



Original Article

Effects of deformation rate variation on biaxial flexural properties of dental resin composites

Naresh Kumar, PhD^a, Muhammad S. Zafar, PhD^{b,*}, Waheed M. Dahri, BDS^c,
Muhammad A. Khan, PhD^d, Zohaib Khurshid, MRes^e and Shariq Najeeb, MSc^f

^a Department of Science of Dental Materials, Dow International Dental College, Dow University of Health Sciences, Karachi, Pakistan

^b Department of Restorative Dentistry, College of Dentistry, Taibah University, Almadinah Almunawwarah, KSA

^c Department of Prosthodontics, Bibi Aseefa Dental College, Shaheed Mohtarma Benazir Bhutto Medical University, Larkana, Pakistan

^d Department of Dental Materials, Khyber College of Dentistry, Peshawar, Pakistan

^e Department of Prosthodontics and Implantology, College of Dentistry, King Faisal University, Al-Ahsa, KSA

^f Department of Restorative Dental Sciences, Al-Farabi Colleges, Riyadh, KSA

Received 15 February 2018; revised 19 April 2018; accepted 23 April 2018; Available online 6 June 2018



المخلص

أهداف البحث: تهدف هذه الدراسة إلى تحديد قوة الثني ثنائي المحور لمواد الراتنج المركبة في معدلات تشوه محددة.

طرق البحث: تم اختيار اثنين من الهجانن الدقيقة من مواد الراتنج المركبة [فلتك زي ١٠٠ ريسنورائف تي ام (زي ١٠٠) وفلتك زي تي ام ٢٥٠ (زي ٢٥٠)]، واثنين من مواد الراتنج المركبة المعبأة عن طريق النانو (فلتك سبريم اكس تي بودي وفلتك عالي الشفافية). كما تم تصنيع عينات على شكل أقراص (١٢ × ١ مم) باستخدام قوالب نايلون مقسمة، من أجل تحديد معامل الثني. وتم اختبار تصنيع عينات على شكل قضبان (٢٥ × ٢ مم) من كل مادة. وتم اختبار العينات لاحتساب قوة الثني ثنائي المحور ومعامله في الظروف الجافة والظروف الرطبة بعد أسبوع و١٣ أسبوعاً و٥٢ أسبوعاً.

النتائج: سُجّلت أعلى قوة ثني ثنائي المحور لـ "زي ٢٥٠" (١٦٢ +/- ١٩ ام بي أي) ويتبعها فلتك عالي الشفافية (١٥٤ +/- ١٦ ام بي أي) و "زي ١٠٠" (١٥٠ +/- ١٨ ام بي أي) وفلتك سبريم اكس تي بودي (١٣٦ +/- ١٨ ام بي أي). وأظهرت الزيادة في معدل التشوه وجود نمط واضح بزيادة قوة الثني ثنائي المحور في المواد. بعد أسبوع واحد من الغمر، كانت قوة ثني ثنائي المحور ١٢٦ +/- ١٨ لـ "زي ١٠٠" و ١٢٤ +/- ١٧ لـ "زي ٢٥٠"، التي كانت أعلى من فلتك سبريم اكس تي بودي ٩٩ +/- ١٦ ام بي أي وفلتك عالي الشفافية

١١٥ +/- ١٩ ام بي أي في ظروف مماثلة. انخفض المعامل الانثنائي للعينات المغمورة لمدة أسبوع واحد بشكل واضح مقارنة بالعينات الجافة؛ "زي ١٠٠" من ١٨.٣ ± ١.٢ ا.ج. بي أي إلى ١٥.٧ ± ٠.٨ ا.ج. بي أي، و"زي ٢٥٠" من ١٦.٧ ± ٠.٨ ا.ج. بي أي إلى ١٣.٣ ± ١.٤ ا.ج. بي أي، وفلتك سبريم اكس تي بودي من ١٣.٧ ± ٠.٦ ا.ج. بي أي إلى ١١.٠ ± ٢.١ ا.ج. بي أي وفلتك عالي الشفافية من ١٢.٧ ± ٢.٣ ا.ج. بي أي إلى ١٠.٤ ± ١.٠ ا.ج. بي أي.

