. The UDMA network has a lower viscosity that facilitates the migration of free radicals, increasing crosslinking, which subsequently increases the FS values.³¹

E reflects stress transmission between the restoration and remaining tooth structure, signifying resistance to elastic deformation that could compromise the bonded interface's integrity.¹⁴ Resin-based materials that have a high elastic modulus produce more rigid restorations; therefore, the elastic modulus is intimately linked to the hardness.¹⁸ The E of the resin-based materials should have a nearly intermediary elastic modulus of the dentin that will be replaced by 18.6 (MPa).³⁴ In the present study, the E values correlated with filler content, as bulk-fill CRs and dual-polymerize resin-core materials presented higher E values. High inorganic contents have been associated with homogenous E and KH, and FS values that produce strength restorations in depth.^{12,17}

Despite replicating feasible laboratory conditions, the present study did not account for real clinical challenges like mouth movement, tooth positioning, and light-tip distance..

Note that the mechanical properties are affected by the material type and composition.¹⁶ The dual-polymerize resin-core materials showed satisfactory results, similar in most cases or superior to the dual-polymerize resin cements.. In addition, adequate polymerization at deeper locations supports the indication for cementing fiberglass post. Comprehensive analyses of the physical and mechanical properties of the resin core materials suggest that rehabilitation of an endodontically treated tooth can be achieved using the same material.^{13,30} The present study suggests in vivo evaluation of resin-based materials longevity is warranted through clinical trials assessing restoration survival, success rates, and material behavior over time.

Due to their superior efficiency and effectiveness achieved by eliminating multiple material interfaces, dual-polymerized resin-core materials offer potential for time-saving restorations, encompassing post cementation, core build-up, and final prosthesis cementation, as demonstrated by their suitable physical and mechanical properties for restoring endodontically treated teeth.^{28,30}

CONCLUSIONS

Based on the findings of this present study, the following conclusions can be drawn:

- 1) The light-activation with a five minute delay after mixing the dual-polymerize resin-based materials significantly reduced post-gel shrinkage and maintained all tested mechanical properties.
- 2) Dual-polymerize resin-core materials present mechanical properties similar to those of bulk-fill CRs, guiding the performance of core build-up with a single material.
- 3) Dual-polymerize resin materials presented a higher depth of polymerization than bulk-fill CRs and similar or better mechanical properties than dual-polymerize resin cements used for cement fiberglass posts and final indirect restorations.

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TABLES

Table 1. Resin cements and bulk-fill resin composites tested

Materials		Type	Manufacturers	Primary Components	Weight	Volume	Lot Number	Shade
Clearfil DC	Core Plus	Dual-polymerize resin-core material	Kuraray	Inorganic fillers range from 0.01 μm to 20 μm . Paste A: Bis-GMA, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, Colloidal silica, dl-Camphorquinone, Initiators and Pigments. Paste B: TEGDMA, Hydrophilic aliphatic dimethacrylate, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, Aluminum oxide filler, and Accelerators.	Not specified	52%	5MO392	Dentin

LuxaCoreZ-Dual	Dual-cure resin-core material	DMG	Mean of filler size is about 0.02 - 2.4 µm. Barium glass, pyrogenic silicic acid, nanofiller, and zirconium dioxide in a Bis-GMA-based matrix from dental resin.	70%	50%	241962	A3
Rebilda DC	Dual-polymerize resin-core material	VOCO	Bis-GMA, UDMA, BHT and benzoyl peroxide.	Not specified	Not specified	2131566, 2143447, 2032447	Dentine
Allcem Core	Dual-polymerize resin-core material	FGM	Base paste: metacrylate monomers such as BisGMA, Bis EMA and TEGDMA, camphoroquinone, coinitiators, barium-aluminium-silicate glass microparticles and nano-particles of silicon dioxide, inorganic pigments and conservers. Catalyst paste: methacrylic monomers, dibenzoyl peroxide, stabilizers and Barium -Aluminium-silicate glass micro-particles.	62%	Not specified	150920, 170322, 130922.	A2

RelyX Universal	Dual-polymerize resin cement	3M ESPE	Catalyst Paste: YbF3, UDMA, Silane treated glass powder, TEGDMA, Silane treated silica, Initiator, pigments, e.g., Titanium dioxide, HEMA. Base Paste: TEGDMA, Silane treated silica, UDMA, Hydroperoxide.	Not specified	Not specified	8402901, 8452689	AO3
RelyX U200	Dual-polymerize resin cement	3M ESPE	Mean of filler size is about 12.5 µm. Base past: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, ionitiator components, stabilizers and reheological additives. Catalyst paste: methacrylate monomers alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments and rheological additives.	70%	Not specified	8299171. 8629419	A2
Allcem Dual	Dual-polymerize	FGM	Base paste: metacrylate monomers such as BisGMA, Bis EMA and TEGDMA, camphoroquinone,	66-67%	Not specified	300622, 1710022, 130122	A2

resin
cement

coinitiators, barium-aluminium-silicate glass microparticles, particles of silicon dioxide, inorganic pigments and preservatives. Catalyst paste: methacrylate monomers, dibenzoyl peroxide and stabilizers aluminium-silicate glass microparticles.

Filtek	One	Bulk-fill	3M ESPE	Non-agglomerated/non-aggregated 20nm silica filler, a non-agglomerated/non-aggregated 4 to 11nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20nm silica and 4 to 11nm zirconia particles) and a YbF3 filler consisting of agglomerate 100nm particles. Contains AFM (dynamic stress-relieving monomer), AUDMA, UDMA and DDDMA.	76.5%	58.5%	NE88322NF43624	A3
Bulk Fill		resin composite						

Opus Bulk Fill APS	Bulk-fill resin composite	FGM	Active ingredients: urethane-dimethacrylic monomers, stabilizers, photoinitiating composition (APS) and co-initiators. Inactive ingredients: inorganic load of silanized silicon dioxide (silica), stabilizers and pigments.	79%	Not specified	190122	A3
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Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, Urethane dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; BHT, butylated hydroxytoluene; YbF₃, ytterbium trifluoride; APS, Advanced Polymerization System; AUDMA Aromatic urethane dimethacrylate; AFM, addition fragmentation monomers; DDDMA, 1, 12-Dodecanediol dimethacrylate.

