

## Evaluation of long-term bond strength and selected properties of self-adhesive resin cements

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**Abstract:** This study evaluated the shear bond strength (SBS) of self-adhesive resin cements (SARCs) to dentin and their physical-chemical properties. Five commercial SARCs were evaluated [SmartCem®2 – DENTSPLY (SC2); BisCem® – Bisco (BC); SeT PP® – SDI (SeT); Relyx U100® – 3M ESPE (U100) and YCEM® SA - Yller (YCEM)]. The SARCs were evaluated for SBS to dentin (n = 10) after 24 h, 6 months, and 12 months. The dentin demineralization caused by acidic monomers was observed by SEM, and pH-neutralization of eluate was observed for 24 h. Degree of conversion (DC), rate of polymerization (Rp), flexural strength (FS), and elastic modulus (E) were evaluated. Immediate SBS of SC2, SET, U100, and YCEM were statistically higher than that of BC (p < 0.001). After 12 months, all SARCs showed reduced SBS values and U100 showed values similar to those of SET and YCEM, and higher than those of BC and SC2 (p = 0.001). Demineralization pattern of SARCs was similar. At 24h, all SARCs showed no differences in the pH-value, except BC and U100 (p < 0.001). YCEM showed the highest Rp. U100, YCEM, and SC2 showed statistically higher FS (p<0.001) and E (p < 0.001) when compared with SET and BC. U100 and YCEM showed the best long-term bonding irrespective of the storage period. A significant reduction in SBS was found for all groups after 12 months. SBS was not shown to be correlated with physical-chemical properties, and appeared to be material-dependent. The polymerization profile suggested that an increased time of light activation, longer than that recommended by manufacturers, would be necessary to optimize DC of SARCs.

**Keywords:** Resin Cements;

## Introduction

The clinical success of an indirect restorative procedure partly depends on the luting technique used to create a link between the restoration and the tooth.<sup>1</sup> Self-adhesive resin cements (SARCs) have attained great popularity among clinicians<sup>2</sup> because they simplify adhesive luting procedures by: reducing the number of steps of adhesive systems; reducing technique sensitivity, and making the cementation process simpler and faster.<sup>3,4,5</sup> Furthermore, the use of SARCs reduces the occurrence of postoperative sensitivity, improves the retention of glass-fiber posts,<sup>6</sup> and produces satisfactory bonding to dentin and indirect restorative materials.<sup>3</sup> However,



the mechanical performance of SARC has been shown to be material-dependent and probably correlated to their chemical composition.<sup>7,8,9</sup>

The self-adhesive properties of these SARC are attributed to the presence of acidic methacrylate monomers capable of demineralizing and infiltrating into the dental substrate,<sup>10,11</sup> resulting in micromechanical interlocking. The first SARC introduced was RelyX Unicem® (3M ESPE, Germany) and the bond strength of RelyX Unicem®, has been evaluated in several studies,<sup>5,9,10,12,13,14,15,16,17,18</sup> with results comparable with those of multi-step luting agents available at present.<sup>10,12,18</sup> However, the long-term storage of these SARC to show long-term dentin bond effectiveness<sup>12,14,15,17</sup> and mechanical stability<sup>19</sup> has rarely been evaluated.<sup>12,14,15,17</sup> Mechanical stability is related to high pH-neutralization over time, which in turn renders the polymer more hydrophobic and less prone to hydrolytic degradation.<sup>19,20</sup> Nevertheless, to the best of our knowledge, the relationship between bonding longevity and pH-over-time has not yet been evaluated. The degree of conversion and the mechanical properties of SARC have also been assessed,<sup>19,21,22</sup> but no studies have evaluated the relationship between bonding longevity and their properties. Despite the satisfactory dentin bond strength behavior reported for RelyX Unicem®, bond strength can vary among SARC due to differences in composition, such as acid

monomer concentration, filler particle content and photoinitiator system used. Good long-term bonding capacity is desired for clinical long-term success.<sup>1,11</sup>. Although the characteristics of Relyx Unicem® are well known, detailed information about the chemical reaction and pH-buffering effect of most of the other SARC now available is still lacking.<sup>11</sup>

Thus, the aim of this study was to evaluate the long-term bond strength to dentin, pH-neutralization, kinetics of polymerization, and mechanical properties produced by five commercial SARC. The hypothesis tested was that there would be difference between the bond strengths to dentin and other material properties produced by self-adhesive luting agents.