الاستنتاجات: تنخفض قوة ومعدلات انحناء مواد ترميم الأسنان المصنوعة من الراتنج عند غمرها في وسط مائي لحين تشبعها بالماء. وبعد الوصول إلى التوازن فإن الوسط المستخدم للغمر لا يؤثر على المواد التركيبية. إن تأثيرات معدل التشوه على قوة الثني ثنائي المحور في مواد ترميم الأسنان المصنوعة من الراتنج ليست ذات قيمة.

الكلمات المفتاحية: مواد طب الأسنان؛ الخواص الميكانيكية؛ مُعامل؛ طب الأسنان الترميمي

Abstract

Objectives: This study aimed to determine the biaxial flexural strength (BFS) of resin composite materials at distinct deformation rates.

Methods: Two micro-hybrid [Filtek Z100™ Restorative (Z100), Filtek™ Z250 (Z250)] and two nano-filled [Filtek™ Supreme XT Body (FSB), Filtek™ Supreme Translucent (FST)] composite resins were selected. Disc-shaped (12 × 1 mm) specimens were fabricated using nylon split moulds. Bar-shaped specimens (25 × 2 × 2 mm) were fabricated from each material to determine the flexural modulus.

* Corresponding address: Department of Restorative Dentistry, College of Dentistry, Taibah University, P.O. Box 2898, Almadinah Almunawwarah, KSA.

E-mail: drsohail_78@hotmail.com (M.S. Zafar)

Peer review under responsibility of Taibah University.



The specimens were tested for BFS and flexural modulus under dry and wet conditions after 1, 13, and 52 weeks.

Results: The highest BFS was recorded for Z250 (162 ± 19 MPa), followed by FST (154 ± 16 MPa), Z100 (150 ± 18 MPa), and FSB (136 ± 18 MPa). The materials exhibited a clear trend of increase in BFS with deformation rate. Following immersion for 1 week, the BFS was 126 ± 18 MPa for Z100, and 124 ± 17 MPa for Z250, which were higher than those of FSB (99 ± 16 MPa) and FST (115 ± 19 MPa) under comparable conditions. There was a remarkable reduction in the flexural moduli of the specimens immersed for 1 week compared to those of the dry specimens: Z100 (from 18.3 ± 1.2 GPa for dry specimen to 15.7 ± 0.8 GPa after immersion for 1 week), Z250 (from 16.7 ± 0.8 GPa to 13.3 ± 1.4 GPa), FSB (from 13.7 ± 0.6 GPa to 11.0 ± 2.1 GPa) and FST (from 12.7 ± 2.3 GPa to 10.4 ± 1.0 GPa).

Conclusion: This study concludes that the BFS and flexural moduli of resin-based dental restoratives decline when they are immersed in an aqueous medium until saturation with water. However, after equilibrium is established, the immersion medium does not affect the restorative materials further. Variations in deformation rate did not have a significant effect on the BFS of resin-based dental restoratives.

Keywords: Dental materials; Mechanical properties; Modulus; Restorative dentistry

© 2018 The Authors.

Production and hosting by Elsevier Ltd on behalf of Taibah University. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

Introduction

Restorative dental materials form the basis for replacement of tooth structure lost due to disease or injury. Given this significance, a variety of direct aesthetic restorative dental materials has been developed; however, no restorative material possesses ideal properties. For example, silicate cement, the first tooth-coloured restorative material, was introduced in the 1870s; it did not gain much acceptance due to its brittleness, quick erosion, and marginal discoloration.¹ The first polymeric, tooth-coloured polymethyl methacrylate (PMMA)-based composite was introduced in the 1940s. Although, it was relatively better in terms of aesthetics, it exhibited inferior properties such as high polymerisation shrinkage, poor bonding, and mismatch of thermal expansion coefficients.² Subsequently, Rafael L. Bowen used high-molecular weight epoxy and methacrylate derivatives to overcome the deficiencies associated with PMMA-containing composites.³ The advancement by Bowen significantly helped industries develop novel resin-based composites (RBCs) as restorative materials comprising resin and inorganic fillers.

