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Evaluation of the Physicochemical Properties of A Hydrophilic Cement

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Abstract: *The aim of this study was to evaluate the physical-chemical properties of EndoREZ endodontic sealer. In the study the properties of hardening time, solubility, dimensional changes and cement flow were divided into two groups according to the norms and tests of the American Dental Association (ADA) specification nº 57. Group 1, using a mixing tip compared to Group 2 with equal volume amounts of the pastes. It was observed that the cement EndoREZ in both groups presented similar flow between each other and according to the ADA determination (greater than or equal to 25mm). However, the cement did not set completely. The solubility and dimensional stability tests could not be performed. In view of the study, it was found that EndoREZ cement met the minimum requirement suggested by the ADA for its flow, but it was difficult to meet the specification nº57 of ADA about the hardening time, making it impossible to carry out solubility and dimensional stability tests.*

Keywords: *Endodontics; root canal; physical and chemical properties.*

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I. Introduction

The success in the endodontic treatment depends on the infection control in the root canal space, which is reached by an adequate cleaning, modeling, and hermetic sealing of the canals. The incorrect filling can carry to the endodontic failure, although it made an accurate canal prepare[1]. The endodontic treatment motivated by new technologies and techniques crossed the limits. However, even with all scientific development, some concepts remained: the main canal treatment goal is still the bacterial reduction of the root canal, such as an infection prevention[2]. The achievement of a complete filling, as well as a correct cleaning and modeling of the root canals, is the main way to achieve a satisfactory endodontic treatment in the long term3. Thought the gutta-percha is the most used material in the root canal filling, it does not join to the dentin walls, needing an endodontic cement to an adequate root sealing 5.

In 1984, a series of standards was created to analyze endodontics materials, published by the American Dental Association (ADA). This is of fundamental importance, since there are now standardized procedures for verifying the properties of these materials. The specification nº 57 of the ADA for endodontic materials determines the following tests for the evaluation of properties: flow, film thickness, hardening time, radiopacity, solubility disintegration and dimensional stability6.

The cement endodontics can be classified in resinous cement, cement-based on zinc oxide and eugenol, cement containing calcium hydroxide, cements based on glass ionomer. All have different properties and clinical performance6,7.

The resinous type of cement was developed for the filling of the root canal system presented better clinical results6. Among these, cement-based on methacrylates, such as the EndoREZ (Ultradent Products, Inc. South Jordan, Utah-USA) cement, a cement-based on methacrylate urethane resin (UDMA). According to the manufacturer (Ultradent- Produtcs, Inc)8,9, cement has hydrophilic characteristics (hermetic sealing in wetted channels), direct application in a few seconds, sealing, self-curing, radiopaque, has the ability to save time, biocompatible, adheres to resinous materials, can be removed in case of core preparation and retreatment.

The cement presents two parts of storage. In the compartment 1:Resin monomers: TEGDMA, urethane dimethacrylate, bisglycerol dimethacrylate phosphate; Loading particles: bismuth oxychloride, calcium lactate petahydrate, and silica. In compartment 2: resin monomers: TEGMA, diurethane diethyl acrylate, bisclicerol dimethacrylate phosphate; Charge particles: bismuth oxychloride, calcium lactate pentahydrate, silica, and initiators: diethanol-tolyimino, bisphenol (2,4,6 trimethyl benzoyl), phosphate oxide.

Probably the initiator of the photoactivated reaction is p-tolimine diethanol and the chemical reaction is bisphenol.10 Regardless of the technique used to prepare the root canal, microorganisms or their toxins are still present inside the root dentin. Although the bacteria are not completely eliminated, a hermetic seal allows the treatment to be successful. 11 The work aims to evaluate the physical-chemical properties of the EndoREZ endodontic cement, analyzing the hardening time, flow, solubility, and dimensional change of the cement using mixing tips in comparison with equal amounts in the volume of the pastes.

II. Methodology

The experiment was carried out at the Clinical School of Dentistry at UESPI, where the hardening time, solubility, dimensional change, and flow test of the EndoREZ cement were evaluated, using the protocol of Specification nº 57 of the ADA, which was separated into groups. In group 1 tests were performed using a mixing tip compared to group 2, in which tests were carried out with equal amounts in folder volumes, both composed of five samples.

