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Research Article



Influence of inorganic composition and filler particle morphology on the mechanical properties of self-adhesive resin cements

Marina Rodrigues Santi ,¹ Rodrigo Barros Esteves Lins ,¹
Beatriz Ometto Sahadi ,¹ Giovanna Corrêa Denucci ,² Gabriela Soffner ,¹
Luís Roberto Marcondes Martins ¹

¹Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

²Department of Cariology, Operative Dentistry and Dental Public Health, Indiana University School of Dentistry, Indianapolis, IN, USA



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Santi MR, Lins RBE, Sahadi BO, Denucci GC,
Soffner G, Martins LRM

*Correspondence to

Marina Rodrigues Santi, DDS, MSc

PhD Student, Department of Restorative
Dentistry, Piracicaba Dental School, University
of Campinas, Avenida Limeira 901, Areião,
Piracicaba, SP 13414-903, Brazil.
Email: marina.rsanti@gmail.com

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Conflict of Interest

No potential conflict of interest relevant to this
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Methodology: Santi MR, Sahadi BO, Soffner G;
Project administration: Santi MR, Sahadi BO;
Software: Lins RBE; Supervision: Martins LRM;

ABSTRACT

Objectives: This study aimed to evaluate the influence of inorganic composition and filler particle morphology on the mechanical properties of different self-adhesive resin cements (SARCs).

Materials and Methods: Three SARCs including RelyX Unicem-2 (RUN), Maxcem Elite (MAX), and Calibra Universal (CAL) were tested. Rectangular bar-shaped specimens were prepared for flexural strength (FS) and flexural modulus (FM) and determined by a 3-point bending test. The Knoop microhardness (KHN) and top/bottom microhardness ratio (%KHN) were conducted on the top and bottom faces of disc-shaped samples. Sorption (Wsp) and solubility (Wsl) were evaluated after 24 hours of water immersion. Filler morphology was analyzed by scanning electron microscopy and X-ray energy dispersive spectroscopy (EDS). FS, FM, %KHN, Wsp, Wsl, and EDS results were submitted to 1-way analysis of variance and Tukey's *post-hoc* test, and KHN also to paired *t*-test ($\alpha = 0.05$).

Results: SARC-CAL presented the highest FS value, and SARC-RUN presented the highest FM. SARC-MAX and RUN showed the lowest Wsp and Wsl values. KHN values decreased from top to bottom and the SARCs did not differ statistically. Also, all resin cements presented carbon, aluminum, and silica in their composition. SARC-MAX and RUN showed irregular and splintered particles while CAL presented small and regular size particles.

Conclusions: A higher mechanical strength can be achieved by a reduced spread in grit size and the filler morphology can influence the KHN, as well as photoinitiators in the composition. Wsp and Wsl can be correlated with ions diffusion of inorganic particles.

Keywords: Inorganic fillers; Mechanical properties; Morphology; Resin cements

INTRODUCTION

Self-adhesive resin cements (SARCs) have gained popularity due to simpler applications since this type of material doesn't require any pre-treatment such as etching, priming, and bonding to achieve adhesion [1]. Besides the facilitated application, SARCs are indicated in most

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ORCID iDs

Marina Rodrigues Santi 
<https://orcid.org/0000-0003-3608-2389>
Rodrigo Barros Esteves Lins 
<https://orcid.org/0000-0002-8224-6578>
Beatriz Ometto Sahadi 
<https://orcid.org/0000-0001-5129-5599>
Giovanna Corrêa Denucci 
<https://orcid.org/0000-0002-6970-0377>
Gabriela Soffner 
<https://orcid.org/0000-0003-2343-0137>
Luís Roberto Marcondes Martins 
<https://orcid.org/0000-0001-6376-4540>

procedures that involve unfavorable and difficult clinical circumstances, such as deep dentin, subgingival preparation, fiber glass post cementation, and challenging isolation, owing to the improved mechanical properties and lower solubility (Wsl) [2]. However, due to SARCs composition complexity, Miotti *et al.*, [3] in a recent systematic review and meta-analysis, reported that conventional multistep resin cements have better mechanical properties and adhesive performance. The complex composition of SARCs is based on: functional acidic monomers, conventional methacrylate monomers, activator-initiator systems, and inorganic filler particles, where the mechanism of adhesion is reached through the balance between each component [2]. What differs from the commercially available SARCs are especially the functional acid monomers and the concentration of each component, which requires experimental studies to categorize this material [1-3].

