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Evaluation of the Microhardness of Different Types of Bulk Fill Resins

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ABSTRACT

The constant search for the ideal restorative material causes the new formulations to aim for an increase of 4 to 5 mm, getting an improvement of its mechanical properties, for example, the organic and inorganic matrix of other materials. With this, the objective of the present study was to evaluate the microhardness of different brands of bulk fill resin. In order to perform the research, 30 specimens were prepared, with group 1 with 10 specimens of the conventional composite resin Opallis (FGM). Group 2 with 10 specimens of Oppus Bulk Fill composite resin (FGM). And group 3 with 10 specimens of the Tetric N-Ceram Bulk fill resin (Ivoclar Vivadent), all made from a preset silicon matrix. Each group was photopolymerized on the upper surface at the time indicated by the manufacturer, stored in an oven with distilled water under 37 ° C for 24 hours. After this period, they were measured with a digital caliper and submitted to the Vickers microhardness test at 5 random points on the upper surface. The values recorded by the microdurometer were taken to the statistical analysis using ANOVA test with Tukey post-test, in which it showed a statistical difference between Bulk fill Tetric N-Ceram (Ivoclar Vivadent) and Opallis (FGM) with p value<0.001, and a statistical difference between Bulk Fill Tetric N-Ceram (Ivoclar Vivadent) and Opus Bulk Fill (FGM) with a p value of 0.001. There was no difference between Opallis and Opus Bulk fill (FGM). It was concluded that Bulk Fill Tetric N- Ceram resin (Ivoclar Vivadent) obtained a lower microhardness when compared to Opallis (FGM) and Opus Bulk Fill (FGM) resins.

Keywords: Composite Resins, Hardness tests, Physical Propertie.

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I. Introduction

The science of dental materials has the need to follow the considerations that are associated with its selection and its use [1].

It is undeniable how much has been seen about the improvement of restorative materials. With each new material it is noticeable the modifications made in order to satisfy the new demands found in the day-to-day clinical. Composite resins, for example, have evolved significantly since they were first introduced in dentistry, about 50 years ago, in which they are clinically applicable due to their adhesiveness and their ability to adapt to dental tissues [2]-[3].

These resins are currently the most used dental materials in direct restorations, whether in anterior or posterior teeth. Since its advent and with the development of acid conditioning, composite resins have undergone varied modifications in their chemical composition in order to improve their mechanical properties and consequently their clinical performance. The changes occurred in both the organic and inorganic matrix [4].

The heterogeneous market for dental materials is often driven by consumer demand for faster and easier procedures. As an example of this, there has been a growing popularity among dental practitioners of so-called "single fill" materials, which allow restorations with thick layers of up to 4 or 5 mm [5].

Therefore, composite resins called Bulk fill were launched, which enabled professionals to insert a single increment into the dental cavity, reducing clinical time. The properties of these materials, such as their fluidity, translucency, low modulus of elasticity and low polymerization contraction, cause the margins of the cavity to be sealed with greater precision, improving the durability of the work [6].

Bulk fill resins do not require polymerization with increased irradiation, this fact does not affect the contraction of polymerization and the adaptation to the cavity or the degree of conversion, considered vital for clinical success [7]. They have a low modulus of elasticity and an efficient adhesion to the tooth structure, essential characteristics for an effective transmission of tensions between the bodies, causing them to behave as a single body [8]. Some mechanisms ensure sufficient cure and adequate mechanical properties of this material due to the use of new monomers based on methacrylate, use of chemical modulators of the polymerization reaction, new photoinitiator systems, increased translucency and the reinforcement of the inorganic phase by glass fibers [9]. With greater translucency, resin composites allow more photopolymerizing light to penetrate

deeply which possibly causes greater polymerization of the resinous monomers [10].

According to the manufacturers, this new material allows the application of increments of up to 5mm thick, with a uniform degree of conversion throughout the increment and reduced contraction and polymerization stress [11]-[14].

