REVISTA DE ODONTOLOGIA DA UNESP

ORIGINAL ARTICLE

Rev Odontol UNESP, Araraquara. nov./dez., 2011; 40(6): 285-289

Comparison of the radiodensity of luting materials

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Feitosa FA, Oliveira M, Rodrigues JA, Cassoni A, Reis AF. Comparação da radiodensidade de materiais cimentantes. Rev Odontol UNESP. 2011; 40(6): 285-289.

Resumo

O objetivo desse estudo foi avaliar a radiodensidade de 7 cimentos resinosos: Panavia F (PA- Kuraray Medical), Enforce (En- Dentsply Latin America), Ecolink (Ek-Ivoclar Vivadent), Rely X ARC (Re-3M ESPE), Rely X Unicem (Un-3M ESPE), Multlink (Mk-Ivoclar Vivadent) e Variolink II (Vk-Ivoclar Vivadent). Os cimentos foram manipulados de acordo com as instruções dos fabricantes e inseridos em uma matriz acrílica de $1 \times 1 \times 10$ mm^3 ($n = 8$) e foto ativados por 40 segundos com luz halógena (750 mW.cm^{-2}, Optilux 501). Após 24 horas de armazenamento no escuro em umidade relativa à 37 °C por 24 horas, os espécimes foram posicionados sobre filmes radiográficos juntamente com uma escala de alumínio. Os filmes foram expostos ao raio X com 60 kV, 10 mA, com a distância foco filme de 10 cm por 0,7 segundos. Os filmes foram revelados e a radiodensidade dos espécimes foi avaliada qualitativamente por ranqueamento utilizando-se os degraus da escala de alumínio, atribuindo do escore 1 para o mais radiolucente ao escore 10 para o mais radiopaco. Os dados foram analisados pelo teste de Kruskal-Wallis e de Dunn ($p < 0,05$). A mediana obtida para cada cimento foi: Pa= 2^a, En= 3^a; Ec= 3^a; Re= 3,5^a, Un= 4^{ab}; Mk= 7^b; Vk = 8^b (medianas seguidas por diferentes letras indicam diferença estatística significante). Pôde-se concluir que os cimentos resinosos apresentam diferentes níveis de radiodensidade. O Panavia F, Enforce, Ecolink, and Rely X ARC foram os mais radiolucentes, o Unicem apresentou radiodensidade intermediária, e o Multilink e o Variolink II foram os cimentos que apresentaram maior radiopacidade.

Palavras-chave: Cimentos dentários; cimentos de resina; radiografia; raio X.

Abstract

The aim of this study was to evaluate the radiodensity of 7 resin cements: Panavia F (PA- Kuraray Medical), Enforce (En-Dentsply Latin America), Ecolink (Ek-Ivoclar Vivadent), Rely X ARC (Re- 3M ESPE), Rely X Unicem (Un-3M ESPE), Multlink (Mk- Ivoclar Vivadent) and Variolink II (Vk- Ivoclar Vivadent). The cements were mixed according to manufacturer's instructions and inserted into $1 \times 1 \times 10$ mm^3 ($n = 8$) acrylic molds and photo-activated for 40 seconds with halogen light (750 mW.cm^{-2}, Optilux 501). After 24 hours of storage in relative humidity at 37 °C in a dark box for 24 hours, the specimens were positioned on the X ray films with an aluminum scale. The film was exposed to 60 kV and 10 mA X ray, with a focus-film distance of 10 cm for 0.7 seconds. The films were revealed and the radiodensity of the specimens was evaluated qualitatively by ranking according to the scale steps, with rank 1 for the most radiolucent to rank 10 for the most radiopaque. The data were submitted to Kruskal-Wallis and Dunn tests ($p < 0.05$). The median obtained for each cement was: Pa= 2^a, En= 3^a; Ec= 3^a; Re= 3.5^a, Un= 4^{ab}; Mk= 7^b; Vk= 8^b (medians followed by different letters differ among them). It can be concluded that resin cements showed different degrees of radiodensity. Panavia F, Enforce, Ecolink, and Rely X ARC were the most radiolucent, Unicem presented intermediary radiodensity, and Multilink and Variolink II were the most radiopaque cements.

