Journal of Dental Biomaterials
Shiraz University of Medical Sciences, Shiraz, Iran

Journal of Dental Biomaterials. 2016;3(1)

Original Article

Hertzian Load-bearing Capacity of Hybrid and Nano-hybrid Resin Composites Stored Dry and Wet

Farmani S[a], Orandi S[b], Sookhakiyan M[a], Mese A[c]

[a]Student Research Committee, Shiraz Dental School, Shiraz University of Medical Sciences, Shiraz, Iran
[b]Postgraduate Student , Department of Prosthodontics, School of Dentistry, Shiraz University of Medical Sciences, Shiraz, Iran
[c]Department of Prosthodontics, School of Dentistry, Dicle University, Diyarbakir, Turkey

ARTICLE INFO

Article History:
Received 5 November 2015
Accepted 2 February 2016

Key words:
Hertzian Indentation
Load-bearing Capacity
Resin Composites

Corresponding Author:
Ayse Mese,
Department of Prosthodontics,
School of Dentistry,
DicleUniversity,
Diyarbakir, Turkey
Tel: +90 505 4845819
Email: amese@dicle.edu.tr

Abstract

***Statement of Problem**:* Hertzian indentation test has been proven to be an efficient and reliable alternative upon Vickers hardness test. This method has been used to test dental ceramics, amalgams, glass ionomers and luting cements.There is limited published information about the load-bearing capacity of resin composites using Hertzian indentation test.

Objectives: To investigate the load-bearing capacity of hybrid and nano-hybrid resin composites stored dry or wet up to 30 days, using Hertzian indentation test.

Materials and Methods: Three resin composites were used: two nano-hybrids (Filtek Supreme, and Luna) and one hybrid, (Rok). A total of 108 disc-shaped specimens (1mm thick x 10 mm diameter) were prepared using polyethylene mould. The specimens of each material were randomly divided into 6 groups of 6 (n=6) and stored at 37^{0}C either in distilled water or dry for 1, 7 and 30 days. The specimens were tested using Hertzian jig aligned in the universal testing machine. The specimen was placed on the top of a disc-shaped substrate. The load was applied at the center of each specimen and the load at the first crack was recorded. Data were analyzed by ANOVA, Tukey'sand student's t-test using SPSS version 18.0.

Results: Three-way ANOVA showed a significant interaction between all the factors (p = .0001). The load bearing capacity of almost all materials reduced significantly in the wet condition in comparison with the dry condition (p = .0001). After seven days of immersion in distilled water, Filtek Supreme had significantly lower values than those of Rok and Luna, there was no significant differences between materials in the dry condition.

Conclusions:In contrast to dry condition, the load-bearing capacity of specimens stored in distilled water decreased significantly over the 30 days of immersion. The load bearing capacity of nano-hybrid composites tested in this study was shown to be comparable with that of the hybrid composite.

Cite this article as: Farmani S, Orandi S, Sookhakiyan M, Mese A.Hertzian Load-bearing Capacity of Hybrid and Nano-hybrid Resin Composites Stored Dry and Wet. J Dent Biomater, 2016;3(1):162-168.

Introduction

Increasing demands for cosmetic dentistry and new developments in resin composites and adhesives have guided many dentists to use these materials, instead of amalgam, to restore posterior teeth. Over the last two decades, much research has been conducted to enhance the aesthetics and strength of resin composites.

Dental composites are composed of a polymeric matrix, reinforcing fillers, silane coupling agent for binding the filler to the matrix, and chemicals that stimulate or control the polymerization reaction [1,2]. Since their introduction, filler particle sizes incorporated in the resin matrix have reduced from the traditional to the nano particles to improve the mechanical properties of resin composites [3]. Today resin composites are mostly classified as hybrid, micro-hybrid, nano-hybrid, and nanoFill [3]. The particle sizes are 0.04-5 µm in hybrid, 0.04-1 µm in micro-hybrid, 5 - 75 nm in nano-hybrid, and 0.6 µm to 1.4 µm in nanofill composites [4].

