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Evaluation of physical-mechanical properties of self-adhesive versus conventional resin cements

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Aim: The purpose of this study was to compare the microhardness, diametral tensile strength, compressive strength and the rheological properties of self-adhesive versus conventional resin cements. **Methods:** Specimens of a conventional (RelyX ARC) and 3 self-adhesive (RelyX U200, Maxcem Elite, Bifix SE) types of resin cements were prepared. The Knoop test was used to assess the microhardness, using a Microhardness Tester FM 700. For the diametral tensile strength test, a tensile strength was applied at a speed of 0.6 mm/minute. A universal testing machine was used for the analysis of compressive strength and a thermo-controlled oscillating rheometer was used for the Rheology test. One-way ANOVA and Tukey's test ($\alpha=0.05$) were used for data analysis. **Results:** According to microhardness analysis, all the cements were statistically similar ($p>0.05$), except for Maxcem that presented lower hardness compared with the other cements in relation to the top surface ($p<0.05$). In the diametral tensile strength test, Relyx U200 and RelyX ARC cements were statistically similar ($p>0.05$), presented higher value when compared to the Maxcem and Bifix cements ($p<0.05$). The compressive strength of RelyX ARC and Maxcem Elite cements was statistically higher than RelyX U200 and Bifix cements ($p<0.05$). Regarding the rheology test, Maxcem Elite and RelyX ARC cements showed a high modulus of elasticity. **Conclusions:** The self-adhesive cements presented poorer mechanical properties than conventional resin cement. Chemical structure and types of monomers employed interfere directly in the mechanical properties of resin cements.

KEYWORDS: Cementation. Dental materials. Flexural strength. Longevity. Resin cements.

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Introduction

The performance of resin cement on the luting procedure and their mechanical properties are essential requirements for the clinical success of indirect restorations[1]. Moreover, due to its low solubility, high bonding strength, better physical and mechanical properties such as high values of fracture toughness, tensile strength and compression, resin cements are frequently used in the cementation of ceramics[2-4]. Nevertheless, the broad variety of brands and types of resin cements makes the selection of material difficult for the dentist.

Resin cements were developed to be used in the cementation of indirect restorations and intra-radicular pins; thus, they contain different types of monomers that connect to each other during the polymerization reaction[5]. Therefore, the cement composition is generally a mixture of dimethacrylate monomers, such as: BisGMA (bisphenol A diglycidil Dimethacrylate), TEGDMA (triethylene glycol dimethacrylate), UDMA (Urethane Dimethacrylate) and inorganic fillers which vary according to the trademarks and initiator. Silica or high-molecular-weight oligomers can also be added to modify rheological properties and achieve optimum handling characteristics[6]. However, the manufacturers of several materials often do not entirely disclose details of cement composition.

The dual-cure resin cements are available on the dental market and are considered practical to use, because they combine benefits such as working time and the mechanical properties of both light cure and chemical cure resin cements[7]. However, the chemical polymerization associated with photopolymerization provides better monomer conversion[8]. Furthermore, these cements can be classified in two categories according to adhesive cementing technique: conventional and self-adhesive resin cements. The self-adhesive resin cements are considered to be easier to use, once do not require any pretreatment of dental substrate[9]. The bonding mechanism of conventional resin cement depends on the type of adhesive used in combination with this system, whereas the bonding mechanism of self-adhesive resin cements to dental tissues depends on chemical reactions among acid monomer or phosphoric acid ester with calcium of enamel and dentin[10]. In addition, the clinical indications of self-adhesive and conventional resin cements are resembling. However, differences in chemical compositions may lead to dissimilar mechanical properties[11].

These cements must be selected according to the clinical conditions of each case, the physical properties of the indirect restorative material and the physical and biological characteristics of the cementitious materials, such as adhesiveness, solubility, resistance and biocompatibility[12]. Hence, for clinical success with long term follow-up, it is important that the cementitious material exhibits mechanical stability, since the filling is subjected daily to mechanical forces such as mastication, and also to parafunctional habits such as bruxism and tightening[13].