It is already known that an ideal restorative material must imitate the mechanical and physical structural characteristics of dentin and enamel and to analyze the effectiveness of new materials mechanical tests are made, an example is the bending test, in which all types of stresses (compression, shear and traction) that act simultaneously are seen, due to the dynamic nature of the tensions existing in these masticatory movements [15]. With this, it is worth emphasizing the importance of such tests, as they are a way to simulate these movements and ensure that these materials, when well used and studied, have good applicability in the clinical environment.

Another mechanical test is that of microhardness, one of the most important physical properties. Clinically it is seen the importance of the study of microhardness since this property is related to the resistance to deformation and malleability of the restorative material [15]. In other words [16], reported that the hardness of a material can be defined as its ability to resist indentation or penetration.

Thus, in view of the growing appearance of new resins of the Bulk fill type, there is a need for new in vitro studies of their mechanical properties, evaluating the real effectiveness of this material in its different brands offered in the market. In addition, it is important to analyze the influence of the composition of each brand on the microhardness of Bulk fill resins since their composition is directly linked to their mechanical behavior in the oral cavity. Because it is a recent material, the literature is still very controversial regarding its performance and clinical longevity.

The objective of the present study was to verify the surface microhardness of different brands of composite resins of the Bulk fill type, analyzing the composition of the charge particles of these resins before the manufacturer's information and to expand the knowledge about the new materials present in the dental market due to the single increment technique.

II. Materials and Methods

A. Experimental Design

The experimental units were composed of 30 specimens made with three types of composite resins: a conventional composite resin and two Bulk fill resins for the tests and described in Table I.

B. Preparing the Specimes

The specimens were made with a low rigidity rubber matrix OMT189 (Odeme, Luzerna/SC) of cylindrical shape, with internal measures of 5.0 mm in diameter and 2.0 mm in height. Increments of conventional direct composite resin and Bulk fill with spatula for Suprafill N°1 composite resin (Millenium- Golgran, São Paulo, Brazil) were inserted.

At the end of the filling of the silicone matrix, the increment was leveled with a polyester matrix (Prevén) and glass plate (Golgran), to obtain a flat surface prior to polymerization, which was performed with the photopolymerizer Emitter B (Schuster). The polymerization time of each group of specimens is described in Chart II, according to the manufacturer's recommendations.

TABLE I: Information on the Composite Resins That Were Used in the Study

Composite	Charge particle size	Organic matrix	Inorganic matrix	Cargo %
Opallis -FGM	0,5 µm	BIS GMA,BIS EMA,UDMA, TEGDMA	Barium Glass-Aluminum Silicate Signpost, Silicon.	78,5-79,8% by weight
Opus Bulk Fill- FGM	0,7 a 1,0 µm	UDMA, stabilizers, co-initiators and photoinitiators	Silicon dioxide, stabilizers and pigments	76,5% by weight
Tetric N-Ceram Bulk Fill-Ivoclar Vivadent	0,04 a 3 µm	Bis-GMA, Bis-EMA e UDMA	Barium glass, ytterbium trifluoride, mixed oxide, prepolymer	75-77% by weight

TABLE II: Polymerization Time of Each Group according to the Manufacturer

Group	Power	Polymerization time
Group 1- Opallis (FGM)	1000-2000 Mw/cm²	40 seconds
Group 2- Opus Bulk Fill (FGM)	1000-2000 Mw/cm²	30 seconds
Group 3- Tetric N-CeramBulk-Fill (Ivoclar Vivadent)	1000-2000 Mw/cm²	10 seconds

C. Microhardness Test

For the microhardness assay Vickers was using the Shimadzu HMV – Micro Hardness Tester microdurometer (Tokyo, Japan) with specimen positioned below the indentation tip. The applied load was 100g (98 N) for 10 seconds, being performed in five random points of the surface of the specimen that were later seen through a microscope, with an increase of 40x. The arithmetic means obtained from the five evaluations were used for the statistical analysis.

