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Diametral tensile strength and film thickness of an experimental dental luting agent derived from castor oil

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ABSTRACT

The need to develop new dental luting agents in order to improve the success of treatments has greatly motivated research. Objective: The aim of this study was to evaluate the diametral tensile strength (DTS) and film thickness (FT) of an experimental dental luting agent derived from castor oil (COP) with or without addition of different quantities of filler (calcium carbonate - $CaCO_3$). Material and Methods: Eighty specimens were manufactured (DTS N=40; FT N=40) and divided into 4 groups: Pure COP; COP 10%; COP 50% and zinc phosphate (control). The cements were mixed according to the manufacturers' recommendations and submitted to the tests. The DTS test was performed in the MTS 810 testing machine (10 KN, 0.5 mm/min). For FT test, the cements were sandwiched between two glass plates (2 cm^2) and a load of 15 kg was applied vertically on the top of the specimen for 10 min. The data were analyzed by means of one-way ANOVA and Tukey's test (α=0.05). Results: The values of DTS (MPa) were: Pure COP- 10.94±1.30; COP 10%- 30.06±0.64; COP 50%- 29.87±0.27; zinc phosphate- 4.88±0.96. The values of FT (μm) were: Pure COP- 31.09±3.16; COP 10%- 17.05±4.83; COP 50%- 13.03±4.83; Zinc Phosphate- 20.00±0.12. One-way ANOVA showed statistically significant differences among the groups (DTS - p=1.01E-40; FT - p=2.4E-10). Conclusion: The experimental dental luting agent with 50% of filler showed the best diametral tensile strength and film thickness.

Key words: Dental prosthesis. Biocompatible materials. *Ricinus communis*. Calcium carbonate. Zinc phosphate cement. Tensile strength.

INTRODUCTION

In the past, traditional dental luting agents such as zinc phosphate were commonly used for cementation of crowns[21]. In spite of disadvantages, such as solubility, lack of retention and low pH[7], this luting agent has been successfully used[26] and was the most researched cement for over a century. Nowadays, some new adhesive luting agents have been tested in order to reduce microleakage, increase retention, and improve physical properties.

Currently, vegetable polyurethanes, which combine the versatility of polymer formulation with the global concern in producing new biomaterials through resources that preserve the environment, became one of the main studied categories of

materials[2]. In dentistry, membrane material[20], sealers[17,23,24] and irrigating solution[18] were developed from vegetable polyurethane obtained from castor oil. Additionally, since this polyurethane has been described as being biocompatible, osteoconductor, osteoinductor, antimicrobial, osseointegrable and absorbable[8,16], it has been used in bone prosthesis, alveolar healing and plastic surgery[16]. In a study conducted by Camargo, et al.[3] (2010), the castor oil bean showed less inflammatory response in subcutaneous tissue of rats when compared with calcium hydroxide cement.

This polyurethane has elasticity, good compatibility, versatility, composition and structure that can be modified in accordance with specific requirements[27], characteristics that enable great applicability in the biomedical area and open a new field to the development of dental luting agents. In addition to the innumerable uses of the castor oil, the plant from which it is extracted can be found in many parts of the world. It is greatly exploited in Brazil and India[9], and can be produced on a large scale, which makes it interesting from an economic and ecological point of view.

In the oral environment, dental luting agents must withstand masticatory and parafunctional stresses in different clinical situation[6,13]. They should maintain their integrity while transferring stresses from crowns or fixed partial dentures to tooth structure. Once the retention of crowns is intimately related to mechanical properties of the dental luting agents, the strength of these materials associated to the ability of resisting crack propagation[4], which causes ruptures, can predict the clinical success. Resistant dental luting agents provide better stress distribution, less probability of compressive or tensile failures and great probability of clinical success[25].

Diametral tensile strength (DTS) is a mechanical property that must be assessed[4] because several cements are extremely friable and have a susceptibility to mechanical failure[28]. This test is widely used due to its relative simplicity and reproducible results. Additionally, it is the most common method for assessing the tensile strength of friable materials because it avoids the difficulties inherent to the flexural tensile strength test[1].