Therefore, in order to assess the ability of the material to withstand these types of stress, mechanical tests such as compressive strength and diametral traction are increasingly applied in research. In addition, to evaluate the surface of the material, degree of conversion, rate of wear and other properties, a microhardness mechanical test is performed. Tests such as rheology are extremely important to understand the behavior of the material. Thus, the purpose of this study was to evaluate the microhardness, diametral tensile strength, compressive strength and the rheological properties of self-adhesive versus conventional resin cements. The null hypothesis tested was: there are no significant differences in physical-mechanical properties between the conventional resin cement and the self-adhesive resin cements.

MATERIALS AND METHODS

In order to carry out the experiment, samples were prepared from specific matrices, which were made with resin cements (Table 1).

Table 1. The materials used in the study and their composition according to their manufacturers

Resin Cements (Lote No)	Manufactures	Type	Resin Matrix
RelyX ARC (N502901)	3M ESPE (3M/ESPE,St Paul, MN, USA)	Dual-cured Conventional cement	Bis-GMA, TEGDMA
RelyX U200 (1518200193)	3M ESPE (3M/ESPE,St Paul, MN, USA)	Dual-cured Self-adhesive	Bis-phenol-A-bis-(2-hydroxy-3-methacryloxypropyl), Ether Bis-GMA, TEGDMA
Maxcem Elite (5925082)	Kerr (Orange,CA, USA)	Dual-cured Self-adhesive	GPDM, HEMA
Bifix SE (1621136)	Voco GmbH, (Cuxhaven. Germany)	Dual-cured Self-adhesive	Bis-GMA,UDMA,Gly-DMA, Phosphatemonomers

Bis-GMA bisphenol A dimethacrylate; TEGDMA triethylen glycol dimethacrylate; GPDM glycerol phosphate dimethacrylate; HEMA hydroxyethyl methacrylate; UDMA urethane dimethacrylate; Gly-DMA glycerol dimethacrylate.

For the microhardness analysis, the samples were made from a circular Teflon matrix with 2 mm of height and 8 mm of diameter. For the preparation of each sample (total of 4 samples), the cement was introduced into the single-increment circular matrix which was pressed between two polyester strips and glass coverslips, so the surfaces would be smooth. Afterwards, photoactivation was carried out with a LED dental curing light device (Elipar Freelight 2, 3M ESPE, USA) with irradiance of 800 W^2 for 20 seconds. Immediately after being made, the samples were removed from the matrix and incubated at 37°C for 24 hours. After this period, the Knoop microhardness test was performed on the top and base surfaces, using an FM 700 microhardness tester (Future Teck Kanagawa, Japan), applying a load of 25 grams for 30 seconds. By activating the penetrator of the equipment, a compression was applied to the sample, which generated an indentation (diamond-shaped geometric print) on its surface. Three indentations were made on each top and base surface of each sample. Then, the Knoop hardness average was obtained for each sample by applying the values found with the indentations with the equation: $KNH = C \times c / d^2$.

Where: KNH is the Knoop hardness value; C (constant) = 14.230; c = 25 grams; d is the length of the longest diagonal of the indentation.

A cylindrical matrix (1 ml insulin syringe, SR Insulin U-100 Luer Slip) with cylindrical specimen (8 mm high and 2 mm diameter) was used to test the mechanical resistance to diametral tensile. The material was handled properly and inserted into the matrix with a plastic spatula. The test was performed 24 hours after the preparation. After the resin cement was inserted into the tube, the photopolymerization was carried out, following the manufacturer's instructions. Each pick was then removed from the syringe with an exploratory probe that pushed it out of the tube. All samples were previously assessed in a 30x magnification optical microscope (OPMI pico®, Carl Zeiss, Oberkochen, Germany) to verify their structural integrity. Ten samples of each resin cement and its respective control and experimental groups were made for each group. The samples were individually attached to the ends of a special traction device and mounted on the Instron universal testing machine, model 4411 (Instron Inc. Canton, MA, USA). The strength was tested by applying tensile forces at a speed of 0.6 mm/min.

