Evaluation of mechanical properties of composite resins with shelf life expired. First test: volumetric shinkrage

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Objective: To evaluate the volumetric shrinkage of different brands of composite resin 6 months after the expiration date, using a video/imaging system (Acuvol, Bisco). **Methods:** Composite resin increments of approximately 15μL were placed on a teflon pedestal and the initial sample perimeter was checked. After resting for 3 min, the increments were light cured and the perimeter was checked through the video/imaging and a computer software, before and after light-curing. The difference between both measurements was calculated to achieve the volumetric shrinkage percentage. **Results:** The mean volumetric percentage values were 2.8% for Group I, 2.66% for Group II, 2.56% for Group III, 2.58% for Group IV, 2.3% for Group V, and 2.26% for Group VI. After statistical analysis using Student *t* test, no significant differences were found between the groups. **Conclusion:** It was concluded that the volumetric shrinkage of the composites tested does not change 6 months after the expiration date. **Keywords:** Dental materials. Date of validity of products. Dental restoration, permanent.

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INTRODUCTION

The free volumetric contraction, which occurs during the polymerization of the composite resins, can generate marginal slits, favoring microleakage, displacement of the restoration and postoperative sensitivity.

Composite resins currently available on the market are, for the most part, based on silorane or methacrylate. The polymerization process occurs differently between the materials, the polymerization of the silorane-based composite resin occurs by means of a mechanism of opening of cationic rings, while methacrylate based resins, polymerize through free radical mechanisms, Thus, the silorane-based resin presents a mean volume contraction of approximately 0.9%, while the methacrylate composite resins have a volume contraction between 2.3 and 3%. With this, it is observed that the percentage of free volumetric contraction is directly related to the composition of the material. However, there are not many report on the influence of material storage time on this property.

The percentage of free volumetric contraction can be measured laboratorially when the photoactivated composite is not adhered to any surface that may influence the result. There are several methods for if measuring this contraction. However, in the present study, a mechanism video image was used, provided by the Acuvol test machine (Bisco, Schaumburg, USA), to present advantages such as the practicality and the ability to monitor the volumetric contraction during the entire polymerization process.

In some cases, composite resin used in dentistry has the expiration date expired before the end of the material; however, there are not many reports in the literature associating the loss of properties of the composite resin with its period of validity established by the companies.

Considering the importance of the polymerization contraction of composite resins in the longevity of the restoration performed, the objective of this work was to evaluate the polymerization contraction of resins composite of different commercial brands with expiration date up to 6 months.

The null hypothesis was that, after the period of validity, established by the manufacturers, there was changes in the physical and mechanical properties of the composite resins, among them, alteration of the volumetric contraction.

MATERIALS AND METHODS

For the study, 3 different brands of methacrylate-based composite resins were selected, being two micro-hybrid resins (4 seasons, Ivoclar Vivadent; and Opallis, FGM) and a nanoparticulate composite resin (Filtek Z350, 3M ESPE) within expiration date and 6 months after this period.

However, some criteria were used for the inclusion of composite resins, within expiration date or not, in the research, as well as the storage location of the material, which should be stored in places with adequate temperatures according to each manufacturer, and the amount of material remaining in the package (tubes containing little material were discarded).

After the selection of the composite resins, they were divided into 6 groups: for the GI, the contraction percentages of the Opallis composite resin (FGM) were evaluated within the validity period. In the GII, the composite resin Opallis (FGM) with the expiration date expired in 6 months.

For the GIII, the 4 Seasons composite resin (Ivoclar Vivadent) was selected within the validity period and in the GIV, the same resin with the validity period exceeded in 6 months.

The nanoparticulate composite resin Filtek Z350 was selected for groups V and VI; in GV, the material was used within the period of validity and in GVI after the period of validity.

To determine the percentage of free volumetric contraction of the composite resins, a test machine using a video and image mechanism, named Acuvol (BISCO) (Fig 1), was used.

This machine, in addition to containing inside a video camera (Sony XC75-X), which is responsible for the filming of the samples in real time, is coupled to a computer, which has specific software (MIOD Detection Technologies), which captures and digitizes the images of the samples.

When starting the computer program, it displays three measurement tables: one for measuring the volume of the material, another for measuring the percentage of volumetric shrinkage and one for measuring the brightness (Fig 2).

For each of the selected resins, before and after expiration, 5 specimens (n = 5) were evaluated.

