EGYPTIAN DENTAL JOURNAL
www.eda-egypt.org
VOL. 68, 1003:1012, JANUARY, 2022
PRINT ISSN 0070-9484 • ONLINE ISSN 2090-2360
CONSERVATIVE DENTISTRY AND ENDODONTICS
Submit Date : 24-10-2021 • Accept Date : 22-12-2021 • Available online: 30-01-2022 • DOI : 10.21608/edj.2021.102634.1837

EFFECT OF THERMAL AGING ON HARDNESS AND MODULUS OF ELASTICITY OF DIFFERENT DENTAL RESIN COMPOSITES: AN INVITRO STUDY

Weam Douidar [*], Abdallah F. Elsadany [**] *and* Asmaa M. Abdallah[*]

ABSTRACT

Objectives: Nanoindentation technique was used to evaluate the effect of thermal aging on Young's modulus (*E*) and nanohardness (*H*) of bulk-fill and fiber reinforced resin composites.

Materials and methods: Three resin composites that fall into the categories of conventional (GrandioSo, Filtek Z350 XT; Z250), bulk-fill (tetric Evo ceram, flowable SDR Flow) and fiber reinforced (Ever-X posterior, Alert) were evaluated in this study. Ten disc specimens (8 mm × 2 mm) were prepared from each material. The *E* and *H* were determined by a nano-mechanical tester (UMT 1, Bruker, Santa Barbara, CA, USA) equipped with a Berkovich diamond indenter tip both before and after thermocycling 5,000 times in distilled water (5°C-55°C). Data were collected and statistically analyzed using one-way analysis of variance and paired *t*-test.

Results: All materials were significantly different from each other regarding Hardness before and after themalcycling with the highest means for GrandioSo and Alert. It was found that GrandioSo and Alert had the highest modulus values. But no significant differences between Tertic evo ceram, Z250, and Ever X. All of the evaluated composites showed a significant drop in hardness as a result of thermal cycling, while only a minor reduction(insignificant) in elastic modulus was observed.

Conclusion: E and H were significantly increased when the filler content increased. H was significantly affected by thermal cycling. *E* was slightly affected by thermal cycling.

KEYWORDS: Nanohardness, nanoindentation, resin composites, thermal cycling, Young's modulus.

* Lecturer, Department of Operative Dentistry, Faculty of Dentistry, Mansoura University, Mansoura, Egypt.
** Assistance Professor of Dental Biomaterial, Restorative Dental Science Department, Vision Colleges, Riyadh, Saudi Arabia.

INTRODUCTION

Due to the aesthetic characteristics of dental composite resin, it is the preferred material among patients and dentists [1]. To overcome their disadvantages, such as polymerization shrinkage and fracture, attempts are made to improve the physical and mechanical qualities of dental composite resin[2-4]. This needs a detailed examination of their constituents (the resin matrix, the filler, the filler-resin interface) and their effects on material properties. Various research has studied this in attempt to enhance composite properties by changing filler size, amount, or chemistry of the organic matrix [4,5].

Developments in dental composite resin filler and polymer technology have resulted in a wide range of materials with appropriate characteristics for each clinical situation [4]. However, using dental composites in high-stress areas can be challenging for dentists, because bulk fracture is still one of the most common causes of failure [2,6] . Bulk fill composites with improved physical and mechanical properties as well as a higher cure depth (4mm) were introduced to improve the clinical performance of composite resin restorations. They are used in the posterior teeth to withstand higher masticatory stresses, reduce treatment time, and to reduce the risk of air inclusion and moisture contamination [7]. They're also said to improve light transmission and reduce cuspal deflection [7,8].