It is believed that there are no factual differences between particle sizes of micro-hybrid and nano-hybrid composites. The logic behind this claim is that adding more commercialised nanoparticles and nanoclusters bounded in the resin matrix of micro-hybrid composites results in an optimized nano-hybrid with possible pre-polymerized resin fillers [5]. Therefore, these two types of composites should have the same load bearing capacity under the Hertzian indentation test.

Immersion of resin composites in the solution in laboratory over the period of time simulates the oral environment and is known to be responsible for the degradation of the materials, leading to loss of their mechanical properties [6-8]. Some studies reported that water storage led to a decrease in the elastic modulus [9], flexure strength [10], tensile strength [11], and fracture toughness of composites [12]. On the other hand, some others have shown no change or an increase in flexure strength [13] and fracture toughness [14] after aging in distilled water.

The Hertzian indentation in which a spherical indenter is used to apply a load to a flat surface has been proven to be a simple and clinically-relevant test method [15]. This method has been employed to test the dental ceramics [16], dental amalgams [17,18], glass ionomer restoratives [19] and resin-based luting cements [20].

Aging in distilled water leads to a significant change of material's strength as water is an existing element in the matrix and also the media of reaction [21]. It has been shown that aging in artificial saliva produces a consistent degradation by means of sorption and solubility of more leachable ions [22]. On the other hand, it is reported that although exposure to the air begins the loss of water, thus shrinking and generating cracks, load bearing capacity is not greatly affected [22]. Wang *et al.* in their study of measuring load-bearing capacity of ceramic-reinforced glass ionomer cement kept wet and dry found that the failure load was relatively stable for air-stored specimens but it showed a significant decrease for wet-stored specimens [22]. Chenglin *et al*. also showed that load-bearing capacity of all-ceramic crowns was reduced due to cement aging in distilled water [23].

Based on limited information about the load-bearing capacity of resin composites stored wet and dry using Hertzian indentation test, the aim of the current study was to investigate the load-bearing capacity of hybrid and nano-hybrid resin composites stored dry or wet up to 30 days, using Hertzian indentation test.

Materials and Methods

Specimen preparation

Three resin composites were tested (Table 1) in two conditions (wet and dry) and three time intervals (1,7 and 30 days). A total of 108 disc-shaped specimens of 1mm ± 0.1 mm thick and 10 mm in diameter were prepared using polyethylene mould. Resin composite was placed in the mould using a plastic instrument, packed gently with hand pressure and pressed between two plastic matrix strips and glass slabs to extrude the excess material. The top glass slab was removed and the resin composite was cured according to the manufacturer's recommended exposure times using an LED curing light at a wavelength range of 440–480 nm and output of 1500 mW/cm2 (Radii plus; SDI, Melbourne, Vic., Australia). The excess material around the mould was removed by wet grinding both sides of the specimen with 600-grit silicon carbide paper. Then, the specimen was removed from the mould and the thickness of each specimen was adjusted to 1 (± 0.1) mm using a digital caliper (Mitutoyo Company, Japan). The specimens of each material were randomly divided into 6 groups of 6 (n = 6) and stored at 37◦C either in distilled water or dry for 1,7 and 30 days. After each time interval, the specimens were tested as explained below.

Specimen testing

The test set-up and conditions have been previously described by Wang *et al.* [17]. The specimen was placed on the top of a disc-shaped substrate, 5-mm thick ×10-mm diameter (nylon 6,6 [30 % glass fiber-reinforced polyamide]), with an elastic modulus of 10 GPa, (Good Fellow Cambridge, Huntingdon, UK). The jig was aligned to the loading axis of the universal testing machine (Zwick/Roll Z020; Zwick GmbH, Ulm, Germany). The load was applied at the center of each specimen at a cross-head speed of 1 mm/min. It was recorded at the first audible detection of a crack.