The axial compression test used a cylindrical matrix (1 ml insulin syringe, SR Insulin U-100 Luer Slip) whose sample was cylindrical in shape (8 mm height and 2 mm diameter). For each group, 10 samples of each resin cement and its respective control and experimental groups were made. The material was handled properly and inserted in the matrix with a plastic spatula. The test was performed 24 hours after the preparation. Samples were taken to the Instron universal testing machine, model 4411 (Instron Inc. Canton, MA, USA). After obtaining the necessary loads for the rupture of the samples, the compressive strength was calculated: Compressive strength = load/ $\pi.r^2$ (MPa), where: π= 3.14 (constant) and r = cylinder base radius.

The rheology test used a thermo-controlled oscillating rheometer (Thermo Scientific HAAKE RheoStress 6000 Design) driver, version 13. The parallel plate model with a diameter of 35mm was used to measure the rheological properties of the materials. The space between the plates was 1 mm. The material was handled according to the manufacturer's instructions and inserted into the plate. The average initial temperature was 25°C and the final temperature reached 250°C. The test time ranged from 0 to 600 seconds. Therefore, the test was performed at an angular frequency of 100 to 0.01 rad.s^{-1}, determining, due to sinusoidal voltage, the viscosity and the modulus of viscosity. In a dynamic oscillatory shear test with an oscillating frequency (ω) and the phase difference (δ) between stress (σ) and strain (y), the strain and the stress in a complex formula are as follows: Strain $y(t) = y_0\, e^{i(\omega t)}$, stress $\sigma(t) = \sigma_0\, e^{i(\omega t+\delta)}$. The complex shear modulus, G*, is defined as stress over strain $G^* = \frac{\sigma(t)}{\gamma(t)} = \frac{\sigma 0}{\gamma 0} e^{i\delta} = \frac{\sigma 0}{\gamma 0} (\cos \delta + i \sin \delta) = G' + iG''$. Where G' is the real (storage) shear modulus and G" is the imaginary (loss) shear modulus. The magnitude of the complex modulus is given by: $|G^*| = \sigma_0/\gamma_0 = \sqrt{(G')^2 + (G'')^2}$ and the complex viscosity n^* = G*/ = ω. G' is a measure of stored energy without phase difference between the stress and strain, and represents the elastic component of the material. In contrast, G" represents the viscosity of the materials, and it is a measure of the energy lost as heat. The ratio G"/G' is the loss tangent, tan δ, which represents the ratio of the viscous part to the elastic part (energy loss/energy stored) of the materials. The G', G", *n**, and tan δ of the composite specimens were measured, and the relationships between these measured values and the resin matrix formulations of the experimental composites were investigated[14].

After reaching the results, the values were tabulated and submitted to a statistical analysis. First, it was evaluated whether the data presented a normal distribution and homoscedasticity, so that the One-way analysis of variance (ANOVA) and Tukey's range test could be applied. The level of significance was 5% ($p < 0.05$).

RESULTS

Table 2 shows the microhardness values of all the cements analyzed. In this test, all the cements were statistically similar ($p > 0.05$), except for Maxcem that presented lower hardness compared with the other cements in relation to the top surface ($p < 0.05$). For all cements, the microhardness values of the top surface were higher than the basal area.

Table 2. Averages (standard deviation) of microhardness of resin cements

Cement	Microhardness TOP	Microhardness BASE
RelyX U200	41.2 (1.3)^A	35.7 (1.4)^B
RelyX ARC	43.9 (2.1)^A	37.8 (0.8)^B
Bifix SE	42.0 (0.7)^A	35.4 (2.1)^B
Maxcem Elite	36.6 (0.3)^B	34.3 (1.3)^B

Means followed by the same capital letter in the column do not present significant statistical difference ($p > 0.05$).

Table 3 brings the diametral tensile values of all the cements analyzed. The RelyX U200 and RelyX ARC presented the highest values ($p > 0.05$). A significant difference was observed between Bifix and Maxcem in comparison to the other cements analyzed ($p < 0.05$).

Table 3. Averages (standard deviation) of Diametral Tensile Strength of resin cements.

Cement	Diametral Tensile Strength
RelyX U200	155.6 (19.7)^A
RelyX ARC	144.3 (12.4)^A
Bifix SE	97.2 (10.6)^C
Maxcem Elite	116.8 (11.8)^B

Means followed by the same capital letter in the column do not present significant statistical difference. ($p > 0.05$).