Table 2. Post-gel shrinkage (mean $\pm$ standard deviation) as per group of different material types for each light curing protocol (immediate and after five minutes)

Material type	Resin-base materials	Post-gel Shrinkage - %	
		Immediate	5 min
Dual-polymerize resin-core materials	ClearFill DC Core Plus	0.67 $\pm$ 0.05 Bb	0.42 $\pm$ 0.06 Aa
	LuxaCore Z	0.78 $\pm$ 0.04 Cb	0.57 $\pm$ 0.06 Ba
	Rebilda DC	0.55 $\pm$ 0.06 Bb	0.38 $\pm$ 0.03 Aa
	Allcem Core	0.63 $\pm$ 0.06 Bb	0.50 $\pm$ 0.07 Aa
Dual-polymerize cements	Rely X Universal	0.46 $\pm$ 0.05 Ab	0.36 $\pm$ 0.05 Aa
	Rely X U200	0.46 $\pm$ 0.05 Ab	0.38 $\pm$ 0.04 Aa
	Allcem Dual	0.59 $\pm$ 0.05 Bb	0.49 $\pm$ 0.07 Ba
Bulk-fill resin composites	Filtek One Bulk Fill	0.69 $\pm$ 0.08 C	-
	OPUS Bulk Fill APS	0.71 $\pm$ 0.05 C	-

Different letters expressed significant differences; capital letters used for comparing material type for each light-curing protocol; lower-case letters used for comparing light-curing protocol for each material.

Table 3. Flexural strength and elastic modulus (mean $\pm$ standard deviation) as per group of different material types for each light curing protocol (immediate and after five minutes)

Material type	Resin-base materials	Flexural strength (MPa)		Elastic modulus (MPa)	
		Immediate	5-min	Immediate	5-min
Dual-polymerize resin-core materials	ClearFill DC Core Plus	99.7 $\pm$ 11.2 Bb	108.1 $\pm$ 8.8 Ba	9.5 $\pm$ 1.2 Ba	10.4 $\pm$ 1.0 Ba
	LuxaCore Z	84.4 $\pm$ 8.8 Ca	87.8 $\pm$ 6.9 Ca	7.0 $\pm$ 0.9 Da	7.1 $\pm$ 0.7 Da
	Rebilda DC	128.3 $\pm$ 12.4 Aa	130.1 $\pm$ 7.7 Aa	8.4 $\pm$ 1.2 Ca	8.5 $\pm$ 1.0 Ca
	Allcem Core	114.3 $\pm$ 10.9 Ba	116.1 $\pm$ 9.0 Ba	8.2 $\pm$ 1.0 Ca	8.3 $\pm$ 1.0 Ca
Dual-polymerize resin cements	Rely X Universal	85.3 $\pm$ 6.1 Ca	91.2 $\pm$ 9.6 Ca	3.4 $\pm$ 0.3 Ea	3.6 $\pm$ 0.3 Ea
	Rely X U200	88.8 $\pm$ 8.1 Ca	93.3 $\pm$ 16.6 Ca	6.8 $\pm$ 0.3 Da	6.9 $\pm$ 0.9 Da
	Allcem Dual	104.8 $\pm$ 11.7 Ba	111.1 $\pm$ 9.0 Ba	7.5 $\pm$ 0.8 Da	7.7 $\pm$ 0.7 Da
Bulk-fill resin composites	Filtek One Bulk Fill	112.0 $\pm$ 11.8 B	-	10.0 $\pm$ 1.2 B	-
	OPUS Bulk Fill APS	109.0 $\pm$ 11.3 B	-	11.8 $\pm$ 1.0 A	-

Different letters expressed significant differences; capital letters used for comparing material type for each light-curing protocol; lower-case letters used for comparing light-curing protocol for each material.

Table 4. Depth of polymerization(mean $\pm$ standard deviation) as per group of different material types for each light curing protocol (immediate and after five minutes)

Material type		Resin-base materials	Depth of Polymerization- mm	
			Immediate	5-min
Dual-cure materials	resin-core	ClearFill DC Core Plus	5.08 $\pm$ 0.03 Aa	5.11 $\pm$ 0.07 Aa
		LuxaCore Z	5.03 $\pm$ 0.18 Aa	5.01 $\pm$ 0.23 Aa
		Rebilda DC	5.16 $\pm$ 0.49 Aa	5.09 $\pm$ 0.40 Aa
		Allcem Core	5.02 $\pm$ 0.35 Aa	5.07 $\pm$ 0.39 Aa
Dual-polymerize cements	resin	Rely X Universal	5.04 $\pm$ 0.08 Aa	5.08 $\pm$ 0.13 Aa
		Rely X U200	5.13 $\pm$ 0.26 Aa	5.15 $\pm$ 0.04 Aa
		Allcem Dual	5.15 $\pm$ 0.26 Aa	5.15 $\pm$ 0.09 Aa
Bulk-fill resin composites		Filtek One Bulk Fill	3.27 $\pm$ 0.35 B	-
		OPUS Bulk Fill APS	3.16 $\pm$ 0.25 B	-

Different letters expressed significant differences; capital letters used for comparing material type for each light-curing protocol; lower-case letters used for comparing light-curing protocol for each material.

FIGURES

Figure 1. Means and standard deviation of KH values (N/mm²) for core materials, dual-polymerize resin cements and bulk-fill resin composites. Linear regression of KH values according to depth of measurement. Logarithmic regression coefficient (R^2) for all resin-based materials.

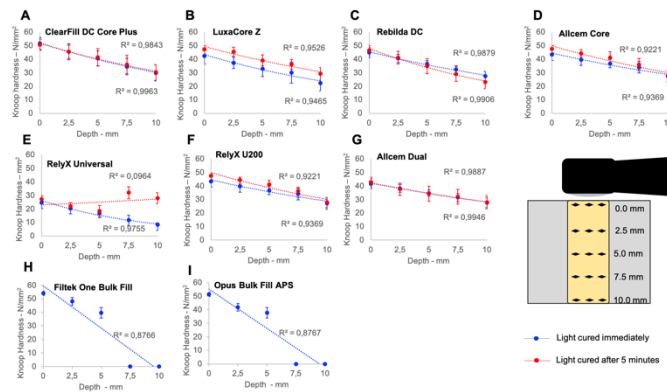
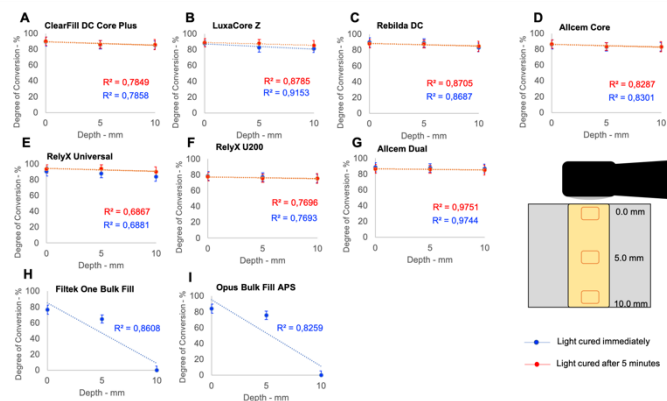


Figure 2. Means and standard deviation of DC values (%) for core materials, dual-polymerize resin cements, and bulk-fill resin composites. Linear regression of DC values according to depth of measurement. Logarithmic regression coefficient (R^2) for all resin-based materials.