Statistical Analysis

Data were analyzed using SPSS version 18.0 (SPSS Inc., Chicago, IL, USA). Three –way ANOVA was used to assess the interaction effects between the three factors. One-way ANOVA, Tukey's and student's t test were employed for sub-group analysis comparing the materials, times and conditions individually. $p < 0.05$ was considered statistically significant.

Table 1: Materials used in the present study

Name	Manufacturer	Type	Matrix/Filler (type, size and vol%)	Lot no./shade
Filtek Supreme XTE	3M /ESPE, St Paul, MN, USA	Nano-hybri resin composite	Resin: Bis-GMA,UDMA ,TEGDMA, PEGDMA, Bis-EMA Filler: combination of nonaggregated 20 nm sillica filler,non aggregated 4-11 nm zirconia filler & aggregated zirconia cluster filler(0.6-10 µm);59 vol%	N564759/A2
Luna	SDI, Vic, Australia	Nano-hybri resin composite	Resin: UDMA, Bis-EMA, TEGDMA Filler :SAS,AS 0.02-2 µm ,200-400nm ;61 vol%	130951T/A2
Rok	SDI, Vic, Australia	Hybrid resin composite	Resin: UDMA, TEGDMA, Bis-EMA Filler: SAS, AS, (0.04 -2.5 µm); 67.7 vol%	140454Z/A3

Bis-GMA=bisphenol a glycidyldimethacrylate,TEGDMA=triethylene glycol dimethacrylate, PEGDMA=Poly(ethylene glycol) dimethacrylate, UDMA = urethane dimethacrylate ,SAS= Strontium alumino silicate, AS= amorphous silica, Bis-EMA= bisphenol a Ethylmethacrylate,

Results

Three way ANOVA showed a significant interaction between the following factors; material ×time, material ×condition, and condition × time (p = .0001). Results of one-way ANOVA and pair comparison are shown in Table 2. The effect of time on materials was condition dependent. Over the time, wet condition significantly decreased the load bearing capacity of almost all materials in comparison with dry condition (p = .0001) except for Luna after 1 day of immersion in dry condition that showed slightly lower values than wet condition. At one day of wet condition, Luna (199 ± 15.6) showed significantly (p = .011) greater values than those of Rok (147.3 ± 37.4), while in the dry condition, there was no significant difference between the three materials.

After 7 days of wet condition, Filtek Supreme (115.1 ± 22.1) had significantly lower values than

Table 2: Means and standard deviations (±) of Hertzian load capacity for all tested materials, times and conditions (n = 6)

Material \ Condition	Wet			Dry		
	1D	7D	30D	1D	7D	30D
Rok	A 147.3 ± 37.4	A 141.± 16.1	AB 115 ± 2.3	A 171.9 ± 13.9	A 212 ± 18.8	A 221 ± 54.9
Filtek.supreme	AB 162.6 ± 11.5	B 115.1 ± 22.1	A 95.4 ± 31.1	A 193.6 ± 20.3	A 214.5 ± 5.4	B 155.3 ± 2.1
Luna	B 199 ± 15.6	A 167 ± 8.5	B 169.3 ± 5.3	A 186.5 ± 6.5	A 207.2 ± 11.1	A 215 ± 7.3

Different upper case shows significant differences in each column.
D=Day

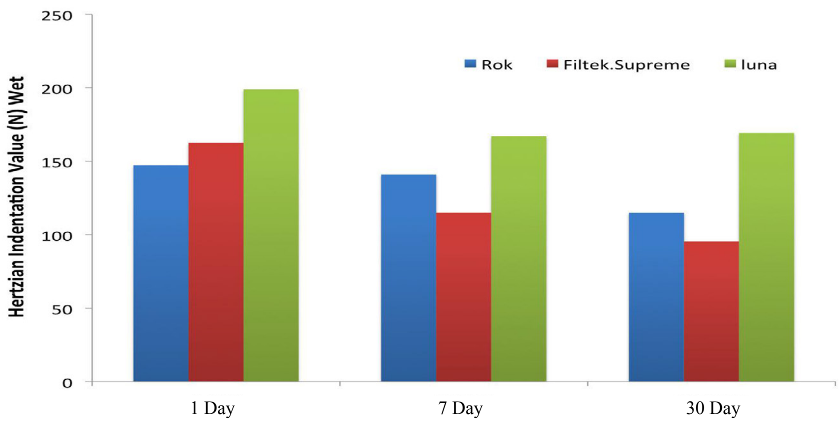

Figure 1: Load-bearing capacity values for specimens stored in wet condition up to 30 day