The acid monomers can vary from carboxylic or phosphoric chains and the concentration must be balanced to achieve demineralization of the tooth substrate but at the same time avoid excessive hydrophilicity in the final polymer, because the water in the polymeric network may act as a plasticizer and reduce the longevity of the material [4,5]. However, functional acid monomers may cause delayed initial polymerization, and low pH conditions present in the acidic components of SARCs may impair radicals and salts formation when benzoyl peroxide and tertiary amines are used as initiators in the reaction [4-8]. Thus, sorption (Wsp) and Wsl tests have been widely used to indicate hydrophilic and hydrophobic balance in the material, and the microhardness test has been successfully used as an indicator of relative polymerization extent [1,4,7].

On the other hand, the inorganic fillers are used to neutralize resin acidity during the reaction by delivering ions, so it must be a combination, especially from glass, ytterbium, and colloidal silica [4]. Barium glass has been added for radiopacity, amorphous silica has been introduced for improved handling conditions, and ytterbium has been added for an esthetic effect [5]. Also, it must be considered that the quantity, shape, and size of the filler particles have become more diverse, and it can influence the stress transmitted to the structures during the masticatory forces leading to cracks [6]. Due to this fact, microscopy morphology analyses are necessary to revise the classification of the materials and, flexural strength (FS) and flexural modulus (FM) serve as basic parameters for mechanical behavior evaluations [5].

The literature regarding SARCs, concerns mainly RelyX Unicem (3M Oral Care, St. Paul, MN, USA) since it was the first material of this group in the market [4,9]. This material is composed of Phenyl-P as the acid functional monomer and Sodium p-toluene sulfonate as the initiator system, however, it has been reported that this resin cement has shown to be less effective when bonding to enamel and dentin compared to other widely used conventional resin cement systems [1,8,9]. Owing to the fact that the properties of SARCs are dependent on the conversion of monomers to polymers and the concentration of the acid functional group; besides, the initiator system may interfere with the SARCs performance, as observed in the Maxcem Elite resin cement (Kerr, Orange, CA, USA) and Calibra Universal (Dentsply Sirona, York, PA, USA) that was launched in the market with different monomers and initiator systems [4,10]. However, the acid-based reaction of these resin cements is also controlled by physical factors such as water content and ion Wsl, therefore, some different inorganic particles have been included in the composition depending on the manufacturer [4].

Thus, considering the advances in technology and the different compositions of products available, laboratory researchers are still necessary to characterize and categorize this class

of materials. Thereby, the objective of this *in vitro* study was to evaluate the FS, FM, Knoop microhardness (KHN), Wsp, Wsl, and filler particle character of 3 different SARC. The null hypotheses were: (1) FS and FM would not be different between the SARC; (2) filler morphology would not influence KHN values, and (3) Wsp and Wsl values would not increase after 24 hours of water storage.

MATERIALS AND METHODS

The following 3 SARC were selected for the study: RelyX Unicem 2 (RUN); MaxCem Elite (MAX); and Calibra Universal (CAL). The material, manufacturers, lot number, and composition of the tested composites are presented in **Table 1**.

FS and FM

Rectangular bar-shaped specimens (25 mm in length, 2 mm in thickness, 2 mm in width) were prepared according to ISO4049 [11]. The SARC ($n = 12$) were inserted into a stainless-steel mold and a glass plate was positioned. Then, the specimen was light-cured in the center followed by the ends for 20 seconds each time according to the manufacturer's instruction, using a light-curing unit (Valo; Ultradent Products, Inc., South Jordan, UT, USA) with a radiant emittance of 1,200 mW/cm² and tip size of 9.5 mm (± 0.1 mm). Afterward, a 1,200-grit abrasive paper was used to remove excesses of resin cement and the specimen was kept in water for 24 hours at 36°C. For the 3-point bending test, the samples were fixed in a universal testing machine (Instron, Norwood, MA, USA) and loaded until fracture with a crosshead speed of 1.0 mm/min [1,6,12].