ARTIGO 2

*Artigo enviado para Operative Dentistry

Radiopacity and filler content of dual-cure resin core material, dual-cure resin cement, and bulk-fill resin composites used for restoring endodontic treated teeth

ABSTRACT

OBJECTIVE. This study evaluated the effect of filler content and composition on the radiopacity level of different resin-based materials used to cement fiberglass posts (dual-cure resin cements or dual-cure resin core materials) and build the core (bulk-fill resin composites or dual-cure core resin materials) restoring endodontically treated teeth.

METHODS AND MATERIALS. Nine resin-based materials were tested as follows: Four dual-cure resin core materials: Allcem Core, FGM; LuxaCore Z, DMG; Rebuilda DC, VOCO, and Clearfil DC Core Plus, KURARAY; three dual-cure resin cements: RelyX Universal, 3M Oral Care; RelyX U200, 3M Oral Care, and Allcem Dual, FGM, and two bulk-fill resin composites: OPUS Bulk Fill APS, FGM, and Filtek One Bulk Fill, 3M Oral Care. Thermogravimetric analysis (TGA, %) was performed to determine the filler content. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) were used to evaluate the filler morphology and composition. Radiopacity was measured using digital radiographs ($n = 10$) according to ISO 4049 standards. TGA and radiopacity data were subjected to one-way analysis of variance (ANOVA) followed by Tukey test ($\alpha = .05$).

RESULTS. All the tested resin-based materials satisfied the ISO 4049 radiopacity requirements. The bulk-fill resin composites and dual-cure resin core materials had higher filler contents than dual-cure resin cements ($P < 0.001$). Allcem Core exhibited more homogeneous fillers without agglomerates, whereas RelyX Universal presented only nanoparticles. The OPUS Bulk Fill APS, Allcem Dual, and Allcem Core, made by the same manufacturer, presented similar sizes, shapes, and distributions of filler content. Energy-dispersive X-ray spectroscopy analysis revealed a consistent filler composition including Barium (Ba), Aluminum

(Al), Ytterbium (Yb), Fluorine (F), Strontium (Sr), Bromine (Br), and Zirconium (Zr), which contribute to the radiopacity of all the tested materials.

CONCLUSIONS. Dual-cure resin core materials exhibited a filler content similar to that of bulk-fill resin composites and higher than that of dual-cure resin cements, except for LuxaCore Z and Allcem Core. The radiopacity levels of the dual-cure resin core materials were similar to those of the bulk-fill resin composites, which indicates that the presence of elements with high atomic numbers achieved the minimum requirement of 4 mm of aluminum according to ISO Standards 4049 for all the tested materials.

Clinical relevance: Dual-cure resin core materials presented similar filler content, composition, handling characteristics and radiopacity to the bulk-fill resin composites. This suggests their potential suitability for core build-up following endodontic treatment and cementing the fiberglass post. Further investigation is warranted to assess their performance compared to traditional core materials

Keywords: dual-cure resin core materials, bulk-fill resin composite, dual-cure resin cement, radiopacity, filler content, scanning electron microscopy.

INTRODUCTION

The restoration of endodontically treated teeth continues to be a challenge because of the variability of clinical protocols involving different tooth conditions and the diversity of resin-based materials.^{1,2} In several cases, the amount of coronal structure is insufficient to provide retention in direct or indirect restoration.^{1,3} In these cases, to provide adequate retention, utilizing fiberglass posts associated with core reconstruction becomes necessary.^{1,4} Until now, for restoring endodontically treated teeth, dual-cure resin cement has been the first option to cement the fiberglass post associated with coronal reconstruction using conventional or bulk-fill resin composites.^{1,5} Simplifying the restorative procedure, minimizing the number of materials used, and reducing the number of interfaces are desirable goals in the restoration of endodontically treated teeth.. The use of dual-cure resin core material has been promoted by using a single material to do both procedures, which reduces the number of interfaces and saves time.^{5,6} The

in vitro evidence suggests that the resin-based core build-up material that has higher filler content showed the highest elastic modulus, which indicates stiffness, thereby improving the fracture resistance of the endodontically treated teeth compared to conventional resin composites.^{7,8}

Secondary caries located at the gingival margin restoration is a frequent cause of restoration failure.⁹ Adequate radiopacity diagnose recurrent caries at the gingival margin, but can also facilitate the identification of other potential issues such as ¹⁰ poor adaptation, contact with adjacent teeth, interfacial gaps, and voids present in the restoration.¹¹⁻¹³ The difficulty in discriminating the radiopacity levels between fiberglass posts, resin cements, and core materials may contribute to the complexity of evaluating the restorative procedures of endodontically treated teeth.¹⁴⁻¹⁸ The radiopacity of dental materials has been established according to ISO 4049 and is generally expressed in terms of equivalent aluminum thickness (mm).¹⁹ A resin-based material produced by the manufacturer must have the same or greater radiopacity value than the specified thickness of material to aluminum step wedges.^{20,21} Digital radiography images can measure the gray values on a scale of 0 to 255 through software, which provides a quantitative analysis of the material radiopacity.²¹ In this study, ImageJ software has been used to evaluate the mean gray values of the digital radiography of different resin-based materials.^{17,18}

The radiopacity level of resin-based materials can be influenced by various parameters such as composition, polymer matrix content, filler particle size, filler concentration, and thickness variations.^{19,22} Individual filler fraction analyses using thermogravimetric analysis (TGA) may better explain the physical-mechanical performance of resin-based materials.²³ Filler particle characterization using X-ray dispersive energy (EDS) microanalysis and scanning electric microscopy (SEM)²⁴ can produce a combination of methodologies to characterize and explain the radiopacity level of different resin-based materials.

