CONCILIUM, Vol. 23, Nº 3, 2023
DOI: 10.53660/CLM-854-23B14
ISSN: 0010-5236

Tensile strenght of a dental cement based on copaíba oil-resin

Resistência à tração de um cimento odontológico a base de óleo-resina de copaiba

Received: 2023-01-11 | Accepted: 2023-02-12 | Published: 2023-03-02

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ABSTRACT

This study aimed was to compare the tensile strength of a dental cement based on copaiba resin-oil with the temporary cement RelyX Temp NE (3M, Sumaré-SP, Brazil) in bisacrylic resin crowns. Ten human third molars ,standardized prepared to total crowns, recived temporary crowns cemented with the copaiba cement and the others ten with RelyX Temp NE (3M, Sumaré-SP,Brazil). The twenty samples passed by twenty-four hours immersed in artificial saliva, and then the crowns were tractioned in a universal testing machine at 1.0 mm/min. The crowns cemented with copaiba resin-oil showed tensile strength statistically higher when compared to those cemented with RelyX Temp NE (3M, Sumaré-SP,Brazil).

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Keywords: Dental materials; Dental cement; Phytotherapy;

RESUMO

O objetivo desse trabalho foi comparar a resistência a tração de um cimento odontológico a base de óleo-resina de copaiba com a do cimento temporário RelyX Temp NE (3M, Sumaré-SP, Brasil) em coroas provisórias de resina bisacrílica. Dez terceiros molars humanos preparados, de forma padronizada, para coroa total, receberamcoroas provisórias cimentadas com o cimento de copaíba e outros dez,com o RelyX Temp NE (3M, Sumaré-SP, Brasil). As vinte amostras passaram vinte e quatro horas submersas em saliva artificial, e em seguida, as coroas foram tracionadas em uma máquina universal de ensaios a 1.0 mm/min. As coroas cimentadas com o cimento de óleo-resina de copaíba apresentaram estatisticamente maior resistência à tração quando comparadas às cimentadas com o RelyX Temp NE (3M, Sumaré-SP, Brasil).

Palavras-chave: Materiais dentários; Cimentos dentários; Fitoterapia;

INTRODUÇÃO

The cementation of temporary crowns, an important stage of oral rehabilitation, usually made with self-cured acrylic resin, as well as the definitive ones, made of metal or ceramics, requires specific care regarding the properties of temporary cement, which vary in terms of fluidity, setting time, retention and film thickness (SHEKAR, GIRIDHA & SUHAS, 2010). Cementing agents can provide different levels of retention, which depends on multiple factors such as intraoral pH, occlusal forces, and temperature changes in the oral environment (MORIS et al., 2015).

Zinc oxide eugenol cement has properties such as sufficient strength for retention of the temporary restoration, complete sealing of the crown, and easy removal when necessary, making it excellent for the cementation of acrylic resin crowns. Eugenol is a phenolic compound and is the main constituent of clove oil. Despite its anti-inflammatory and antimicrobial properties, exposure to high concentrations of this product has a cytotoxic and neurotoxic effect, which becomes undesirable when used on living pulps. Moreover, this agent has chemical interactions with resin monomers that cause the delay or inhibition of polymerization of resins used in cores and blocks of cement (ARORA et al., 2016; GALAZI et al.,2015).

Different researches involving the substitution of eugenol in a zinc oxide eugenol-based temporary cement is justifiable for the aforementioned reasons. Thus, and seeking an alternative with more favorable characteristics for the patient, for the development of this research copaiba oil was used, a phytotherapeutic from a tree native to the Amazon region with therapeutic potential in dentistry (GARRIDO et al., 2004). Because of this, the association between zinc oxide and copaiba has proven possible and effective, with results such as low cytotoxicity, and antimicrobial and anti-inflammatory activity (VASCONCELOS et al.2008).