KHN

Disc-shaped samples (10 mm in length and 2 mm in thickness) were prepared for each SARC ($n = 6$). The material was inserted into a silicon mold, covered with a polyester strip, and light-cured (Valo; Ultradent Products, Inc.) according to the manufacturer's instruction. The samples were polished with grit abrasive papers (#600 and #1,200) to obtain a flat surface. In order to evaluate the entire area of the disk-shaped sample, the specimen was divided into 4 quadrants and 5 indentations were made on the top and bottom (according to **Figure 1**) under a load of 50 gram-force (gf) and a dwell time of 10 seconds using digital microhardness

Table 1. Specifications of the materials used in the study.

Material and manufacturer (#lot number)	Composition
RelyX Unicem 2 - 3M Oral Care (St. Paul, MN, USA) (#3579029)	- Functional acid monomers: 1-Benzyl-5-phenylbarbituric acid, 2-Propenoic acid, 2-methacryl-oxethyl phenyl hydrogen phosphate (Phenyl-P). - Conventional methacrylate monomers: Di- and Tri-Methacrylate resins. - Activator-initiator systems: Sodium p-toluenesulfonate. - Inorganic components: Calcium hydroxide; titanium dioxide; Silane-treated silica, glass fiber, sodium persulfate, and Tert-butyl peroxy-3,5,5-trimethylhexanoate (43 vol%).
Maxcem Elite - Kerr (Orange, CA, USA) (#7293230)	- Functional acid monomers: Glycerol phosphate dimethacrylate (GPDM), Glycerol 1,3-dimethacrylate (GDM). - Conventional methacrylate monomers: Urethane Dimethacrylate, Di- and Tri-Methacrylate resins. - Activator-initiator systems: 1,1,3,3-tetramethylbutyl hydroperoxide. - Inorganic components: fluoroaluminosilicate glass, ytterbium fluoride, barium glass filler, fumed silica (46 vol%).
Calibra Universal - Dentsply Sirona (York, PA, USA) (#180108)	- Functional acid monomers: Phosphoric acid modified acrylate resin. - Conventional methacrylate monomers: Urethane Dimethacrylate; Di- and Tri-Methacrylate resins. - Activator-initiator systems: Organic Peroxide Initiator; Camphorquinone (CQ) Photoinitiator; Phosphene Oxide Photoinitiator; Accelerators; Butylated Hydroxy Toluene; UV Stabilizer. - Inorganic components: Barium Boron FluoroAluminoSilicate Glass; Titanium Dioxide; Iron Oxide; Hydrophobic Amorphous Silicon Dioxide Particles of inorganic filler range from 16 nm to 7 μm, average particle size 3.8 μm. (48.7 vol%)

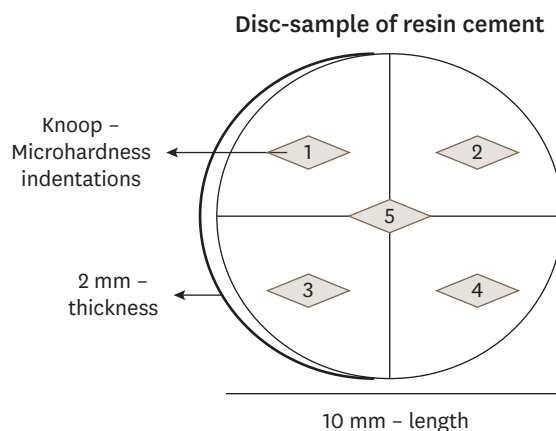


Figure 1. Schematic representation of microhardness indentations.

equipment (FM-ARS 900; Future-Tech Corp., Tokyo, Japan) to obtain mean KHN (kgf/mm²). In addition, the microhardness ratio between top and bottom was calculated (%KHN) [1,13].

Wsp and Wsl

Wsp and Wsl tests were performed according to ISO4049:2000 [11]. A total of 5 disk-shaped samples were prepared for each group ($n = 5$). The SARC was inserted into the circular silicon mold (6 mm in length and 1 mm thickness), covered with a mylar strip and light-cured according to the manufacturer's instructions. Then, specimens were carefully removed from their molds and the excess was removed using 1,000 grit silicon carbide paper [11,14]. Lastly, the samples were protected from the light and stored at 36°C for 24 hours.