D. Organization and Analysis of Data

Data were analyzed using the Statistical Package for the Social Sciences (SPSS, Chicago, IL, USA.), Version 20.0. A descriptive analysis of the data was performed, presenting them with mean, standard deviation, 95% confidence interval, minimum and maximum value. To verify the normality pattern of the variables, the Shapiro Wilk test was performed and for homogeneity the Levene test was performed. It was verified that the variables followed the normality pattern with p-value of the tests greater than 0.05. Thus, the test selected for the associations was the One Way ANOVA test, with Tukey's post-test. Values of $p<0.05$ were considered significant.

III. Results and Discussion

In the present study, the mechanical surface microhardness test was used to evaluate this property in different types of Bulk fill resin (Opus Bulk Fill-FGM and Tetric N-Ceram-Ivoclar Vivadent) and compare it to the microhardness of a conventional resin (Opallis-FGM).

Table I shows the average distribution of the values evaluated in three types of composite resins, one conventional and the last two Bulk fill.

It is observed in Table III that the microhardness was higher for the conventional resin Opallis (FGM), with an average of 65.3, followed by Opus Bulk fill (FGM), with an average of 61.6. Tetric N-Ceram Bulk fill (Ivoclar Vivadent) obtained the lowest average (54.1).

According to some authors, there is still no consensus on an ideal hardness value for composite resins. However, a study showed that, for composite resins, the value considered correct would be a hardness value greater than 50 VHN – Vickers hardness number [17]. Corroborating with these authors, this study showed that the resins studied here have acceptable surface hardness values.

When submitted to the ANOVA association test with Tukey's post test, the three groups were compared as follows: Opallis (FGM) and Opus Bulk fill (FGM) showed a mean difference of 3.7. Opallis (FGM) and Tetric N-Ceram Bulk fill (Ivoclar Vivadent) with an average difference of 11.2. And then the Opus Bulk fill (FGM) and Tetric N-Ceram Bulk fill (Ivoclar Vivadent) had an average difference of 7.5. All with a standard error of 1.7, as shown in Table IV.

When comparing Opallis (FGM) and Opus Bulk fill (FGM) there was no difference between them. However, Tetric N-Ceram (Ivoclar Vivadent) was inferior both when

compared to Opallis (FGM) with $p<0.001$, and when compared to Opus Bulk fill (FGM) with $p=0.001$, demonstrating the lowest microhardness between the studied groups.

The Vickers microhardness test, which was used in this study, produces an impression in the composite resin as a result of diamond penetration in the form pyramid with a square base and an angle of 136o between opposite faces. The diamond fixed to the microdurameter penetrates the composite resin under a load and for a pre-established time [18]. For Soprano [19], this test is the most suitable for composite resins due to its greater stability and should be used preferably to the detriment of other tests when trying to verify the hardness of resin composites.

The study resins were selected according to the following criteria: Opallis and Opus Bulk fill, both from FGM, are national brands that are easy to access and low cost for the clinician, and Tetric N-Ceran Bulk fill (Ivoclar Vivadent) was selected by to be a globally recognized brand, being the object of several studies with mechanical tests.

Thus, the specimens were made according to the standard of dental materials developed by the International Standards Organization (ISO) 4049. In which they were submitted to the Virkers microhardness test, and each specimen received a load through indentation tip at 5 random points, thus obtaining averages taken to statistical analysis in order to ascertain their effectiveness with a focus on microhardness, a crucial property for the success of restorations.

Studies have shown that the improvement of single-fill restorative materials was due to the modification of its organic and inorganic matrix. The microhardness of composite resins is influenced by the volume/weight of inorganic particles but also by the constitution of the polymeric matrix as well as by the morphology, size, and distribution of filler particles [20]-[21]. However, there is little information about the morphology and the amount of filler present in the composites in relation to their effects on these properties [22].