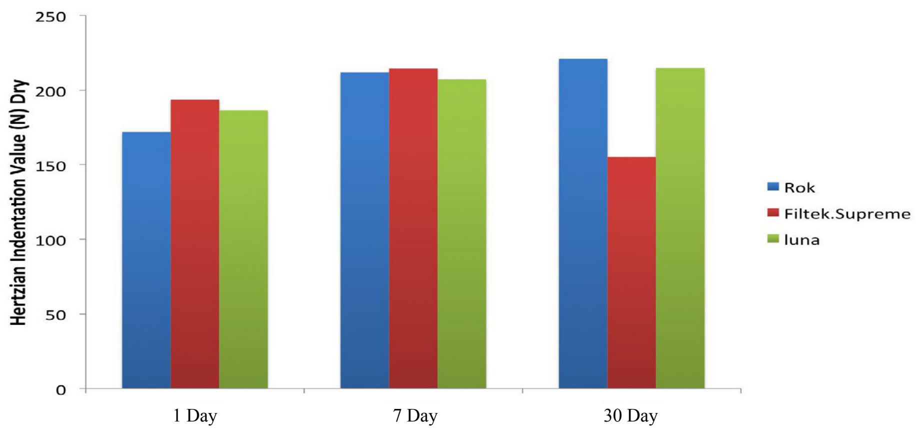

Figure 2: Load-bearing capacity values for specimens stored dry up to 30 day

those of Rok (212 ± 18.8; *p* =.037) and Luna (167 ± 8.5; *p* =.0001), but there was no significant difference between materials in dry condition. At 30 days of wet condition, Luna (169.3 ± 5.3) revealed significantly greater values than those of Filtek Supreme (95.4 ± 31.1; *p* =.0001), and in dry condition Filtek Supreme (155.3 ± 2.1) showed significantly lower values than those of Rok (221 ± 54.9; *p* =.002) and Luna (215 ± 7.3; *p* =.002). The differences between times in each condition are graphically shown in Figure 1 and 2.

Discussion

Results of this study showed that the load bearing capacity of nano-hybrid composites is comparable with or even higher than that of hybrid composite depending on the aging and storage condition. Wet condition significantly decreased the load bearing capacity of almost all materials in comparison with dry condition (*p* =.0001). A possible explanation for these results would be related to water sorption of the materials in the wet environment. Due to water uptake by the polymer, the interface between the resin matrix and the filler may collapse. In addition, water uptake causes a softening effect on the polymer matrix by swelling the network and reducing the frictional forces between the polymer chains [24,25] and reduces the strength of the material.

The load bearing capacity of all materials reduced consistently over the period of time up to 30 days which could be due to plasticization and that the further water uptake may cause cracking or degradation of either the resin matrix or filler/matrix interface [26-28]. Rok, as a hybrid resin composite, showed a lower baseline strength (171.9 ±13.9) compared to nano-hybrid materials but increased significantly after 30 days of being stored dry (221 ± 54.9). In contrast, the wet stored specimens showed a consistent decrease up to 30 days. Long-term water sorption has been shown to be lower for UDMA than for Bis-GMA and TEGDMA due to the presence of hydrophilic ether linkages in TEGDMA [7]. These observations might contribute to the very consistent behavior of Rok during storage.

