# Comparison of the Mechanical Properties and Push-out Bond Strength of Self-adhesive and Conventional Resin Cements on Fiber Post Cementation

MR Santi • RBE Lins • BO Sahadi • JR Soto-Montero • LRM Martins

### Clinical Relevance

The mechanical behavior of self-adhesive resin cements is not superior to conventional resin cements.

### **SUMMARY**

Objectives: The purpose of this study was to compare the mechanical properties and pushout bond strength of self-adhesive resin cements (SACs) and a conventional resin cement (CRC).

Methods and Materials: Eighty bovine incisors were divided into four groups for cementation of a fiberglass post (Whitepost - FGM Dental Group, Coral Springs, FL) with different resin cements: three SACs (Maxcem Elite, MAX - Kerr; Calibra

Jorge Rodrigo Soto-Montero, DDS, MS, PhD, invited professor,

Universal, CAL - Dentsply; and RelyX Unicem 2, RUN - 3M Oral Care) and one CRC (RelyX Ultimate, RXU - 3M Oral Care). The groups were subdivided into two groups each (n=10) for evaluation of the push-out bond strength test (POBS) after 24 hours of water storage or after thermal aging (5000 cycles), following 24 hours of storage. The failure modes were evaluated using a stereomicroscope. Flexural strength (FS) and modulus of elasticity (EM) were determined using a three-point bending. Also, pH of the cements

University of Costa Rica, School of Dentistry, Department of Restorative Dentistry, Montes de Oca, SJ, Costa Rica

Luís Roberto Marcondes Martins, DDS, MS, PhD, professor, Piracicaba Dental School, University of Campinas, Piracicaba, SP Brazil

http://doi:10.2341/21-015-L

<sup>\*</sup>Marina Rodrigues Santi, DDS, MS, PhD student, Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

Rodrigo Barros Esteves Lins, DDS, MS, PhD, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

Beatriz Ometto Sahadi, DDS, MS, PhD student, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

<sup>\*</sup>Corresponding author: Avenida Limeira 901, Areião, São Paulo, Brazil 13414-903; e-mail: mrs.santi@hotmail.com

was measured over 48 hours and filler morphology was observed by scanning electron microscopy. Appropriate statistical analyses were performed by SPSS 21.0 (SPSS Inc., Chicago, IL, USA), with a significance level set at 5%. Results: RXU presented the highest POBS at both evaluation times. Among the SACs, RUN and CAL presented significantly lower POBS than MAX in cervical and middle-thirds at the 24-hour evaluation, and in all root regions after thermocycling. Adhesive failure between the cement and dentin were the most prevalent fractures at both times evaluated. MAX presented the lowest FS and RUN showed the highest EM. The pH reached the minimal point at the 30-minute evaluation for RXU and MAX. For RUN and CAL, the minimal pH was observed at the 60-minute evaluation. RXU and RUN presented spherical and regular filler particles, while MAX and CAL presented irregularly shaped and sized filler particles.

Conclusions: The mechanical behavior of SACs is not superior to CRC; however, among all the SACs evaluated, MAX presented the highest POBS and stability after thermocycling evaluation. MAX also reached the closest neutral pH after 48 hours. Therefore, SACs with low initial pH and strong neutralization reactions are recommended, because these characteristics may lead to better mechanical properties and stability.

# INTRODUCTION

The luting procedure is one of several factors that lead to clinical success of fiberglass post (FRC) cementation.<sup>1,2,3</sup> Conventional resin cements (CRC) are the usual material of choice because of their good mechanical properties. 4,5 However, CRCs are technique sensitive because of the numerous steps involved, such as acid etching the root canal, and the application of an adhesive system. These steps can be affected by operator mistakes, causing misplacement of the FRC. 6,7,8 In addition, adhesion into the radicular dentin is challenging because of polymerization shrinkage stresses resulting from a high configuration factor, and the limited access and visibility.<sup>2,3</sup> Hence, in response to clinical concerns, a new kind of self-adhesive resin cement (SAC) was developed to simplify the luting procedure and promote good adhesion in clinical situations where there is low mechanical retention.

Commercially available, SACs adhere to tooth structure without intermediary adhesives or etchants, combining ease of application with mechanical and adhesive properties similar or higher to those of CRCs. <sup>1,8</sup> Nonetheless, besides the mentioned simplified technique, the main difference between SACs and CRCs are the composition and bonding mechanism to the dental structure. <sup>3,8,9</sup> In short, traditional SACs include acidic functional monomers to promote etching of the mineralized tissues, <sup>8,9</sup> a dual cure polymerization mechanism, and fillers that neutralize the low pH of the cement. <sup>7,8</sup>

Adhesion by SACs occurs when the acid-functionalized monomers such as methacrylate with carboxylic or phosphoric acid-groups etch the enamel and dentin, while also producing a chemical bonding to the calcium on hydroxyapatite of the substrate. <sup>9,10</sup> Based on this mechanism, it is important to analyze the changes of the pH of the cements during setting and curing, because high hydrophilicity leads to water sorption, degradation, and consequently compromises the mechanical stability of the material. <sup>9,10,11</sup> Another potential effect of cement on the outcome of restorations is related to the composition of each resin cement, which can affect the flexural strength (FS) and elastic modulus, which in turn are indicators of the ability to resist stresses without deformation or fracture. <sup>10-13</sup>

Recently a review about the current status of clinical studies on SACs found that only a few studies compared the performance of SACs with that of CRCs, and none of the studies reported retention loss for either cement type. However, there is scarce literature on the behavior of SACs, and how the differences of the mechanical properties produced by the alteration in the composition of a SAC compared to a CRC affect the performance of this material. Hence, the comparative analysis of SACs and CRCs becomes clinically relevant and can provide important information to clinicians, by characterizing the behavior of the cements. 4,11,14

Thus, the aim of this study was to evaluate the pushout bond strength (POBS) of fiberglass posts cemented with SACs and CRCs to root dentin after 24 hours and after thermocycling, and to compare the flexural strength, modulus of elasticity, pH-neutralization behavior, and filler morphology. The null hypotheses were: 1) the POBS of the SACs would not be different to that of the CRCs; 2) the POBS of the evaluated cements would not change after thermocycling; 3) there would not be differences on the flexural strength and modulus of elasticity of the evaluated cements; and 4) the pH of the evaluated cements would not change during the setting reaction.