Although the radiopacity of resin-based materials has been investigated extensively,^{12,17,18,25,26} few studies have evaluated the relationship between the filler characteristics and the radiopacity of current resin-based materials used for restoring endodontically treated teeth. Furthermore, to the best of our knowledge, no study has evaluated the radiopacity of different resin core materials.

Therefore, this in vitro study aimed to evaluate the radiopacity of different types of resin-based materials using a digital radiography system, determine the relationship between filler composition and radiopacity, and compare the radiopacity values expressed as the equivalent thickness of aluminum. The null hypotheses tested are as follows: the tested dual-cure resin core materials would present a radiopacity level that meets the requirements of ISO 4049 and the filler content of the dual-cure resin core materials would not differ from the resin cements and bulk-fill resin composites, and consequently from the radiopacity levels.

METHODS AND MATERIALS

Study design

Nine resin-based materials used for restoring endodontically treated teeth were tested in this study, which includes: four dual-cure resin core materials: Allcem Core (FGM, Joinville, Brazil); Clearfil DC Core Plus (Kuraray, Tokyo, Japan); LuxaCore Z (DMG, Hamburg, Germany), and Rebilda DC (VOCO, Cuxhaven, Germany); three automix dual-cure resin cements: RelyX Universal (3M Oral Care, St Paul, USA); RelyX U200 (3M Oral Care, St Paul, USA), and Allcem Dual (FGM, Joinville, Brazil), and two bulk-fill resin composites: OPUS Bulk Fill APS (FGM, Joinville, Brazil), and Filtek One Bulk Fill (3M Oral Care, St Paul, USA). The composition and light-curing procedures provided by the manufacturers are listed in Table 1. All the materials were tested for filler content (%) through thermogravimetric analysis (TGA), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS) to evaluate the morphology of the inorganic filler.

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) was used to obtain the filler mass fractions (%) of all the tested materials ($n = 3$).²³ The change in mass of the resin-based materials was measured as a function of temperature. Approximately 0.005 mg of each sample was light-activated for 40 s. Subsequently, the specimen was placed in a thermogravimetric analyzer (model TGA 55, TA Instruments, New Castle, Delaware, USA) and immersed in standard NETZSCH alumina 85 mL crucibles attached to the thermos-analytical unit (TGA-50, Netzsch-Thermische Analyse, Selb, Germany) with a TA System Controller

(TASC 414/2, New Castle, DL, USA) at a temperature range of 10–900 °C and a heating rate of 10 °C/min for approximately 80 min under synthetic air dynamics (50 mL/min). Thereafter, only the filler remained, and the filler content was calculated by considering the % of the remaining mass. The data were graphically analyzed at the onset of the E/T curve.²³

Scanning Electric Microscopy Analysis (SEM) / Energy Dispersive Spectroscopy (EDS)

The morphology and size of the fillers were evaluated using scanning electron microscopy (SEM; TESCAN, Brno, Czech Republic). A unique paste of the resin-based material, 0.3 g of each base and catalyst, was inserted into a conical bottom centrifuge tube in polypropylene (Falcon, Vitchlab, Wertheim, Germany) in 5 mL of acetone and centrifuged for 2 min at 1,000 rpm; this process was repeated three times. The remaining material mass was immersed thrice in 5 mL of chloroform P.A. (Alphatec, Rio de Janeiro, Brazil) and centrifuged as described above to further wash and eliminate the matrix.²⁷ The filler content was then smeared over the aluminum stubs and sputter-coated with gold/palladium in high vacuum (QR 150ES, Quorum, Laughton, East Sussex, United Kingdom). The specimens were examined under a scanning electron microscope (VEGA 3 LMU; Tescan, Brno, Czech Republic) operating at 20.00 kV. The images were obtained at 5,000 ×, 10,000 ×, and 20,000 × magnification. The particle composition was determined using the obtained powder and observed using an SEM/EDS integrated analysis system (INCA X-ACT model 51-ADD0007, Oxford, Abingdon, Oxfordshire, United Kingdom). The analyses were performed at working distance of 14.9 mm, 20 kV, and variable spot sizes to obtain a 30 s lifetime and 20 % dead time. For each specimen, one scan analysis of 4 s was performed at a magnification of 5000 x, 10000 x, and 20000x.

Radiopacity

Digital radiographs were obtained using standardized specimens measuring 5 mm in length, 2 mm in width, and 2 mm in thickness. The specimens were positioned over a phosphor plate (size 2, Durr Dental), and radiographically exposed using radiography equipment (Timex 70 E, Gnatus, Ribeirão Preto, Brazil) exposing the specimens to 0.07 s at 70 kV and 7.0 mA. The aluminum

scale, with a thickness ranging from 1 to 10 mm was increased every 1 mm (Odeme, Santa Catarina, Brazil) and placed on the matched plate having a focal length of 20 cm. Radiopacity was determined using the ImageJ software.^{11,28-30} Five locations were determined for each sample, proportional to its length, and the mouse cursor was positioned to collect the radiopacity value. The average of five calculated values was used as the radiopacity level for each specimen. A value was assigned to each pixel to represent a specific gray shade. These pixel values range from 0 to 255, where 0 represents the darkest gray or black and 255 represents the lightest gray or white.

Statistical Analysis

Radiopacity and TGA data were tested for normal distribution (Shapiro-Wilk) and equality of variances (Levene's test), followed by parametric statistical tests. One-way analysis of variance (ANOVA) was performed to identify the differences in the tests. Tukey post-hoc test was used for multiple comparisons between the tested materials. All the tests were performed at a statistical significance level of $P < 0.05$. The SEM and EDS analyses were performed descriptively.

RESULTS

TGA, Filler content (%)

The mean filler content in weight (%) and standard deviation values of the light-activated resin-based materials are shown in Figure 1. One-way ANOVA revealed a significant difference between the resin-based materials ($P < 0.001$). Tukey's test showed that the bulk-fill resin composite and most of the dual-cure resin core materials had higher filler contents than the dual-cure resin cement. Both the bulk-fill resin composites, Clearfil DC Core Plus and Rebilda DC, had the highest filler content, and RelyX Universal had the lowest filler content in weight ($P < 0.001$).