Several studies in the literature have shown superior results for conventional resins in relation to Bulk fill resins when performing the microhardness test, in line with the results of the present study. As for example, the studies by Alkhudhairy [23], who even varying the light intensity of light curing units showed that conventional resins had better average results. The studies by Kelic *et al.* [24], who, by varying the depth of the specimens of different bulk fill resins compared to the conventional one, these were superior in terms of Vickers microhardness, and studies by Nascimento *et al.* [25], who compared several brands of Bulk fill and yet none was superior to the conventional one, however, all the brands studied by them presented acceptable microhardness values, confirming the effectiveness of the restorative materials offered in the market.

When comparing the mechanical properties in class I cavities restored with four types of bulk fill resin and one of the conventional type, this author observed that, given all the properties studied, there were divergences between conventional and bulk fill resins, an example was that the bulk fill in terms of its microhardness was inferior when compared to a conventional resin, in agreement with the present study [13].

TABLE III: DESCRIPTIVE ANALYSIS OF CENTRAL MEASUREMENT VALUES AND MICROHARDNESS VARIABILITY IN THREE DIFFERENT RESIN TYPES

	n	Média	DP*	Confidence interval 95%		Minimum value	Maximum value
				minor	biggest		
Opallis -FGM	10	65,3	3,1	63,0	67,5	60,1	67,7
Opus Bulk fill -FGM	10	61,6	4,0	58,7	64,4	57,5	67,4
Tetric N-Ceram - Ivoclar Vivadent	10	54,1	4,4	50,9	57,2	44,7	60,1
Total	30	60,3	6,0	58,0	62,5	44,7	67,7

TABLE IV: VERIFICATION OF MICROHARDNESS IN DIFFERENT TYPES OF RESIN

Resin	Average difference	standard error	Confidence interval 95%		p-value*
			Minor	Biggest	
Opallis (FGM) - Opus Bulk fill(FGM)	3,7	1,7	-0,6	8,0	0,104
Opallis (FGM) - Tetric N- Ceram (Ivoclar Vivadent)	11,2	1,7	6,9	15,5	**<0,001**
Opus Bulk fill(FGM) - Tetric N- Ceram (Ivoclar Vivadent)	7,5	1,7	3,2	11,8	**0,001**

Similar results were found by Leprince *et al*. [5] and Ilie, Bucuta and Draenert [26], when evaluating the mechanical properties of bulk fill and conventional resins, observed that the bulk fill type resins were inferior in relation to the conventional ones, emphasizing the caution that must be taken when using the new composites on the occlusal surface, as these, in view of their studies, are more subject to greater degradation in the cavity over time, in addition to their greater translucency affecting the aesthetics of the restoration. Therefore, it is necessary to use an occlusal layer with conventional resin.

Due to the chronology of the history of bulk fill resins, it is worth remembering that they were once considered dentin resins, since they would not be suitable for resisting the masticatory forces received by the restoration.

Son *et al*. [27] submitted five Bulk fill and two conventional resins to the microhardness test, realizing that the bulk fill type decreased the microhardness value from top to bottom of the specimen more considerably than conventional resins.

Agreeing with Garoushi *et al*. [28] who analyzed the degree of conversion and microhardness of conventional resins, fiber-reinforced resins and bulk fill resins, calling attention to Tetric Evo Ceram Bulk Fill, which had the lowest microhardness, in addition to having a drastic decrease in the microhardness value obtained in the upper surface to the lower surface of the specimens. This is also in line with Flury *et al*. [29] who showed in their study a significant decrease in microhardness in TetricEvoCeram with increasing increment thickness when compared to other Bulk fill resins.

This can be explained because the degree of conversion was lower near the bottom surface, therefore the hardness of the material decreased with increasing depth of increment, due to the lower amount of light transmission [28].

The same authors cited above explained this fact because the single increment resins had low filler content and consequently lower microhardness and greater polymerization than those with high filler content, recommending operator care when choosing the single filling material, analyzing the filler content of these composites.

