Thermocycling effect on microhardness of laboratory composite resins

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Abstract
The aim of this study was to evaluate the thermocycling effect on microhardness of laboratory composite resins. 30 disks were fabricated, 5 mm of diameter and 2mm of width, using 3 laboratory resins: G1 (n=10) - RESILAB MASTER (Wilcos-Brasil), G2 (n=10) - Vita VMLC (VITA Zahnfabrik-Germany), and G3 (n=10) - Vita Zeta (VITA Zahnfabrik-Germany). Vickers microhardness (HV) of all specimens was evaluated using a microhardness tester FM-700 (Future Tech- 50 g/10s). The specimens were measured before and after the thermocycling (3.000 times and 12.000 times - 5° /55°C±1). The microhardness values before cycling were (mean±SD): G1: 55.50±4.6; G2: 35.54±2.5; G3: 27.97±1.6.; after 3.000 thermocycles: G1: 55.54±3,9; G2: 29.92±2,73; G3:21.01±1.4 and after 12.000 cycles G1:54.27±3.2; G2: 30.91±1.6. G3: 23.81±0.9. Variance analysis (ANOVA) and Tukey's test was accomplished (p<0,05), the highest microhardness values were observed in G1; G2 and G3 showed reduction of microhardness values. It was concluded that, after thermocycling, the tested laboratory composites resins are susceptible to the decrease of surface microhardness.

Key Words:
composite resins, microhardness, thermocycling

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Introduction

The search for techniques of indirect restorations based on polymers increased due to the evolution of restorative materials and the use of adhesive conservative's procedures[1], just when the ceramic restorations present limitations as abrasiveness, difficult of reparation, brittle and sensitivity in the technique[2]. These materials provided alternative ways for clinicians to overcome some inherent deficiencies of direct composites restorations, including polymerization shrinkage, inadequate polymerization in deep interproximal areas and restoration of proximal contacts and contour[3]. In spite of the similar composition, indirect composite restoration is thought to have superior mechanical properties[4].

The success of any dental restorative material depends upon its physical, chemical and mechanical properties[5-7]; thus, second generation composite resins feature several differences in those terms compared to first generation composite resin because it was magnified the flexural strength, wear resistance, elastic modulus, and polymerization shrinkage. However, fracture, abrasion and discoloration of laboratory processed composite resins are significant problems in clinical use[8-10].

To improve the mechanical properties of laboratory composite resins, inorganic particles are added in its composition which promotes resistance, increases the rigidity, and reduces dimensional changes. When they are warmed and cooled, also reduce the shrinkage, the radiopacity; improve the aesthetic and the manipulation[11]. The organic matrix influences mechanical properties of some materials[10]. Also, the organic filler has an effect on the mechanical characteristics of the material; and the reduction of the matrix volume reduces the shrinkage of polymerization and the process of wear[4].

It demonstrated that the uptake of water by the matrix was related to a reduction in the mechanical properties of the composite resin[12-13]. Under oral conditions the temperature changes induced by eating, drinking and breathing. These changes may cause some failures of the composites resins cohesion because of the thermal expansion differences between the matrix and inorganic particles[14].

Laboratory simulations of clinical service are often performed to analyze dental materials properties, so clinical trials and time consuming are less. Thermocycling is an in vitro process often represented in these simulations; it may vary considerably and, with few exceptions, is always used without reference in vivo observations. Thus, to standard the conditions is necessary to allow comparison among the reports[6,15].

Few studies assessed the influence of thermocycling on the microhardness of composite resin. No reports have been found about the necessary number of thermocycles to simulate the use-time of a material in vivo, so it was proposed that 10000 cycles might represent a one year of service[16]. In front of this, the present study selected two amounts of cycles to be tested, 3000 that is around of what is commonly find on literature and 12000 which is above of that considered to represent one year of use.

The aim of this study was to evaluate the thermocycling influence on the microhardness of indirect composite resins.

Material and Methods

Three laboratory composite resins were selected for the experiment: Resilab Master (Wilcos-Brazil), VITA VM LC and VITA ZETA LC (VITA Zahnfabrik-Germany).

For this study were prepared thirty resin specimens (n=10) which were fabricated using a stainless steel matrix with an internal diameter of 5mm and depth of 3mm.