Fiber reinforcement is another way to improve the physical and mechanical qualities of conventional dental composites while also increasing fracture resistance. This is accomplished by stress transfer from the matrix to the fibers, which was dependent on the length and diameter of these fibers. Garoushi et al. investigated the effect of fiber inclusion in dental composites and discovered that the physical properties of composite materials were significantly improved [9] . For the success of final restorations, it is necessary to have a thorough understanding of the mechanical properties of dental materials. Composite restorative materials' clinical behavior and lifespan are determined by their mechanical characteristics [10], which are influenced not only by their compositions but also by changes in the oral cavity[11] . Water sorption in resin composites, as well as its negative consequences such as hydrolytic deterioration of the resin matrix and matrix-filler interface debonding, has a detrimental impact on the physicomechanical characteristics of these materials and, as a result, their long-term survival[12]. Mechanical parameters such as Young's modulus and hardness can be used to estimate a material's resistance to occlusal forces [13]. Young's modulus is a measure of a material's elastic stiffness. Restorative materials with a greater Young's modulus can withstand deformation and cuspal fracture, particularly in posterior teeth with higher stresses; however, materials with a lower Young's modulus deform more under occlusal forces, resulting in catastrophic fracture[14,15]. As a result, Young's modulus and the reliability of restorative dental materials are linked[16]. The Young's modulus that is most desired is one that is comparable to that of dentin [17].

Hardness is defined as a material's resistance to persistent indentation or penetration [18]. It's used to show how resistant a material is to wear and abrasion when compared to opposing tooth structure or materials [19]. Wear affects restorations on a daily basis, both during use and during cleaning [16]. Surface hardness is important since abrasion is the cause of wear. As a result, one of the most important requirements, particularly in posterior stress-bearing areas, is the hardness of the restorations [20]. Hardness is a mechanical characteristic that should be considered when defining restorative materials because of its link to other physical qualities [21].

Nanoindentation is a precise and accurate technique for determining the local mechanical properties of very small amounts of material, such as hardness and elasticity modulus [22]. Most earlier investigations on hardness testing were done at a microscopic level [23-25]. However, in this study, hardness was assessed at the nanoscale utilizing modern technology and the nanoindentation technique. Several dental researches have suggested that nanoindentation can be used to characterize the mechanical properties of resin composites and tooth structures [21,26].

The purpose of this research was to evaluate the hardness and elastic modulus of fibre reinforced and bulk-fill resin-composites before and after thermal cycling in comparison to conventional resin-composites by nanoindentation. The null hypotheses were: (1) there was no significant difference in nanohardness between the tested resin composites before and after thermal ageing; (2) there was no significant difference in nanoelastic modulus between the tested resin composites before and after thermal ageing.

MATERIALS AND METHODS

Table 1 represents the restorative materials that were evaluated. The restorative materials were handled according to the manufacturer guidelines, and all specimen's preparation procedures were accomplished by one operator.

Half-split stainless-steel round mold with 8 mm diameter and 2 mm thickness [27] was used to make 10 composite specimens of each composite type. Mold was put on a glass slide covered by Mylar strip and separating medium was applied to the mold walls with a brush, then composite material was applied to the mold cavity according each type (e.g., flowable SDR Flow by injecting technique and SureFil SDR with a plastic instrument). After that, glass slide covered with Mylar strip was put on the top of the mold. Curing was carried out from the top and the bottom of the specimens before removal from the mold. Curing was achieved by light-emitting-diode LED curing unit for 20 secs with four overlapping light exposures to cure the entire length of specimen. Wavelength range of LED curing unit was between 430-485 nm and output intensity was at 1200 mW/cm^2.

TABLE (1): Specifications of tested resin composite materials

Commercial Name	Composite Type	Manufacturing	Chemical composition	Filler loading (wt %)
GrandioSo	Conventional Nanohybrid	Voco, Cuxhaven, Germany	Matrix: Bis-GMA, Bis EMA, TEGDMA Filler: glass ceramic fiber, functionalized silicon dioxide nano particles	89%
Ever-X Posterior	fiber reinforced bulk fill	GC, Tokyo, Japan	Matrix: Bis-GMA, TEGDMA,PMMA Filler:short E glass fibers filler, barium glass	74.2%
Filtek™ Z350 XT	Conventional Nanofill	3M ESPE, St Paul,MN,USA	Matrix: Bis-GMA, UDMA, Bis-EMA, TEGDMA Filler: silica nanofiller (5–75 nm), zirconia/silica nanocluster (0.6–1.4 μm)	72.5%
Alert	Condensable Dental Hybrid Composite	Jeneric/Pentron, Wallingford, CT	Bis-GMA, UDMA, TEGDMA, THFMA, Filler: Silica and micrometer scale glass fiber .	84%
Tetric Evo ceram	Nanohybrid bulk fill	Ivoclar Vivadent, Chicago, USA	Matrix: Bis-GMA, UDMA, Bis-EMA Filler: Barium glass	80%
Z250	Conventional Microhybrid	Ivoclar Vivadent, Chicago, USA	Matrix: BIS-GMA, TEGDMA, UDMA, functionalized dimethacrylate. Filler: Zirconia/silica	76%
Flowable SDR Flow	Flowable bulk-fill	DENTSPLY Caulk, Milford, Delaware, USA	Matrix:modified UDMA, TEGDMA, EBPDMA, pigment, photoinitiator, barium and strontium alumino-fluoro-silicate glasses, Silicon Dioxide—Amorphous, Strontium. Aluminosilicate Glass.	68%