Keywords: Dental cements; resin cements; radiography; X ray.

INTRODUCTION

Resin luting agents are used for cementing indirect restorations[1] and are commonly used for the cementation of fiber posts[2]. Radiopacity is one of the prerequisites for resin luting cements especially when they are selected for seating indirect restorations[1] and posts, enabling the detection of marginal defects, contour of restoration, contact with adjacent teeth, cement overhangs, interfacial gaps, and secondary caries[3-5].

Radiographic assessment is frequently used to detect occlusal and proximal primary and recurrent caries. Restorative materials with radiodensity lower than enamel and dentin are difficult to distinguish from dental caries and it can be mistaken interpreted as caries lesions[6]. Many studies have reported that to improve their clinical detection, the minimum radiopacity level of composite resin restorations should be higher than that of dentine or slightly in excess than that of enamel[6].

Most fiber posts present lower radiopacity than dental structures, and the use of highly radiopaque resin cements becomes important in the rehabilitation of endodontically treated teeth with fiber posts ref. This property improves the radiographic evaluation of fiber posts, which should be closely adapted to root canal spaces, with the canal walls being surrounded by a thin and uniform film of resin cement[2].

The radiodensity of resin cements can be influenced by the filler content and its composition. The use of components with high atomic number result in a radiopaque materials. The most used radiopaque elements are barium, ytterbium, yttrium trifluoride, zirconium, lanthanum, aluminum, potassium, and strontium present in the resin cement, and their proportion are determinants of the material radiopacity[7,8]. Also, studies have found a linear correlation between load percentage and the radiodensity of resin materials[9,10]. Since a radiopaque resin cement is desired to improve diagnosis contrast with dental structure and this property depends on filler composition that varies on manufactures formulations, the aim of the present study was to compare the radiodensity of 7 resin cements currently available in the market. The null hypothesis was that there is no difference in the radiodensity of the resin cements.

MATERIALS AND METHODS

This study evaluated seven resin luting cements (n = 8): Rely X Unicem (A2, 3M ESPE, St. Paul, MN, USA), Variolink II (Transparent, Ivoclar Vivadent, Schaan, Liechtenstein), Ecolink (A2, Ivoclar Vivadent, Schaan, Liechtenstein), Enforce (A2, Dentsply Latin America), Rely X ARC (A3, 3M ESPE, St. Paul, MN, USA), Panavia F (Kuraray Medical, Kurashiki, Japan) and Multilink (Ivoclar Vivadent, Schaan, Liechtenstein). The experimental units were 56 specimens, eight samples of each of lutting cement. Composition, batch number and manufacturers of the luting agents are listed in Table 1.

Table 1. Materials, manufacturers, batch number and composition of the lutting cement

Material (Batch #)	Composition	Manufacturers
Panavia F (51136)	Paste A: 10-MDP,silanated silica hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacriyate photo-iinitiator, dibenzoyl peroxide. Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate monomer, BPO	Kuraray Medical, Kurashiki, Japan
Enforce (692278)	Base paste: TEGDMA, Boron glass, Aluminum Silicate and Silanized Barium, Silanized Pyrolytic Silica, CQ, EDAB, BHT, Mineral Pigments, DHEPT. Catalyzing Paste: Titanium Dioxide, Silanized Pyrolytic Silica, Mineral Pigment, Bis-GMA, BHT, EDAB TEGDMA, Benzoyl peroxide.	(Dentsply Latin America, Petropolis, RJ, Brazil)
Ecolink (H16038)	Paste of dimethacrylates, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments Bis-GMA; TEGDMA; urethane dimethacrylate; benzoyl peroxide	(Ivoclar Vivadent, Schaan, Liechtenstein)
Rely X ARC (FMGG)	Bis-GMA, TEGDMA polymer, zirconia/silica filler	(3M ESPE, St. Paul, MN, USA)
Unicem A2 (233749)	Powder: glass powder, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators.	(3M ESPE, St. Paul, MN, USA)
Multlink (FL-9494)	Dimethacrylate and HEMA, Barium glass filler, Ytterbium trifluoride, Silicon dioxide filler initiators, stabilizers, and pigments.	(Ivoclar Vivadent, Schaan, Liechtenstein)
Variolink II Transparent (J26921)	Paste of dimethacrylates, inorganic fillers (silica, barium glass) Ytterbium trifluoride, initiators, stabilizers and pigments	(Ivoclar Vivadent, Schaan, Liechtenstein)