Resin-based composites have been widely accepted in dental practice due to key benefits such as minimal need of tooth

preparation⁴ and aesthetic properties.⁵ However, they exhibit a number of drawbacks including polymerisation shrinkage,⁶ bond strength,^{7,8} and inferior fracture toughness.⁹ Since the development of RBCs, extensive research has been carried out to improve their clinical life with focus being mainly on the development of new monomers¹⁰ and fillers based on particle size, content, and silanisation.¹¹ One of the most important developments in this field during the past decade has been the incorporation of nanotechnology in RBCs.^{12–14} Nanotechnology is the manufacturing and handling of materials and structures in the size range 0.1–100 nm using a number of physical or chemical techniques.¹⁵ It is believed that lower filler particle size and broader particle size distribution enable enhanced filler loading, thereby reducing polymerisation shrinkage and improving mechanical properties such as flexural and tensile strength.^{16,17}

Biaxial flexural strength (BFS) has been employed by researchers to evaluate the mechanical properties of restorative materials.^{18,19} The key benefit of using BFS as a parameter is that tensile stress is mostly applied on the central loading area, eliminating edge failures. The disc-shaped specimens (12×1 mm) used to test the BFS simulate the average width of molars. Additionally, BFS testing is not influenced by specimen geometry and flaw directions.²⁰

Clinically, restorative materials undergo variable stresses in a cyclic pattern due to functional masticatory forces. The stresses encountered vary in several aspects including anatomical features, physiological chewing patterns, diet, and individual variations.²¹ For instance, patients with para-functional habits such as bruxism may exert higher stress on restorations than would physiological forces of mastication alone.²² Nevertheless, the strength of RBCs is determined under a constant deformation rate. The international standard for testing polymer-based restorative and luting materials (ISO 4049) recommends a narrow range of deformation rates (0.5–1.0 mm/min) to test flexural strength.¹⁶ However, a constant deformation rate may reflect the precise behaviour. Moreover, according to ISO 4049, 2000, it is recommended that the specimen be stored in distilled water for 1 week prior to being tested for flexural strength. However, long-term water storage at body temperature is essential in order to simulate real clinical environment and reveal any water-induced degradation effects. This study was based on the hypothesis that the RBCs would exhibit no difference in BFS at varying deformation rates under storage conditions. Thus, this study aimed to determine the BFS of RBCs at distinct deformation rates (0.01, 0.1, 1.0, and 10.0 mm/min) and up to 1 year of water storage regime. In addition, the effect of nanoclusters and micro-hybrid fillers on the BFS of RBCs has been comparatively analysed.

Materials and Methods

Materials

The current study used four resin-based dental restorative composite materials (A3 shade): two micro-hybrid materials [Filtek Z100™ Restorative (Z100) and Filtek™ Z250 (Z250)] and two nano-filled materials [Filtek™ Supreme XT Body (FSB) and Filtek™ Supreme Translucent (FST)]. In terms of

resin chemistry, Z250, FSB, and FST had similar types of resins such as triethyleneglycol dimethacrylate (TEGDMA), bisphenol-A-diglycidyletherdimethacrylate, bisphenol-A-polyethylene glycol dietherdimethacrylate, and urethane dimethacrylate (Table 1). In contrast, Z100 comprised only TEGDMA and bisphenol-A-diglycidyletherdimethacrylate.

The inorganic components of Z100 and Z250 were zirconia silica fillers (84.5 wt.% and 66.0 vol.%, and 84.5 wt.% and 60.0 vol.%, respectively), with particle size in the range 0.01–3.5 μm and averaging 0.6 μm . The FSB had individually dispersed silica nanoparticles (8.0 wt.%, 5–20 nm), and zirconia and silica nanoclusters (71.0 wt.%, 0.6–1.4 μm) constituting total filler content of 79 wt.%. The FST fillers consisted of silica nanoparticles (40.0 wt.%, \sim 75 nm) and silica nanoclusters (30.0 wt.%, \sim 0.6–1.4 μm), constituting filler content of 70.0 wt.% and 57.5 vol.% in total.

Specimen preparation for BFS testing

For each material, 120 disc-shaped (12 \times 1 mm) specimens were synthesised using nylon moulds. A split design was used to facilitate stress-free removal of the specimens. Briefly, RBC paste was weighed accurately (0.24 \pm 0.005 g) using an analytical micro-balance (Mettler AE163, Mettler-Toledo Ltd, Leicester, UK) prior to insertion into the mould. The mould was placed on a strip of cellulose acetate and filled with RBC in a single increment. It was then covered with another strip of cellulose acetate, and the RBC was thumb-pressed.²³ Each specimen was light-cured (23 \pm 2 $^{\circ}\text{C}$, 20 s on each side) using a quartz-tungsten-halogen lamp (Optilux-501, Kerr, Orange, USA). A light guide was used to hold the curing tip to ensure concentric alignment of the curing light. The intensity of the curing light was calculated (\sim 780–880 mW/cm²) using a radiometer (Coltolux C-7900 Coltene/Whaledent Inc, Mahwah, USA) before each set of samples was irradiated. The cellulose strips were discarded after the specimens were removed from the moulds. Each specimen was inspected carefully, and those exhibiting any defects such as presence of air bubbles, voids, or visible cracks were excluded.