Radiopacity

The mean and standard deviation radiopacity values of all the resin-based materials are shown in Figure 2. One-way ANOVA showed a significant influence for the resin-based materials ($P < .001$). Tukey's test showed that all the tested resin-based materials had radiopacity levels higher than the required 4 mm

aluminum step-wedge, which demonstrated that all tested materials had radiopacity levels recommended by ISO 4049 (Figure 3).

SEM analysis / EDS

The morphologies of the filler particles in the resin-based materials are shown in Figure 4. The evaluated materials contained irregularly agglomerated particles of different sizes. Allcem Core presented more homogeneous fillers without agglomerates, whereas RelyX Universal exhibited only nanoparticles in the catalyst paste. Furthermore, Clearfil DC Core Plus, RelyX U200, and LuxaCore Z had the largest particle sizes. The Filtek One Bulk Fill resin composite contained rounded nanocluster particles, unlike the resin cements (RelyX Universal and RelyX U200) from the same manufacturer. However, the OPUS Bulk Fill APS resin composite presented a similar size, shape, and distribution of filler content as the dual-cure resin cement (Allcem Dual) and dual-cure resin core material (Allcem Core) from the same manufacturer.

The results of the EDS analysis in Figure 5, showed that oxygen (O, 8) and silicon (Si, 14) followed by carbon (C, 6), aluminium (Al, 13) and Ba were the prevalent elements present in the filler content of all the tested resin-based materials. RelyX Universal also contains Bromine (Br, 35) fluorine (F, 9), Yb, F, and Sr. The RelyX U200 contained sodium (Na, 11), phosphorus (P, 15), calcium (Ca, 20) and Sr, La, F. Filtek One Bulk Fill contained Zr and Yb.

DISCUSSION

The first null hypothesis that all the tested dual-cure resin core materials, dual-cure resin cements, and bulk-fill resin composite had radiopacity levels higher than 4.0 mm of aluminum was accepted. However, the second hypothesis that the dual-cure resin core materials presented filler content similar to that of bulk-fill resin composites, but higher filler content than dual-resin cement was rejected. Dual-cure resin core materials presented a radiopacity similar to that of the bulk-fill resin composites, thereby indicating a high atomic number and higher radiopacity than the dual-cure resin cements.

Resin-based materials possess different radiopacities; consequently, they can perform differently in radiographic monitoring.⁹⁻¹¹ The intrinsic radiopacity of resin-based materials depends, in part, on the atomic number of their constituent atoms. Resin-based materials tend to be more radiopaque if they contain a high

number of atoms, such as zirconium (Zr, 40), Iodine (I, 53), barium (Ba, 56), and Ytterbium (Yr, 70). The presence of chemical elements such as zinc (Zn, 30), strontium (Sr, 38), yttrium (Y, 39), lanthanum (La, 57), Zr, Yr, Ba, v increases the radiopacity.^{12,31} All the materials had a higher radiopacity than the required 4 mm aluminum scale. Ba was the most commonly used elemental filler in the test material. However, resin-based materials produced by 3M Oral Care had Sr, La and Yr. The bulk-fill resin composites contained elements with high atomic numbers such as Zr, Y, and Yr. Therefore, the radiopaque filler particles influenced the radiopacity level obtained by all the tested resin-based materials, which made their radiopacity equivalent to or greater than that of the 4 mm aluminum step-wedge. This finding demonstrated that all materials had higher radiopacity to dentin, whereas the ISO (4049-2019) standards recommend 1 mm aluminum.^{16,20}

While radiopacity of resin-based materials is crucial for clinical assessment, it primarily aids in visualizing potential issues like interfacial gaps. Dentin itself exhibits natural radiopacity, making it difficult to distinguish the interface between core material and dentin solely based on radiographs.^{15 15} This is important to diagnose marginal gaps, recurrent caries, marginal overhangs extending to subgingival regions, and delineating tissues and structures such as dentin, enamel, reconstruction of coronal portion using fiberglass post, and crown.^{9,20} The complications associated with cementing fiberglass post and core build-up can be monitored and prevented when using a radiopaque material.^{11,28,30} Additionally, it can aid in identifying any excess that needs to be removed to prevent periodontal inflammation or other complications.⁵ Using radiopaque cement helps identify the contour of fiberglass posts on a radiograph for long-term monitoring restorations.^{11,19} The material selection to be used for restoring endodontically treated teeth must permit visibility in the radiographs.³¹ Radiopacity is subject to ISO standards and all the materials tested meet the minimum requirements of 4 mm aluminum.

Previous studies have highlighted the importance of filler content in luting resin-based materials, higher levels are associated with increased rigidity and mechanical resistance.⁵ Moreover, the higher filler load is associated with elevated viscosity, which hinders the injection of the material into the root canal causing voids and gaps, ultimately contributing to recurrent caries.³¹ The dual-

cure resin core materials, predominantly based on methacrylate resin presented a higher filler content, endowing them superior mechanical properties compared to conventional resin cements.⁷ The TGA analysis performed in the present study suggest that dual-cure resin core materials exhibited similar filler content to bulk-fill resin composite. While this aligns with previous research demonstrating a connection between filler content and similar resin-based materials.²⁹ The accuracy of the TGA method itself can be questioned, particularly considering the observed discrepancies between our results and manufacturer specifications. These discrepancies suggest potential underestimation, highlighting the need for further investigation with potentially more reliable methods. Furthermore, the sample size of N=3 per group is relatively small; while acceptable for the specific parameter measured being very precise methodology, where the results give no significant difference when working with a sample size of 5 or 10 as occurred in the pilot test. Moreover, the higher percentage of filler content with high atomic numbers significantly influences radiopacity, with resin cements containing a greater percentage of filler being more radiopaque, aiding in clinical detection.^{19,29} It was observed that Clearfill DC Core Plus and Rebilda DC exhibited more filler content, and barium particles, resulting in higher radiopacity values. In contrast, RelyX U200 and RelyX Universal lack Ba, but contain Zr, Sr, and Yb. Ensuring that these materials exhibit radiopacity values within the standard range is essential for clinicians to diagnose interfacial gaps, voids, defects in adaptation, discrepancies in restoration contours, and lack of contact with neighboring teeth.^{10,11}