Solubility Test: For this test, 05 circular Teflon molds were used, 1.5 mm thick and 7.75 mm internal diameter, for each cement. The molds were placed on a thin cellophane sheet supported by a 40 X 80 X 5mm glass plate. The cement was manipulated and placed inside the mold. Then, a waterproof nylon thread with a diameter of approximately 0.5 mm was inserted into the mass of the softened cement. Then, another glass plate was placed, with dimensions equal to the one placed under the cement, surrounded by cellophane foil, on the mold filled with material. On this set, a weight of 100 g was placed. The set was transported to a greenhouse, with a temperature of 37 ± 2 ° C and relative humidity of 95 ± 5%.

After a time interval three times greater than the hardening time of the tested cement. The set was transported to a greenhouse, with a temperature of 37 ± 2 ° C and relative humidity of 95 ± 5%. The specimens would be suspended, two by two, by fixing the nylon threads inside plastic containers with a lid containing 7.5 ml of distilled and deionized water, and taken to an oven at 37 ± 2 ° C for 24 hours. Later, the samples would be rinsed with distilled and deionized water, the excesses removed with an absorbent tissue, kept in a dehumidifier for 24 hours and after that time a new weighing would be performed. However, the tested cement did not take prey in the given period, making it impossible to continue the experiment for the solubility test.

Dimensional Change Test: Cylindrical Teflon molds 3.57 mm high x 3.0 mm in diameter was made for this test. The molds were placed on a glass plate, 26 mm wide x 75 mm long and 1.5 mm thick, covered with cellophane, and fixed to it with useful wax. The molds were filled with the studied cement and then a microscope slide was placed on them, also covered with cellophane, with light pressure. The set was maintained in this position with the aid of a C-shaped clamp. Five minutes after the start of the mixing, the set was taken to the oven at 37 ° C and 95% relative humidity, for a period of time corresponding to three times. The hardening time of the tested cement according to the manufacturer's guidance. After this period, the sample surfaces would be sanded, under irrigation with distilled water, and removed from the molds. Their lengths would be measured with the aid of a digital caliper, thus obtaining their initial length. After being placed in containers containing 2.24 ml of distilled and deionized water, closed, the containers would be taken to the greenhouse, at 37 ° C and 95% humidity, for a period of 30 days. After this time interval, the samples would be removed from the containers, dried with absorbent paper, and their lengths were measured again with the aid of a digital caliper, thus obtaining the final length of the samples. However, the tested cement did not take prey in the given period, making it impossible to continue the experiment for the dimensional change test.

Flow test: In carrying out the flow test, a 3.0 ml glass Luer syringe was adapted to receive 0.5 ml of manipulating cement. The cement was placed in the center of a glass plate, 180 ± 5 s after the beginning of the manipulation, a second plate of the same dimensions was placed on the cement and, on this last one, an additional weight, totaling 120 grams After 10 minutes, the larger and smaller diameters of the discs obtained with the flow were measured with a digital caliper. Five repetitions were performed for each group studied and the arithmetic mean of the values found was obtained. The results were taken for statistical analysis using the Mann-Whitney test.

III. Results

According to the experiment carried out, in the hardening test, there was no final setting of EndoREZ cement in both groups even after 72 h of material handling. In the present study, the tests of solubility and dimensional stability could not be performed, due to the fact that cement did not take final prey in any of its presentations. The EndoREZ cement manipulated with the use of the mixer and form equal parts in volume by the pastes allowed a flow according to specification nº 57 of ADA12, which proposes that the ideal flow for an endodontic cement is equal to or greater than 25 mm of diameter, like the table 1.

Table 1. Flowing of the EndoREZ cement using different manipulating methods of pastes A and B

Samples	Group 1 (mixingtips)	Group 2 (equal amounts in the volume of the pastes)
SAMPLE 1	25,20 mm	29,80 mm
SAMPLE 2	26,64 mm	22.66 mm
SAMPLE 3	24,63 mm	22,48 mm
SAMPLE 4	25,29 mm	27,41 mm
SAMPLE 5	31,44 mm	23,14 mm

There was no significant difference in flow between group 1 (mixing tips) and group 2 (equal amounts in the volume of the pastes), showing that there was no difference regarding the flow of cement in the two handling conditions, according to the table. The results are described in table 2.

Table 2. Statistical difference between the two conditions

Statistical Test	Value
Mann-Whitney	8,00
Z	-0,94
p	0,42

IV. Discussion

The hardening time is the time available for filling after handling the material. This should not be a very short time, so as not to harm the clinical practice, nor so long as to release toxic agents. The setting time serves to provide a working time and adequate consistency for a correct filling of the channel 13,14. The hardening time is the time available for filling after handling the material. This should not be a very short time, so as not to harm the clinical practice, nor so long as to release toxic agents. The setting time serves to have working time and adequate consistency for a correct filling of the channel 13,14. According to the manufacturer, the setting time of the EndoREZ cement is 20 to 30 minutes, where it describes that the photoactivation aims to polymerize the initial surface that is less than 0.3 mm thick, which helps for an immediate restoration.