Subsequently, to calculate the volume (V), specimens were measured using a digital caliper (Mitutoyo Sul Americana Ltda, Suzano, SP, Brazil), and to obtain a constant mass (m_L), they were weighed daily on an analytical balance (JK-180; Chiyo Balance Corp., Tokyo, Japan) and storage in a silica gel desiccator. Afterwards, the samples were submerged in water for 24 hours, and the mass of each sample was recorded again (m^2). Finally, the specimens were dry stored and reweighed to obtain a constant mass (m^3) [1,5,12]. Water Wsp and Wsl were calculated using the following equations, respectively:

$$Wsp = [m2 - m3]/V$$

$$Wsl = [m1 - m3]/V$$

Filler particle characterization

Approximately 1 g of each unpolymerized SARC was immersed in different organic solvents. The SARC was centrifuged at 1,000 rpm in 99.5% acetone and 99.8% chloroform for 5 minutes and immersed in absolute ethanol for 24 hours followed by air-dried overnight at 36°C [15]. Afterwards, the powder of the resin cements was divided into 2 groups according to the microscopy analysis.

To analyze the filler morphology ($n = 3$), the powder was placed over metallic stubs, sputter-coated with gold (SDC 050 Sputtercoater; Bal-Tec, Balzers, Liechtenstein), and analyzed by scanning electron microscopy (SEM – JEOL, JSM-5600LV, Tokyo, Japan) at 1,200× and 3,000× magnification. The 3,000× images were exported to Image J software (National Institute of Health, Bethesda, MD, USA) to measure the length and width of 20 random particles per image,

totalizing 60 particles ($n = 60$) per group to calculate the mean size of the filler content.

Also, to identify the chemical elements ($n = 3$), the powder was fixed in plastic stubs and sputter-coated with carbon (MED 010; Oerlikon Balzers, Balzers, Liechtenstein). The energy dispersive X-ray analysis (EDX) was performed with a 100-second live time spectrum (voltage: 15 kV; dead time 20%–25%; working distance 20 mm) [16].

Statistical analyses

Data were analyzed for normal distribution and homoscedasticity (Kolmogorov-Smirnov and Levene, respectively). FS, FM, %KHN, Wsp, Wsl and energy dispersive spectroscopy (EDS) were analyzed by one-way analysis of variance (ANOVA) and Tukey's *post-hoc* test, and KHN by paired *t*-test. Statistical analyzes were performed by SPSS 21.0 (SPSS Inc., Chicago, IL, USA), with a significance level of 5%.

RESULTS

FS and FM

Table 2 presents the results of FS and FM of each resin cement evaluated. CAL presented the highest FS values compared to RUN and MAX ($p < 0.004$), which differed from each other ($p = 0.001$). However, RUN presented the highest FM values compared to MAX and CAL ($p < 0.001$), which did not differ from each other ($p > 0.05$).

KHN

The KHN of each resin cement was evaluated on top and bottom surfaces, and the results are presented in **Figure 2A**. For all resin cements, the microhardness values statistically decreased from the top to the bottom ($p < 0.049$). **Figure 2B** represents the top/bottom ratio of all resin cements. No statistical differences were observed ($p > 0.05$).

Wsp and Wsl

Wsp and Wsl values are presented in **Figure 3**. For both methods, CAL presented the highest values compared to RUN and MAX ($p < 0.041$ and $p < 0.047$, respectively). RUN and MAX did not differ from each other for both methods ($p > 0.05$).

Filler particle characterization

Table 3 and **Figure 4** show inorganic elements identified by energy-dispersive X-ray spectroscopy and the morphology of filler particles according to the tested composites. The elemental composition of RUN by EDS shows an elevated amount of aluminum and silica. Zinc, calcium, and lanthanum were also identified. Differently, MAX and CAL presented predominantly silica and barium, however, CAL also showed oxygen and sodium. The elements carbon and silica are present in all SARC tested.

Table 2. Mean and standard deviation values of flexural strength (MPa) and flexural modulus (GPa) of the evaluated resin cements

Product	Flexural strength (MPa)	Flexural modulus (GPa)
RUN	87.58 (5.7) ^b	8.14 (0.8) ^a
MAX	71.20 (4.3) ^c	4.82 (0.5) ^b
CAL	96.47 (6.4) ^a	5.02 (0.7) ^b

Mean values represented with different letters are significantly different at 5%, according to one-way ANOVA with Tukey *post-hoc* test.