### **METHODS AND MATERIALS**

Four resin cements were included in the study: three SACs (Calibra Universal [CAL], Dentsply Sirona, York,

PA, USA; MaxCem Elite [MAX], Kerr, Orange, CA, USA; and RelyX Unicem 2 [RUN], 3M Oral Care, St Paul, MN, USA) and one dual cure CRC RelyX Ultimate 2 (3M Oral Care), as described in Table 1.

# **Push-Out Bond Strength Test**

For POBS evaluation, the procedure was validated, and the required sample size was calculated based on a pilot study. Bovine incisors with closed apex, were used for POBS evaluation. The crowns of the teeth were sectioned 2 mm below the cement-enamel junction, and root canals with a remaining length between 16 and 18

mm were selected for use in the study, to standardize the working length at 15 mm (±1 mm). Then, the thickness of the circumferential coronal dentin was measured at four positions (mesial, distal, buccal, and lingual) with a digital caliper (Mitutoyo, Suzano, SP, Brazil). The root canals with a coronal dentin thickness of 2 mm (±0.5 mm) were selected. The process was repeated until 80 specimens that complied with the established criteria were obtained. The selected root canals were divided into four groups for POBS evaluation of each cement. The groups were further subdivided into two groups for each cement (n=10), one for POBS evaluation 24 hours after specimen preparation, and

Material (lot number)	Classification	Light Cure Time (seconds for each surface)	Composition	
RelyX Ultimate - 3M ESPE, St Paul, MN, USA (#6018295)	CRC	40	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers. Catalyst paste: methacrylate monomers. Fillers (43 vol%): Silanated fillers; Alkaline (basic) fillers; Initiators: Sodium toluene-4-sulphinate, disodium peroxodisulphate Tertbutyl 3,5,5 Trimethylperoxyhexanoate	
RelyX Unicem 2 - 3M Oral Care, St Paul, MN, USA (#3579029)	SAC	20	Base Paste: Silane-treated glass powder, 2-Propenoic acid, 2- methacryl-oxyethyl phenyl hydrogen phosphate (Phenyl-P), 2- methyl, 1,10-[1-(hydroxymethyl)-1,2-ethanodiyl] ester, triethylene glycol dimethacrylate (TEGDMA), silane-treated silica, glass fiber, sodium persulfate, and Tert-butyl peroxy-3,5,5-trimethylhexanoate.  Catalyzer paste (43 vol%): Silane-treated glass powder, dimethacrylate substitute, silane-treated silica, Sodium p-toluenesulfonate, 1-Benzyl-5-phenylbarbituric acid, calcium salts, 1,12-Dodecanediol dimethacrylate, calcium hudroxide, and titanium dioxide.	
Calibra Universal - Dentsply Sirona, York, PA, USA (#180108)	SAC	10	Urethane Dimethacrylate; Di- and Tri-Methacrylate resin: Phosphoric acid modified acrylate resin (Dipentaerythrite penta-acrylate monophosphate: PENTA-P); Barium Borofluoroaluminosilicate Glass; Organic Peroxide Initiator; Camphorquinone (CQ) Photoinitiator; Phosphene Oxide Photoinitiator; Accelerators; Butylated Hydroxy Toluene UV Stabilizer; Titanium Dioxide; Iron Oxide; Hydrophobi Amorphous Silicon Dioxide Particles of inorganic filler range from 16 nm to 7 µm, average particle size 3.8 µm total filler 48.7% by volume.	
MaxCem Elite - Kerr, Orange, CA, USA (#7293230)	SAC	10	Multifunctional DMAs, GPDM, proprietary Redox initiators and photo-initiators, barium, fluoroaluminosilicate, and fumed silica (46% by volume)	

the other group was tested after thermocycling aging following 24 hours of storage.

Cleaning and shaping of root canals were performed by crown-down technique, using K-type files #80 (Dentsply Maillefer, Ballaigues, Switzerland). Working length was set 1 mm short of the apical foramen, based on the clinical length of the roots. Apical enlargement was performed manually using a #70 file. Afterward, the specimens were washed with 0.5% sodium hypochlorite solution and 17% EDTA for 1 minute each. Sealing of the root canals was performed by lateral condensation of gutta percha cones (Dentsply Sirona, York, PA, USA), using a calcium hydroxide cement (Sealer 26; Dentsply Sirona). The coronal access of the specimens was sealed with Coltosol (Coltene, Rio de Janeiro, RJ, Brazil) and stored at relative humidity for seven days in an oven (Fanen, Guarulhos, SP, Brazil) at 37°C.15 After storage, 12 mm (±1 mm) of intracanal gutta percha were removed using the specific bur of the selected glass-fiber post system (White Post DC #3, FGM, Joinville, SC, Brazil) in a low-speed handpiece (Kavo, Kerr, SP, Brazil) in a single perpendicular movement. After preparation, the root canals were rinsed with distilled water to remove remaining gutta percha and dried using absorbent paper tips (Dentsply Maillefer). 15 Prior to cementation, the fiber post was cleaned with 70% alcohol according to manufacturer's instructions.