In this study, two tested nano-hybrid resin composites, Luna and Filtek Supreme, performed differently with the highest values of load bearing capacity for Luna. This performance could be affected by the material's composition such as filler volume percentages, filler type and sizes, prepolymerized resin fillers, type of resin matrix, and the ratio of high molecular monomer (Bis-GMA) to the low molecular ones (UDMA, TEGDMA, Bis-EMA) [26,29-31]. Some studies showed that with increasing urethane content, the solubility values of resin composites decreased; this may imply that the degree of conversion and rate of cure are higher for Urethane based composites compared to BisGMA-based ones [32,33]. This may support the lower load bearing capacity of Filtek Supreme (BisGMA-based composite) than Luna (low molecular-based monomers). It has also been reported that decrease in the filler volume results in greater water sorption [34,35] which may be another reason for lower load bearing capacity of Filltek Supreme (59 vol%) in wet condition compared to Luna (61 vol%). In addition, based on the manufacturer's claim, Filtek Supreme has PEGDMA [Poly (ethylene glycol) dimethacrylate] as part of its resin matrix; this was used to replace a part of the TEGDMA component to moderate the shrinkage in Filtek Supreme XTE restorative.

Hertzian indentation test has been considered an effective simulator of oral loadings with two variables: contact load and indenter radius [15]. A spherical indenter is used to apply the load to a flat surface [17]. Two advantages of Hertzian indentation test upon Vickers hardness test are its ball shape indenter compared to the indenter tip in Vickers hardness and the substrate is used under the specimens [17]. The ball shape indenter prevents the contact stress field produced by the indenter tip, and the substrate acts as the elasticity of the dentin under the restoration to absorb the main forces applied to the specimen [17].

Conclusions

Within the limitations of this study, the following conclusions are drawn. Nano-hybrid composites tested in this study were shown to have comparable or even higher load bearing capacity than that of hybrid composite. In comparison with dry condition, specimens stored in distilled water showed significantly lower load bearing capacity. Time was found to be an effective factor. Further long-term studies are needed to compare the effect of aging on the load bearing capacity of resin composites more objectively.

Acknowledgements

The authors thank the Vice-Chancellor of Shiraz University of Medical Sciences for supporting this research (Grant# 9617).The authors thank Dr. Mehrdad Vossoughi Statistician in Shiraz Dental School for his help with the statistical analysis, and SDI and 3M/ESPE for providing the materials used in this study. The authors also thank Mrs. Marjan Bagheri for her valuable help throughout the experiment.