## Methodology

### Materials used

Table 1 shows the five commercially available self-adhesive resin cements (SARC) tested in this study and data about their composition. All materials were processed according to the respective manufacturer's instructions, and the equal volume of pastes A and B were mixed for 30 s before testing for all analyses.

The light-cure mode was used for all specimen preparation and the light activation procedures were carried out using a light-emitting diode unit (Radii; SDI, Bayswater, Australia) with 1,000 mW/cm<sup>2</sup> irradiance.

**Table 1.** Materials tested in this study (manufacturer information).

Code	Materials	Manufacturer	Composition
SC2	SmartCem2	Dentisply (Konstanz, Germany)	UDMA Resin, EBPADMA Urethane Resin, di-and tri- functional diluents, PENTA, Proprietary photoinitiating system, proprietary self-cure initiating system, 69% filler by wt.
BC	Biscem	Bisco (Schaumburg, USA)	Bis (Hydroxyethyl methacrylate) phosphate, tetraethylene glycol dimethacrylate, dental glass
SET	SeT PP	SDI (Bayswater, Australia)	Fluoro-aluminosilicate glass, urethane dimethacrylate, camphorquinone, acidic monomer
U100	Relyx U100	3M-ESPE (St. Paul, USA)	Glass powder, methacrylated phosphoric acid esters, Triethylene glycol dimethacrylate, Silane treated silica, sodium persulfate cupric acetate
YCEM	YCEM AS	Yller Biomaterials (Pelotas, Brazil)	A: Dimethacrylate , acidic monomer , glass particle , silica nanoparticle B: Dimethacrylate, initiators, glass particle, silica nanoparticle

### Bond strength to dentin

Fifty bovine incisors ( $n = 10$ ) were embedded in acrylic resin, and their buccal surfaces were wet-ground to expose middle depth dentin. The dentin surfaces were wet-polished with 600-grit SiC abrasive papers for 60 s. The bovine incisors were ultrasonically cleaned in distilled water for 10 min. Dentin moisture was controlled with the use of absorbent paper until no water was observed on surface. Polyvinylsiloxane molds (thickness 0.5 mm) with a cylindrical orifice (diameter 1.5 mm) were placed on the dentin surfaces. The orifices were filled with SARCs and the molds were covered with polyester strips and glass slides, and photoactivated for 20 s. The specimens were stored in distilled water at 37 °C for 24 hours, 6 months or 12 months. The shear bond strength test was performed using a mechanical testing machine (EMIC DL 500, Brazil) at a crosshead speed of 0.5 mm/min until failure. A steel wire (diameter 0.2 mm) was looped around the cylinder and aligned with the bonding interface. Shear bond strength (SBS) values were obtained in MPa. Failure modes were classified at 100 X magnification and pre-testing failures were assigned a value of 0 MPa. Representative failed specimens were analyzed by scanning electron microscopy (SEM – JEOL JSM-6610/LB, USA)

### Demineralization pattern on dentin

The dentin surface of bovine incisors ( $n = 1$ ) was prepared as previously described for the SBS test. Dentin moisture was controlled with absorbent paper until no surface water was observed. The dentin surfaces were filled with the SARCs, and after 2 min were thoroughly washed and ultrasonically cleaned in acetone and distilled water for 30 min, and dry-stored at 37 °C for 48 h for the purpose of obtaining the demineralization pattern on unground dentin, caused by the acidic monomers of SARCS. The control groups consisted of dentin surfaces only polished and etched with 37 % phosphoric acid for 15 s. The demineralization pattern promoted on unground dentin was observed by SEM.

### Potential hydrogen (pH) of the eluate

The SARCs were poured into a disc-shaped polyvinylsiloxane mold (thickness 2 mm, diameter 20 mm) and light photoactivated between glass slides for

20 s on each side. The discs ( $n = 3$ ) were individually immersed in 10 mL of distilled water (pH = 6.0) and pH readings of the eluate were taken with a digital pHmeter (An2000; Analition, Ribeirão Preto, Brazil) at time intervals of 1, 5, 15, 30, 60, 120, 240, 360, and 1,400 minutes after immersion.