After curing of each specimen; the mold was opened and the excess composite was removed. The specimens were polished with Sof-Lex discs (3 MESPE, Seefeld, Germany) in a decreasing order of abrasiveness (Coarse 55m, medium 40m, fine 24m, and ultrafine 8m) using a low-speed hand piece at 4.000-5.000 rpm (standard finishing). To remove any remaining surface debris, the polished surfaces were water-rinsed for 60 seconds with an air-water syringe, and the specimens were cleaned in an ultrasonicator (Power sonic 405, Hwashin Technology Co, Korea) before being stored in distilled water for 24 hours.

At each specimen; five point were selected: one in the middle and the others at a distance of 3mm in the four direction, nanoindentations via a Berkovich diamond indenter tip with a nominal radius of 100 nm was used with a nano-mechanical tester (UMT 1, Bruker, Santa Barbara, CA, USA) to measure nanohardness and nanoelastic moduli of these points. The system was calibrated with a fused silica block with an elastic modulus of 80 GP and a Poisson's ratio of 0.2 to get an accurate indenter area function and ensure instrument compliance. The tests were carried out at a constant temperature of 26 ± 1 °C, with loading and unloading rates of 0.2 mN/s and a dwell time of 10 seconds. 30 mN was chosen as the maximum load.

The composite specimens were then thermocycled 5,000 times between 5°C and 55°C in distilled water. Each temperature had a 15-second dwell period, with a 15-second transfer time between the water baths. This was accomplished using a thermos-cycler device (Model 1100, SD Mechatronik, Feldkirchen-Westerham, Germany). After thermocycling nanohardness and elastic modulus were retested at another five point at least 0.5mm far from previous points.

Data were collected and tabulated and statistically analyzed by an IBM compatible personal computer with SPSS Statistical Package of Social Science version 20 (SPSS Inc. Realesed 2011. IBM SPSS statistics for windows, version 20.0, Armnok, NY: IBM Corp.). Two types of statistical analysis were used:

1) Descriptive statistics were expressed in mean ($\bar{x}$), standard deviation (SD).

2) Analytic statistics: for each mechanical property;

a) One-way ANOVA was used to determine statistical significance between groups and post hock test (Tukey' Kramer) was used for multiple comparisons, if there was significant difference between groups.

b) t-test was used to determine statistical significance between composite before and after thermal aging.

RESULTS

Nanohardness (GPa) and Elastic modulus (GPa) of all tested composites groups means ± standard deviations are presented in Tables (2 and 3) and Figures (1 and 2) respectively.

As shown in table 2 and figure 1, the highest nanohardness (GPa) mean± standard deviation value was reported for GrandioSo (before thermocycling (1.72 ± 0.063), after thermocycling (1.59 ± 0.081)); followed by Alert (before thermocycling (1.6 ± 0.066), after thermocycling (1.51 ± 0.07)); then Tetric Evo ceram (before thermocycling (1.49 ± 0.07), after thermocycling (1.37 ± 0.064)); after that Z250 (before thermocycling (1.32 ± 0.062), after thermocycling (1.24 ± 0.055)); in sequence Ever X Posterior (before thermocycling (1.17 ± 0.051), after thermocycling (0.97 ± 0.076)); then Z350 (before thermocycling (0.95 ± 0.053), after thermocycling (0.89±0.08)). The lowest nano hardness mean value appeared in Flowable SDR Flow (before thermocycling (0.78 ± 0.03), after thermocycling (0.69±0.02)). One-Way ANOVA revealed there was significant difference in nanohardness between different tested composite groups (P=0.000). t-test (table 2) revealed that the nanohardness of all composite groups after thermocycling was markedly

significantly lower than that before thermocycling (P=0.000).

