

Original

Effects of critical thermal cycling on the flexural strength of resin composites

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Abstract: We compared flexural strength (FS) in four resin composites before and after three protocols for thermal cycling aging. Four resin composites were evaluated: Enamel Plus Hri, Gradia Direct Posterior, Grandioso, and Grandioso Flow. Sixty specimens ($2 \times 2 \times 25$ mm) were fabricated using a split metallic mold and light-cured for 30 s. The specimens were then randomly divided into four groups and tested using one of the following thermal cycling procedures: 1) storage in deionized water for 24 h (control group), 2) 15,000 cycles, 3) 30,000 cycles, and 4) 45,000 cycles. Each thermal cycling procedure was conducted between 5°C and 55°C, with a dwell time of 30 s. All specimens were subjected to a three-point bending test, to determine FS (0.5 mm/min). "Material" and "thermal aging" were significantly associated with FS ($P < 0.001$). A statistically significant interaction between the two factors was also detected ($P < 0.001$). In the non-aged groups, nanohybrid composites had the highest FS. FS significantly decreased after thermal cycling protocols in all composites tested. Gradia composite exhibited decrease in FS only after 45,000 cycles. In contrast, FS significantly decreased in the Grandioso Flow composite at 15,000 cycles. The

trend in the decrease varied among composites, and the decrement in FS was not proportional to baseline values. (J Oral Sci 57, 137-143, 2015)

Keywords: aging; flexural strength; mechanical properties; resin composite; thermal cycling.

Introduction

Resin composites are widely used for direct and indirect restorations of anterior and posterior teeth because of their esthetic, physical, and mechanical properties (1,2). Substantial improvements have been made in the field of resin composites during the last few decades, so restorative dentists currently have a wide range of materials available for use in clinical practice. However, the long-term success of a composite resin depends on its properties, particularly its resistance and durability in the oral environment (3).

Differences among materials in the monomer system, polymer matrix, filler composition, matrix-filler coupling chemistry, and the concentration, type, size, and distribution of particles may account for variation in mechanical performance and could explain variation in resistance to chemical and mechanical degradation (4,5). Moreover, dynamic conditions of the oral cavity, such as mastication forces, occlusal habits, dietary factors, fluctuations in humidity and temperature, and pH changes in saliva, contribute to uncontrollable factors that affect the longevity of material (6,7).

Composite materials may be damaged by deterioration

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Table 1 Summary of the resin composites tested

Material (Group)	Shade	Composition	Classification	Batch n.	Manufacturer
Enamel Plus HRi (ENA)	UE2	Resin: Diurethandimethacrylate, BisGMA, 1,4- butandioldimethacrylate Filler: surface-treated nano zirconium oxide particles with high refractive index; new type of filling glass with high refractive index (Wt%: 80% - Vol%: 63%)	Nanofilled	2008005653	Micerium, Avegno, Genova, Italy
Gradia Direct Posterior (GRAD)	P-A2	Resin: Methacrylate monomers Filler: Prepolymerized filler; silica; fluoro-alumino-silicate glass average silica and fluoro-alumino-silicate glass particles (Wt%: 77% - Vol%: 65%)	Microfilled resin hybrid composite	1001081	GC Corp., Tokyo, Japan
Grandioso (GRAND)	A2	Resin: BisGMA, BisEMA, TEGDMA Filler: Glass ceramic filler; functionalized silicon dioxide nanoparticles; pigments (iron oxide, titanium dioxide) (Wt%: 89% - Vol%: 73%)	Nanohybrid	1116129	VoCo, Cuxhaven, Germany
Grandioso Flow (GRAFL)	A2	Resin: BisGMA; TEGDMA; HEDMA Filler: Functionalized SiO ₂ nanoparticles with glass ceramic particles (Wt%: 81% - Vol%: 65%)	Nanohybrid	1106503	VoCo; Cuxhaven; Germany

Wt%: Filler content weight; Vol%: Filler volume.