Considering the medicinal properties of copaiba oil-resin, results found using *Copaifera officinalis*, *Copaifera mutijuga* (PIERI et al.,2012; SIMÕES et al.,2016), and tests to retention evaluation of temporary cement (LOPES et al., 2018), this work aimed to compare the tensile strength of a dental cement based on copaiba resin-oil with the temporary cement RelyX Temp NE (3M, Sumaré-SP, Brazil), which was selected due to the proximity of the formulation with the patent of the endodontic cement based on zinc oxide and copaiba developed by Garrido et al.,2004, used as the starting point of this research.

MATHERIAL AND METHODS

Before the beginning of the study, the research protocol was approved by the Research Ethics Committee of UFAM, under the protocol #4.249,803 and #36885820.8.0000.502 in compliance with the Helsinki Declaration. Twenty human third molars from biorepository, extracted for orthodontic reasons, free of caries, cracks and previous restorations were collected. The teeth were washed in water for 24h and kept in distiled water until use.

The development of the cement, according to specification number 30 of ANSI/ADA (2013), was premised on obtaining a zinc oxide and copaiba-based cement with the following characteristics: working time of 1 to 2 minutes, final post-manipulation appearance as a creamy and homogeneous mixture of shiny appearance with cement thread formation of 12 to 19 mm before breaking when raised on the glass plate, initial setting time of 5 minutes and final setting time of 24 hours. The components of the cement are described in table 1 below:

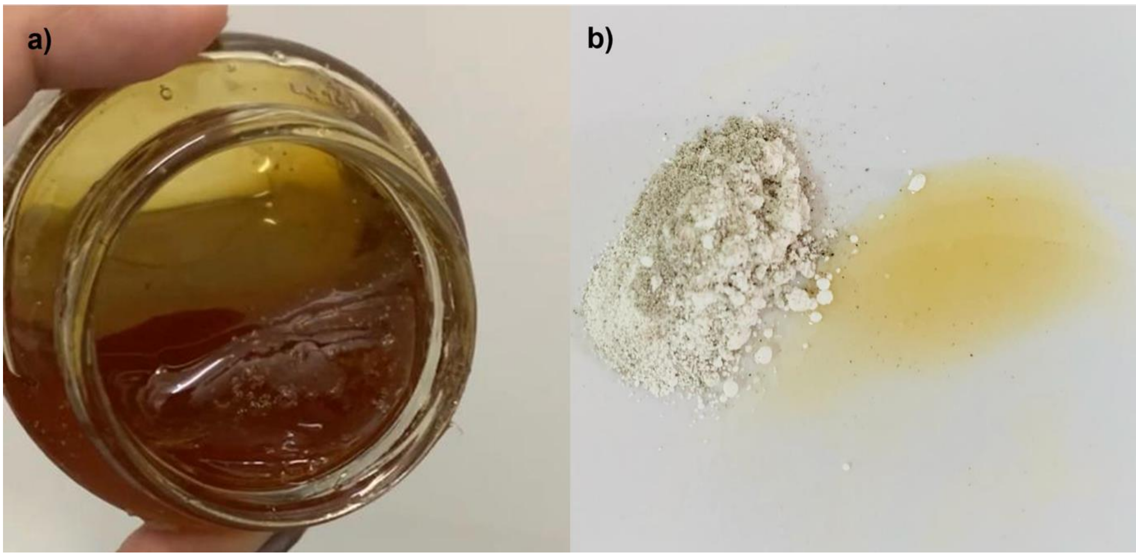
Table 1 - Composition of the provisional cement formulation

Solid component Part 1	Liquid component Part 2
Calcium hydroxide PA (Maquira®, Maringá – PR, Brasil)	Polyethylene glycol (Dinâmica Química Contemporânea, Indaiatuba, SP, Brasil)
Barium sulphate PA (ACS científica, Sumaré – SP, Brasil)	Copaiba oil-resin
White pitch cholophonia (Flora essências naturais, Manaus-AM, Brasil)	Glacial acetic acid PA (Dinâmica Química Contemporânea, Indaiatuba, SP, Brasil
Zinc oxide PA (SSWhite®, São Cristóvão, RJ, Brasil)	
Anhydrous sodium tetraborate (Casa do Químico, Manaus-AM, Brasil)	

For the optimization of the formulation, was used the Initial Design - D-optimal (Design Expert) type of planning. The statistical model of factorial planning was applied, with two independent variables and four dependent variables (responses), and this algorithm provided 16 runs in total, which were tested one by one until the working and setting times proposed in the objectives were obtained.