Eight specimens of each of luting cement were prepared in an acrylic split mold with a cross-section of 1 × 1 mm and 10 mm depth. Schematic representation of the procedures used for specimen preparation is presented in Figure 1.

Luting cements were mixed following manufacturers' instructions and applied into the mold with a lentulo drill (Dentsply/Maillefer - Maillefer Instruments AS, Ballaigues, Switzerland). The resin luting cements were irradiated from the top surface for 40 seconds using a visible light-curing unit (Optilux 501, Sybron Kerr, Danbury, CT, USA) with a power output of 750 $mW.cm^{-2}$, except for the chemically cured luting agent (Multilink) that was set to the chemical cure for 10 minutes. After polymerization, the clamp was opened and specimens were removed from the molds and stored in relative humidity at 37 °C in a dark box for 24 hours, prior to radiographic procedures.

The specimens were randomly placed on seven periapical dental films (Ektaspeed, Eastman Kodak Co., Rochester, NY, USA) with a ten-step aluminum step wedge as a control. Radiographic exposure was taken using a dental X ray (Spectro 70X Electronic, Dabi Atlante, Ribeirão Preto, SP, Brazil) set at 60 kV, with a current of 10 mA and a standard exposure time of 0.7 seconds. Focus-film distance was kept constant at 10 cm. The films were processed manually in a tank with the same holder. The lightproof wrappers were removed from the films, the films were placed on the holder and the film holder was immersed in the developing solution for 1 minute, moving the holder up and down several times to break up air bubbles. After that, the film holder was removed, rinsed in water for 20 seconds and immersed in the fixing solution for 10 minutes for unexposed silver crystals to be removed from the films. Upon the completion of fixation, the film holder was immersed in fresh circulating water for 5 minutes and dried in X ray film drier.

Two independent and calibrated examiners (Kappa intra- and inter-examiner higher than 0,83/IBM SPSS 19, SPSS Inc., IBM Company, Armonk, NY, USA) blindly evaluated the radiographs (Figure 1) using a standardized illumination source in a dark room. Scores from 1 to 10, from the most radiolucent to the most radiopaque in the aluminum scale (millimeters of Al/mm specimen), were given to each specimen by comparing then to the Al stepwedge.

The median score of each examiner was used in the non-parametric Kruskal-Wallis test to compare the resin luting cements and the Dunn Test (BioEstat 5.0 - Belem, Brazil) was used to determine the paired differences.

RESULTS

Results for the radiodensity measurements, median scores, amplitude, lower and higher values and statistical analysis are displayed in Table 2. Kruskal-Wallis and Dunn test revealed significant differences among materials ($p < 0.05$). The lowest radiodensity were recorded for Panavia F, Enforce, Ecolink, and Rely X ARC, which did not differ from each other. On the other hand, Multilink and Variolink II were the most radiopaque cements. Rely X Unicem showed an intermediary radiopacity and presented no significant differences from the other resin cements.

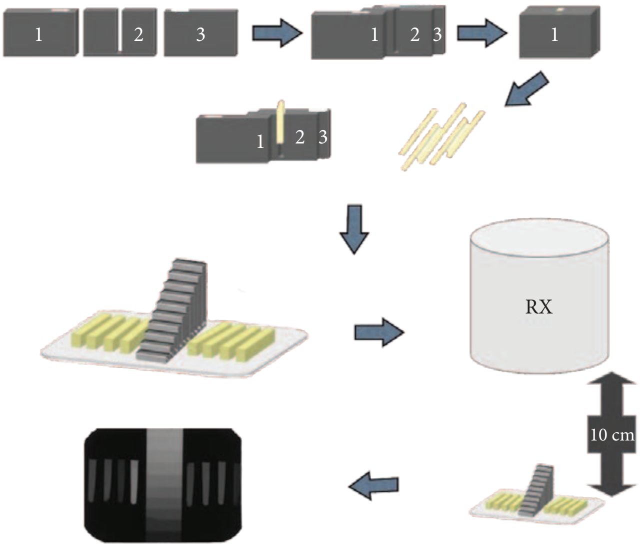

Figure 1. Schematic representation of the procedures used for specimen preparation.