Filler content plays a critical role in determining the performance of dental materials, particularly when used to lute fiberglass post, fixed prosthesis, and core build-up.⁴ The higher the percentage of filler, the better these materials can support stresses, which is crucial to sustain the load on them during function.³¹ Additionally, higher filler percentages can influence post-gel shrinkage and subsequently increase the fracture resistance.⁵ Comprehensive analyses of the physical and mechanical properties of dual-cure resin core materials highlight their potential for supporting the rehabilitation of endodontically treated teeth.²⁴ These materials facilitate the cementing of fiberglass posts, reconstruction of core build-up, and helps to perform crown cementation with reduced number of the interfaces, thereby offering a convenient and efficient solution.³ Moreover, the

presence of higher filler content may lead to an increase in the fracture resistance of weak teeth.^{7,23} Ultimately, understanding the composition of resin-based materials through methodologies such as TGA provides valuable insights into their mechanical properties and performance.²³

The composition of the resin matrix significantly influences the rheological and viscoelastic properties of the resin-based materials, impacting their flow characteristics and overall performance.^{7,7} The present study aligns with previous research²⁴ demonstrating that while dual-cure core materials may have similar content to resin composites, the specific matrix composition plays a critical role in determining their rheology and viscoelasticity. . The addition of monomers with low molecular weight enhances the flexural properties and reduces viscosity in these materials.^{5,24} The use of dual-cure resin core materials with high modulus elasticity, can enhance the fracture resistance of endodontically treated teeth and serve as an alternative for one-stage fiberglass post-placement and core build-up.²³

CONCLUSIONS

Within the limitations of this in vitro study the following conclusions were drawn:

- The dual-cure resin core materials have filler percentages similar to those of bulk-fill resin composites.
- The radiopacity of all the tested resin-based materials fulfilled the minimum requirement of 4 mm of aluminum according to the ISO Standards 4049.
- The high atomic number of the elements present in all the tested resin-based materials explains the higher radiodensity.
- The filler content and radiopacity of the dual-cure resin core materials varied among the tested products. LuxaCore Z and Allcem Core exhibited lower values compared to Filtek One Bulk Fill and OPUS Bulk Fill APS.

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Figures

Figure 1. Means and standard deviation of filler content in weight values (%) for core materials, dual-cure resin cements and bulk-fill resin composites. Different letters mean significant difference; upper case letter used for comparison between materials for each light curing protocol; and lower case letter used for comparison between light curing protocol for each resin-based material.

Figure 2. Means and standard deviation of radiopacity values for dual-cure resin core materials, dual-cure resin cements and bulk-fill resin composites; upper case letter used for comparison between materials for each light curing protocol; and lower case letter used for comparison between light curing protocol for each resin-based material.

Figure 3. Radiopacity level of different tested resin-based materials: A. Clearfill DC Core Plus, B. LuxaCore Z, C. Rebilda DC D: Allcem Core, E. RelyX Universal, F. RelyX U200, G: Allcem Dual, H. Filtek One Bulk Fill, I: OPUS Bulk Fill APS.

Figure 4. Scanning electron microscopy of filler content in the dual-cure resin core materials, dual-cure resin cements, and bulk-fill resin composites tested - 20000× magnification.

Figure 5. Energy dispersive spectroscopic analysis of the filler particles of dual-cure resin core materials, dual-cure resin cement, and bulk-fill resin composites tested.

Table

Table 1. Resin-based materials tested.

Materials	Type	Batch Number	Shade	Components	Weight %/ Volume %	Manufacture
Clearfil DC Core Plus	Dual-cure resin core material	5MO392	Dentin	Inorganic fillers range from 0.01 μm to 20 μm . Paste A: Bis-GMA, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, colloidal silica, dl-camphorquinone, Initiators and Pigments. Paste B: TEGDMA, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, aluminum oxide filler and accelerators.	74/52	Kuraray, Osaka, Japan
LuxaCore Z	Dual-cure resin core material	241962	A3	Mean of filler size is about 0.02 - 2.4 μm . Barium glass, pyrogenic silicic acid, nanofiller and zirconium dioxide in a Bis-GMA based matrix from dental resin.	70/50	DMG, Ridgefield Park, NJ, USA
Rebilda DC	Dual-cure resin core material	2131566, 2143447, 2032447	Dentin	Bis-GMA, UDMA, BHT and benzoyl peroxide.	71/57.3	VOCO, Cuxhaven, Germany

Allcem Core	Dual-cure resin core material	150920, 170322, 130922.	A2	Base paste: methacrylate monomers such as BisGMA, Bis EMA and TEGDMA, camphoroquinone, coinitiators, barium-aluminium-silicate glass microparticles and nano-particles of silicon dioxide, inorganic pigments and conservers. Catalyst paste: methacrylic monomers, dibenzoyl peroxide, stabilizers and barium-aluminium-silicate glass micro-particles.	70/62	FGM, Joinville, SC, Brazil
RelyX Universal	Dual-cure resin cement	8402901, 8452689	AO3	Catalyst Paste: YbF3, UDMA, Silane treated glass powder, TEGDMA, silane treated silica, Initiator, pigments, e.g., titanium dioxide, HEMA. Base Paste: TEGDMA, silane treated silica, UDMA, hydroperoxide.	72/50	3M Oral Care, MN, St Paul, USA
RelyX U200	Dual-cure resin cement	8299171, . 8629419	A2	Mean of filler size is about 12.5 µm. Base past: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers and rheological additives. Catalyst paste: methacrylate monomers alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments and rheological additives.	70/43	3M Oral Care, MN, St Paul, USA
Allcem Dual	Dual-cure resin cement	300622, 1710022, 130122	A2	Base paste: methacrylate monomers such as BisGMA, Bis EMA and TEGDMA, camphoroquinone, coinitiators, barium-aluminium-silicate glass microparticles, particles of silicon dioxide, inorganic pigments and preservatives. Catalyst paste:	66/57	FGM, Joinville, SC, Brazil

				methacrylate monomers, dibenzoyl peroxide and stabilizers aluminium-silicate glass microparticles.		
Filtek One Bulk Fill	Bulk-fill resin composite	NE88322NF4 3624	A3.5	Non-agglomerated/non-aggregated 20nm silica filler, a non-agglomerated/non-aggregated 4 to 11nm zirconia filler, an aggregated zirconia/silica cluster filler (comprised of 20nm silica and 4 to 11nm zirconia particles) and YbF3 filler consisting of agglomerate 100nm particles. Contains AFM (dynamic stress-relieving monomer), AUDMA, UDMA and DDDMA.	76.5/58.5	3M Oral Care, MN, St Paul, USA
OPUS Bulk Fill APS	Bulk-fill resin composite	190122	A3	Active ingredients: urethane-dimetacrylic monomers, stabilizers, photoinitiator composition (APS) and co-initiators. Inactive ingredients: inorganic load of silanized silicon dioxide (silica), stabilizers and pigments.	79/68	FGM, Joinville, SC, Brazil

Bis-GMA, bisphenol A-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, Urethane dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; BHT, butylated hydroxytoluene; YbF3, ytterbium trifluoride; APS, Advanced Polymerization System; AUDMA Aromatic urethane dimethacrylate; AFM, addition fragmentation monomers; DDDMA, 1, 12-Dodecanediol dimethacrylate.