To capture the images, each increment of the material was taken directly from the tube by a calibrated investigator, with a volume of approximately 15μL, ranging 0.5 above or below the established volume of 15 μL and positioned on a Teflon pedestal located about 10 cm from the video camera lens, and illuminated by lamps whose light prevents it from interfering with the influence of external light on the polymerization of the material (Fig 3).

To determine the initial volume of the sample, a material flow time of about 2 minutes after the positioning of the same material on the aforementioned pedestal, pre-determined through a pilot study was determined, and after that period, an initial image was captured and the sample perimeter was circumvented by a virtual line, thus defining the initial volume of the sample.

Subsequently, the material was photoactivated by LED light apparatus, 1mm away from the sample surface. To make the standardization of this distance, a metallic device was made, being coupled to the tip of the polymerizer, forming an angle of 90o with the surface of the increment.

Ten minutes after the polymerization of the material, another line skirted the material, thus determining its final volume, given by the value established by the software, which can be observed on the computer screen.

With the two lines represented in the same image, it was possible to visualize the contraction of the material, calculated automatically by software. (Fig 4).

After obtaining the results of the 30 samples, 5 of each material, the values were submitted to statistical analysis by the student t test.

Figure 1: Set of machines for measurement of volumetric contraction and computer contained in specific software.

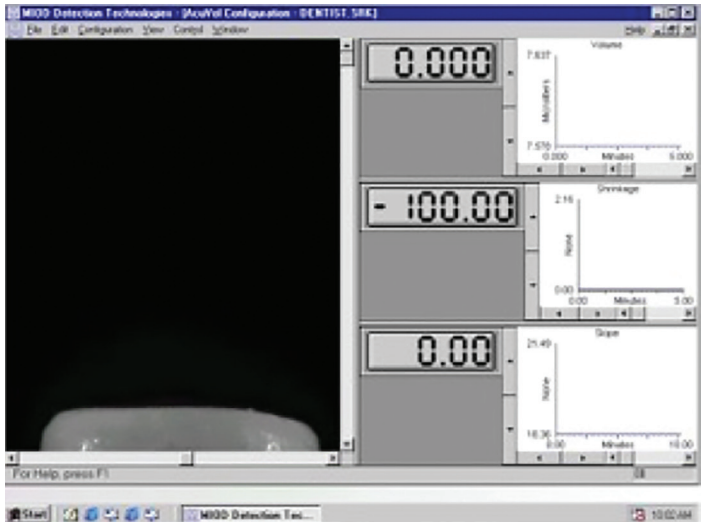

Figure 2: Initial screen of the software after previous configuration.

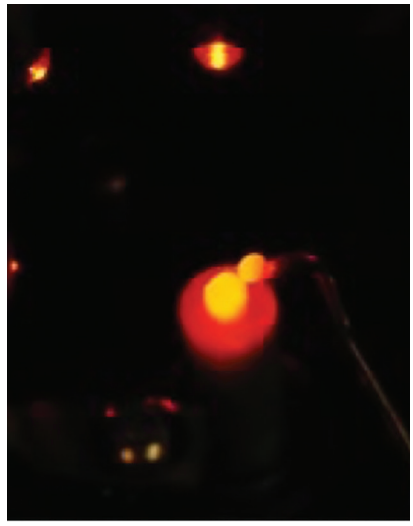

Figure 3: Increment of composite resin being positioned on the Teflon pedestal, illuminated by light-curing inhibition lamps from the external medium.

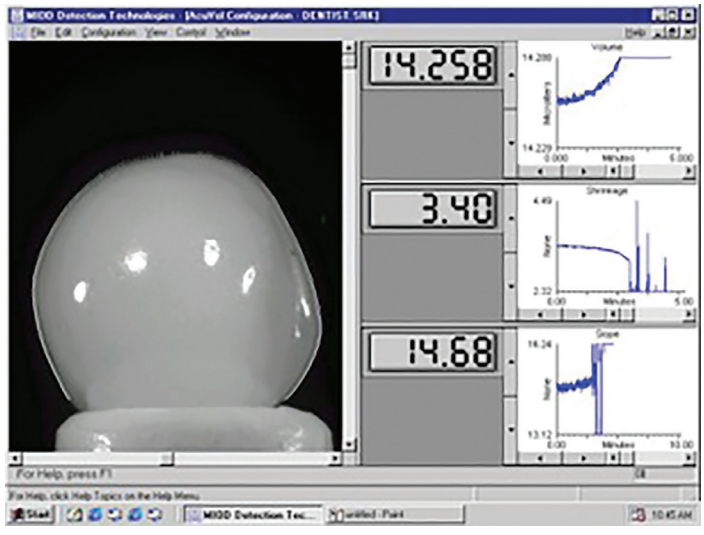

Figure 4: Final image of software with final value of volumetric contraction percentage.