Table 4 presents the compression values of all the cements analyzed. The RelyX ARC showed the highest compressive strength without statistical difference to Maxcem ($p>0.05$). The lowest value was obtained for Bifix with significant difference for the other cements analyzed ($p<0.05$).

Table 4. Averages (standard deviation) of Compressive Strength of resin cements

Cement	Compressive Strength
RelyX U200	190.8 (17.3)^{B}
RelyX ARC	261.3 (16.9)^{A}
Bifix SE	176.9 (18.1)^{C}
Maxcem Elite	233.5 (19.1)^{A}

Means followed by the same capital letter in the column do not present significant statistical difference. ($p>0.05$).

According to the rheology test, was verified that the resin cements RelyX ARC and Maxcem Elite presented high Elasticity Modulus (EM). Both presented high EM at the beginning of the test. In addition, the four cements had similar behavioral characteristics over the period of 100 seconds, remaining practically until the end of the rheological test (Figure 1,2).

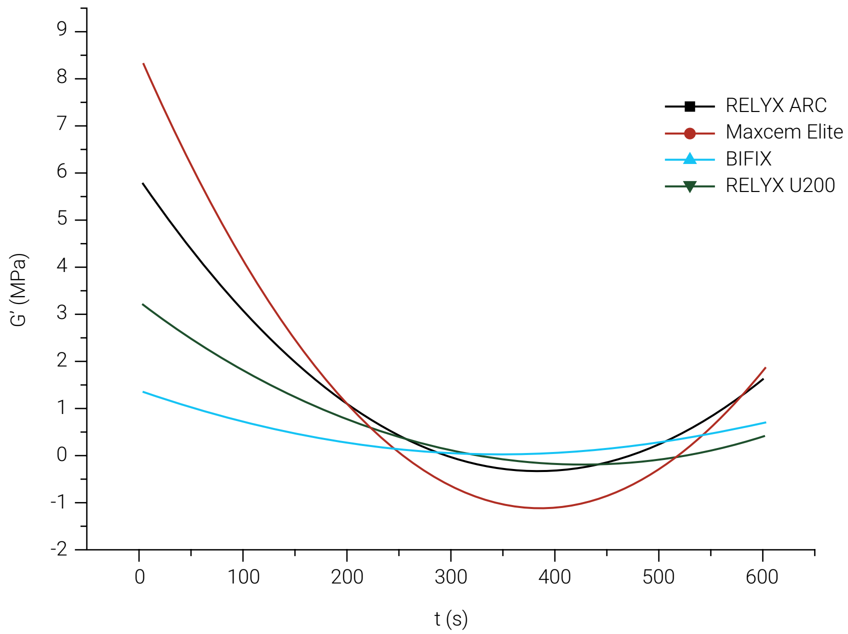
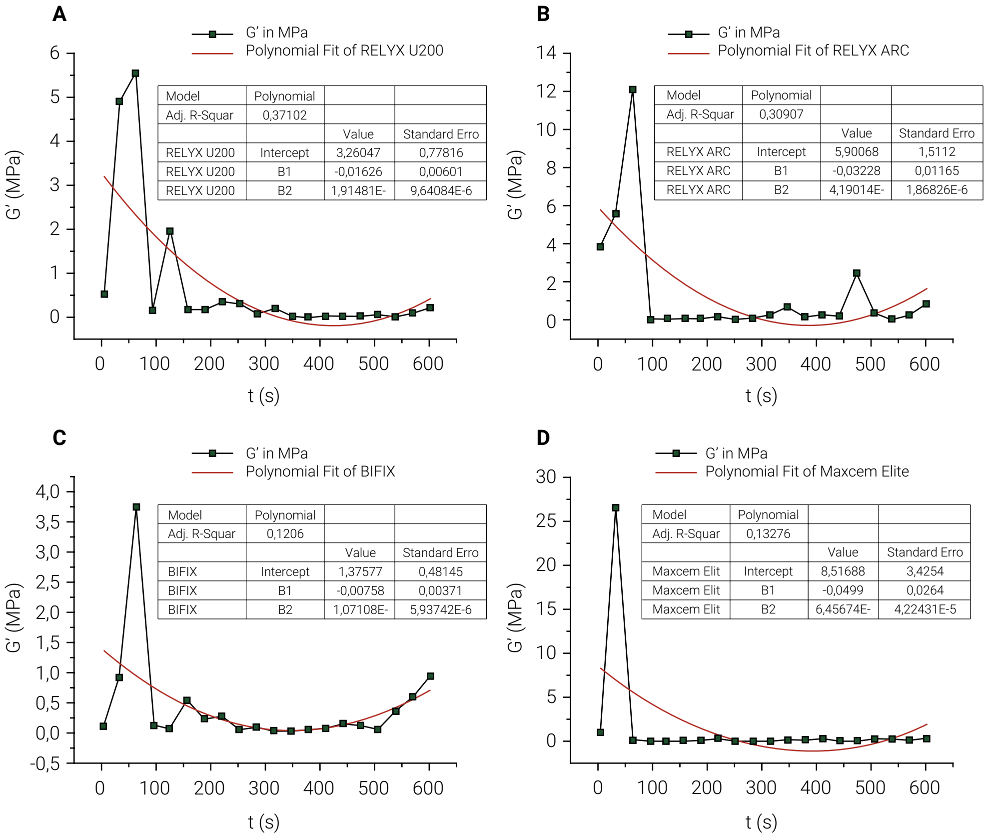