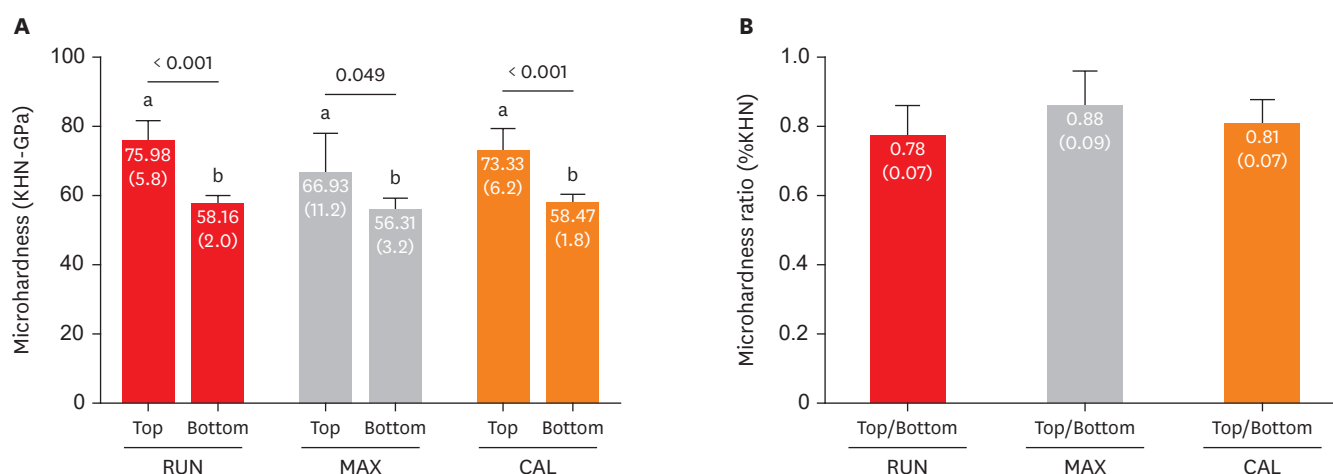


Figure 2. (A) Knoop Microhardness of the evaluated SARC; (B) Knoop microhardness top/bottom ratio. Mean values and standard deviation represented with different letters are significantly different at 5%, according to paired *t*-test. Lowercase letters compare top and bottom for each resin cement.

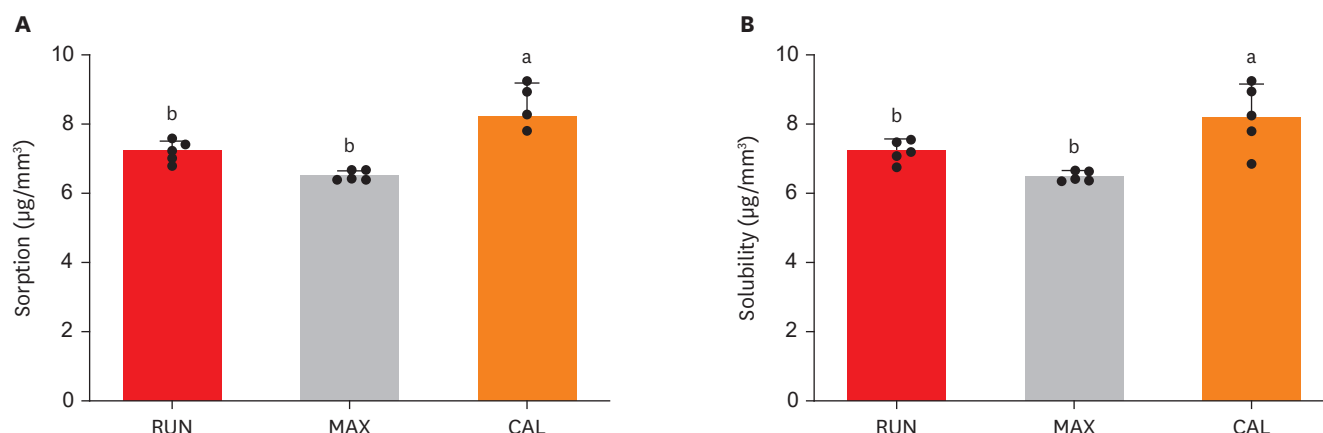


Figure 3. Mean values and standard deviation of sorption (A) and solubility (B) characteristics after 24 hours immersion in distilled water. Lowercase letters compare the resin cements.

Regarding the filler particle size and morphology, the SARC-MAX and RUN presented irregular-sized, predominantly large and splintered, in a range of 9.23 μm and 8.11 μm , respectively. However, SARC-CAL showed a statistical difference, presenting the smallest and regular-sized filler particles among all composites evaluated, ranging around 5.95 μm .