### Kinetics of polymerization (KP)

The polymerization reaction of the SARCs was evaluated using real-time Fourier transform infrared (RT-FTIR) spectroscopy with an attenuated total reflectance device that was composed of a horizontal diamond crystal. To evaluate the degree of conversion (DC) and rate of polymerization ( $R_p$ ), the SARCs were dispensed on the diamond crystal ( $n = 3$ ,  $\sim 3 \mu\text{L}$ ). For the materials, readouts were taken by IResolution software (SHIMADZU, USA) using a monitoring scan mode, at a rate of 1 scan per second, at Happ-Genzel apodization, and a  $4 \text{ cm}^{-1}$  resolution. The sample scanning was performed simultaneously with the photoactivation for 60 s. Analysis was performed at a controlled room temperature of 23 °C ( $\pm 2$  °C) and 60% ( $\pm 5\%$ ) relative humidity. The DC (%) and  $R_p$  ( $\text{s}^{-1}$ ) were calculated as previously described,<sup>23,24</sup> considering the intensity of carbon-carbon double-bond stretching vibration (peak height) at  $1,635 \text{ cm}^{-1}$ , and as an internal standard, symmetric ring stretching at  $1,710 \text{ cm}^{-1}$  was used from the polymerized and unpolymerized samples. The  $R_p$  was recorded as the data-curve fitting performed by Hill three-parameter non-linear regression.

### Flexural strength (FS) and elastic modulus (E)

Bar-shape specimens ( $n = 10$ ) measuring  $10 \times 2 \times 2 \text{ mm}$  were made in a metal mold, and light polymerized between glass slides in two windows for 20 s on each side. After 24 h storage in distilled water at 37°C, in the dark, the bar-shaped dimensions were measured with a digital caliper; the flexural strength and elastic modulus<sup>24</sup> were obtained in a three-point bending test on a mechanical testing machine (EMIC DL500) using a span of 8 mm and a crosshead speed of 0.5 mm/min.

### Statistical analysis

The property data of each SARC were individually submitted to one-way Analysis of Variance (ANOVA).

When data failed the normality or equal of variance tests, data were analyzed using ANOVA on Ranks. Pairwise multiple comparison procedures were carried out using the Tukey test. A significance level of  $\alpha = 0.05$  was set for all analyses.

## Results

The dentin bond strength results are shown in Table 2. Statistical analysis showed that the factors “SARCs” and the “storage period” were both significant ( $p < 0.001$ ). Irrespective of the material, a decrease in bond strength was observed after 6 and 12 months when compared with 24 h. Whereas, with regard to time, irrespective of the storage period evaluated, materials U100 and YCEM presented similar SBS values, and higher than those of the other SARCs, except for SET that showed values similar to those of YCEM. When the immediate (24 h) bond strength of SARCs was compared, the materials SC2, SET, U100, and YCEM showed similar SBS values; SC2 and SET were similar to those of BC ( $p < 0.001$ ). However, BC showed median values equal to 0 MPa for all storage periods, because of the high number of pre-test failures:

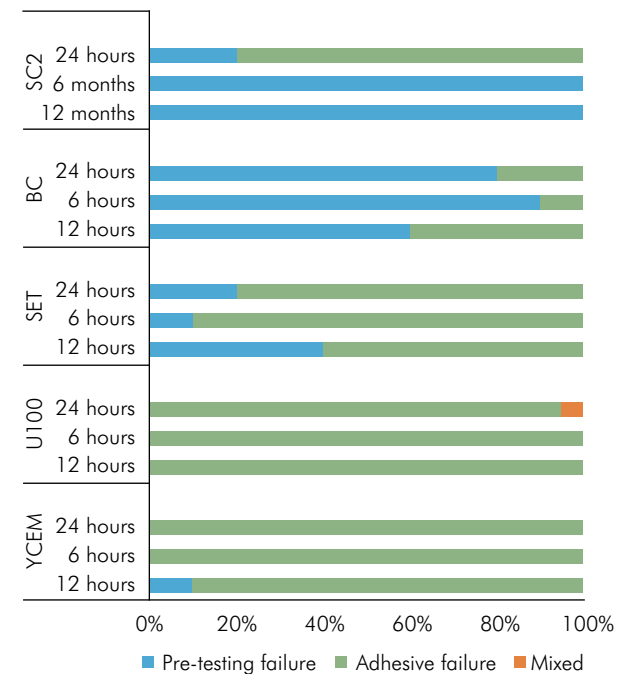
78% of specimens failed before the test. After 6 months of water storage, only material SC2 showed a decrease in the long-term bond strength values ( $p < 0.001$ ), and no bond to dentin (0 MPa). After 12 months of water storage, all SARCs showed reduced bonding effectiveness when compared with 24 h, except BC; however, U100 bond strength values were similar to those of SET and YCEM, and significantly higher than those of BC and SC2 ( $p < 0.001$ ). Predominance of interfacial (adhesive) failures was detected for U100, YCEM, and SET; and pre-testing failures for BC and SC2 in all storage periods (Figure 1). At 6 and 12 months, increasing pre-testing failures were observed for materials BC, SC2, and SET (Figure 1). Although SET had similar bond strength values to those of U100 and YCEM, it showed 40% of pre-testing failures after 12 months. The SEM micrographs of the representative failed specimen in the SBS test at 45x and 500x magnifications are illustrated in Figure 2 A and B, respectively. Insert Figures 1 and 2