With a metallic spatula, the laboratory composite resins were condensed into the matrix, in two layers, carrying a initial polymerization for 4 minutes (EDG - LUX-RESILAB MASTER and VITA ZETA LC, *Spectramat* - VITA VM LC). After the condensation of the second layer, a polyester strap was placed on the resin, to obtain a smooth surface. The final polymerization was made according to manufacturer's instructions, as follow:

RESILAB MASTER was processed in a light-curing source with wave length between 400 and 500nm and a maximum temperature that not exceed the 50ºC. In this case the equipment of the first layer polymerization, with the maximum thickness of 1,5 mm was carried out, during 4 min and the final polymerization for 12 min (EDG-LUX-Brazil).

VITA VMLC was processed using the light-curing Spectramat (Ivoclar Vivadent/ Liechtenstein), carrying a final polymerization per 10 minutes. For VITA ZETA LC was used a light-curing source with wave length of 400-500 (EDG-LUX-Brazil), carrying a final polymerization for 10 minutes.

The specimens were embedded in acrylic resin, and the surfaces were polished using from 220 to 600-grits SiC paper on a roating disc at 150 revolutions/min under water cooling (mechanic polishment) (POLI PAN-2/PANAMBRA, Brazil) with a diamond paste of 6, 3 and 0,25µm. After that, they were stored in distilled water 37°C for 24 hours.

The surface of each specimen was evaluated at 0, 3000 and 12000 thermal cycles with baths at 5ºC/55ºC±1 with a dwell time of 30s in each bath by means of Vickers (HV) hardness with a microdurometer FM-700 (Future Tech), with a load of 50 g / 10 seconds. For each surface, three indentations were carried out to obtain the hardness average for each sample.

The microhardness data were submitted to statistical analysis, using a two-way ANOVA and Tukey's test (pd"0.05) it was using the Statistical program Software for Windows (StatSoft, Incorporation, version 5,5, 2000, Tulsa, OK) and Statistix for Windows (Analytical Software, Incorporation, version 8,0, 2003, Tallahase, FL, USA).

Results

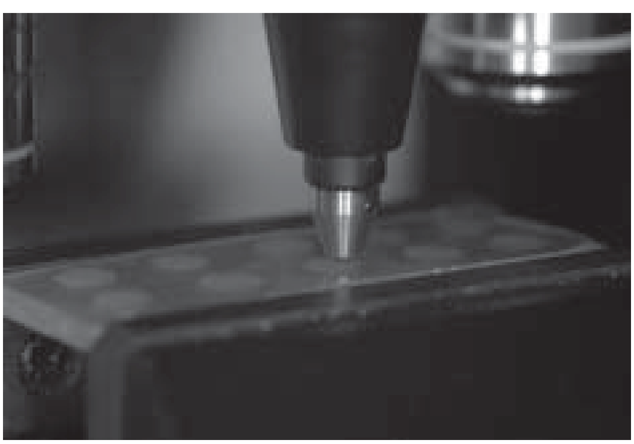
The results have shown microhardness average values for

each type of laboratory composite resin with or without thermocycling (Table 1). It was verified statistically significant of the resin effect (p=0.001), thermocycling effect (p=0.001) and the interaction (p=0.002) between them (Figure 1).
Tukey's test (5%) showed that there are statistically significant differences among resins. In RESILAB MASTER, the thermocycling (3000 and 1200 cycles) did not produce any alteration on microhardness values; while in VITA VM LC and VITA ZETA LC produced a reduction on microhardness values after 3000 cycles. No statistical difference was showed between 3000 and 12000 cycles in all the groups (Table 1).

Table 1: Means and standard deviation (n=10) of the microhardness data (NHV).

	Resins		
Thermocycling	**RESILAB MASTER**	**VITA VM LC**	**VITA ZETA LC**
Without	55.50±4.64 a, A	35.54±2.49 a, B	27.97±1.61 a, C
3000	55.54±3.96 a, A	29.92±2.73 b, C	21.01±1.36 b, D
12000	54.27±3.11 a, A	30.91±1.51 b, C	23.81±0.99 b, D

Means followed by different lower case letters in each column and upper case letters in each row differ significantly at a 5% significance level according to the Tukey's test.