DISCUSSION

The use of SARC has increased because the reduced technique sensitivity might improve clinical success; however, among all the SARC on the market, the mechanical behavior and properties can vary due to the percentages, composition, dimensions, and shape of the fillers, besides the acidic monomers. Thus, FS and FM are necessary tests for characterization of the cements, in addition to being an indicative method of the ability to support masticatory forces, avoiding microleakage and prosthetic dislodgement [6].

According to the findings of this study, FS and FM were different between SARC, therefore, the first null hypothesis was rejected. **Table 2** shows that the SARC-CAL presented the

Table 3. Elements detected and filler content of self-adhesive resin cements

Product	Filler particle size mean	Elements detected
RUN	8.11 μm (1.4) ^a	C; Zn; Al; Si; P; Ca; La
MAX	9.23 μm (1.5) ^a	C; Al; Si; Ba
CAL	5.95 μm (0.9) ^b	C; O; Na; Al; Si; Ba

Mean values represented with different letters are significantly different at 5%, according to one-way ANOVA with Tukey *post-hoc* test.

C, Carbon; Zn, Zinc; Al, Aluminum; Si, Silica; P, Phosphor; Ca, Calcium; La, Lanthanum; Ba, Barium; O, Oxygen; Na, Sodium.

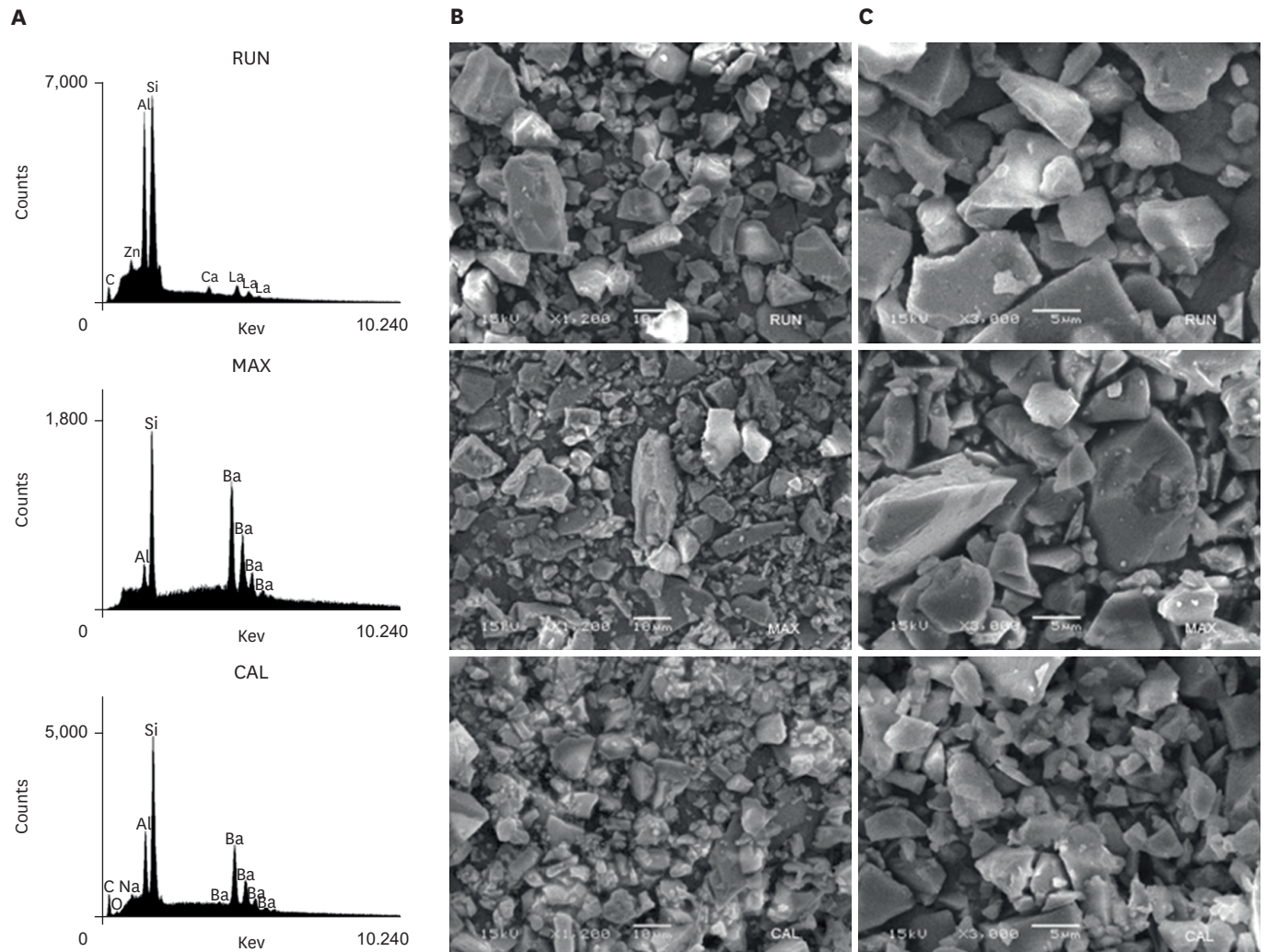


Figure 4. Energy dispersive X-ray analysis charts showing the elemental composition of filler particles (A), and scanning electron micrographs of filler particles at $\times 1,200$ (B) and $\times 3,000$ (C) magnifications.

C, Carbon; Zn, Zinc; La, Lanthanum; Al, Aluminum; Ba, Barium; O, Oxygen; Na, Sodium; Si, Silica.

highest value of FS, and the representative filler morphology image (**Figure 4**) shows that this resin cement presented the smallest and more regular shaped filler particles among all composites evaluated. Hence, these results can be correlated with previous results in the literature that reported that higher mechanical strength can be achieved by similar particles size and a reduced spread in grit size [6,15,17] since the SARC-CAL also presented the highest percentage of inorganic fillers in the composition compared to MAX and RUN (**Table 1**). However, the acceptable values of FS are considered minimum of 50 MPa by ISO4049 as

type 2, class 3 luting materials, and all the evaluated resin cement reached this value. The explanation of these results may also be correlated with the particle size.