In addition to the mechanical properties, physical properties, such as film thickness (FT) of luting agents, can directly affect long-term clinical success. Dental cements should not exhibit a very high FT. Current ISO standards require a FT at the time of seating inferior to 25 μm for water-based luting cements[12], and no greater than 50 μm for resin-based cements[11]. Low FT can improve seating and decrease marginal gaps; whereas improved marginal adaptation can also reduce plaque accumulation, periodontal disease and cement dissolution[29]. So, the purpose of this study was to compare the DTS and FT of an experimental polyurethane dental luting agent derived from castor oil with traditional zinc phosphate cement.

MATERIAL AND METHODS

The materials with their nomenclature, manufacturers and composition are listed in Figure 1. All materials were proportioned and mixed according to the manufacturers' instructions. Forty specimens were manufactured for DTS test and 40 specimens for FT test.

The castor oil polyurethane (COP) was supplied

Groups	Manufacturer	Composition
Pure COP*	Poliquil Araraquara- Polímeros Químicos Ltda, Araraquara, São Paulo, Brazil	Polyol: tri-functional polyester (castor oil) 370 mgKOH/g Prepolymer: MDI**
COP* 10%	Poliquil Araraquara- Polímeros Químicos Ltda, Araraquara, São Paulo, Brazil	Polyol: tri-functional polyester (castor oil) 370 mgKOH/g Prepolymer: MDI** Powder (filler): $CaCO_3$ 10% w/w
COP* 50%	Poliquil Araraquara- Polímeros Químicos Ltda, Araraquara, São Paulo, Brazil	Polyol: tri-functional polyester (castor oil) 370 mgKOH/g Prepolymer: MDI** Powder (filler): $CaCO_3$ 10% w/w
Zinc Phosphate	SS White, Rio de Janeiro, Rio de Janeiro, Brazil	Powder: Zinc oxide + Magnesium oxide Liquid: Phosphoric acid + aluminum hydroxide + Zinc oxide + Distilled water

Figure 1- Experimental groups, manufacturers and compositions of materials used in this study

* COP= Castor oil poliurethane; ** MDI= methylene diphenyl disocyanato

in sachet. Pure COP contained the prepolymer and polyol separately, while COP 10% and COP 50% included another separation containing 10% or 50% in weight of filler (calcium carbonate - $CaCO_3$), respectively. The quantity of filler, in % weight, was determined by the manufacturer in relation to the sum of the polyol and the prepolymer weights. The mixing method of COP groups includes 2 min of manual mixing into the sachet and other 2 min of mixing with a spatula on a Teflon plate to obtain the final material (Figure 2).

Zinc phosphate was mixed within 90 s with a spatula following the incremental technique. The proportion of 1.4 g/0.4 mL recommended by the manufacturer was converted to 1.4 g of powder/1.54 g of liquid.

DTS Test: it was conducted with 40 cylindrical specimens (6.0 mm in diameter x 3.0 mm in height) divided into the 4 experimental groups (n=10). Immediately after the mixing, the materials were inserted in a Teflon mold, which was put in a mechanical press under constant load in an atmosphere of 100% relative humidity at 37°C for 1 h[1]. Subsequently, all specimens were finished and polished with 400-grit abrasive SiC papers (BuehlerMet Abrasive Papers, Buehler, Lake Bluff, IL, USA) and were stored in distilled water at 37°C for 24 h before the mechanical test. The specimens were subjected to a compressive load (10 KN) in a universal testing machine (MTS-810 Material Test System, Eden Praire, MN, USA) at a crosshead speed of 0.5 mm/min until fracture. The results were recorded and transformed in tensile values (MPa) by the computer software (Test Star II, International Business Machines Corporation – IBM, Armonk, NY, USA), connected to the system. The results were submitted to one-way ANOVA and Tukey's test (α=0.05) in order to compare the values of DTS among the 4 experimental groups.