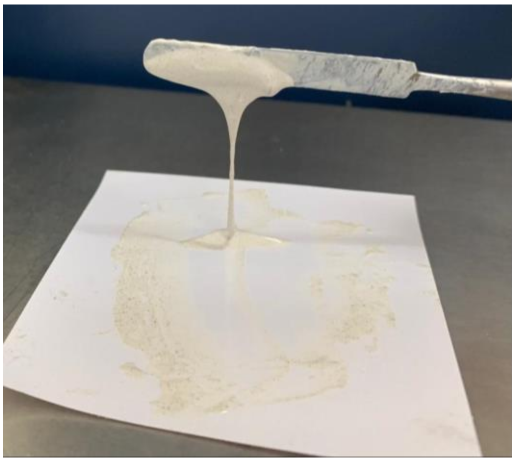
The ingredients from Part 1 after being weighed on a precision balance (Shimadzu, Barueri-SP, Brazil) were mixed in a 10 mL Bécker r and transferred to a mixing sheet. Then, the copaiba resin and polyethylene glycol were added properly homogenized with the ingredients from part 1, using a flexible spatula #24 (Figure 1).

Figure 1 - (a) Copaiba residue; (b) Ingredients from part 1 and part 2 before spatulation



Each formulation was evaluated soon after its preparation for the following characteristics: working time of 1 to 2 minutes, after several attempts by changing the volume of components, with the observation of the mixture every 30 seconds before the cement starts to crack on the spreadsheet. The final post-manipulation appearance, as well as specifications (ANSI/ADA,2013), was a creamy, homogeneous mixture with a glossy appearance with cement thread formation of 12 to 19 mm before breaking when elevated on the mixing sheet (Figure 2).

Figure 2 - Aspect of the cement after 1 minute of manipulation



An initial setting time of approximately 5 minutes was reached by vertically inserting a 1.5 mm diameter heparin-free capillary tube every 60 seconds until there were no more needle impressions on the mixture and a final setting time of approximately 24 hours, considering the appearance of the cement as a stiff and brittle mass after this period. The results were statistically evaluated using the Mann-Whitney test and a rain cloud plot was constructed for the data.

The teeth were embedded in acrylic resin using PVC tubes of 25 mm in diameter by 25 mm (Jet Set - Clássico Artigos Odontológicos - São Paulo, SP, Brazil) with the positioning of 3 mm apical to the cementoenamel junction (CORRER et al.,2002). The included teeth were placed in a laboratory pressure cooker at 0.6 kg/cm2 for finalization of the polymerization to avoid expansion of the acrylic and stored in saline.

The preparations for full crowns were performed by a single operator, using a delineator (Parallelometer B2 model, BIO ART, São Carlos, SP, Brazil) adapted to a high speed (DABI ATLANTE, Campinas, SP, Brazil) under refrigeration. Conical diamond tips with rounded top FG 4138 (KG Sorensen, Rio de Janeiro, RJ, Brazil) were used, performing 1.8 mm of wear and ending in a rounded shoulder angle (Figure 3). The occlusal surfaces were cut with a double-face diamond disk (American Burrs - Palhoça, SC, Brazil) at the height of the main grooves (15).