As shown in table 3 and figure 2, the highest Elastic modulus (GPa) mean ± standard deviation value was reported for GrandioSo (before thermocycling (25.9 ± 0.19), after thermocycling (24.6 ± 0.2)); followed by Alert (before thermocycling (24.2 ± 0.21), after thermocycling (23.9 ± 0.188)); then Tetric Evo ceram (before thermocycling (21.12 ± 0.13), after thermocycling (20.65 ± 0.17)); after that Z250 (before thermocycling (19.65 ± 0.124), after thermocycling (19.03 ± 0.18)); in sequence Ever X Posterior (before thermocycling (19.23 ± 0.15), after thermocycling (18.82 ± 0.16)); then Z350 (before thermocycling (16.98 ± 0.094), after thermocycling (15.99 ± 0.14)). The lowest elastic modulus mean value appeared in Flowable SDR Flow (before thermocycling (15.29 ± 0.105), after thermocycling (14.69 ± 0.13)). One-Way ANOVA revealed that there was significant difference in elastic modulus between different composite groups (P=0.000). The modulus of elasticity of Tertic evo ceram was insignificantly higher than that of Z250, which was insignificantly stiffer than Ever X. t- test (table 3) revealed that there was no significant difference in elastic modulus of all composite groups after thermal aging.

TABLE (2): Mean nanohardness and standard deviation for the tested composite before and after themal cycling.

Commercial Name	Nanohardness (GPa) means ± standard deviations		P VALUE
	Before	After	
GrandioSo	1.72 ± 0.063^{aA}	$1.59 \pm .081^{b}$	0.000
Ever-X Posterior	1.17 ± 0.051^{aB}	0.97 ± 0.076^{b}	0.000
Z350	0.95 ± 0.053^{aC}	0.89 ± 0.08^{b}	0.000
Alert	1.6 ± 0.066^{aD}	1.51 ± 0.07^{b}	0.000
Tetric Evo ceram	1.49 ± 0.7^{aE}	1.37 ± 0.064^{b}	0.000
Z250	1.32 ± 0.062^{aF}	1.24 ± 0.055^{b}	0.000
Flowable SDR Flow	0.78 ± 0.03^{aG}	0.69 ± 0.02^{b}	0.000

Means with the different small superscripted letters in the same row and the different capital superscripted letters in the same column demonstrated statistically significant differences ($p \leq 0.05$).

TABLE (3): Mean elastic modulus of elasticity and standard deviation for the tested composite before and after thermal cycling.

Commercial Name	Elastic modulus (GPa) means ± standard deviations		P value
	Before	After	
GrandioSo	25.9 ± 0.19^{aA}	24.6 ± 0.2^{a}	0.153
Ever-X Posterior	19.23 ± 0.15^{aB}	18.82 ± 0.16^{a}	1
Z350	16.98 ± 0.094^{aC}	15.99 ± 0.14^{a}	0.570
Alert	24.2 ± 0.21^{aD}	23.9 ± 0.188^{a}	1
Tetric Evo ceram	21.12 ± 0.13^{aB}	20.65 ± 0.17^{a}	1
Z250	19.65 ± 0.124^{aB}	19.03 ± 0.18^{a}	0.976
Flowable SDR Flow	15.29 ± 0.105^{aE}	14.69 ± 0.13^{a}	0.92

Means with the different small superscripted letters in the same row and the different capital superscripted letters in the same column demonstrated statistically significant differences ($p \leq 0.05$).

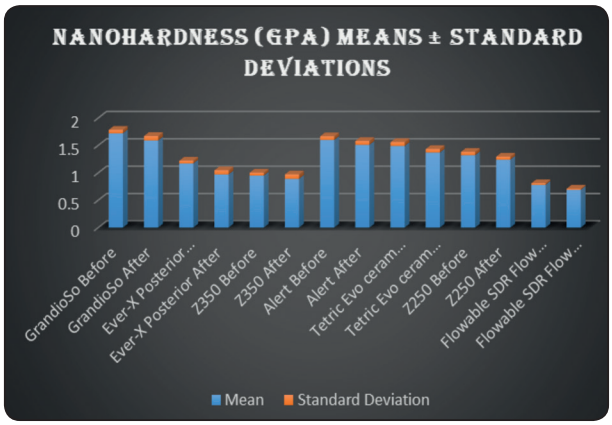

Fig. (1): Mean nanohardness and standard deviation for the tested composite before and after themal cycling