of the matrix and fillers or by mechanical and environmental loads, interfacial debonding, microcracking, and filler particle fracture, which could reduce the survival probability of composite restorations *in vivo* (4,8). In laboratory studies, thermal cycling (TC) is a widely used aging procedure for simulating thermal changes that occur in the oral cavity during eating, drinking, and breathing (9,10). Numerous *in vitro* studies of the mechanical performance of dental composite materials after TC showed that artificial aging protocols accelerate degradation of materials, which significantly degrades mechanical properties (4,8,10-16). In contrast, Smisson et al. found no significant differences in the mechanical properties (flexural strength [FS] and bond strength) of a hybrid resin composite tested under five different TC protocols (17). However, an important limitation of these previous studies is that only one TC protocol was used, which complicates the analysis and comparison of responses of materials to physiological aging. The authors of a recent review concluded that there was no standardized TC protocol to reproduce aging conditions in the oral cavity (10).

The aims of the present *in vitro* study were thus to compare FS in four composite materials and analyze the impact of three different TC protocols on aging behavior. The null hypotheses were that the FS of composites is not significantly influenced by 1) the type of resin composite or 2) TC regimen.

Materials and Methods

Preparation of composite resin specimens

Four light-curing resin composites, differing in resin and filler chemistry and composition, were investigated, namely, Enamel Plus HRi (ENA; Micerium, Avegno, Italy), Gradia Direct Posterior (GRAD; GC Corp., Tokyo, Japan), Grandioso (GRAND; VoCo, Cuxhaven, Germany), and Grandioso Flow (GRAFL; VoCo). Their properties are shown in Table 1.

Sixty specimens of each composite resin were prepared using a stainless steel mold with the dimensions specified by International Organization for Standardization (ISO) standard 4049/2000 (i.e., 25 × 2 × 2 mm) and positioned over a polyester strip (18). Resin composites were packed into the mold, covered by an acrylate strip, and smoothed with a glass slide to achieve a uniform surface finish. Three overlapping sections of the composite were light-cured for 20 s with a curing light (Bluephase C8; 800 mW/cm² output; Ivoclar Vivadent AG, Schaan, Liechtenstein).

After irradiation, excess material was carefully removed with a scalpel blade. Specimen dimensions were confirmed using a digital caliper (series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA). The specimens were then placed into deionized water for 24 h.

Thermal cycling

The specimens were randomly divided into four groups

Table 2 Multiple comparisons for the factor “type of composite”

		Diff of Means	<i>P</i>	95% Confidence Interval	
				Lower Limit	Upper Limit
ENA	GRAFL	39.5278	<0.0001	31.0009	48.0547
	GRAD	15.4333	<0.0001	6.9064	23.9602
	GRAND	-27.7124	<0.0001	-36.2393	-19.1855
GRAFL	ENA	-39.5278	<0.0001	-48.0547	-31.0009
	GRAD	-24.0945	<0.0001	-32.6214	-15.5676
	GRAND	-67.2403	<0.0001	-75.7671	-58.7134
GRAD	ENA	-15.4333	<0.0001	-23.9602	-6.9064
	GRAFL	24.0945	<0.0001	15.5676	32.6214
	GRAND	-43.1457	<0.0001	-51.6726	-34.6189
GRAND	ENA	27.7124	<0.0001	19.1855	36.2393
	GRAFL	67.2403	<0.0001	58.7134	75.7671
	GRAD	43.1457	<0.0001	34.6189	51.6726

Table 3 Multiple comparisons for the factor “thermal aging”