RESULTS

Five samples of each of the composite resins included in the study were evaluated, the individual results of each one are shown in the tables below (Tables 1, 2 and 3).

There was not a wide variation in the values of the average percentage of volumetric contraction in the composite resins tested. For group I, we found 2.8% of contraction on average,; when the values of group II are observed, we found the value of 2.66%.

In GIII, the mean value of volumetric contraction was 2.56%, while in GIV, the mean value was 2.58%. In group V, 2.3 was the average percentage found, and finally in GVI, we observed 2.26% of mean contraction.

After statistical evaluation of the results by Student's t test ($p < 0.05$), it was observed that there was no statistical difference between new resins and expiration time between each of the resins evaluated.

Table 1: Opallis (FGM) resin.

SAMPLE NUMBER	% VALUES OF GI	% VALUES OF GII
1	2.9	2.8
2	2.8	2.7
3	2.6	2.5
4	2.8	2.7
5	2.9	2.6

Percentage values of contraction of groups I and II.

Table 2: 4 Seasons (Ivoclar Vivadent) resin.

SAMPLE NUMBER	% VALUES OF GIII	% VALUES OF GIV
1	2.8	2.3
2	2.5	2.6
3	2.6	2.6
4	2.3	2.7
5	2.6	2.7

Percentage values of contraction of groups III and IV.

Table 3: Z350 (3M ESPE) resin.

SAMPLE NUMBER	% VALUES OF GV	% VALUES OF GVI
1	2.3	2.1
2	2.3	2.3
3	2.5	2.2
4	2.1	2.4
5	2.3	2.3

Percentage values of contraction of groups V and VI.

DISCUSSION

After evaluation of the results obtained in the present research, no changes were observed in the percentage of volumetric contraction.

It is observed in the literature evaluation of the degree of conversion, microhardness and surface roughness of 4 composite resins used 6 months before and after the period of validity. After the tests and statistical evaluation of the results, the only property in which there was any statistical difference was the microhardness, showing a decrease of the values after the expired date.

The video and image mechanism for measurement of free contraction of the composite resin presents advantages, when compared to the mercury dilatometer, as the visualization on the computer screen of the volumetric contraction in real time during the polymerization process.

Authors evaluated the influence of the amount of charge on the volumetric contraction of 3 different resins composed by the video image system (Acuvol). In addition, they evaluated whether the system used is a simple and easy to use system for volumetric shrinkage measurement. After the tests, the authors concluded that, in addition to the amount of load interfering with the percentage of contraction, the acuvol system actually presents itself as a simple and practical method to obtain the percent values of volumetric contraction.

It was evaluated the volumetric contraction of 3 different composite resins in 2 video and image mechanisms to observe if the 2 reproduce the same values. To measure the volumetric contraction of the 15 samples of each of the selected composite resins, 2 video and image mechanisms (Acuvol - Bisco, and a DSA10 Mk2 - DSAS analysis system)

The authors concluded that both devices were presented as an easy method to evaluate the volumetric contraction of composite resin, with no difference between the two.

Another factor that some authors have been researching to evaluate if it influences the polymerization contraction is the type of light used for the photoactivation of the material. In the case of the present study, an LED light source was used.

Study evaluated the polymerization contraction and polymerization tension of photoactivated resins with LED light and halogen light (QTH). To measure the contraction values of the material, the authors used the stress calibration method and the contraction stress was measured using a photoelastic analysis. After statistical evaluation of the results by the ANOVA and Tukey tests, the authors observed that when using the conventional mode of polymerization, there is no difference between the contraction voltage values found in the photopolymerizable resins tested in both light sources used.

However, there is a disagreement among the authors mentioned above: after evaluating the influence of the light unit on the polymerization contraction of different composite resins using LED light, halogen light and a third curing light unit (LCU), the authors observed that the contraction of polymerization in Z100 and Z250 (3M ESPE) resins was lower when using the LED light source when compared to halogen light.

CONCLUSION

Considering that, after a statistical evaluation of the results obtained, no difference was observed between the volumetric contraction of the composite resins when compared within the validity period and with the period of validity exceeded in 6 months, the null hypothesis of the work was rejected.

Therefore, it is concluded that the period of 6 months after the limit of validity, established by the manufacturers, does not alter the free volumetric contraction of the tested composite resins.

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