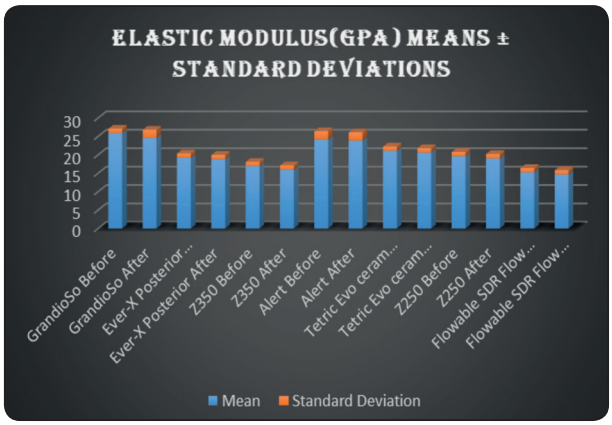

Fig. (2): Mean elastic modulus of elasticity and standard deviation for the tested composite before and after thermal cycling

DISCUSSION

The mechanical properties of composite restorative materials are essential for identifying and anticipating clinical efficacy and long-term effectiveness[13]. Dental treatment success is dictated on a complete understanding of the mechanical properties of dental tissues and materials, as well as biological, chemical, physical, and pathophysiological aspects. The mechanical properties of dental tissues and materials must be assessed before biocompatible dental materials may be developed[28]. Elastic modulus (E) and hardness (H) are the two mechanical properties that are most commonly determined using indentation procedures [29]. The resistance of a material to indentation or penetration is its hardness. It has been used to assess a material's wear resistance when stresses such as occlusal loading are applied. The elastic modulus of a material describes its relative stiffness and its ability to stretch without deformation under constant loading [30]. As a result, studying the modulus of elasticity and hardness is crucial to understanding the clinical behavior of different biomaterials.

Based on analyzing load displacement response during indentation, A nanoindentation method has been developed for assessing the mechanical characteristics of resin composites at the nano scale [12,31]. Nanoindentation is a well-known and widely used method for determining a material's local mechanical properties, such as hardness and Young's modulus. Some features that promote the use the nanoindentation for assessing the mechanical properties of materials includes: small amount of material is required for specimen preparation, no need to image the indentation area, the load and displacements can be continuously recorded during indentation, and the capability of changing the testing factors (e.g., applied load, loading and unloading rates, time, and indenter geometry) [13,32]. Bulk fill resin composite materials are becoming increasingly popular due to their easier procedures for filling posterior restorations in a single increment, as compared to the multi-increment procedures required by conventional resin composites. Indeed, manufacturers and recent scientific papers show that the primary benefits of this restorative procedure are increased cure depth and minimal polymerization shrinkage[33,34]. Therefore, the dental materials used in this study were either bulk fill (fiber reinforced, or nano filled) or conventional dental composite.

Storage in water and thermal cycling are the most popular techniques for aging resin-based materials. Thermocycling is an experimental technique in which thermal changes that are very comparable to actual oral circumstances are replicated. The test samples are immersed in hot and cold distilled

water during the thermocycling process to imitate temperature cycles in specific numbers and durations. The temperature gradient and the water absorbed by dental materials during the thermocycling process affect material characteristics leading to surface damage to composites, and the thermal stress induced between composite constitutions can cause microcracks, according to earlier study [35,36] so, it's critical to assess the mechanical properties of dental materials that have been exposed to the thermocycling process. Based on a study by Morresi et al[37], ten thermocycles are equivalent to a day of clinical service. Thus, 1000 thermocycles selected in this study to simulate 100 days of clinical service.

Filler loading, filler size, and shape all have an impact on mechanical and surface properties of dental composites. Larger filler sizes tend to give stiffer materials, and irregular filler geometries are more probably result in composites with improved mechanical properties[38]. Filler (loading, size, form, and distribution) and organic matrix all have a major effect on hardness[39,40]. The degree of conversion is also said to have a direct impact on hardness[41]. Furthermore, the increased contact surface area among the nanofillers and the resin matrix improves the hardness of the materials[42].