Figure 3 - Tooth after axial grinding and with the finished preparation

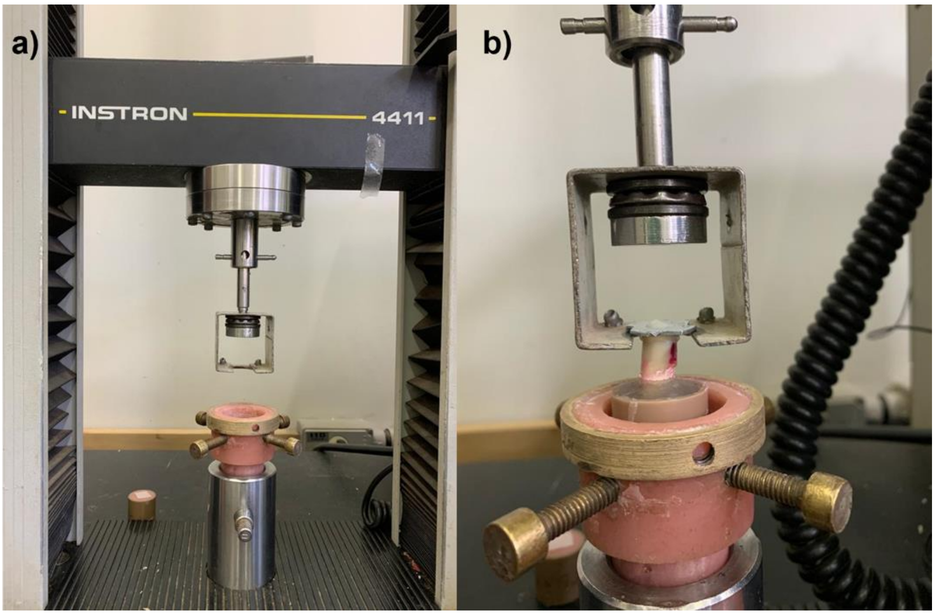
After preparation, the teeth were wrapped in vaseline with polyester strips forming a ring 2 mm from the tooth. Then, PROTEMP bisacrylic resin (3M - Sumaré, SP, Brazil) was added and a galvanized 5/16 claw nut was inserted at the upper end 4 mm deep into the resin.

Before cementation, all teeth were cleaned with a prophy dental brush with a pumice paste and water for 30 seconds. Subsequently, the teeth were rinsed under running water for 30 seconds and dried with compressed air jets. In each group ten teeth was cemented with RelyX Temp NE, and the formulation. After inserting the cement on the bisacrylic resin crowns, a pneumatic cylinder (Norgren Martonair - São Paulo, SP, Brazil) was used with a 5 kg load for 5 minutes on the metal device present in the occlusal region (Figure 4).

Figure 4 - Cementation of temporary crowns.

Excess cement was removed and the teeth were placed in a plastic container with artificial saliva (Bioexata, Manaus, AM, Brazil) composed of sodium chloride, potassium chloride, and pure calcium citrate, magnesium chloride, sodium benzoate, and carboxymethylcellulose. The container was then placed in an oven (Heraeus - Barueri, SP, Brazil) at 37°C for 24 hours. The teeth with cemented crowns, soon after being removed from the oven, were dried with paper towels and fixed in the testing machine (Figure 5) Instron 4411 (Instron - Cambridge, UK) with a load cell of 500N.

Figure 5 - (a) 500N load cell and gripper positioned on the universal testing machine; (b) Tooth adapted to the testing machine