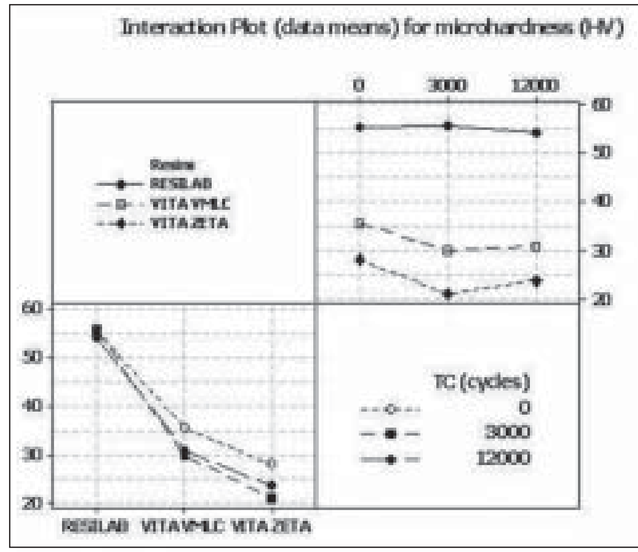


Fig. 1 - *Average graph of experimental conditions*

Discussion

The hardness can be described as the capacity of a material to resist indentations under constant load or abrasion[17]. The microhardness is a non destructive laboratorial test, specifically located, that supplies fundamental data about the material[18]. However, according to Harrison and Draughn[19] and Lappalainem et al.[20], the microhardness does not have relationship between hardness and wear resistance of composites. Kawai[21] has already found the existence of a direct relation between hardness and resistance in resins.
Condon[22] told that properties of composites are influenced by type, size, load particles volume and degree of linking to resinous matrix. The type of matrix and degree that conversion occurs during the polymerization can also influence mechanical properties. Despite indirect resin materials had presented similar mechanical properties, a time theoretically that its compositions are almost identical, being constituted basically of oxygen, aluminum, silicon and barium, according to Mandikos[17].
In this study, all used resins presented ceramic particles in its inorganic matrix. For the three materials, values of microhardness before thermocycling had been superior of 20HV. The RESILAB MASTER and the VITA ZETA LC material micro-hybrid materials, while that VITA VM LC is a microparticle resin.
Microparticle of resins with less percentages of filler particles in its composition, has demonstrated low resistance to fracture, rigidity and fatigue strength, when is comparing to resins with more load[23-26]. A direct relationship between the particle content and the microhardness can be observed, resins RESILAB MASTER, VITA VM LC, VITA ZETA LC approximately present average percentages of inorganics particles of 53%, 48%, 44%, in weight, respectively. It was observed that VITA ZETA LC had lower microhardness values before being submitted to thermo-cycling, that might had occur due its lower content of inorganic fillers.
The materials durability can be affected by the thermocycling[14]. Water absorption affects the mechanical properties of composites for hydrolytic degradation[27]. It can also cause microfracture in the interface between fillers and resin matrix, besides induce superficial stress because of high temperature gradient variation close to the surface[5,12,28]. In our study, we observed a great reduction in the values of microhardness for VITA VM LC and VITA ZETA LC resins with 3000 cycles, while RESILAB MASTER did not show any

reduction in these values after any thermocycling effect (Table1). No statistical difference was showed between 3000 and 12000 cycles in all the groups (Table 1). Thermocycling with 12000 cycles might not have decreased the composite hardness when compared with 3000 cycles, because probably there is a stable level where the composites tested do not suffer alteration for changes in temperature.

The main difficult of this study was to determine the number of cycles that the specimens were submitted, because this value is still not established in literature. The effect of thermocycling on others properties laboratorial composites should be investigated. Standard test conditions such as specimen type, dwell times and storage must also be established so that data from different studies can be compared and analyzed. Finally, fracture toughness, surface roughness and flexural strength of these materials should be investigated in future tests.

According to the methodology developed in this study it is possible to conclude that G1 presented highest values of microhardness when is compared to G2 and G3. Thermocycling was directly related to the reduction of microhardness values for the G2 and G3, with 3000 cycles; however G1 did not showed statistical difference after thermocycling; and no statistical difference was showed between 3000 and 12000 cycles in all the groups.

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