The current study showed significant difference between all tested materials regarding nano hardness either before or after aging. The mean values were in the following order: Grandio > Alert > Tetric evo ceram> Z 250 > Ever x post >Z350 > flowable SDR. In current study nanohardness, values of composite were highly related to composite's filler loading which are Grandio (89%by weight) > Alert (84%) > Tetric evo ceram (80%) > Z 250 (76%)> Ever x post (74.2)>Z350 XT (72.5) > flowable SDR (68 %). These findings are in agreement with prior research, which found that the filler content had a considerable impact on the material's mechanical properties[43]. Therefore, the first hypothesis was accepted.

Thermocycling can have an impact on the material's durability[44]. Water absorption degrades composites leading to microfracture at the interface between the fillers and the resin matrix, as well as causing superficial stress due to high temperature gradients near the surface)45(. In our study, we observed a significant reduction in the values of nanohardness after thermal cycling for each restorative material. This result is in accordance with Fan et al. [46].

The degradation effect of thermocycling on resin composite nanohardness is thought to be caused by the softening of the hydrophilic monomer in the resin matrix due to heat and water, followed by the expansion of polymer chains and a decrease in friction forces between the polymer chains. Furthermore, dental composites can be degraded by hydrolysis of the siloxane bond, resulting in the loss of filler particles [47].

It was found that GrandioSo and Alert had the highest modulus values. The modulus of elasticity of Tertic evo ceram was insignificantly higher than that of Z250, which was insignificantly stiffer than Ever X. The reinforced filler particles, which provided the essential strength to the composite materials, were responsible for the difference in modulus of elasticity. Our findings support those of El-Safty [13] and others[48], who found a positive correlation between stiffness and filler content in dental composites.

The results obtained in this study indicated that thermal cycling revealed insignificant reduction in Elastic modulus for tested materials. This result may be explained by the elastic modulus is a primary property that directly proportional to inter-atomic or inter molecular forces of the material and not affected by the low temperature range used during heating cycle (5–55 ◦C) [49].

There are arguments over the impact of water aging on the mechanical properties of restorative composite materials, such as E & H. De Moraes et al. [50] who reported that after 6 months of water

storage, the elasticity modulus and hardness of resin composites were decreased. However, Yap et al. [15] found no differences in the modulus of elasticity or hardness of several resin composites after 30 days in water. Another study [51] found that storing composites in water increased the elastic modulus, whilst another [52] found that it decreased the elastic modulus. Other studies concluded; no change in Young's modulus of resin composites after water storage at room temperature [53,54]. Varied compositions of the examined composite materials and different testing conditions (storage times, storage mediums, and temperature gradients) may be responsible for this controversy.

CONCLUSIONS

1. Increasing filler content improves the mechanical properties of dental composites (elastic modulus and nanohardness).
2. Thermal cycling (5-55^0) has detrimental effects on nanohardness of dental composites.
3. Thermal cycling (5-55^0) has little effect on Elastic modulus of dental composites.