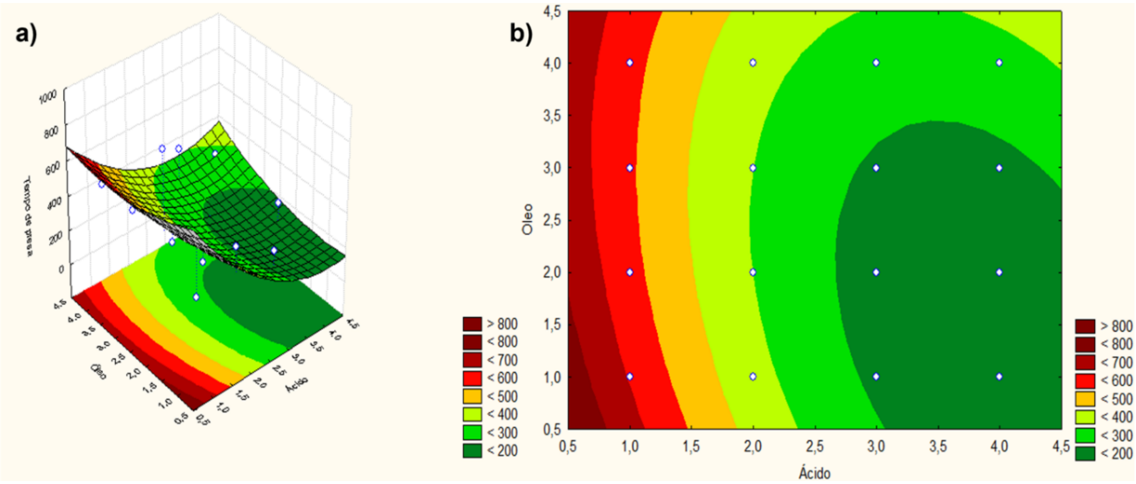
The data obtained in the experiments, during the formulation improvement phase, were expressed by mean and standard deviation. The model proposed in the Experimental Design with the aid of Statistica (StatSoft) software was statistically analyzed by the ANOVA test. For the data obtained from the tensile test, the Mann-Whitney test was used, the statistical software used was R version 4.0.2, using the software Rstudio (RStudio, PBC), version 1.1.4, with various packages (TIDYVERSE, GTSUMMARY, GT, sjPlot, and HNP).

RESULTS

The central compound planning performed in this study had the purpose of predicting the optimal level for the responses working time and setting time, from the statistically significant factors obtained in the fractional factorial. To compare with the previous results, we left the analysis of the factorial design to the end of the results, thus pointing out in practice corroborating data.

The numbers obtained for working and setting times by varying components of the formulation were entered into the D-optimal formulation optimization software, and response surface graphs were generated that demonstrated the influence of varying components in the formulation, as in the example of figure 6 below:

Figure 6 - (a) 3D plot (X=%Oil versus Y=%Acid) of the proposed quadratic model for the Hold Time response; (b) 2D plot (X=%Oil versus Y=%Acid) of the proposed quadratic model for the Hold Time response.



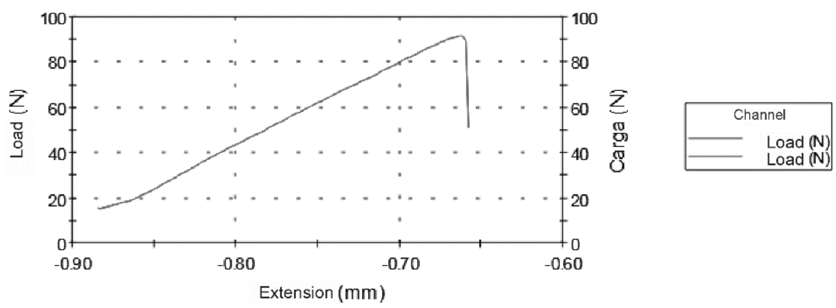
After the tensile tests were completed, the values were tabulated and are available in table 2.

Table 2 - Necessary load values, in Newtons (N), to remove the crowns recorded by the testing machine.

Sample	Copaiba cement	RelyX Temp NE(3M)
1	55,25	18,43
2	91,60	25,25
3	45,93	19,42
4	38,98	18,27
5	57,15	16,38
6	32,66	32,35
7	33,64	32,42
8	69,29	18,83
9	33,62	13,60
10	30,59	20,52

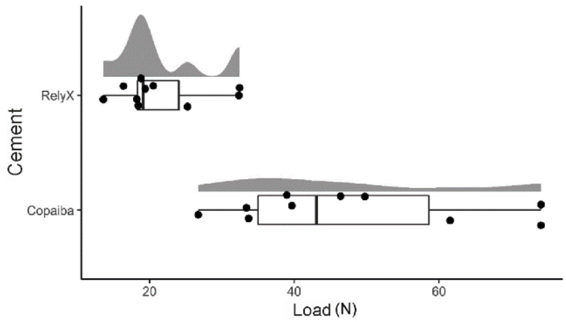
The highest value recorded in the tensile strength test of all samples was obtained by a crown cemented with zinc oxide and copaiba cement, illustrated by the graph generated by the universal testing machine (figure 7) where the red growing line represents the increase in force necessary to remove the crown, the apex of the curve the exact moment of removal, and the vertical decreasing line, the decrease in the force of the machine because the cement line was already completely broken.