Despite MAX and RUN do not present small and regular shaped filler particles such as CAL, **Figure 4** shows that these resin cements have irregular small and large particles, and the combination of the sizes promotes a good adaptation filling all the spaces in the resin matrix, improving the fracture of resistance [18,19]. The literature reports a greater variability for FS and FM of resin cements with studies reporting higher, similar, and lower values than those determined in this study [3,6,12]. The FM observed for RUN (8.14 GPa) was higher when compared to MAX and CAL, being the closest to the modulus of dentin (12 to 20 GPa) as recommended, to provide similar deformation under high masticatory force [6]. This may be attributable to the type of inorganic fillers present or to the degree of polymer conversion [6].

The SARC's evaluated in this study present 43% to 49% of volume, and it is a combination of barium fluoroaluminoborosilicate glass, strontium calcium aluminosilicate glass, quartz, colloidal silica, ytterbium fluoride, and other glass fillers present in **Table 1**, and they have multiple purposes during adhesion mechanism [4,6,18]. These particles can be released locally, and/or it is partially dissolved to neutralize the resin acidity of the functional acidic monomers, because residual acidity may unbalance the chemical reaction, causing deactivation of the amine initiator, reducing the curing rate, and consequently compromising polymerization and mechanical properties such as FS and FM [2,4]. As another option to prevent acidity unbalance, different photoinitiators were included in the composition, like Sodium p-toluenesulfonate present in RUN and tetramethylbutyl hydroperoxide in MAX, thus, it can be assumed that despite the percentage volume of filler particles, the different photoinitiations may also influence FS and FM [18].

Regarding the curing mode, microhardness is a relative indicator of the extent of polymerization, and factors such as the photoinitiators should be considered as well to discuss the findings [8]. In this study, the top surface of the disc specimens was light exposed directly and formed a large number of free radicals thru the absorption by the photoinitiators resulting in higher values of KHN, however, the attenuation and dispersion of the light may be influenced by the material thickness, resulting in lower values at the bottom surface [7,8]. Therefore, it can be assumed that polymer formation is dependent on light reflection and scattering for the reaction process, which is also influenced by the quantity and size of particles present in the composition [7,15].