Figure 1. Rheological behavior of the four cements analyzed. EM(MPa) and time in seconds(s)



Model	Polynomial		
Adj. R-Squar	0,37102		
		Value	Standard Erro
RELYX U200	Intercept	3,26047	0,77816
RELYX U200	B1	-0,01626	0,00601
RELYX U200	B2	1,91481E-	9,64084E-6

Model	Polynomial		
Adj. R-Squar	0,30907		
		Value	Standard Erro
RELYX ARC	Intercept	5,90068	1,5112
RELYX ARC	B1	-0,03228	0,01165
RELYX ARC	B2	4,19014E-	1,86826E-6

Model	Polynomial		
Adj. R-Squar	0,1206		
		Value	Standard Erro
BIFIX	Intercept	1,37577	0,48145
BIFIX	B1	-0,00758	0,00371
BIFIX	B2	1,07108E-	5,93742E-6

Model	Polynomial		
Adj. R-Squar	0,13276		
		Value	Standard Erro
Maxcem Elit	Intercept	8,51688	3,4254
Maxcem Elit	B1	-0,0499	0,0264
Maxcem Elit	B2	6,45674E-	4,22431E-5

Figure 2. (A) Rheological behavior of RelyX U200 cement. EM(MPa) and time in seconds(s); (B) Rheological behavior of RelyX ARC cement. EM(MPa) and time in seconds(s); (C) Rheological behavior of Bifix SE cement. EM(MPa) and time in seconds(s); (D) Rheological behavior of MaxCem Elite cement. EM(MPa) and time in seconds(s)

DISCUSSION

The physical and mechanical properties investigated were different among the tested resin cements. The results could be related to the different chemical composition of the materials. Thus, in the present study, the null hypothesis that there are no significant differences in physical-mechanical properties between the conventional resin cement and the self-adhesive resin cements was rejected.

High values of the Knoop microhardness test may be related to high conversion degree values and to other factors such as high crosslink density in the polymer matrix, chemical composition and material translucency[15].Regarding the microhardness test, in the present study, all the cements had a top surface value higher than the base surface value, 24 hours after being made. This superiority from the top to the base was also demonstrated in the study by Arraias et al.[16] (2010).

This superiority of the top over the base is related to the absorption of light during the photopolymerization process. The top receives higher incidence of light and consequently acquires higher hardness values. In addition, some studies point to a reduction of hardness due to the partial absorption of light caused by the thickness and opacity of aesthetic restorative materials, such as porcelain and metal crowns, for example[17,18].