Resin Cement Luting—For the RXU, a drop of a onestep adhesive (Single Bond Universal, 3M Oral Care) was applied on the FRC post surface and in the canal wall according to manufacturer's recommendation. For SACs, the fiber post and the canal wall did not receive any pretreatment, according to the manufacturer's instructions. For the CRC, the cement was mixed and inserted into the canal using the intracanal needle tip of a Centrix system (DFL; Rio de Janeiro, RJ, Brazil). For CAL, RUN, and MAX, the cements were applied using the self-mixing tip supplied by the manufacturers. In all groups, the FRC was placed into the root canal and while excess cement was removed, a digital pressure was applied for one minute. The cements were light cured according to the manufacturers' recommended time at 0 mm distance, using a light curing unit (Valo, Ultradent Products Inc; South Jordan, UT, USA) with a radiant emittance of 1200 mW/cm<sup>2</sup>. Classification, brand name, composition, and exposure time of the evaluated materials are presented in Table 1. A schematic representation of the configuration of the root canal after the luting procedure is presented in Figure 1.

The specimens were stored in a dark oven for 24 hours in 100% relative humidity at 36°C. After storage, specimens were sectioned using a slow-speed cutting machine (Isomet 1000, Buehler, Uzwil, Switzerland) into

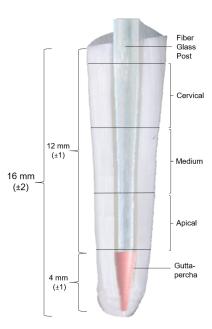


Figure 1. Schematic representation of the fiberglass post cementation and the slices obtained for the Push-Out Bond Strength.

three, 1-mm-thick slices from each radicular third. 16,17 The thickness of the obtained slices was measured and registered, and the POBS test (n=10) was realized. For the group treated by thermocycling aging following 24 hours of water storage, in the cycling machine (MCT2-AMM2, São Paulo, SP, Brazil), the specimens were submitted to 5000 cycles in water at 5°C (±1°C) to 55°C (±1°C), 30 seconds at each temperature and a transfer time of five seconds, simulating six months of aging. After thermal cycling, the roots were sliced following the aforementioned protocol and the POBS was measured. 18 The POBS evaluation (n=10) was performed using a universal testing machine (Instron, Norwood, MA, USA) at 1.0 mm/min of speed. 16

### **Failure Mode**

After POBS evaluation, the debonded specimens were recovered and observed using a stereomicroscope (Carl Zeiss Inc, Oberkochen, Germany) at 40× to categorize the failure mode. The observed patterns were: (I) adhesive failure between the cement and the dentin; (II) adhesive failure between the cement and the post; (III) cohesive failure in the cement; (IV) cohesive failure in the post; and (V) mixed failures consisting in a combination of two or more failure modes. 17,18

### Flexural Strength and Modulus of Elasticity

For flexural strength and modulus of elasticity measurement, the specimens (n=15) were prepared according to ISO 4049. The specimens were molded

using a customized mold (2×2×25 mm), light cured (Valo, Ultradent Products, Inc) and finished using a 1200-grit abrasive paper to remove flanges. After finishing, the specimens were stored in water for 24 hours at a temperature of 36°C. <sup>19,20,21</sup> The bars were fixed in a three-point-bending test rig (fin distance 20 mm) of a universal testing machine (Instron-Norwood, MA, USA) and loaded until fracture with a crosshead speed of 1.0 mm/min.

# pH Measurement

The resin cements (n=3) were filled into a silicone mold (diameter 20 mm, thickness 2 mm) and a Mylar strip was positioned on the top of the surface to prevent the formation of an air inhibition layer. The specimens were light cured according to manufacturer's recommendation. Afterwards, the samples were individually immersed in 10 mL of distilled water with a pH-electrode (An2000, Analion, Ribeirão Preto, SP, Brazil) on the top, and evaluated over 1-, 5-, 15-, 30-, 60-minutes, 24 hours and 48 hours, protected from light to exclude any curing influence.<sup>20</sup> After each pH evaluation, the distilled water was changed, measured (baseline pH=6) and the pH electrode was recalibrated at pH 4 and pH 7.

# Filler Morphology

For each material, 60 mg of unpolymerized cement paste was placed in a plastic tube (n=1). The unpolymerized cements were dissolved in 6 mL of acetone (99.5%, Merck KGaA, Darmstadt, Germany) and subjected to agitation for 1 minute. After mixing, the obtained suspension was centrifuged for 5 minutes at 14,000 rpm, and the supernatant was discarded. Then, 6 mL of chloroform 99.8%, (Merck KGaA) was used to redisperse the sediment, and the mixing and centrifugation procedures were repeated. The supernatant was discarded, and the remaining sediment of filler particles was redispersed in 6 mL of absolute ethanol (Merck KGaA). Three 20-µL drops of the obtained suspension were placed in a metallic stub and covered in a desiccator for 24 hours.<sup>22</sup> The stubs were sputter-coated with gold in a vacuum evaporator (MED 010, Bal-Tec, Balzer, Liechtenstein) and observed using a scanning electron microscope (SEM) (JSM-5600, JEOL Inc, Peabody, USA) at 1000× magnification.

# **Statistical Analysis**

Data were tested for normality and homoscedasticity (Shapiro-Wilk and Levene test). The POBS was analyzed by three-way analysis of variance (ANOVA, factors: cement, root region, and evaluation time) and

Bonferroni *post hoc* test ( $\alpha$ =0.05). Flexural strength and modulus of elasticity were tested by one-way ANOVA and Tukey *post hoc* test for resin cement comparison. The pH was analyzed by one-way repeated measures ANOVA (factor cement) and Bonferroni *post hoc* test ( $\alpha$ =0.05). Failure mode data were submitted to the Pearson chi-square statistical test. Analyzes were performed by SPSS 21.0 (SPSS Inc., Chicago, IL, USA), with a significance level set at 5%.