References

1. Klapdohr S, Moszner N. New inorganic components for dental filling composites. Monatshefte für Chemie. 2005;136:21-45.
2. Wassell R, Walls A, McCabe J. Direct composite inlays versus conventional composite restorations: 5-year follow-up. J Dent. 2000;28:375-382.
3. Wilson N, Wilson M, Wastell D, *et al*. A clinical trial of a visible light cured posterior composite resin restorative material: five-year results. Quintessence Int. 1988;19:675.
4. Sensi LG, Strassler HE, Webley W, *et al*. Direct composite resins. Inside Dentistry. 2007;3:76.
5. Ilie N, Hickel R. Investigations on mechanical behaviour of dental composites. Clin Oral Investig. 2009;13:427-438.
6. Ferracane J, Marker V. Solvent degradation and reduced fracture toughness in aged composites. J Dent Res. 1992;71:13-19.
7. Shin MA, Drummond JL. Evaluation of chemical and mechanical properties of dental composites. J Biomed Mate Res. 1999;48:540-545.
8. Soderholm K-J, Roberts M. Influence of water exposure on the tensile strength of composites. J Den Res. 1990;69:1812-1816.
9. Ferracane JL, Hopkin JK, Condon JR. Properties of heat-treated composites after aging in water. Dent Mater. 1995;11:354-3588.
10. Drummond J, Miescke K. Weibull models for the statistical analysis of dental composite data: aged in physiologic media and cyclic-fatigued. Dent Mater. 1991;7:25-29.
11. Fujishima A. Evaluation of environmental durability in water of light cure composite resins by the direct tensile test. Jpn J Dent Mater. 1988;7:44-61.
12. Mair L, Vowles R. The effect of thermal cycling on the fracture toughness of seven composite restorative materials. Dent Mater. 1989;5:23-26.
13. Drummond J, Savers E. In vitro aging of a heat/pressure-cured composite. Dent Mater. 1993;9:214-216.
14. Pilliar R, Vowles R, Williams D. The effect of environmental aging on the fracture toughness of dental composites. J Dent Res. 1987;66:722-726.
15. Lawn BR, Deng Y, Thompson VP. Use of contact testing in the characterization and design of all-ceramic crownlike layer structures: a review. J Prosthet Dent. 2001;86:495-510.
16. Deng Y, Lawn BR, Lloyd IK. Characterization of damage modes in dental ceramic bilayer structures. J Biomed Mater Res. 2002;63:137-145.
17. Wang Y, Darvell B. Failure mode of dental restorative materials under Hertzian indentation. Dent Mater. 2007;23:1236-1244.
18. Darvell B. Development of strength in dental silver amalgam. Dent Mater. 2012;28:e207-e217.
19. Wang Y, Darvell B. Failure behavior of glass ionomer cement under Hertzian indentation. Dent Mater. 2008;24:1223-1229.
20. Bagheri R, Vojdani M, Mogharabi S, *et al*. Effect of home bleaching on the mechanical properties of resin luting cements using Hertzian indentation test. J Investig Clin Dent. 2015;6:234-239.
21. Crisp S, Lewis B, Wilson A. Characterization of glass-ionomer cements 1. Long term hardness and compressive strength. J Dent. 1976;4:162-166.
22. Wang Y, Darvell B. Hertzian load-bearing capacity of a ceramic-reinforced glass ionomer cement stored wet and dry. Dent Mater. 2009;25:952-955.
23. Lu C, Wang R, Mao S, *et al*. Reduction of load-bearing capacity of all-ceramic crowns due to cement aging. J Mech Behav Biomed Mater. 2013;17:56-65.
24. Ferracane J, Berge H, Condon J. In vitro aging of dental composites in water—effect of degree of conversion, filler volume, and filler/matrix coupling. J Biomed Mater Res. 1998;42:465-472.
25. Ferracane J, Condon J. Degradation of composites caused by accelerated aging. J Dent Res. 1991;70:480.

26. Bagheri R, Tyas MJ, Burrow MF. Comparison of the effect of storage media on hardness and shear punch strength of tooth-coloured restorative materials. Am J Dent. 2007;20:329.
27. Asmussen E. Softening of BISGMA-based polymers by ethanol and by organic acids of plaque. Scand J Dent Res. 1984;92:257-261.
28. Wassell R, McCabe J, Walls A. Subsurface deformation associated with hardness measurements of composites. Dent Mater. 1992;8:218-223.
29. Bagheri R, Fani M, Ghasrodashti AB, *et al*. Effect of a home bleaching agent on the fracture toughness of resin composites, using short rod design. J Dent. 2014;15:74.
30. Ferooz MG, Azadeh N, Berahman N, *et al*. The role of home bleaching agent on the fracture toughness of resin composites using four-point bending test. J Dent Biomater. 2014;1:9-15.
31. Taha NA, Ghanim A, Tavangar MS. A comparison evaluation of mechanical properties of resin modified glass ionomer with resin composite restoratives. J Dent Biomater. 2015;2:47-53.
32. Schmidt C, Ilie N. The effect of aging on the mechanical properties of nanohybrid composites based on new monomer formulations. Clin Oral Investig. 2013;17:251-257.
33. Deepa C, Krishnan VK. Effect of resin matrix ratio, storage medium, and time upon the physical properties of a radiopaque dental composite. J Biomater Appl. 2000;14:296-315.
34. Kim K-H, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthet Dent. 2002;87:642-649.
35. Kim K, Park J, Imai Y, *et al*. Microf racture Mechanisms of Dental Resin Composites Containing Spherically-shaped Filler Particles. J Dent Res. 1994;73:499-504.