Table 2. Median, amplitude, lower and higher score of radiopacity of resin luting cement (in mm Al/mm specimen)

	Median (Dunn*)	Amplitude	Lower	Higher
Panavia F	2.0^{A}	2	1	3
Enforce	3.0^{A}	2	2	4
Ecolink	3.0^{A}	2	3	5
Rely X ARC	3.5^{A}	7	1	8
Unicem	4.0^{AB}	4	1	5
Multlink	7.0^{B}	2	4	6
Variolink II	8.0^{B}	5	5	10

*Values followed by different letters are significant different from each other.

DISCUSSION

Metallic restorations are easily detected on radiographs because they are highly radiopaque. On the other hand, polymers are radiolucent, and difficult to be detected. Adequate radiopacity of restorative materials is important for a dentist to clearly delineate between the restoration and the tooth structure. It's important not only to detect any extruding material, but also to allow the diagnosis of secondary caries, the detection of voids, and gaps[11].

In the cervical region, resin cement overhangs are easier to detect, because the remaining tooth structure is reduced in this surface, due to the tooth anatomic configuration and materials' radiodensity is greater[12]. However, with the increasing use and indication of fiber posts, which are radiolucent materials, resin cements with adequate radiodensity are important to detect the adaptation and presence of fiber posts within the root canal.

The present study compared the radiodensity of seven resin cements and significant differences were observed among materials. Thus, the null hypothesis was rejected. Panavia F, Enforce, Ecolink and Rely X ARC cements presented the lowest radiodensity values, followed by Rely X Unicem, which presented intermediate values, and Variolink II and Multilink, which presented the highest radiodensity values among the resin cements.

ANSI/ADA Specification Nº 27 and ISO 4049:2000 requires that a resin-based composite material needs to present radiopacity equivalent to 1 mm of aluminum, which is approximately equal to natural tooth dentin[13]. In the test, a 1-mm–thick, disk-shaped sample of the resin-based composite is placed alongside a 1-mm–thick piece of aluminum, and a radiograph is taken. The film is developed, and a photographic densitometer is used to determine the different optical densities of the resin-based composite material and the aluminum. If the optical density of the image of the resin-based composite material is higher than or similar to that of the image of the aluminum, the material complies with the standard.

All resin cements showed radiopacity higher than 1 mm of Al and are in agreement with ANSI/ADA[13] Specification Nº 27 and ISO 4049:2000[14] to resin-based composite[13]. However, the ISO for root materials[14] requires a minimal radiopacity equivalent to 3 mm of Al., and only the Panavia F resin cement showed the median of radiodensity lower than 3 mm of aluminium.

Although the comparison of composite, enamel and dentin radiodensity of previous investigations with the present study is not feasible because of differences in specimens thickness and methods for imaging analyzing, in agreement with our results, Tsuge[1] (2009) described a higher median of radiodensity (millimeters of Al for a 2 mm specimen) for Variolink II (9.9) compared to Panavia F (2.3), and Rely X ARC (4.6), and the high radiopacity values of Variolink II was attributed to the incorporation of Ytterbium trifluoride (YbF_3) into the Base paste. Both Variolink II and Multilink resin cements evaluated in this study present barium glass filler and ytterbium trifluoride in its contents, both of which present high atomic numbers (56 and 70 respectively) and a high filler volume. The median of millimeters of Al for these cements respectively were 8.0 and 7.0. The self-adhesive cement Rely X Unicem did not differ from the other cements, presenting intermediary radiodensity. Although the radiodensity reported for Unicem in the technical profile is 2.43 mm of Al, in the present study it showed a higher value with median of 4.0 mm of Al. This result may be due the presence of strontium (atomic number 38), silica and glass powder.