### **RESULTS**

# **Push-out Bond Strength Test**

The mean POBS values of all the evaluated cements are presented in Table 2. The ANOVA for POBS found that the resin cement (p<0.001), root region (p<0.001) and evaluation time (p<0.001) significantly influenced the results, as well as the triple interaction between all factors (p=0.025). Comparison between cements showed that the POBS of the CRC-RXU was higher than the SACs, in all radicular regions and evaluation times (p<0.024), except for MAX in cervical and middle thirds at the 24-hour evaluation, and at the cervical region after thermocycling (\$\phi > 0.05\$). Among the SACs, RUN and CAL presented lower POBS than MAX in the cervical and middle thirds at the 24-hour evaluation, and in all root regions after thermocycling (\$\phi<0.048)\$. In general, the POBS at the apical third was lower than at the cervical region (p<0.038), except for CAL and RUN after thermocycling. Thermocycling produced a reduction on the POBS of all the cements and radicular thirds (p<0.048), except for CAL and RXU in the middle and cervical root regions, respectively.

### **Failure Mode**

The frequency of failure mode distribution is represented in Figure 2. The Pearson chi-square statistical test indicated that the association between resin cement and failure mode distribution with statistical differences [ $x^2(42) = 129.87$ ; p<0.001].

There was a lower occurrence (adjusted residual < -1.96) for the type of failure within the following groups: in 24-hour, type I and III (RXU type I [RUN], type II [CAL], and type V [MAX]); and after thermocycling, type II (RXU), type II and V (RUN), type II (CAL), and type V (MAX). On the other hand, there was a greater occurrence (adjusted residual > 1.96) for the failure type within the groups: in 24-hour, type II and V (RXU), type III (CAL), and type II and III (MAX); and after thermal cycling, type I (RXU), type I (RUN), and type I (MAX).

Table 2: Mean (SD) Push-out Bond Strength (MPa) of the Evaluated Resi	n Cements According to Evaluation
Time and Root Region <sup>a</sup>	_

Type of Cement	24 Hours			Thermocycling (5000 cycles following 24 hours storage		
-	Cervical	Middle	Apical	Cervical	Middle	Apical
RXU	89.96 (18.7)	79.67 (7.6)	69.93 (15.1)	84.08 (12.7)	54.55 (15.7)	55.74 (10.1)
	Aa	ABa	Ва	Aa	Ba*	Ba*
RUN	69.13 (14.9)	53.15 (14.2)	52.20 (7.5)	24.55 (9.7)	20.00 (4.6)	16.20 (3.3)
	Ab	Bb	Bb	Ac*	Ac*	Ac*
MAX	78.91 (18.5)	78.74 (25.1)	64.37 (9.1)	53.90 (11.9)	41.14 (11.08)	47.37 (15.6)
	Aa	Aa	Bb	Aa*	Bb*	ABb*
CAL	44.92 (15.5)	32.56 (11.5)	41.80 (13.4)	27.96 (8.9)	29.42 (13.8)	30.05 (8.9)
	Ab	Вс	ABc	Ab*	Ac	Ac*

Abbreviations: CAL, Calibra Universal; MAX, Maxcem Elite; RUN, RelyX Unicem; RXU, RelyX Ultimate.

# Flexural Strength and Modulus of Elasticity

The mean flexural strength (MPa) and modulus of elasticity (GPa) values are presented in Figure 3A and 3B, respectively. MAX cement presented the lowest flexural strength among all cements (p<0.001) and there were no significant differences between CAL, RUN, and RXU. RUN showed the highest modulus of elasticity of all cements (p<0.001), followed by RXU. There were no significant differences between MAX and CAL.

### pH Neutralization

The pH profiles of all tested materials are presented in Figure 4. The one-way repeated measures ANOVA for pH detected significant influence of the material (Table 3). All resin cements presented an initial decrease of the pH that reached the minimal point at the 30-minute evaluation for RXU and MAX. For RUN and CAL, the minimal pH was observed at the 60-minute evaluation. However, for all the cements, the pH started to increase after reaching the minimal point, and at the 48-hour evaluation, the pH was equal or above the initial measurement.

### Filler Morphology

The SEM images show that there are notable differences in the filler particles' size and shape

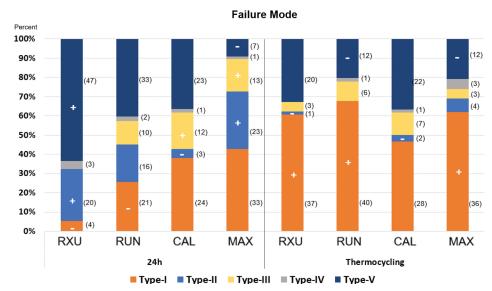


Figure 2. Distribution of failure mode (%) of the resin cements at 24 hours and after thermocycling followed by 24 hours storage. (+) represents higher occurrence of each failure mode in each resin cement; and (-) represents lower occurrence of each failure mode in each resin cement. (#) represents the number of occurrences in percentage. mode: (I) adhesive Failure between the cement and the dentin; (II) adhesive failure between the cement and the post; (III) cohesive failure in the cement; (IV) cohesive failure in the post; and (V) mixed failures consisting of a combination of two or more failure modes. Abbreviations: CAL, Calibra Universal; MAX, Maxcem Elite; RUN, RelyX Unicem; RXU, RelyX Ultimate.

<sup>&</sup>lt;sup>a</sup> Means followed by similar characters indicate no significant difference. Uppercase letters compare radicular thirds within the same cement and evaluation time (rows). Lowercase letters compare cements within the same root region and evaluation time (columns).

<sup>\*</sup>Indicates significant differences between evaluation times for the same root region and cement.