The SEM micrographs of the demineralization pattern on dentin are shown in Figure 3 (A–G). The positive control (37% phosphoric acid) showed typical etched dentin with open dentinal tubules (Figure 3 A)

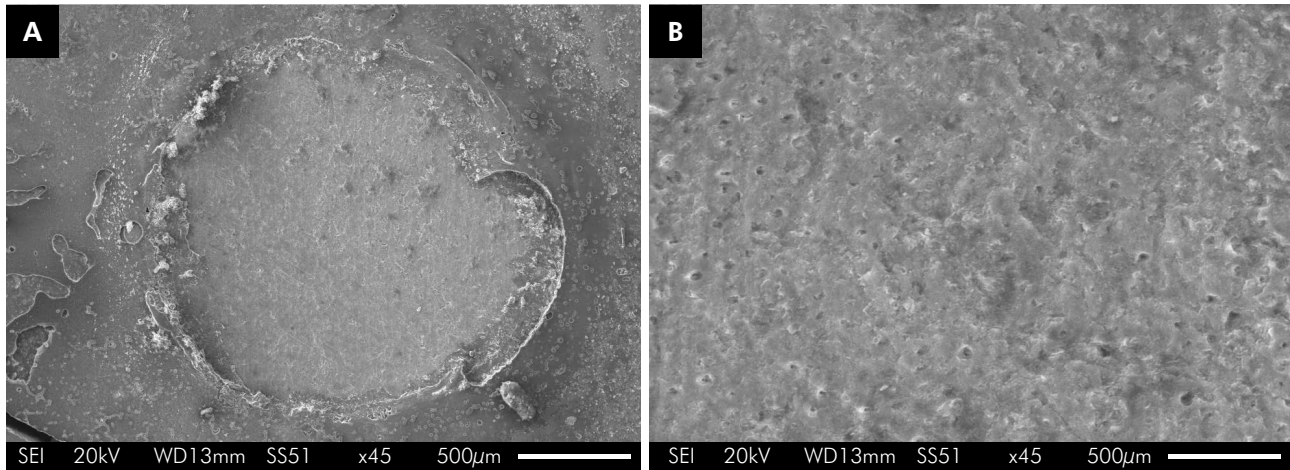
**Table 2.** Shear bond strength (SBS) median values in MPa and the number of pre-testing failures/total number of tested specimens (ptf/n).

SARCs	Time of evaluation (SBS - ptf/n)			Pooled average
	24h	6m	12m	
SC2	<sup>AB</sup> 5.0 <sup>a</sup>	<sup>B</sup> 0.0 <sup>b</sup>	<sup>C</sup> 0.0 <sup>b</sup>	<sup>CD</sup> 0.0
	04/20	10/10	10/10	
BC	<sup>B</sup> 0.0 <sup>a</sup>	<sup>B</sup> 0.0 <sup>a</sup>	<sup>BC</sup> 0.0 <sup>a</sup>	<sup>D</sup> 0.0
	16/20	09/10	06/10	
SET	<sup>AB</sup> 4.8 <sup>a</sup>	<sup>A</sup> 4.1 <sup>ab</sup>	<sup>ABC</sup> 0.8 <sup>b</sup>	<sup>BC</sup> 3.8
	04/20	01/10	04/10	
U100	<sup>A</sup> 8.4 <sup>a</sup>	<sup>A</sup> 10.1 <sup>a</sup>	<sup>A</sup> 5.1 <sup>b</sup>	<sup>A</sup> 7.9
	0/20	0/10	0/10	
YCEM	<sup>A</sup> 8.3 <sup>a</sup>	<sup>A</sup> 6.0 <sup>ab</sup>	<sup>AB</sup> 3.6 <sup>b</sup>	<sup>AB</sup> 5.7
	0/20	0/10	01/10	
Pooled average	5.6 <sup>a</sup>	3.1 <sup>b</sup>	1.1 <sup>b</sup>	

Different superscript capital letters in columns show statistical differences for SARCs ( $p < 0.05$ ); different superscript lowercase letters in rows show statistical differences for storage period ( $p < 0.05$ ).