The quantity and size of particles may influence the extent of polymerization due to the fact that mechanical stress is usually concentrated on protuberances, angles, and irregularities of the filler/matrix interface, thus, the cracks may initiate at these locations, leading to lower values of KHN [16]. Thereby, to avoid this situation, some authors affirm that similar and small particle sizes can improve the bond established between fillers and resin matrix [5,16]. Yet, according to **Table 3**, the resin cement CAL presented the smallest particles, but despite expecting the highest value of KHN, the **Figure 2B** presented no statistical difference between the SARC's evaluated, therefore, the second null hypothesis that filler morphology would not influence KHN values needs to be accepted. These results can be correlated with the combination of fillers size that promoted a great adaptation into the resin matrix [18,19]. However, it is important to mention that despite microhardness results showing no statistical difference, the cavity depth might make it difficult for light to reach the full thickness of the cement layer, thus, mechanical properties may perform differently [8]. Furthermore,

the type of inorganic particles such as silane coupling agent and reactive glass must also be considered to establish the bond between fillers and resin matrix [16,19-21].

According to **Table 3**, all the SARC presented C, Si, and Al, which are the major components to form a glass network, however, the EDS measurements identified other important components to evaluate the basic properties of the materials. Barium (Ba) is present in MAX and CAL, and it is usually added due to the high absorption of short-wavelength X-ray radiation and to develop radiopacity, as well as Lanthanum (La) and Zinc (Zn) for RUN [17,21]. Smaller ions such as Ba have higher diffusion rates and can be eluted faster than La and Zn, however, the water molecules may occupy the available space in the resin matrix, such as microvoids, and consequently, no significant change would exert in the polymer construction [19,20]. Yet the presence of this component on the resin may influence the Wsl and Wsp in a long-time evaluation. Also, the manufactures composition of the evaluated SARCs presented fluoride as an element, and some authors affirm that fluoride is incorporated to release ions for anticaries activity [22], yet the concentration was not sensitive enough to be detected. On the other hand, the SARC-RUN presented Phosphor (P) and Calcium (C), which are elements with a high potential for inducing mineralization, and this may be attributed to the low post-operative sensitivity shown in other studies [23,24].

The evaluation of inorganic fillers is also important to discuss Wsp and Wsl because the excessive hydrophilic character may cause size change (swelling) that compromises the mechanical strength as well as dimensional stability, and it has been shown that the increased filler loading may result in different values [4,5]. In this study, the Wsp and Wsl mean values were different as presented in **Figure 3**, thus, the third null hypothesis that the Wsp and Wsl values would not increase or decrease after 24 hours of water storage was rejected. According to **Table 1**, the SARC-CAL presented a total filler volume of 48.7% while MAX presented 46%, so lower values of Wsp and Wsl would be expected from CAL. However, **Figure 3** showed that MAX presented the lowest value. Thus, despite the filler volume, the results may be correlated with the different concentrations of conventional monomers in the composition.

All SARCs present di- and tri- methacrylate composite according to the manufactures information and monomers that contain hydroxyl (OH), amine (NH₂), amide (NHCO) or carboxylic (COOH) groups are considered hydrophilic and have relatively high polarity and Wsl parameters [5]. As an example, the urethane dimethacrylate (UDMA) is more hydrophilic than the composites with Bis-GMA, Bis-EMA, and TEGDMA [25,26]. Also, it must be considered that each manufacturer uses a different functional acid monomer with a different capacity of hydrophilicity that must be neutralized during the reaction [2,12,25].

Yet, the specification of the monomers, the details of the quantity or percentage of the materials content are not entirely disclosed by the manufacturers, and it is necessary information to fully discuss these findings [5]. Despite the limitations of this study, a lot of different contents present in the composition of SARCs may influence the mechanical properties, thus, this type of resin cements must not be categorized just by the acid monomer or the potential of adhesion, but also by the inorganic fillers and morphology, because each component of SARCs has a specialized function explaining their mechanical behavior.

CONCLUSIONS

Considering the results obtained in this study, higher mechanical strength can be achieved by the reduced spread in grit size. SARC-CAL contained regular-size particles and the highest filler loading, resulting in the highest FS, yet the combination of irregular-size fillers of MAX and RUN promoted intermediated results. However, the protuberances, angles, and irregularities of the particles may influence the microhardness, as well as the photoinitiators present in the composition. Also, the ions diffusion of inorganic particles may alter the Wsl and Wsp values. The SARC-MAX and RUN presented the lowest values compared to CAL, nevertheless, the conventional and acidic monomers must be considered to evaluate these findings.

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