In contrast to the results already shown here, Borges *et al*. [1] showed that the bulk fill resin Tetric N-Ceram Bulk-Fill (Ivoclar Vivadent AG) was superior in terms of its degree of conversion and its microhardness, before being tested with beverages acids when compared to Filtek Z350XT (3M ESPE), a conventional resin. Similar results were obtained in the study by Moharam *et al*. [10].

In addition, reported that the lack of a drastic drop in microhardness found for most bulk fill materials is in line with previous studies and supports manufacturers' claims that bulk fill resins can be used in increments greater than the 2 mm recommended for conventional composite resins, without compromising their properties [26].

Other studies such as Dalkilic *et al*. [30] showed similarities between different resins. Among the results obtained, it was observed that there was no significant difference between the groups, however Tetric N-Ceram Bulk-Fill showed lower average microhardness than Tetric N-Ceram on the lower surface, concluding that the microdozen of the evaluated materials are similar. This agrees with the microhardness values between conventional Opallis (FGM) and single-fill Opus Bulk fill (FGM), which were statistically similar in the study discussed here.

Thus, microhardness depends on the following parameters: material, thickness of the increment, volume and weight of the load and the quality of light transmitted by the curing light. In addition to being directly linked to the translucency of the material.

Bulk fill resins are more translucent than conventional ones, precisely to obtain a greater passage of light through the increment, while following the manufacturer's recommendations regarding light curing time [26].

With regard to the size of the filler particles of the resins, as a result of studies, those with smaller sizes transmit better the passage of light and those with larger sizes, the opposite. This is because the greater the amount of filler and smaller particles, the greater the number of interfaces between the inorganic part and the organic part, which provides an increase in light dispersion due to the different refractive indices between the filler particles and the resin matrix [26].

In view of this, manufacturers use different strategies to improve the composition of these materials and achieve photopolymerization throughout the increment, whether increasing, decreasing or changing the shape of the filler particles.

A different way of increasing depth of cure was followed by Tetric N-CeraBulk fill, introducing a photoinitiator that triggers a faster and deeper polymerization reaction, Ivocerin, which has a more reactive effect than camphorquinone. Furthermore, it boosts the conversion of monomers into polymers with minimal shrinkage. Therefore, the strategy of using a photoinitiator with these characteristics in a bulk fill composite resin would be beneficial for the production of a polymer with good quality, since this composite is often used in 4mm layers [31].

However, even with all these advantages, Tetric N-Ceram Bulk fill (Ivoclar Vivadent) presented the lowest microhardness in this study and in most others discussed, which can be explained by having 75 to 77% by weight and 53 to 55% by volume and particle sizes ranging from 0.04 to 3 μm, being the smallest values among the 3 groups studied here (Table I).

The control group (Opallis – FGM), which showed the best surface microhardness averages (Table 1), can have its result explained due to its filler particle composition with 78.5 to 79.8% in weight and 57 to 58% by volume. Not differing much from the composition of Opus Bulk fill (FGM) with 76.5% in weight and 58.4% in volume, as shown in Table III of this study. Both were statistically similar.

It is worth emphasizing the importance of studying the composition of these new restorative materials because, as seen in several studies, the composition directly influences the mechanical properties of composite resins. In addition, in vitro studies must reliably simulate all these influences of occlusal forces and the stomatognathic system on the composites that appear on the market, since the unsatisfactory mechanical performance of the material reduces long-term stability, which has become a concern for dentists, researchers and developers of dental materials.

IV. Conclusions

Based on data from this study, it is possible to conclude that the conventional composite resin Opallis (FGM) had better surface microhardness values when compared to Tetric N-Ceram (Ivoclar Vivadent). The Opus Bulk Fill (FGM) resin also showed better microhardness than Tetric N-Ceram (Ivoclar Vivadent), but when compared with the conventional Opallis (FGM), no statistical difference was found between them.

There is a need for further studies on new resin composites, since some of their mechanical properties are still inferior in relation to conventional resins. In addition, there are still a few articles on the subject, with little data with statistical difference on this new category of material, mainly with national brands.

Conflict of Interest

Authors declare that they do not have any conflict of interest.

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