ANEXOS

*Figuras do Artigo 2

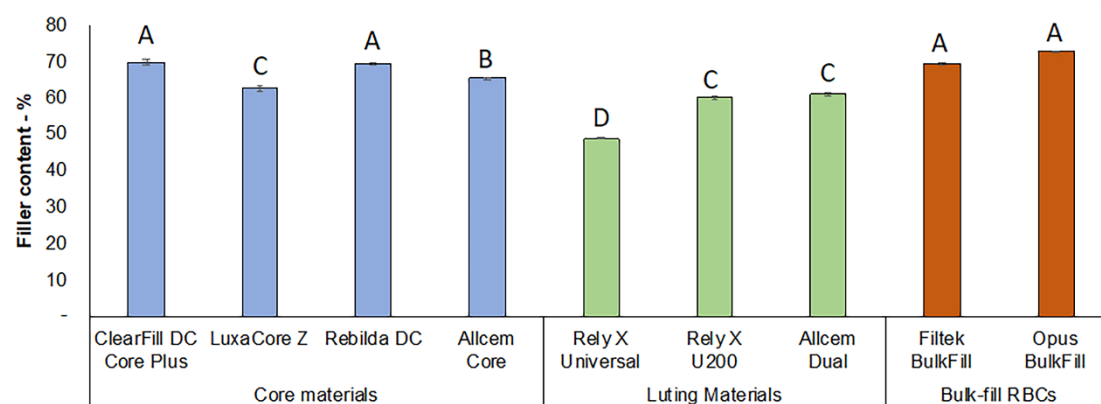


Figure 1. Means and standard deviation of filler content in weight values (%) for core materials, dual-cure resin cements and bulk-fill resin composites. Different letters mean significant difference; upper case letter used for comparison between materials for each light curing protocol; and lower case letter used for comparison between light curing protocol for each resin-based material.

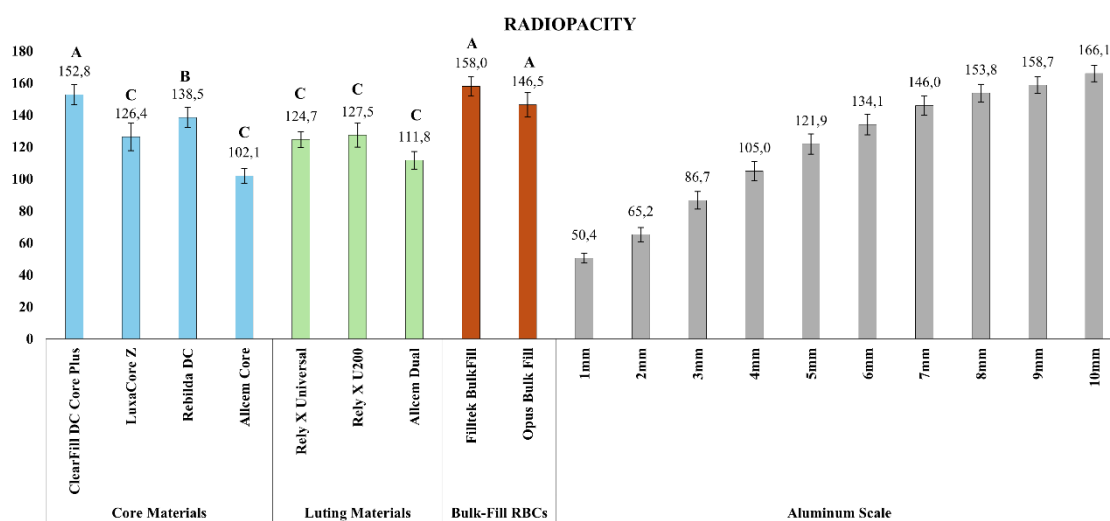


Figure 2. Means and standard deviation of radiopacity values for dual-cure resin core materials, dual-cure resin cements and bulk-fill resin composites; upper case letter used for comparison between materials for each light curing protocol; and lower case letter used for comparison between light curing protocol for each resin-based material.