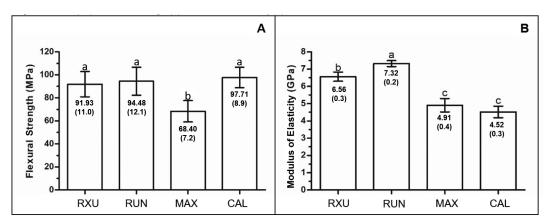


Figure 3. Means (SD) for flexural strength (A) and modulus of elasticity (B) of the resin cements tested. Different letters indicate significant differences according to one-way ANOVA and Tukey post hoc test for resin cement comparison ( $\alpha$ =0.05). Abbreviations: CAL, Calibra Universal; MAX, Maxcem Elite; RUN, RelyX Unicem; RXU, RelyX Ultimate.

between the different cements. For RXU and RUN, the filler particles show similar sizes and shapes (Figure 5A and 5B), although there are evident differences in comparison with MAX and CAL (Figure 5C and 5D). For RXU and RUN, the filler particles are somewhat spherical and show a regular size, while for MAX and CAL, the fillers present irregular shapes and sizes.

### DISCUSSION

The POBS results suggest that using a CRC for post cementation produces a higher bond strength than the evaluated SACs. Considering that, the first null hypothesis, that the POBS of the SACs would not be different from the CRC, was rejected. Despite previous studies indicating that SACs exhibit better mechanical properties for fiberglass post cementation and increase the micromechanical retention and chemical bonding while also exhibiting a greater tolerance to humidity and lower polymerization shrinkage stress compared

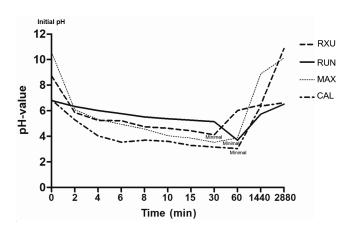


Figure 4. pH profiles of the resin cements over 48-hour period. Abbreviations: CAL, Calibra Universal; MAX, Maxcem Elite; RUN, RelyX Unicem; RXU, RelyX Ultimate.

to CRCs, those claims are not in concordance with the findings of this study.<sup>23,24,25</sup> The SACs are composed of two main components, the conventional monomers (methacrylates) and acid monomers (monomers with carboxylic or phosphoric acid-groups).<sup>8,9</sup> The acidic monomers etch the dental tissues; however, they have lower capacity for demineralization compared to the traditional etch-and-rinse technique, leading to lower hybridization, which compromises the mechanism of adhesion when compared to the CRC approach where the etching and bond are separated.<sup>8,9,26</sup>

Along with the challenges of the self-etching, especially for intraradicular post cementation, the smear layer in the root canal is denser than that of a cavity preparation on coronal dentin, and the viscosity of the cements may compromise the effectiveness of

Table 3: Mean and Standard Deviation of pH Value<sup>a</sup>

Type of Time Measured

Type of	Time Measured					
Cement	Minimal	24 Hours	48 Hours			
RXU	4.11 (0.11)	6.45 (0.20)	10.87 (0.31)			
	C	B	A			
RUN	3.69 (0.30)	5.73 (1.24)	6.52 (0.33)			
	B	AB	A			
MAX	3.53 (0.39)	8.87 (1.74)	10.11 (1.73)			
	B	A	A			
CAL	3.03 (0.04)	6.36 (0.26)	6.61 (0.19)			
	B	A	A			

Abbreviations: CAL, Calibra Universal; MAX, Maxcem Elite; RUN, RelyX Unicem; RXU, RelyX Ultimate.

<sup>&</sup>lt;sup>a</sup>Mean values represented with different letters are significantly different at 5%, according to one-way ANOVA with Bonferroni post hoc test. Uppercase letters compare time evaluation for each resin cement (rows).

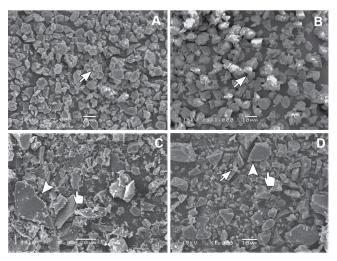


Figure 5. SEM micrographs (1000×) of different resin cements. A, RelyX Ultimate (RXU); B, RelyX Unicem2 (RUN); C, Maxcem (MAX); D, Calibra (CAL). Legend: \$\infty\$, spherical shape fillers with regular size;\$\infty\$, irregular shaped particles with larger size;\$\infty\$, irregular shaped particles with small size

infiltration.<sup>5,6,9</sup> Also, the presence of acidic monomers changes the physicochemical characteristics of the resin cement and can affect the penetrability into the radicular dentin tubules, leading to failure by dislodgement of the post from inadequate adaptation.<sup>7,8,9,27,28</sup> Thus, a lower POBS could also be a reflection of the cement composition and the chemical reaction of each SAC tested.<sup>26,29,30</sup>

Another relevant factor in the analysis of the mechanical properties of resin cements is the degradation of the material. This study showed that all the evaluated cements presented a decrease in the POBS after thermocycling, thus the second hypothesis, that the POBS values would not change, was rejected. This finding may be related to hydrolytic degradation of the resin matrix owing to the effect of contraction and expansion of the composite leading to adhesive failures. Yet, when comparing the resin cements, the SACs showed lower POBS values after thermocycling than the CRCs, thus, it can be noticed that the SACs are generally more susceptible to water sorption and degradation than the CRCs.