FT test: it was conducted with 40 specimens divided into the 4 experimental groups (n=10). The method for determining FT was described in ISO 9917[19,33]. For Pure COP, COP 10% and COP 50%, immediately after the mixing, the dental luting agent was sandwiched between two uniform thickness glass plates (2 cm^2) with faces precisely parallels. A load of 15 kg was applied vertically on the top of the glass plate. After 10 min, the thickness of the two plates with cement was determined using a micrometer (Absolute Digimatic Micrometer 227, Mitutoyo Sul America Ltda, Suzano, SP, Brazil). The FT of the cement was calculated by subtracting the thickness of the glasses without the mixed material from the overall thickness. For zinc phosphate group, after 3 min of the beginning of the mixture, the cement was inserted between the two glass plates and submitted to the FT test as cited above.

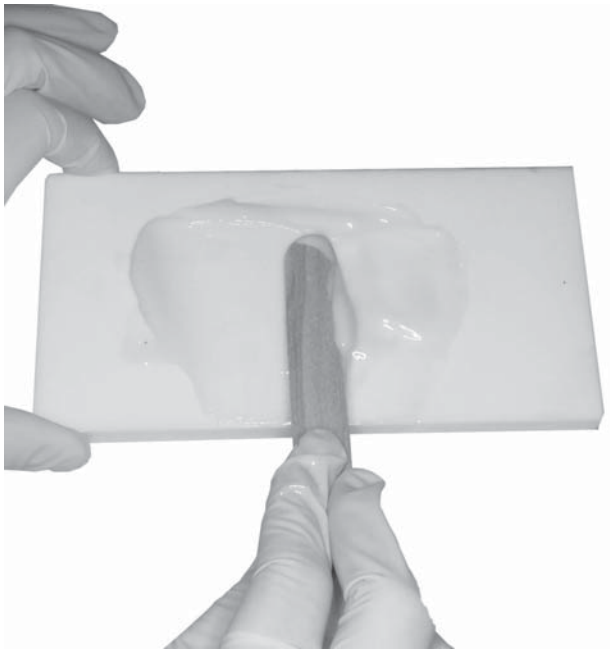

Figure 2- Homogeneous paste of castor oil polyurethane (COP) obtained after manual mixing and mixing with a spatula on a Teflon plate

Table 1- Diametral tensile strength (MPa) of dental luting agents and standard deviations

Groups	DTS
Pure COP*	10.94 (1.30)^{B}
COP 10%	30.06 (0.64)^{A}
COP 50%	29.87 (0.27)^{A}
Zinc phosphate	4.88 (0.96)^{C}

Different uppercase letters indicate significant differences by one-way ANOVA followed by HSD Tukey test
One-way ANOVA (p=1.01E-4)
* COP = Castor oil polyurethane
DTS= Diametral tensile strenght

Table 2- Film thicknesses (μm) of dental luting agents and standard deviations

Groups	Film thickness
Pure COP*	31.09 (3.16)^{C}
COP 10%	17.05 (4.83)^{AB}
COP 50%	13.03 (4.83)^{A}
Zinc phosphate	20.00 (0.12)^{B}

Different uppercase letters indicate significant differences by one-way ANOVA followed by HSD Tukey test
One-way ANOVA (p=2.4E-10)
* COP = Castor oil polyurethane

RESULTS

DTS and FT values of each material are given in Tables 1 and 2.

According to the statistical analysis, all COPs (Pure COP, COP 10% and COP 50%) demonstrated significantly higher ($P<0.05$) DTS than zinc phosphate. The addition of the filler ($CaCO_3$- 10% and 50% w/w) increased DTS. No significant differences ($P>0.05$) were found between the DTS values of COP 10% and COP 50%.