REFERENCE

1. Pallesen U, van Dijken JW, Halken J, Hallonsten AL, Höigaard R. Longevity of posterior resin composite restorations in permanent teeth in Public Dental Health Service: a prospective 8 years follow up. J Dent 2013; 41:297-306.
2. Demarco FF, Corrêa MB, Cenci MS, Moraes RR, Opdam NJ. Longevity of posterior composite restorations: not only a matter of materials. Dent Mater 2012; 28:87-101.
3. Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. Dent Mater 2005; 21:9-20.
4. Cramer NB, Stansbury JW, Bowman CN. Recent advances and developments in composite dental restorative materials. J Dent Res 2011; 90:402-416.
5. Ferracane JL. Resin composite-state of the art. Dent Mater 2011;27:29-38.
6. Da Rosa Rodolpho PA, Donassollo TA, Cenci MS, Loguércio AD, Moraes RR, Bronkhorst EM, Opdam NJ, Demarco FF. 22-Year clinical evaluation of the performance of two posterior composites with different filler characteristics. Dent Mater 2011; 27:955-963.
7. Walter R. Critical appraisal: bulk-fill flowable composite resins. J Esthet Restor Dent 2013; 25:72-76.
8. Moorthy A, Hogg CH, Dowling AH, Grufferty BF, Benetti AR, Fleming GJ. Cuspal deflection and microleakage in premolar teeth restored with bulk-fill flowable resin based composite base materials. J Dent 2012; 40:500-505.
9. Garoushi S, Säilynoja E, Vallittu PK, Lassila L. Physical properties and depth of cure of a new short fiber reinforced composite. Dent Mater 2013; 29:835-841.
10. Mohamad D, Young RJ, Mann AB, Watts DC. Post-polymerization of dental resin composite evaluated with nanoindentation and micro-Raman spectroscopy. Archives Orofacial Sci 2007; 2:26–31.
11. Martos J, Osinaga PW, de Olivera E, de Casto LA. Hydrolytic degradation of composite resins: effects on the microhardness. J Mater Res 2003; 6:599–604.
12. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer network. Dent Mater 2006; 22:211–222.
13. El-Safty S, Akhtar R, Silikas N, Watts DC. Nanomechanical properties of dental rein-composites. Dent Mater 2012; 28:1292–1300.
14. Lamberchts P, Braem M, Vanherle G. Evaluation of clinical performance of posterior composite resins and dentin adhesives. J Oper Dent 1987; 12:53–78.
15. Yap AUJ, Wang X, Wu X, Chung SM. Comparative hardness and modulus of tooth-colored restoratives: a depth-sensing microindentation study. Biomaterials 2004; 25:2179–2185.
16. Braem M, Lamberchts P, van Doren V, Vanherle G. The impact of composite structure on its elastic response. J Dent Res 1986; 65:648–653.
17. Nakayama WT, Hall DR, Grenoble DE, Katz JL. Elastic properties of dental resin restorative materials. J Dent Res 1974; 53:1121–1126.
18. Anusavis KJ. Phillips'science of dental materials. 12th ed. Philadelphia, PA: Saunders; 2013. pp. 63–64.
19. Wassell RW, McCabe JF, Walls AWG. Subsurface deformation associated with hardness measurements of composites. Dent Mater 1992; 8:218–223.

20. Moraes LGP, Rocha RS, Menrgazzo LM, Araujo EB, Yukimitu K, Moraes JCS. Infrared spectroscopy: a tool for determination of the degree of conversion in dental composites. J Appl Oral Sci 2008; 16:145–149.

21. Willems G, Celis JP, Lambrechts P, Braem M, Vanherle G. Hardness and Young's modulus determined by nanoindentation technique of filler particles of dental restorative materials compared with human enamel. J Biomed Mater Res 1993; 27:747–755.

22. Oliver WC, Pharr GM. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiment. J Mater Res 1992; 7:1564–1583.

23. Lazarchik DA, Hammond BD, Sikes CL, Looney SW, Rueggeberg FA. Hardness comparison of bulk-filled/transtooth and incremental-filled/occlusally irradiated composite resins. J Prosthet Dent 2007; 98:129–140.

24. Mousavinasab SM, Meyers I. Comparison of depth of cure, hardness and heat generation of LED and high intensity QTH light sources. Eur J Dent 2011; 5:299–304.

25. Galvao MR, Caldas SG, Bagnato VS, Rastelli AN, Andrade MF. Evaluation of degree of conversion and hardness of dental composites photoactivated with different light guide tips. Eur J Dent 2013; 7:86–93.

26. Xu HHK, Smith DT, Schumacher GE, Eichmiller FC, Antonucci JM. Indentation modulus and hardness of whisker-reinforced heat-cured dental resin composites. Dent Mater 2000; 16:248–254.

27. Standard test method for microhardness of materials". American Society for Testing and Materials ASTM, Annual Book of Standards, v. 3; 1999.

28. McCabe JF and Walls AWG, Applied Dental Materials, Blackwell Publishing, Malden, Mass, USA, 2008.

29. Oliver WC and Pharr GM. Measurement of hardness and elastic modulus by instrumented indentation: advances in understanding and refinements to methodology. J Mater Res 2004:19;3–20.

30. Sabbagh J, Vreven J, and G. Leloup, "Dynamic and static moduli of elasticity of resin-based materials. Dent Mater 2002: 18; 64–71.

31. Drummond JL. Nanoindentation of dental composites. J Biomed Mater Res Part B Appl Biomater 2006; 78:27–34.

32. Oyen ML, Cook RF. A practical guide for analysis of nanoindentation data. J Mech Behavior Biomed Res 2009; 2:396–407.