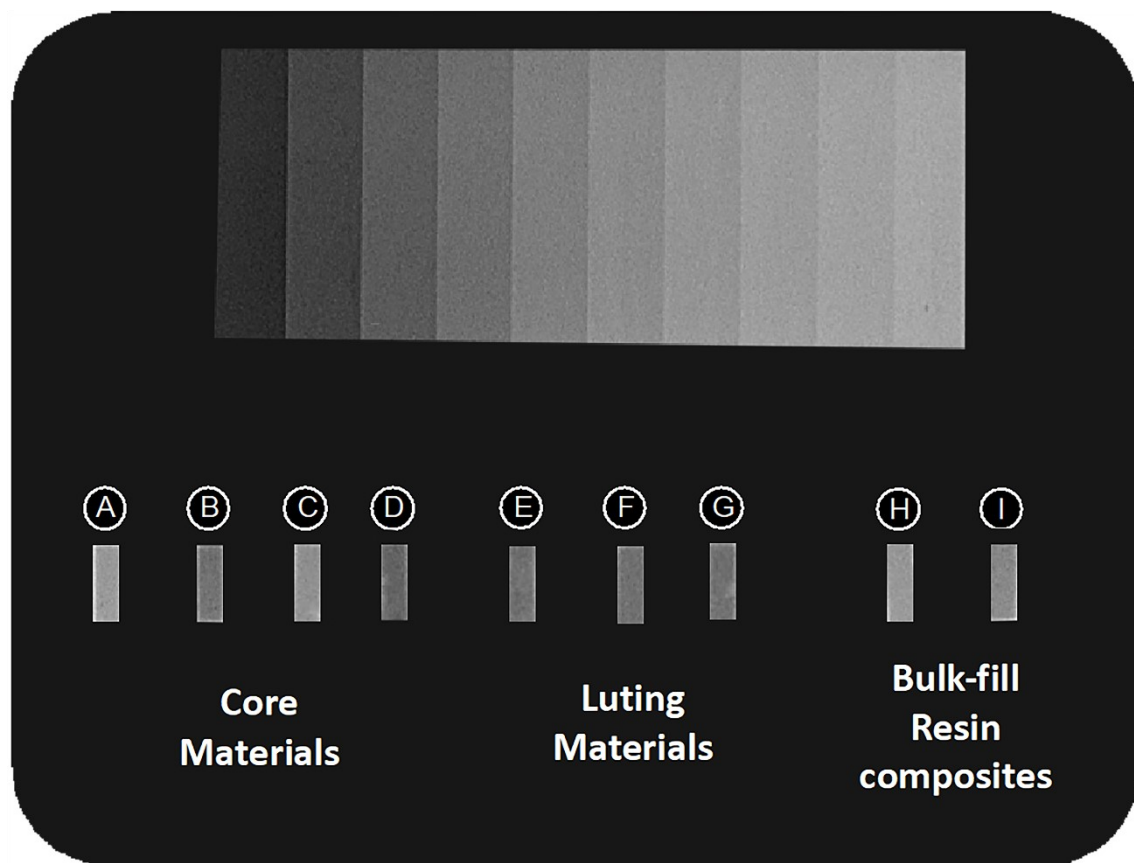


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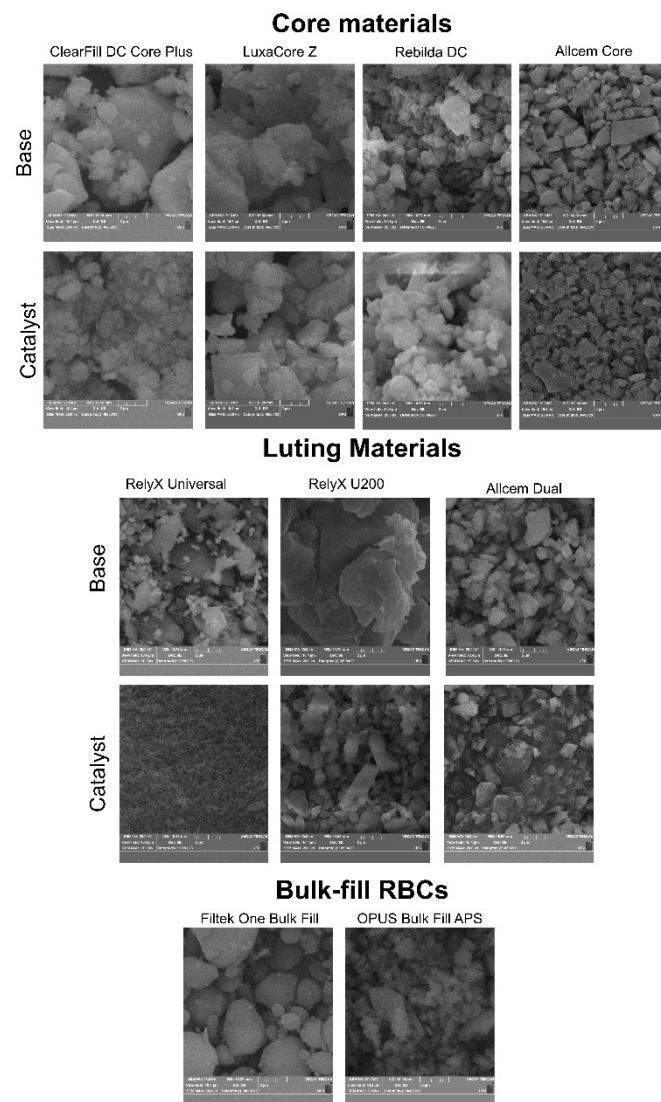


Figure 4. Scanning electron microscopy of filler content in the dual-cure resin core materials, dual-cure resin cements, and bulk-fill resin composites tested - 20000× magnification.

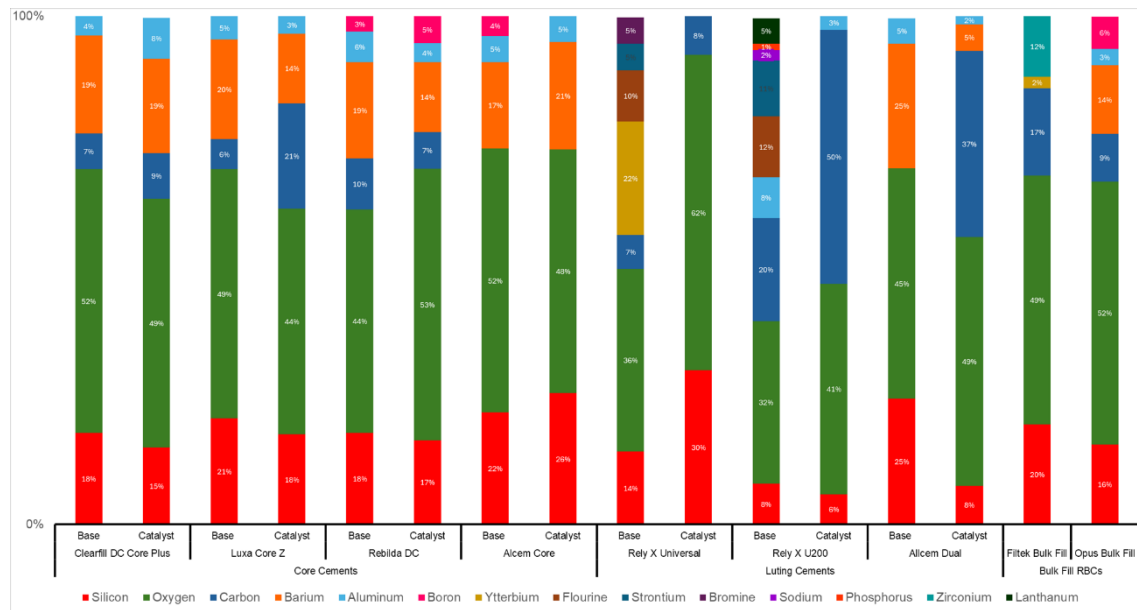


Figure 5. Energy dispersive spectroscopic analysis of the filler particles of dual-cure resin core materials, dual-cure resin cement, and bulk-fill resin composites tested.