As shown in Table 2, FT values of COP 10%, COP 50% and zinc phosphate were inferior to 25 μm; only the Pure COP exceeded this value. There was a positive influence of filler addition in the FT, since the comparisons Pure COP *vs.* COP 10%, Pure COP *vs*. COP 50%, COP 10% *vs.* COP 50% were minor than 0.05 i.e., the mean values of FT diminished as the quantity of filler was incorporated.

DISCUSSION

This research assessed DTS and FT of an experimental polyurethane dental luting agent derived from castor oil, pure or with different quantities of $CaCO_3$, comparing then with zinc phosphate cement.

The zinc phosphate cement and COP differ each other because their viscous and elastic components; zinc phosphate is water-based cement which is more friable after the final setting reaction, while COP is a polymer similar to resinous cements. When submitted to tensile forces, COP shows a curve characteristic of polymers that undergo flow after the linear region of elasticity, with plastic deformation occurring until rupture.

The results showed that the addition of $CaCO_3$ improves the DTS of COP independently of its percentage. Calcium carbonate diminishes the plastic deformation of COP causing an increase in the final resistance since this filler can fill any pores in the matrix, allowing the cement resists to higher loads. The presence of $CaCO_3$ was also proven interesting because it provides radiopacity without affecting biocompatibility[14].

Zinc phosphate cement had the lower DTS (4.88 MPa) when compared with COP. DTS of COPs (COP 10% and COP 50%) was higher than values of glass ionomer cement (18 MPa)[25] and seems to be more close to the literature results of resin cements which (≈40-45 MPa)[6,10,25,26].

With respect to FT, there is no agreement on its minimum value, but values between 50–100 μm seem convenient[19]. The American Dental Association Specification nº. 8 restricts the zinc phosphate FT ranging from 25 μm to 40 μm[5], but literature values of FT of a number of luting materials show that it can range from 10 μm to 152 μm, depending on the nature of the material[22].

The results of this study showed that all evaluated dental luting agents, except Pure COP, had FT means inferior to 25 μm, meeting the relevant ISO standard[11]. The differences found among the FT of the evaluated materials may be explained by the nature and composition of them. While the setting of zinc phosphate cement occurs by means of a water-based reaction with the growth of the crystalline network of the zinc phosphate[10], the polymerization of COP includes a moisture curing kinetics which is determined by a reaction between the isocyanate and hydroxyl groups, showing a volumetric expansion and consequently higher FT. The obtained results of zinc phosphate (20.04 μm) are in agreement previous studies[21]. Pure COP produced FT above the 25 μm recommended by ISO standard.

FT of COP diminished with the addition of $CaCO_3$ making it lesser than that of some resin cements[15]. Considering that the volumetric expansion is linked to the quantity of organic matrix and that the experimental cement may be composed by only organic matrix (Pure COP) or by an organic matrix with mineral filler (COP+$CaCO_3$), the incorporation of filler diminishes the expansion and consequently provides better FT values.

Despite the clinical importance of the two properties evaluated in this study, the assessment of single properties is not sufficient to identify the best dental luting agent. However, if an experimental dental luting agent does not have appropriate FT and DTS it will not allow the prosthesis serves the functions adequately over a long period. Among the various materials used for cementing indirect restorations and fixed dental prostheses, there is not a single one that fulfills all the characteristics considered ideal. Although one of the experimental materials tested in this study provided better results compared with zinc phosphate cement, further research is required. Comparison with other cements and evaluation of other properties, such as adhesion, hydrolytic degradation and microleakage, can be the focus of future researches. Finally, the introduction of new materials, particularly those not derived from petroleum will enrich the available arsenal of luting materials.

CONCLUSION

Within the limitations of this study, it was conclude that:

1) The polyurethane dental luting agent derived from castor oil (COP) with the addition of $CaCO_3$ filler showed better values of DTS and FT when compared with zinc phosphate cement.

2) For both DTS and FT, the best composition of the experimental polyurethane dental luting agent

was the one with the addition of 50% w/w of $CaCO_3$.

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