The recommended photoactivation is 40 s. In research carried out, the author found a setting time of 30 minutes for EndoREZ15 cement, in agreement with the manufacturer. In the present study, the material did not show the final setting time after 72 hours of handling. Dual cure resin cement should be handled on a space without ambient light so that it does not accelerate the polymerization of cement.16 However, in this experiment, even though the EndoREZ cement being manipulated in the presence of ambient light and photoactivated according to the manufacturer (40 s), the Gillmore needle penetrated the cement mass still fluid, without resistance from the polymerized surface layer. After manipulation in a space without ambient light and after light curing for 40 s, the Gillmore needle caused the indentations in the cement EndoRez The resistance to penetration of the needle was observed after 30 minutes (setting time) of the manipulation and light-curing, in research carried out. 15 The author described that a non-polymerized cement layer still persisted in the sample. The experiment was carried out in a dark chamber without light and evaluated according to ADA standards.

The resistance of the needle was detected by finding a hardening time of 28 minutes 17, in agreement with the manufacturer's break time9 (20 to 30 min) and with another study, which was 30 minutes 15. The authors17 described that a non-fully polymerized cement layer still remained in the sample. In the present study, there was no final setting time during the entire experiment. Incomplete polymerization occurs because the exposure of cement to ambient oxygen at the time of manipulation influences the polymerization of resin materials with dual curing, as it reacts with free radicals and slows or inhibits the formation of a well-structured polymer chain 9,16,18, 19. Oxygen reacts with itself or with other radicals, forming inactive products resulting in a layer rich in unpolymerized resin. Previous investigations have shown that oxygen-inhibited layers vary from 4 to 40 µm 18, 20,21. Thus, this polymerization inhibition can impair the properties of the material, therefore, according to the manufacturer's recommendations, there is an importance of using auto tips -mixers, as well as their placement in a Skini syringe with NaviTip tips (specific tips) ensuring that the cement is free from contact with ambient oxygen, since it will be inserted directly into the root canal. The solubility study is necessary because of the possibility of dissolving the material, which can cause the formation of spaces within the filling, or between the filling and the dentin walls, which makes it more susceptible to bacterial infiltration, compromising the treatment carried out22. Solubility should not exceed 3% by weight.12

Researchers evaluated the solubility property of EndoREZ cement through the loss of water weight of the samples over 28 days found a high solubility (9.57%) .17 This result agrees with the study that used the immersion liquids of the cement to the determination of the concentration of metal ions, by means of atomic absorption spectrometry to quantify the ions, also observing a high solubility (5.63%) .15 An endodontic cement should not change by 1% in contraction or 0.1% in expansion12. Dimensional alteration is a property considered necessary for the maintenance of filling inside the root canal and is related to the non-alteration of the material.

However, if there is any change, a quick expansion is better than a contraction, because if the latter occurs, there may be a mismatch between the cement and the channel, causing gaps inside the filling, allowing bacteria to install inside the filling. Same, impairing the sealing and favoring the occurrence of fluids23,24. Research Work observed the cement EndoRez showed a dimensional change of 1.07%, higher than recommended by the ADA standard. The author also reported that in the dimensional change samples, he still found a little superficial layer of unpolymerized cement.15The ability of the cement to drain into spaces not filled with gutta-percha, penetrating the gaps in the root canal is made possible by the flow property, thus, offering adequate sealing to the root canal walls25,26. An endodontic cement has good flow when it reaches a rate greater than or equal to 25 mm.12

Regardless of the handling method, EndoREZ cement presented a flow greater than 25 mm in diameter, according to ADA specification nº 57. However, a study reported that the cement showed no flow, certainly due to the failure to place a force greater than the force of gravity.27 The referred specification recommends the use of a complimentary weight to the plate totaling 120 grams. The authors27 used a vertical flow method, which consists of not applying force.

V. Conclusion

According to the study, it was concluded that: as for the flow, there was a similarity between the manipulation with the mixer and the use of equal parts in volume. Both methods met ADA specification nº 57. EndoREZ cement did not take prey over 72 hours of material handling by the tested methods, also making it impossible to evaluate dimensional stability and solubility. There is a need for ADA to standardize appropriate methodologies to evaluate the physicochemical properties of the new endodontics cement.

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