Among the evaluated SACs, MAX showed the highest POBS after 24 hours of storage with 78.91 ± 15.8 MPa and after thermocycling with 53.9 ± 11.9 MPa. These results may be related to GPDM (glycero-phosphate dimethacrylate) monomer. The GPDM is a phosphoric acid functional monomer that has two polymerizable groups, hence being more reactive and producing a higher chance to co-polymerize with other monomers, compared to the Pent-P monomer in RUN, which only contains one polymerizable group.<sup>33</sup> Also, GPDM has

small monomers with strong hydrophilicity, inducing better dentin wettability on the hybridization process.<sup>33</sup> However, residual hydrophilic monomers can lead to water sorption and significant hygroscopic expansion stresses during and after the setting reaction, resulting in adhesive failures.<sup>10,19,25,29,30</sup>

Regarding the failure mode, adhesive failures between the cement and the dentin (type I) were the most common for all the cements even after thermocycling. This finding could be a result of an ineffective light-curing reaction. Moazzami and others showed that the apical and middle thirds of the root canals do not receive a minimal irradiance of 233 mW/cm², thus, photopolymerization is not adequate because of the reduced light transmission, leaving residual unreacted monomers that act as plasticizers and jeopardize the resistance of the material. 18,37

Because the resistance of the material and the potential failure of cemented restorations are correlated to flexural strength and modulus of elasticity, these properties can be predictors of the clinical performance of resin cements. In this study, there were no significant differences on the flexural strength of the SACs RUN and CAL (94.48 and 68.40 MPa, respectively), and the CRC-RXU (91.93 MPa), although there were differences between RXU and MAX that showed a significantly lowest flexural strength of 68.40 MPa. Based on these results, the third hypothesis was partially rejected. Literature shows a high variability on the flexural strength of resin cements, reporting higher, similar, and lower values than those obtained in this study.<sup>38,39</sup> However, the high variability can be a result of different polymerization protocols on the specimens, and the evaluation parameters that can influence the performance and mechanical resistance of resin cements. 9,28,35

Considering that the flexural strength and the modulus of elasticity are indicators of the ability of the material to resist deformations, resin cements should present a modulus of elasticity similar to that of dentin and the fiber post, to deform in a similar way and resist the applied loads.<sup>5,9,12,19</sup> MAX and CAL exhibited a low modulus of elasticity (4.91 and 4.52 GPa, respectively), while RUN showed a high modulus of elasticity (7.32 GPa) compared to RXU (6.56 GPa). The high modulus of elasticity of RUN could explain the results of failure mode, where a lower rate of type III fractures (cohesive in cement) occurred after thermocycling, which could be expected from a material with a high modulus of elasticity.

Another characteristic that influences the modulus of elasticity and flexural strength is the filler morphology. 38,39 It was observed that MAX contains mostly large filler

particles and accordingly showed a lower modulus of elasticity and flexural strength than RUN and RXU (Figure 3). The lower modulus of elasticity and flexural strength could result from a nonheterogeneous stress distribution between the particles leading to stress concentration, which produces low flexural strength. The stress concentration occurs on irregularities of the filler, angles, and protuberances; thus, the cracks initiate quickly when the filler particles are large and irregular<sup>22</sup> such as present in MAX (Figure 5).

Therefore, the fillers' shape and morphology could explain the highest modulus of elasticity of RUN, which contains smooth, rounded, and homogeneously sized filler particles that increased the fracture strength and improved the stress distribution. This result was followed by RXU, because both cements are developed by the same manufacturer and contain similar filler particles. Also, RUN and RXU presented a low rate of type III failure (Figure 2). This failure pattern can be a result of adequate incorporation of small particles, which allows a high number of fillers to incorporate into the resin matrix, improving the mechanical properties such as modulus of elasticity.<sup>22</sup> On the other hand, the presence of irregularly shaped and sized particles, as observed with CAL (Figure 5) might be preferable, because it could result in tolerance to deformation, resulting in better resistance to masticatory forces and lower fracture rates. 19,22

It must be considered that in resin cements, filler particles such as glass may be added to regulate the acid-base reaction of the cement.9 Thus, the ideal SAC would present a low initial pH for adequate wetting and conditioning, but, once adhesion is achieved, the pH would increase due to the acid-base polymerization reaction, leaving no residual acid monomers.<sup>8,9,11,20</sup> However, the results of this study confirm previous findings that report a heterogeneous pH-neutralization between cements. 10,11,19,20,30 Therefore, the fourth hypothesis was rejected, because for all the evaluated cements an initial pH decrease was observed, reaching a minimal pH after 30 minutes for MAX, and after 60 minutes for the other cements. After that, the pH increased, reaching a close to neutral (for RUN and CAL) or basic (for RXU and MAX) pH after 48 hours.

However, it must be stressed that *in vitro* evaluation of pH changes during the setting reaction has limitations, mainly because it does not consider the interactions between the cement and the tooth, hence failing to include an important element of the acid-base reaction of the cement.<sup>8</sup> However, it still provides relevant information to explain the POBS results. In this study, the SACs CAL and RUN had the lowest

pH after 48 hours, and coincidentally also produced the lowest POBS values after 24 hours of water storage. This could be associated with the fact that after 24 hours, the pH had not been neutralized, making the cement hydrophilic and, consequently, susceptible to hydrolysis.<sup>27,30</sup>

Therefore, the simplification of clinical procedures by elimination of critical steps may compromise the chemical reactions and consequently the mechanical properties of the material. For SACs, the ease of use must be balanced with an adequate composition, and appropriate clinical indications. When a SAC is the preferred clinical choice, it is recommended to select a cement with a high pH neutralization, because this could mean the cement is less prone to degradation and as a result, it can be expected to exhibit better mechanical properties and stability, which are crucial for the clinical success of fiber post cementation.

# **CONCLUSION**

It can be concluded that despite presenting a simplified technique, the mechanical behavior of SACs is not superior to that of CRCs. The CRC-RXU presented high values of POBS even after the thermocycling. Despite the limitations of this study and the variety of the composition of the resin cements, among all the SACs evaluated, MAX presented the highest POBS in 24 hours and after thermocycling evaluation. Also, it reached the closest neutral pH after 48 hours. Therefore, SACs with low initial pH and strong neutralization reactions are recommended, because these characteristics may lead to better mechanical properties and stability.