**Figure 1.** Failure modes



**Figure 2.** SEM micrographs showing the adhesive failure at lower (45 $\times$  – A) and higher (500 $\times$  – B) magnifications

and the negative control (only wet polishing) presented grooves in dentin resulting from the polishing procedure and an unmodified smear layer (Figure 3 B). The SARC<sub>s</sub> showed a similar demineralization pattern (Figure 3 C–G); the smear layer was modified by acidic monomers of the SARC<sub>s</sub> and the dentinal tubules were filled with smear plugs.

Results for the pH analysis are shown in Figure 4. All SARC<sub>s</sub> showed a typical profile of decreased pH-values in the first minutes after immersion, followed by incremental increase in pH during 24 h storage, except for material YCEM that remained constant in the first hours after immersion, and showed subsequent decrease in pH. Significant differences in the pH-value at 24 h were observed among all SARC<sub>s</sub> ( $p < 0.001$ ); U100 presented a higher pH-value than those of the other SARC<sub>s</sub>; and SC2, SET, and YCEM values were similar each other and statistically lower than that of BC group.

Results for the kinetics of polymerization are shown in Figure 5 and Figure 6, and degree of conversion values after light polymerization of 20 s are shown in Table 3. Material YCEM showed degree of conversion similar to that of SC2 ( $p = 0.35$ ) and higher than that of materials U100 and SET ( $p \leq 0.001$ ). Material BC showed values similar to those of all the other SARC<sub>s</sub> ( $p \geq 0.05$ ) at 20 s of photoactivation time (Table 3); however, after 60s of photoactivation time SC2 showed the highest conversion values (Figure 5). Material YCEM showed the highest the

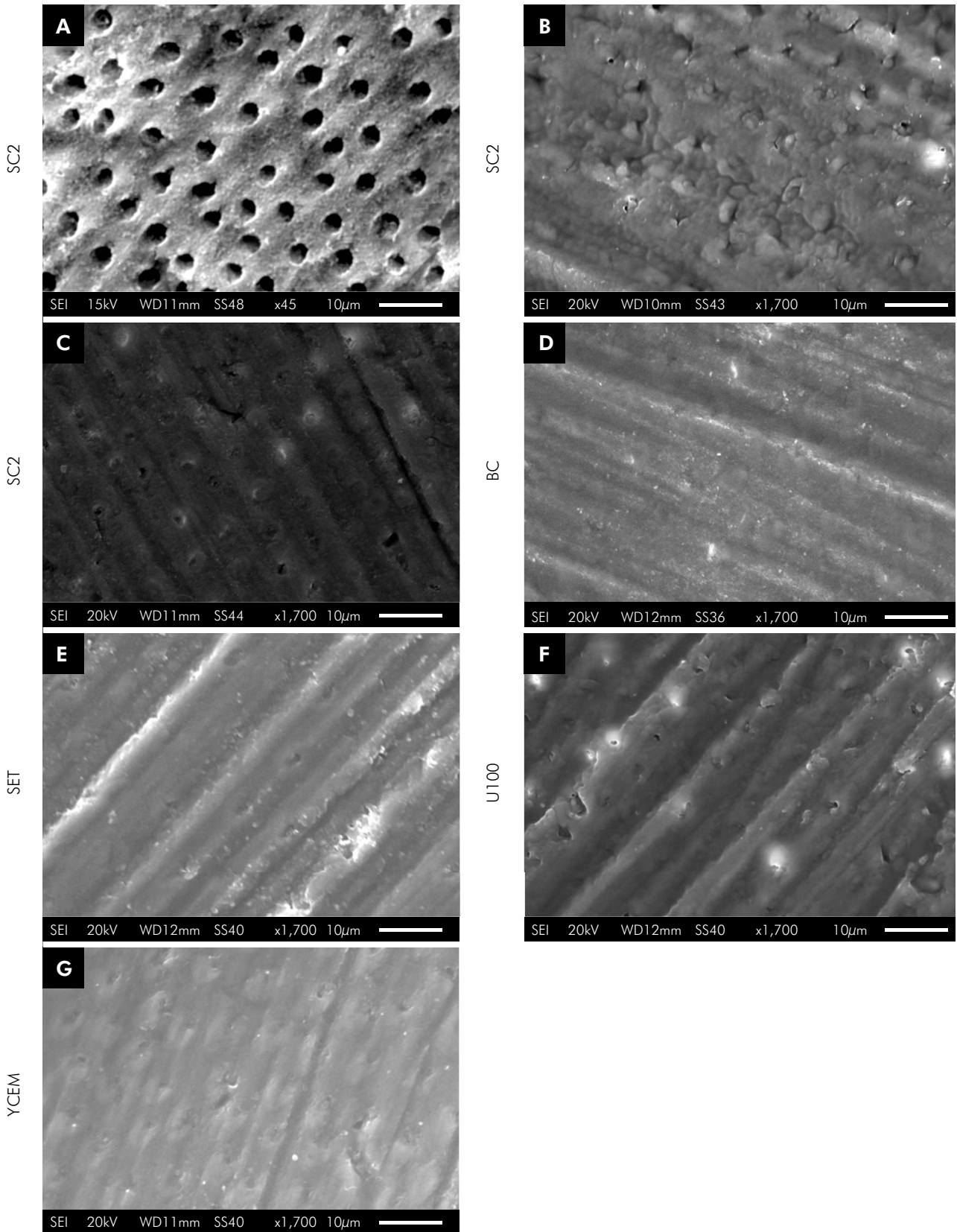
rate of polymerization, while polymerization was slower in the other SARC<sub>s</sub> (Figure 6).