Rely X ARC has monomers of high molecular weight and silica and zirconium fillers that must be the responsible for the median of 3.5 mm of Al observed. Similarly Fonseca et al.[5] (2006) found that Rely X ARC radiodensity are equivalent to a 4 mm of Al.

Evaluating the effect of aging in the radiopacity of resin based materials, Cruvinel et al.[3] (2007) found no significant changes after 384 hours of artificial accelerated aging and described the radiodensity of Enforce as 1.56 mm of Aluminum. In the present study Enforce showed a medium of 3.0 mm aluminiun due to presence of Barium and Aluminum in the formulation. The material radiodensity may be influenced by several factors such as the specimen thickness, the angulation of the X ray beam, the type of X ray film and the age of developing and fixing solutions. These factors were standardized in the current investigation to present no influence in the resin cements evaluation.

Although there are established ISO[14] and ANSI/ADA[13] protocols for determining radiopacity using film-based radiography, these methods are not always followed by researchers. The use of an aluminium step wedge as a reference, which transforms readings of light transmission in the radiograph into an equivalent thickness of aluminium, was first described by Eliasson, Haasken[15] (1979), and has some advantages, such as low cost and the possibility of the qualification of differences in the radiodensity of restorative materials.

In order to solve the limitation of the lower radiodensity of polymers, if compared to metallic restorations, a considerable amount of high-atomic-number compounds have been incorporated in the formulation of resin-based composite dental materials, such as metal oxides, which are radiopaque elements[1,10]. The most important factor that can influence the radiopacity of dental materials is the atomic number of the elements in their constituent materials and the proportion of these elements in the materials composition[8,10]. The most used metal ions are Zinc (Zn-atomic number 30), Strontium (Sr-38), Yttrium (Y-atomic number 39), Zirconium (Zr-atomic number 40), Barium (Ba-atomic number 56), Lanthanium (La-atomic number 57), Ytterbium (Yb-atomic number 70) and Bismuth (Bi-83)[3,9,16].

On the other hand, the incorporation of high amounts of metal oxides in resin cements may be disadvantageous, if the resin-based composite material needs to present low viscosity, like in resin cements. Despite the raise of the cement viscosity by the increase in filler content, the most regular radiopaque composite filler formulations include metal glasses for radiopacity and high SiO_2 content is required for coupling. Then this elements increase solubility, and barium or strontium ions can disrupt the aluminum-silicate network leading to a more intense degradation of dental composites in the oral environment[17,18]. The combination of metallic elements may affect the composite refractive index, as a result, the shade of the material will change from transparent to a whitish or metallic color and the esthetic result may be compromised[17,18].

Since differences in the composition, mainly in the filler content may influence not only the radiopacity, but it can also influence other properties of the resin cements, there is a great variation among commercial brands in order to balance this properties.

According to Tsuge[1] (2009) enamel and dentin radiodensity corresponds to 4.3 and 2.3 mm of aluminum/2 mm specimen respectively. Fonseca et al.[5] (2006) found that enamel and dentin radiodensity corresponds to 3 and 2 mm of aluminum/1 mm specimen respectively and these values are close to the radiodensity of some resin cements evaluated in the present investigation and may difficult clinical follow up evaluations. It is impossible for the clinician to control restorative materials' radiodensity, then, the use of greatly radiopaque resin cements seems advisable for successful clinical evaluation of indirect restorations during clinical follow-up appointments[12].

Thus, all cements presented radiodensity values higher than 1-mm–thick of aluminum and are in accordance to the ANSI/ADA Specification Nº 27, and it can be concluded that all materials demonstrated adequate radiodensity for clinical use.

CONCLUSION

All materials demonstrated adequate radiodensity for clinical use. Panavia F, Enforce, Ecolink, and Rely X were the most radiolucent, Unicem presented intermediary radiodensity values, and Multilink and Variolink were the most radiopaque cements.

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Recebido: 01/12/2011
Aceito: 21/12/2011