33. Nicoleta I, Andreas K, Jürgen D. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin-based composites J Dent 2013: 41;695–702.

34. Julian G, William MP, Julie V, Joseph S, Jacques D, Gaetane L. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent 2014: 42;993–1000

35. Curtis AR, Shortall AC, Marquis PM, Palin WM. Water uptake and strength characteristics of a nanofilled resin based composite. J Dent 2008; 36: 186-193

36. Baracco B, Fuentes V, Garrido MA, González-López S & Ceballos L. Effect of thermal aging on the tensile bond strength at reduced areas of seven current adhesives. Odontology; 2013: 101: 177–185.

37. Morresi AL, D'Amario M, Capogreco M, et al. Thermal cycling for restorative materials: does a standardized protocol exist in laboratory testing? A literature review. J Mech Behav Biomed Mater 2014; 29: 295-308

38. Masouras K, Akhtar R, Watts DC, Silikas N. Effect of filler size and shape on local nanoindentation modulus of resin composites. J Mater Sci Mater Med 2008; 19:3561–6.

39. Bayindir YZ, Yildiz M, Bayindir F. The effect of soft-start polymerization on surface hardness of two packable composites. Dent Mater J 2003;22:610–616.

40. Ildy N, Bayindir YZ, Erdem V. Effect of three different acidic beverages on surface characteristics of composite resin restorative materials. Mater Res Innovat 2010; 14:385–91.

41. Knobloch L, Kerby RE, Clelland N, Lee J. Hardness and degree of conversion of posterior packable composites. Oper Dent 2004; 29:642–649.

42. Mota EG, Oshima HMS, Junior LHB, Pires LAG, Rosa RS. Evaluation of diametric tensile strength and Knopp microhardness of five nanofilled composites in dentin and enamel shades. Stomatologija 2006; 8:67–69.

43. Nathaniel CL and John OB. Wear of nanofilled dental composites at varying filler concentrations J. Biomed Mater Res B Appl Biomater 2015 ;103:424-9.

44. Kawano F, Ohguri T, Ichikawa T, Matsumoto, N. Influence of thermal cycles in water on flexural strength of laboratory processed composite resin. J Oral Rehabil. 2001; 28: 703-7.44.

45. Pereira SMB, Castilho AA, Marocho SMS, Oliveira KMC, Váquez VZC and Bottino MA. Thermocycling effect on

microhardness of laboratory composite resins. Braz J Oral Sci. 6:1372-75.

46. Fan H, Gan X, Liu Y, Xhu Z, Yu H. The nanomechanical and tribological properties of restorative dental composites after exposure in different types of media. J Nanomaterials 2014; 2014:759038.

47. Karimzadeh A, Ayatollahi MR, Shirazi HA. Mechanical properties of a dental nano-composite in moist media determined by nano-scale measurement. Int J Mater Mech Manufact 2014; 2:67–72.

48. Chung KH. The relationship between composition and properties of posterior resin composites. J Dent Res 1990; 69:852– 856.

49. Craig R, Sakaguchi RL and Powers JM. Restorative Dental Materials/ Fundamentals of Materials Science. 13th ed; 2012; Page; 40.

50. De Moraes RR, Marimon JLM, Jochims Schneider LF, Sinhoreti MAC, Correr-Sobrinho L, Bueno M. Effects of 6 months of aging in water on hardness and surface roughness of two microhybrid dental composites. Journal of Prosthodontics. 2008:17:323-26.

51. Sabbagh J, Vreven J, Leloup G. Dynamic and static module of elasticity of resin-based materials. Dent Mater 2002; 18:64–71.

52. Papadogiannis D, Lakes R, Papadogiannis G, Palaghias G, Helvatjoglu-Antoniades M. The effect of temperature on the viscoelastic properties of nano-hybrid composites. Dent Mater 2008; 24:257–266.

53. Chung S, Yap A, Tsai K, Yap F. Elastic modulus of resin-based restorative materials: A microindentation approach. J Biomed Mater Res Part B Appl Biomater 2005; 72:246–253.

54. Janda R, Roulet JF, Latta M, Ruttermann S. The effect of thermocycling on the flexural strength and flexural modulus of modern resin-based filling materials. Dent Mater 2006; 22:1103–8.