Materials U100, YCEM, and SC2 showed significantly higher flexural strength ( $p < 0.001$ ) and elastic modulus ( $p < 0.001$ ) in comparison with materials SET and BC (Table 3).

## Discussion

In this study, according to the findings, the factors “SARC<sub>s</sub>” and “storage period” were both significant, thus research hypothesis was accepted. The effectiveness and the long term bond strength of SARC to dentin may be influenced by several factors such as substrate, operator technique, and composition of luting agents. In complex formulations, such as those of most adhesive resin cements, there may be a synergy between the different material components, such as the functional monomer acid, initiation system, the inorganic filler used.<sup>25,26</sup> This mixture can affect the mechanical properties of the material and should be considered. As self-adhesive resin cements are less technique sensitive, because they do not require preparation of the substrate, their performance seems to be more material-dependent than technique-dependent.

The relatively low shear bond strength values of all SARC<sub>s</sub> could be related to superficial surface interaction with the tooth structure, without dissolving the smear layer.<sup>27</sup> Moreover, these materials need to

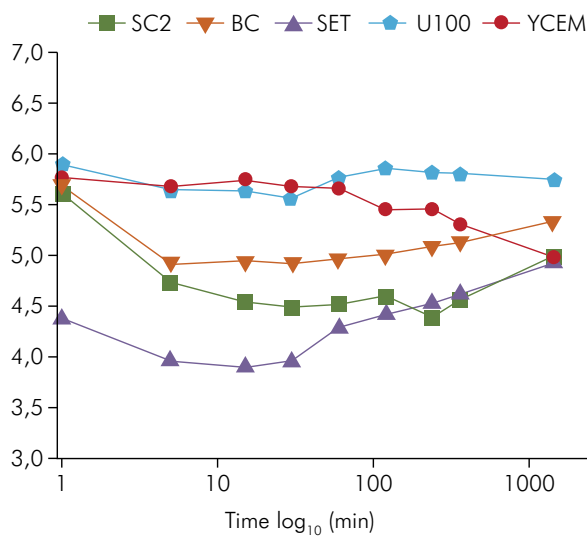


**Figure 3.** SEM micrographs comparing the demineralization pattern on dentin and the morphology created by acid monomers from SARC (C – G), using phosphoric acid 37% as positive control (A) and wet polished specimens as negative control (B)

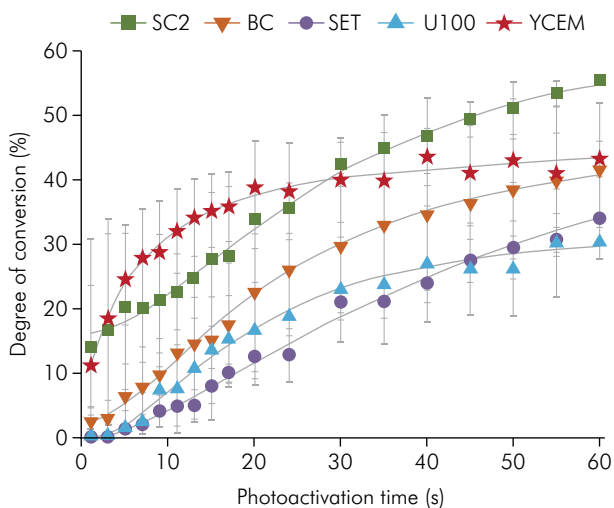
**Table 3.** Means (standard deviations) for the physic-chemicals properties tested.