		Diff of Means	<i>P</i>	95% Confidence Interval	
				Lower Limit	Upper Limit
TC 0	TC 15,000	17.5207	<0.0001	8.9939	26.0476
	TC 30,000	44.6043	<0.0001	36.0774	53.1312
	TC 45,000	64.0660	<0.0001	55.5391	72.5929
TC 15,000	TC 0	-17.5207	<0.0001	-26.0476	-8.9939
	TC 30,000	27.0836	<0.0001	18.5567	35.6105
	TC 45,000	46.5453	<0.0001	38.0184	55.0721
TC 30,000	TC 0	-44.6043	<0.0001	-53.1312	-36.0774
	TC 15,000	-27.0836	<0.0001	-35.6105	-18.5567
	TC 45,000	19.4617	<0.0001	10.9348	27.9886
TC 45,000	TC 0	-64.0660	<0.0001	-72.5929	-55.5391
	TC 15,000	-46.5453	<0.0001	-55.0721	-38.0184
	TC 30,000	-19.4617	<0.0001	-27.9886	-10.9348

($n = 15$) according to the TC procedure, as follows: 1) storage in deionized water for 24 h, without further treatment (control group), 2) 15,000 cycles, 3) 30,000 cycles, and 4) 45,000 cycles. Each TC procedure was conducted between 5°C and 55°C, with a dwell time of 30 s and a transfer time of 5 s between temperature baths (LTC100, LAM Technologies Electronic Equipment, Firenze, Italy) (10).

Flexural strength test

A three-point bending test was performed using a computer-controlled universal testing machine (LMT 150, LAM Technologies Electronic Equipment) at a crosshead speed of 0.5 mm/min. The maximum loads were recorded, and FS was calculated in MPa by using the formula $FS = 3FL / (2BH^2)$, where F is the maximum load (in Newtons), L is the distance between supports in mm, B is the width of the specimen in mm, and H is the height in mm.

Statistical analysis

Statistical analysis was performed using the SPSS software package (Advanced Statistical 11.5 software for Windows, SPSS Inc., Chicago, IL, USA). The Shapiro-Wilk test confirmed the normal distribution of the data, and a parametric approach was used to verify the null hypothesis. Interaction between study variables (type of composite and thermal aging) was tested by two-way ANOVA with Tukey’s post-hoc multiple-comparison test. The significance level was set at $\alpha = 0.05$.

Results

Two-way ANOVA showed that “type of composite” (Table 2) and “thermal aging” (Table 3) had a significant effect on FS ($P < 0.001$). A statistically significant interaction between these factors was also detected ($P < 0.001$). Thermal aging had a significant effect on FS in all four composites (Table 4, Fig. 1). In the GRAFL group, FS was significantly lower after 15,000 and after

Table 4 Mean (SD) flexural strength in resin composites, by TC protocol

	ENA	GRAD	GRAND	GRAFL
TC0	124.7 (23.1) ^{A,b}	83.9 (13.9) ^{A,d}	153.3 (26.1) ^{A,a}	94.4 (19.7) ^{A,c}
TC15,000	111.0 (19.1) ^{A,b}	83.0 (10.4) ^{A,c}	135.6 (28.0) ^{A,a}	56.5 (12.1) ^{B,d}
TC30,000	70.1 (15.0) ^{B,b}	70.8 (12.0) ^{A,b}	110.7 (24.6) ^{B,a}	26.2 (8.4) ^{C,c}
TC45,000	51.6 (11.0) ^{C,a}	57.9 (12.8) ^{B,a}	68.5 (16.5) ^{C,a}	22.1 (8.1) ^{C,b}

Means with the same uppercase letter (in a column) or the same lowercase letter (in a row) do not significantly differ according to Tukey's test ($\alpha = 0.05$).