# **Conflict of Interest**

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 03 July 2021)

# **REFERENCES**

- Rocha AT, Gonçalves LM, Vasconcelos AJC, Matos Maia Filho E, Nunes Carvalho C, & De Jesus Tavarez RR (2017) Effect of anatomical customization of the fiber post on the bond strength of a self-adhesive resin cement *International Journal of Dentistry* 2017 5010712. DOI: 10.1155/2017/5010712
- Ebrahimi SF, Shadman N, Nasery EB, & Sadeghian F (2014)
   Effect of polymerization mode of two adhesive systems on pushout bond strength of fiber post to different regions of root canal
  dentin *Dental Research Journal* 11(1) 32-38.
- 3. Shafiei F, Yousefipour B, & Mohammadi-Bassir M (2016) Effect

- of carbodiimide on bonding durability of adhesive-cemented fiber posts in root canals *Operative Dentistry* **41(4)** 432-440. DOI: 10.2341/15-099-L
- Skupien JA, Sarkis-Onofre R, Cenci MS, de Moraes RR, & Pereira-Cenci T (2015) A systematic review of factors associated with the retention of glass fiber posts *Brazilian Oral Research* 29(1) S1806-83242015000100401. DOI: 10.1590/1807-3107BOR-2015. vol29.0074
- Shafiei F, Memarpour M, & Sarafraz Z (2016) Effect of dimethyl sulfoxide on bond durability of fiber posts cemented with etchand-rinse adhesives *Journal of Adhesive Prosthodontics* 8(4) 251-258. DOI: 10.4047/jap.2016.8.4.251
- Bueno CE, Pelegrine RA, Silveira CF, Bueno VC, Alves Vde O, Cunha RS, Pereira GD, & Paulillo LA (2016) The impact of endodontic irrigating solutions on the push-out shear bond strength of glass fiber posts luted with resin cements *General Dentistry* 64(1) 26-30.
- Mushashe AM, Amaral RO, Rezende CE, Filho FB, Cunha LF, & Gonzaga CC (2017) Effect of sonic vibrations on bond strength of fiberglass posts bonded to root dentin *Brazilian Dental Journal* 28(1) 30-34. DOI: 10.1590/0103-6440201601107
- Manso AP & Carvalho RM (2017) Dental cements for luting and bonding restorations: Self-adhesive resin cements *Dental Clinics of North America* 61(4) 821-834. DOI: 10.1016/j.cden.2017. 06.006
- Ferracane JL, Stansbury JW, & Burke FJ (2011) Selfadhesive resin cements - chemistry, properties and clinical considerations *Journal of Oral Rehabilitation* 38(4) 295-314. DOI: 10.1111/j.1365-2842.2010.02148.x
- Zorzin J, Petschelt A, Ebert J, & Lohbauer U (2012) pH neutralization and influence on mechanical strength in selfadhesive resin luting agents *Dental Materials* 28(6) 672-679. DOI: 10.1016/j.dental.2012.03.005
- Miotti LL, Follak AC, Montagner AF, Pozzobon RT, da Silveira BL, & Susin AH (2020) Is conventional resin cement adhesive performance to dentin better than self-adhesive? A systematic review and meta-analysis of laboratory studies *Operative Dentistry* 45(5) 484-495. DOI:10.2341/19-153-L
- Farina AP, Chiela H, Carlini-Junior B, Mesquita MF, Miyagaki DC, Randi Ferraz CC, Vidal CM, & Cecchin D (2016) Influence of cement type and relining procedure on push-out bond strength of fiber posts after cyclic loading *Journal of Prosthodontics* 25(1) 54-60. DOI: 10.1111/jopr.12271
- Prado M, Marques JN, Pereira GD, da Silva EM, & Simão RA (2017) Evaluation of different surface treatments on fiber post cemented with a self-adhesive system *Material Science Engineering* C, Materials for Biological Applications 77 257-262. DOI: 10.1016/j. msec.2017.03.141
- Weiser F & Behr M (2015) Self-adhesive resin cements: A clinical review Journal of Prosthodontics 24(2) 100-108. DOI: 10.1111/ jopr.12192
- Lima DM, Linhares TS, Lima SNL, Carvalho EM, Loguerico AD, Bauer J, & Carvalho CN (2019) Effect of sonic application of self-adhesive resin cements on push-out bond strength of glass fiber posts to root dentin *Materials (Basel)* 12(12) 1930. DOI: 10.3390/ma12121930