SARCs	pH*	DC (%)**	FS (MPa)	E (GPa)
SC2	4.99 (0.03) <sup>c</sup>	33.88 (4.65) <sup>A</sup>	76.80 (12.10) <sup>A</sup>	2.30 (0.33) <sup>A</sup>
BC	5.33 (0.05) <sup>B</sup>	23.23 (6.28) <sup>AB</sup>	38.00 (4.40) <sup>C</sup>	1.85 (0.33) <sup>B</sup>
SET	4.93 (0.07) <sup>c</sup>	12.42 (4.26) <sup>B</sup>	56.67 (11.5) <sup>B</sup>	1.25 (0.35) <sup>C</sup>
U100	5.76 (0.01) <sup>A</sup>	16.52 (7.49) <sup>B</sup>	77.40 (11.85) <sup>A</sup>	2.60 (0.44) <sup>A</sup>
YCEM	4.98 (0.15) <sup>c</sup>	38.76 (7.21) <sup>A</sup>	84.17 (16.19) <sup>A</sup>	2.70 (0.63) <sup>A</sup>

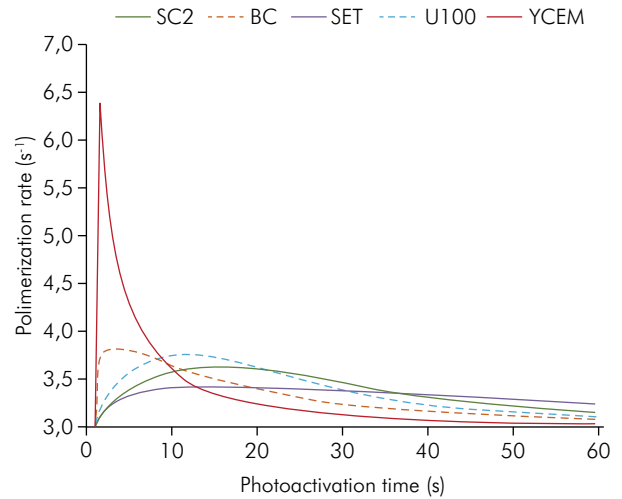
Different superscript capital letters in columns show statistical differences for SARCs ( $p < 0.05$ ). \*pH of the eluate after 24h; \*\*DC after photopolymerization for 20s.



**Figure 4.** pH value of the eluate over a 24-hour storage period



**Figure 5.** Degree of conversion of SARCs during photoactivation time



**Figure 6.** Polymerization rate of SARCs during photoactivation time

be applied with pressure to increase their adaptation to the surface;<sup>28,29</sup> but the shear bond strength testing design with limited contact area, makes this step difficult and promotes high polymerization stress, causing resin shrinkage away from the dentin surface. Furthermore, the pH of self-adhesive resin cements is another factor in their performance and must provide the acidity required for demineralizing the substrate, while avoiding excessive hydrophilicity.<sup>19</sup> The initial drop in pH observed for the self-adhesive resin cements resulted from activating the acid functional monomers that provide greater wettability and promote dental substrate demineralization, thus allowing their infiltration into the demineralized dentin. After the initial drop in pH, the increase during the time observed in this study was in agreement with the results previously described for the commercial SARCs.<sup>19</sup> This phenomenon occurs because these functional monomers are subsequently neutralized by alkaline composites and inorganic components of the tooth structure, such as coming into contact with hydroxyapatite, which neutralizes the reaction of acidic monomers from the hydroxyl groups, making the polymer more hydrophobic and less susceptible to degradation. Although there was an incremental increase in pH during 24 h storage for all SARCs, except for material YCEM, this phenomenon did not seem to be crucial to the longevity of bonding.

SARC Relyx U100 maintained its SBS results after 6 months, probably because it has MDP acid monomer in its formulation, promoting stable adhesion to dentin by both micromechanical interlocking and chemical bonding through the acid groups. Studies have indicated that the interaction between the tooth surface and the adhesive cements may occur through a satisfactory link between  $\text{Ca}^{++}$  hydroxyapatite and acid monomers.<sup>30,31</sup> The acid used in YCEM monomer formulation is unknown. The bonding longevity of this cement was not related to increased pH over time and appeared to be related to quality of the polymer formed and its interaction with the dentin. Indeed, after 12 months all cements showed significant decrease in bond strength.