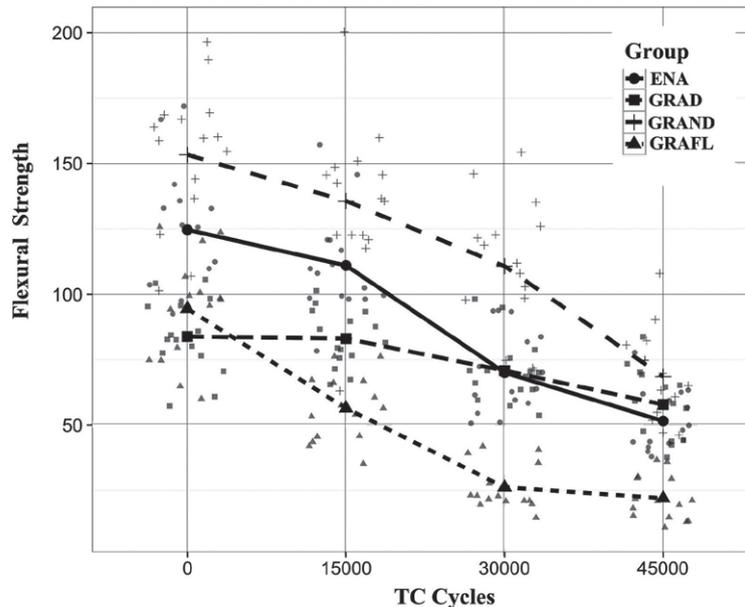


Fig. 1 Flexural strength (MPa) of tested composites, by number of thermal cycling cycles.

30,000 cycles. FS was lower at 30,000 and 45,000 cycles in specimens from the ENA and GRAND groups. In the GRAD specimens, FS was lower only after 45,000 cycles. The type of resin composite significantly affected FS: among the non-aged groups (TC0), FS was highest in the GRAND group and lowest in the GRAD group. Among the three aged groups, the GRAND group maintained the highest FS, while FS was lowest in the GRAFL group (Table 4).

Discussion

FS has been defined as the maximum stress that a material subjected to a bending load can resist before failure. It is regarded as the most important measure of strength for dental materials, as considerable flexural stresses occur during the complex mastication process (4,19). Measurement of long-term FS is essential for a composite resin, as it describes the durability of the material. Degradation of composite resins in the oral environment is attributed

to the resin matrix, filler particles, and hydrolytic instability of the silane coupling agent at the polymer-silica interface (20). The three-point bending test remains the standard for evaluating FS in composites, because of its lower standard deviation and coefficient of variation and the fact that its crack distribution is less complex than that produced by other test designs, such as the biaxial flexural test (21).

The findings of this *in vitro* study suggest that both the evaluated factors (type of composite and thermal aging) have a decisive influence on the FS of resin composites. Thus, both null hypotheses are rejected. Variation in the mechanical properties of dental composites are likely due to differences in the chemical composition of the matrix, fillers, and filler size and distribution (22). Therefore, in the current study we examined composite materials differing in matrix and filler composition, including a nanofilled composite (ENA), a microfilled hybrid composite (GRAD), a nanohybrid composite (GRAND),

and a nanohybrid flow composite (GRAFL).

Many studies have reported that flexural properties are positively correlated with the filler volume of resin composites, i.e., composites with a low filler content (%volume) have low FS (23-26). However, Jiang et al. found that composites with the same matrix and a different filler volume had similar FS (16). Although studies usually define the mechanical behavior of composites in relation to their filler vol%, this variable is more difficult to determine, since it involves previous determination of filler density and must account for variation in filler morphology and molecular composition (21). Therefore, some studies choose filler weight as a variable, to examine the possible correlation with mechanical properties, and have obtained contradictory results. Ilie and Hickel noted that, while the modulus of elasticity increased continuously with filler weight, FS increased only until a filler weight of around 80%, which indicates that increased filler level does not always improve the mechanical properties of resin composites (27). In contrast, Rodrigues et al. reported a weak but significant correlation between FS and filler weight content (21). Adabo et al. found no direct relationship between content of inorganic particles and FS (28). The present results show that FS decreased in the non-aged groups (TC0) in the following manner: GRAND (89% by weight; 73% by volume) > ENA (80% by weight; 63% by volume) > GRAFL (80% by weight; 65% by volume) > GRAD (77% by weight; 65% by volume). Filler volume is therefore not the only factor associated with the mechanical behavior of composites. Instead, the complex chemical composition of composites (matrix, filler size, distribution, filler-resin coupling) is responsible for the different performance characteristics of these materials.