- 16. Lins RBE, Cordeiro JM, Rangel CP, Antunes TBM, & Martins LRM (2019) The effect of individualization of fiberglass posts using bulk-fill resin-based composites on cementation: An in vitro study Restorative Dentistry & Endodontics 44(4) e37. DOI: 10.5395/rde.2019.44.e37
- Shafiei F, Mohammadparast P, & Jowkar Z (2018) Adhesion performance of a universal adhesive in the root canal: Effect of etch-and-rinse vs. self-etch mode *PLoS One* 13(4) e0195367. DOI: 10.1371/journal.pone.0195367
- Bitter K, Meyer-Lueckel H, Priehn K, Kanjuparambil JP, Neumann K, & Kielbassa AM (2006) Effects of luting agent and thermocycling on bond strengths to root canal dentine *International Endodontics Journal* 39(10) 809-818. DOI: 10.1111/j.1365-2591.2006.01155.x
- Saskalauskaite E, Tam LE, & McComb D (2008) Flexural strength, elastic modulus, and pH profile of self-etch resin luting cements *Journal of Prosthodontics* 17(4) 262-268. DOI: 10.1111/j.1532-849X.2007.00278.x
- Madruga FC, Ogliari FA, Ramos TS, Bueno M, & Moraes RR (2013) Calcium hydroxide, pH-neutralization and formulation of model self-adhesive resin cements *Dental Materials* 29(4) 413-418. DOI: 10.1016/j.dental.2013.01.004
- 21. International Organization for Standardization, Technical Committee. ISO/TC 106/SC 1. Dentistry-polymer-based restorative materials (ISO 4049). 4th ed. Geneva: ISO; 2009.
- Sabbagh J, Ryelandt L, Bachérius L, Biebuyck J-J, Vreven J, Lambrechts P, & Leloup G (2004) Characterization of the inorganic fraction of resin composites *Journal of Oral Rehabilitation* 31(11) 1090-1101. DOI: 10.1111/j.1365-2842.2004.01352.x
- 23. Sarkis-Onofre R, Skupien JA, Cenci MS, Moraes RR, & Pereira-Cenci T (2014) The role of resin cement on bond strength of glass-fiber posts luted into root canals: A systematic review and meta-analysis of *in vitro* studies *Operative Dentistry* **39(1)** 31-44.
- Bitter K, Aschendorff L, Neumann K, Blunck U, & Sterzenbach G (2014) Do chlorhexidine and ethanol improve bond strength and durability of adhesion of fiber posts inside the root canal? Clinical Oral Investigations 18(3) 927-934. DOI: 10.2341/13-070-LIT
- Frassetto A, Navarra CO, Marchesi G, Turco G, Di Lenarda R, Breschi L, Ferracane JL, & Cadenaro M (2012) Kinetics of polymerization and contraction stress development in selfadhesive resin cements *Dental Materials* 28(9)1032-1039. DOI: 10.1016/j.dental.2012.06.003
- Hitz T, Stawarczyk B, Fischer J, Hämmerle CHF, & Sailer I (2012) Are self-adhesive resin cements a valid alternative to conventional resin cements? A laboratory study of the long-term bond strength *Dental Materials* 28(11) 1183-1190. DOI: 10.1016/j. dental.2012.09.006
- Chen C, He F, Burrow MF, Xie H, Zhu Y, & Zhang F (2011) Bond strengths of two self-adhesive resin cements to dentin with different treatments *Journal of Medical Biology Engineering* 31(1) 73-77.
- Machry RV, Fontana PE, Bohrer TC, Valandro LF, & Kaizer OB (2020) Effect of different surface treatments of resin relined fiber posts cemented with self-adhesive resin cement on pushout and microtensile bond strength tests *Operative Dentistry* 45(4) E185-E195. DOI:10 2341/19-108-L

 Roedel L, Bednarzig V, Belli R, Petschelt A, Lohbauer U, & Zorzin J (2017) Self-adhesive resin cements: pH-neutralization, hydrophilicity, and hygroscopic expansion stress *Clinical Oral Investigations* 21(5) 1735-1741. DOI: 10.1007/s00784-016-1947-4

- Vrochari AD, Eliades G, Hellwig E, & Wrbas KT (2010) Water sorption and solubility of four self-etching, self-adhesive resin luting agents *Journal of Adhesive Dentistry* 12(1) 39-43. DOI: 10.3290/j.jad.a17539
- 31. Ghavami-Lahiji M, Firouzmanesh M, Bagheri H, Jafarzadeh Kashi TS, Razazpour F, & Behroozibakhsh M (2018) The effect of thermocycling on the degree of conversion and mechanical properties of a microhybrid dental resin composite Restorative Dentistry & Endodontics 43(2) e26. DOI: 10.5395/rde.2018.43.e26
- Celik C, Cehreli BS, Bagis B, & Arhun N (2014) Microtensile bond strength of composite-to-composite repair with different surface treatments and adhesive systems *Journal of Adhesion Science* and *Technology* 28(13) 1264-1276. DOI:10.1080/01694243.2014.89 6069
- Yoshihara K, Nagaoka N, Hayakawa S, Okihara T, Yoshida Y, & Van Meerbeek B (2018) Chemical interaction of glycero-phosphate dimethacrylate (GPDM) with hydroxyapatite and dentin Dental Materials 34(7) 1072-1081. DOI: 10.1016/j.dental.2018.04.003
- Soto-Montero J, Nima G, Dias CTS, Price RBT, & Giannini M (2021) Influence of beam homogenization on bond strength

- of adhesives to dentin *Dental Materials* **37(2)** e347-e358. DOI: 10.1016/j.dental.2020.10.003
- Wang R, Shi Y, Li T, Pan Y, Cui Y, & Xia W (2017) Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin *Journal of Dentistry* 62 72-80. DOI: 10.1016/j. jdent.2017.05.010
- Moazzami S, Kazemi R, Alami M, Attaran E, Mehhary M, Sarmad M, & Shahrokh H. (2012) Light conduction capability of different light-transmitting FRC posts *Journal of Dental Materials* and Techniques 1(2) 40-46.
- Polydorou O, König A, Hellwig E, & Kümmerer K (2009) Long-term release of monomers from modern dental-composite materials *European Journal of Oral Science* 117(1) 68-75. DOI: 10.1111/j.1600-0722.2008.00594.x
- Yoshida K, Condon JR, Atsuta M, & Ferracane JL (2003) Flexural fatigue strength of CAD/CAM composite material and dual-cured resin luting cements American Journal of Dentistry 16(3) 177-180.
- Lu H, Mehmood A, Chow A, & Powers JM (2005) Influence of polymerization mode on flexural properties of esthetic resin luting agents *Journal of Prosthetic Dentistry* 94(6) 549-554. DOI: 10.1016/j.prosdent.2005.09.016