The predominance of adhesive failures for SARCs tested in all storage times indicated that there was damage to the bond between the cement and the substrate. This type of surface interaction has been reported for most of the SARCs on the market.<sup>10</sup> The bond capacity of SARCs can partly or primarily be attributed to their ability to chemically interact with hydroxyapatite. A relatively high proportion of specimens that failed during storage (pre-testing failure) in distilled water or in preparation for the mechanical tests, as seen for materials BC and SC2, may indicate an inability to achieve efficient micromechanical interlocking and chemical bonding, which was reflected in the decrease in bond strength after 6 months of storage.

The degree of demineralization observed by scanning electron microscopy (SEM) showed that the positive control (phosphoric acid etching at 37%) was able to fully expose the dentinal tubules and remove the smear layer. When positive control was used, it was unnecessary to perform previous dental structure etching, demineralization and interaction with substrates, because the monomers were formed by acid radicals along with their molecular chains. Thus, the acidity of these monomers did not seem to be sufficient to cause the same degree of dentin demineralization as caused by phosphoric acid. Furthermore, phosphoric acid etching opens the tubules; generates post-operative sensitivity; promotes excessive demineralization; and poor infiltration of monomers. However, the pattern of

dentin demineralization for phosphoric acid was not necessary to promote the satisfactory adhesion of SARCs to dentin, as observed in long-term SBS data of U100 and YCEM. In Figure 3, particularly in images F and G, it was possible to observe the tubule entrances when materials U100 and YCEM were used.

The acidic pH of SARCs can interfere with the kinetics of polymerization, and the initial drop in pH may account for incomplete polymerization of methacrylate acids. The acid monomers are less reactive, yielding lower rates of polymerization and conversion compared with those of conventional resin cements. Previous studies have shown that the addition of acid functional monomers to unmodified (di) methacrylates may compromise the rate and extent of copolymerization.<sup>32,33,34</sup> This event can be explained by inhibition of the reaction of conversion of the acid groups into free radicals. Terminated radical acid groups are stable, and therefore, less reactive than free radical derivatives of unmodified monomers, thus reducing the speed of polymerization. Furthermore, the degree of conversion (DC) of SARCs occurs by conversion of the monomers into high molecular weight polymers that form the polymeric network. Moreover, the final degree of conversion depends on the chemical characteristics of the monomers in the formulation. A previous study showed that commercial SARCs needed more light exposure time to achieve better degree of conversion results,<sup>35</sup> which explains the low values obtained with up to 20 s of light polymerization.

YCEM had the highest rate of polymerization; this may be related to the photoinitiators used in the formulation, which act as reaction catalysts by lowering the activation energy necessary to initiate the polymerization process, and regenerate rapidly to react with another molecule, camphorquinone. Therefore, the immediate mechanical performance of material YCEM was related to the quality of the polymer formed with increased crosslink density, leading to an increased resistance of resin cements.<sup>36</sup> This mechanism may explain the longevity results of this resin cement. However, a relationship between polymerization and adhesion could not be observed



for U100. The rates of degree of conversion and kinetics of polymerization profiles suggested the importance of highlighting that light activation time should be longer than forty seconds when cements SC2, BC and SET were used.

The properties of flexural strength (FS) and modulus of elasticity (E) indicated the capacity of the cements to resist high chewing forces and avoid the displacement of indirect restorations. Except for material BC, the other cements reached the minimum FS for dual cementing materials (50 MPa) established by ISO 4049, this could be related to the low bond strength values and the large number of premature failures observed for BC.

Further clinical studies are needed to understand the real performance of these materials for bonding longevity. In addition, manufacturers must review their formulations to provide products with improved properties. The authors rejected the null hypotheses since the values for bond strengths to dentin and other material properties produced by self-adhesive luting agents were not statistically similar.

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## Conclusion

All SARCs tested showed a significant reduction in bond strength after 12 months. BisCem and SmartCem2 presented the worst bond strength performance, with no bonding after 24 h and 6 months respectively. Furthermore, irrespective of the storage period materials U100 and YCEM showed the best long-term dentin bond effectiveness associated with a lower rate of pre-testing failures. The polymerization profile analysis suggested that an increased time of light activation, exceeding that recommended by manufacturers, would be necessary to optimize degree of conversion of SARCs especially for SmartCement2, BisCem and Set PP.

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