TC combines hydrolytic and thermal degradation and simulates temperature-related breakdown, by repeated sudden changes in temperature (15). TC may affect microcracks at the interface between the filler and polymer matrix. Moreover, repetitive contraction-expansion stresses are responsible for crack propagation along the resin/dentin interface (8,29). The TC regimen varies greatly among experimental studies and seems to be selected by convenience (10). Only a few studies have investigated FS variation in resin composites after TC aging. They used the following single aging protocols (number of cycles; temperatures; dwell time): Hahnel et al. (2 × 3,000 cycles; 5°C-55°C; 5 min), Gohring et al. (3,000 cycles; 5°C-50°C-5°C; 2 min), Jiang et al. (1,000 cycles; 5°C-55°C; 15 s), Janda et al. (5,000 cycles; 5°C-55°C; 15 s), Souza et al. (5,000 cycles; 5°C-55°C ± 1°C; 30 s), Meric and Ruyter (12,000 cycles; 5°C-55°C;

30 s). These studies agreed that artificial aging dramatically decreases FS of the tested materials (4,11-13,15,16). However, studies examining the association between number of cycles and physiological aging in the oral cavity proposed a thermal cycling protocol with a higher number of cycles. Michalesco et al. (30) suggested that 33,000 cycles simulate 1 year of clinical function. Gale and Darvell (31) proposed approximately 10,000 thermal cycles, with four different baths, for 1 year of physiological aging. Stewardson et al. (32) suggested that 500 cycles, as proposed by the ISO protocol, corresponds to less than 2 months in the mouth. Bayne (33) maintained that it is necessary to perform 50,000 thermal cycles to reproduce 1 year of clinical function. Moreau (34) recently assumed that the equivalent of 50 thermal cycles occurs every day in the oral cavity; therefore, a protocol of 20,000 cycles simulates approximately 1 year *in vivo*. These previous studies suggest a need to analyze the relationship between FS and a high number of aging cycles, through application of different TC protocols. The current study proposed to apply different TC protocols, to evaluate FS variation in different resin composites. Furthermore, as compared with previous studies, we used a substantially higher number of thermal cycles: 15,000, 30,000, and 45,000 cycles. Only one previous study used a higher number of thermal cycles: Moreau et al. investigated mechanical durability of nanocomposites after 10⁵ cycles of TC (5°C-60°C; 15 s). The authors concluded that the aging system did not significantly degrade the FS of composites containing amorphous calcium phosphate nanoparticles (34).

The present results showed that only GRAFL specimens exhibited significant decreases in FS after 15,000 and 30,000 cycles. ENA and GRAND composites were significantly affected only after 30,000 and 45,000 cycles. The GRAD composite showed a significant decrease in FS only after 45,000 cycles. These findings show that FS significantly decreased in all four tested materials between TC0 and TC45,000. However, the trend in the decrease varied among composites, and the decrement in FS was not proportional to baseline values.

ISO 4049 recommends a minimum FS of 80 MPa for dental composite materials, to ensure sufficient durability against mastication forces (4). This value might be indicative of time to restoration failure due to fracture *in vivo*. Our results show that the baseline FS values of all four composites tested were higher than the suggested ISO value. After prolonged artificial aging (TC30,000), the GRAND composite exhibited the best mechanical behavior; after 45,000 cycles, however, values had decreased to below the ISO threshold value, as in the

ENA and GRAD groups.

In conclusion, the present results indicate that the chemical composition of resin composites affects FS. At baseline, FS was highest in the nanohybrid composite, followed by the nanofilled, nanohybrid flow, and micro-filled composites, in that order.

Thermal cycling was an important factor and affected the mechanical properties of the resin composites. It caused a progressive nonlinear decrease in FS in all the tested composites. However, the dental composites were not equally affected by artificial aging: a short TC protocol (TC15,000) resulted in a significant decrease in FS only in the flow composite (GRAFL). In the other resin composites tested, FS significantly decreased only after a high number of cycles (TC30,000-45,000).

Conflict of interest

None of the authors had any conflict of interest (including financial, personal, or other relationships with persons or organizations) regarding this study during the 3 years after its initiation.

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