Figure 7 - Load (N) x Extension (mm) during the removal test of crown number 2 cemented with zinc oxide and copaiba cement.



The load values in RelyX Temp NE cement are lower than in the Copaíba cement, which in turn shows more dispersed values, but with a higher frequency of values above the maximum observed in RelyX TEMP NE (3M) cement, as can be seen in Figure 8.

Figure 8 - Comparison of tensile values of copaiba oxide cement and RelyX TEMP NE (3M).



According to the results in the figure above it is clear that normality was not verified in the data, so the nonparametric Mann-Whitney test for independent samples was used.

The sample data indicate that there is evidence that the copaiba-based cement differs significantly from the other cement. The copaiba cement has higher central tendency values (mean and median) than the other cement.

DISCUSSION

The results are consistent with those of previous studies (GARRIDO et al.,2004; GARRIDO et al.,2010), na acid-base reaction occours between the acidic components of copaiba oil and the alkaline components such as calcium hydroxide and zinc oxide to form a cement. In addition, cement formulated there is zinc oxide and terpenes present in copaiba oil-resin, being thus, the combination of zinc oxide and copaiba for use in dentistry is viable and promising.

Although the industries have managed to replace eugenol in some zinc oxide-based temporary cement with good mechanical characteristics (GALAZI et al.,2015), its high price when compared to other types of cement available has still been a fact that does not go unnoticed. Thus, besides having found a formulation of cement that adds the pharmacological properties of copaiba and the retention in temporary crowns has been higher than the commercial cement tested, the gross cost per use in the cementation of a crown, based on the cost of production of the experimental cement, is 101% lower than that of Relyx TEMP NE (3M), which is advantageous to the dentist and the patient because it is an effective product with a reduced price.

Considering the regulation terms (ANSI/ADA,2013), zinc oxide-based cement should have fine powders with particles of similar size and be subjected to film thickness and solubility tests. However, in this study these tests were not performed because it was not possible to homogenize the white rosin particles, leaving this component larger than the rest of the powder portion, which would generate a premature contact when subjected to the interposition of glass plates and cellophane paper usually used in film thickness tests, causing incorrect reading of the formulation performance as a whole, methodology used in film thickness tests. Similarly, there would be a misinterpretation in a solubility test, because the unreacted particles could solubilize before the rest of the cement.

This study showed that the zinc oxide and copaiba cement had significantly higher tensile strength when compared to the temporary cement Relyx Temp NE (3M). This possible reason are the intense attraction of the particles formed after hydrolysis of the solid components in acidic pH of the liquid components and the interaction of the cement with the internal microporosities of the temporary crown, however, this hypothesis needs to be confirmed with future chemical analyses. The retention values found for this commercial cement are similar to other zinc oxide-based cement without eugenol (LEWINSTEIN et al.,2007, REGO & SANTIAGO,2015). Additionally, according to some studies (ANUSAVICE, SHEN & RAWLS,2013; SEGALLA et al., 1994), the experimental cement retention results are closer to those of zinc polycarboxylate, a material used for permanent cementation.

In conclusion, the copaiba and zinc-phosphate-based cement showed retention better better tensile strength when compared to RelyX TEMP NE. The cost of using the experimental cement for the cementation of one crown was considerably lower than commercial products. Lastly, biological tests ,quality control, process optimization, physicochemical characterization and will be realized before in vivo applications from formulation.

ACKNOWLEDGMENTS

To Dentistry School – UFAM, to the Post-Graduation Program - UFAM and the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior/CAPES - UFAM, to the Pharmaceutical Sciences School – UFAM, and Prof. Dr. Américo Bortolazzi Correr and Marcos Blanco Cangiani to the Dentistry School of Piracicaba – UNICAMP.

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