Diametral tensile strength is a simple method of evaluating tensile forces in cements[19,20]. In relation to the present study, the diametral tensile test found that the RelyX U200 resin cement presented higher value in relation to the Maxcem Elite resin cement, coinciding with the study by Kim et al.[20] (2016). In addition, this difference in behavior can be explained by the varied formulation of the materials, related to the quality of the inorganic polymer phases. Therefore, the resin matrix, inorganic fillers and other components, influence the high mechanical and physical properties of resin cements. Filler particles incorporated in their composition improve their properties, such as elastic modulus, compressive and tensile strength[21,22].

The high EM of the cement is related to the transfer of strain from the restoration to the tooth and demonstrates the capacity of strength against elastic deformation[23]. In the study in question, the cement that presented the highest EM under traction was RelyX ARC. This demonstrates its high mechanical resistance when compared to the other cements analyzed. One of the hypotheses to explain the high mechanical property of RelyX ARC may be due to this cement contains spherical shape filler of different sizes, while other cements have mainly irregular-shaped particles in their composition[24]. Moreover, the high EM of the conventional Relyx ARC cement may be related to the bonding strength. Higher values of bond strength of conventional cements may be due pretreatment of the dentin. This process provides the creation of a real hybrid layer, raising a bonding performances to dentin[25].

Compressive strength of cement is an important factor to predict a restorations resistance against masticatory forces[26]. According to Piwowarczy and Lauer[27] (2003), the evaluation of the degree of compression has been used as an instrument for analyzing the behavior of cement. In their study, the degree of compression of the self-adhesive cements varied between 198.3 MPa and 240.6 MPa, a lower value when compared with the conventional resin cements that were evaluated, which ranged from 244.2 MPa to 325.8 MPa. The study in question found a similar behavior in relation to the superiority of the conventional cement RelyX ARC. This superiority of conventional cement relative to self-adhesive can be explained by the amount of monomer diluent, which is different in the two types of material[21].

The composition of the resinous material directly interferes with its viscosity[28]. It is known that rigid monomers such as BisGMA and UDMA, for example, are fundamental in the formation of more homogeneous and mechanically resistant polymers. Furthermore, due to the high hardness, the density of crosslinks of the polymers increases[29]. However, in addition to the monomers, the content, shape, size distribution and treatment of silane, inorganic filler, filler particles and other factors interfere in the rheological and mechanical properties of the composite[28].

The maximum value of the shear storage modulus achieved for each cement was different. Moreover, this method of the rheology test provides information about how a material changes with time. Thus, was observed the highest G’ of the RelyX ARC and Maxcem in relation to the other cements analyzed from the beginning of the test.

The increase of G' means the progression of the cross-linking and entanglements of polymer chains[29]. This is an important factor, because the lower elastic modulus and resilience of cements may compromise the longevity of brittle restorations, such as all-ceramic restorations[30].

The viscosity of resin cements has an influence on the handling properties of the material. This material has time-dependent properties that affect the working time, setting time and the quality of the cementation[31]. Change of viscosity was monitored according to the increasing shear rate and temperature. However, the viscosity of cements varies considerably among the brands though they were nominally of the same class[28]. The results of this study indicate that RelyX ARC cement showed peaks during the rheometric test. In general, this demonstrates the need for a more cautious handling of this cement, possibly related to its monomeric constitution and viscosity.

The monomeric composition of self-adhesive cements differs from conventional ones. They are composed of acid-functional adhesive resinous monomers, which are a type of monomeric methacrylate that has a phosphoric acid or carboxylic acid grouping in their molecular structure[29]. The presence of those functional monomers may be interfere with the amine initiator and compromises the mechanical properties[5]. Therefore, the inferiority of some self-adhesive cements analyzed in relation to conventional cement may be related to several factors, such as a low capacity of acid monomer corrosion, inorganic polymer phases, reducing surface demineralization; incomplete removal of the smear layer, which promotes weak bonding with the resin intermediate layer and mineral buffering effect on the dentin that neutralizes the cement pH[32-34] .

With the limitations of this study and based on the results obtained, it can be concluded that the physical and mechanical properties investigated were different among the tested resin cements. The self-adhesive cements presented poorer mechanical properties than conventional resin cement. The results suggest that the chemical structure and types of monomers employed interfere directly in the mechanical properties of resin cements. Therefore, clinical trials with longer observation periods are required to confirm the data collected from this study.

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