Prior to testing, all the specimens (n = 120 for each material) were stored in polystyrene cylindrical containers (30 mL) for variable periods of time under different experimental conditions (dry and wet conditions for 1, 13, and 52 weeks) at 37 \pm 1 $^{\circ}\text{C}$. To provide a reproducible reference solution, deionised distilled water was used for the wet storage of specimens throughout the study.²⁴ The storage medium was refreshed weekly for all the specimens to prevent accumulation of leached constituents from the resin composites.

Specimen preparation for flexural modulus testing

For each material, 10 bar-shaped specimens (25 \times 2 \times 2 mm) were fabricated using nylon split moulds. The material was precisely weighed (0.26 \pm 0.005 g) and filled into the mould in a single increment. The surfaces were covered with transparent cellulose acetate strips (0.1 mm) to reduce oxygen inhibition. After the mould was covered with cellulose acetate strips, the RBC was pressed by applying finger pressure. In accordance with ISO 4049, a curing-light tip of 12 mm and an overlapping curing pattern were used to cure the 25-mm long bar-shaped specimens.¹⁶ Briefly, the central part of each specimen was irradiated for 20 s, followed by irradiation of two overlapping positions for 20 s each to ensure that the entire length of each specimen was cured. The curing, inspection protocol, and storage of the specimens were precisely as described in the previous section.

BFS measurement

The BFS of each specimen was determined using variable deformation rates obtained by adjusting the cross-head speed (0.01, 0.1, 1.0, and 10.0 mm/min). Thirty specimens of each material (n = 30) were tested at each speed setting using a ball-on-ring universal mechanical tester (Instron Ltd, High Wycombe, UK). A ball-shaped indenter (3 mm) was centrally loaded on the disc-shaped sample fixed to the knife-edge support beam (10 mm). The load (N) was recorded at the failure of each specimen. The average thickness of the tested specimen at the point of fracture was determined using a micrometer (Moore & Wright, Sheffield, UK). The BFS (MPa) of the tested specimen was calculated using the equation given below.²⁵

$$\sigma_{\max} = \frac{p}{h^2} \left\{ (1 + \nu) \left[0.485 \times \ln\left(\frac{a}{h}\right) + 0.52 \right] + 0.48 \right\}$$

where σ_{\max} stands for the maximum tensile stress (MPa), a is the radius of the knife-edge support beam (mm), p is the measured value of force to fracture (N), h is the thickness of the sample (mm), and ν is Poisson's ratio (0.25 for all RBCs).²⁶

Flexural modulus measurement

The specimens were loaded centrally using a cylindrical roller (3 mm in diameter) across a support span (20 mm) and a universal mechanical tester (Model5544, Instron Ltd, High Wycombe, UK) at a fixed deformation rate of 1.0 mm/min.

Table 1: Description and composition of resin composite materials.

Material (abbreviation)	Type	Fillers	Filler content	Manufacturer
Filtek Z100 (Z100)	Micro-hybrid	Zirconia, silica; 0.01–3.5 μm	84.5 wt.% 66.0 vol.%	3M ESPE, St. Paul, USA
Filtek Z250 (Z250)	Micro-hybrid	Zirconia, silica; 0.01–3.5 μm	84.5 wt.% 60.0 vol.%	
Filtek supreme body (FSB)	Nano-filled	Silica; 5–20 nm nanoparticle (8.0 wt.%); Zirconia silica; 0.6–1.4 μm nanoclusters (71.0 wt.%)	79.0 wt.% 59.5 vol.%	
Filtek supreme translucent (FST)	Nano-filled	Silica; 75 nm nanoparticles (40.0 wt.%) Silica; 0.6–1.4 μm nanoclusters (30.0 wt.%)	70.0 wt.% 57.5 vol.%	

The dimensions (mm) of the fractured sample were measured using a micrometer (Moore & Wright, Sheffield, UK). The load-deflection curve was plotted based on the load (N) and deflection (mm). The flexural modulus (GPa) was calculated using the equation given below.²⁷

$$(E) = \frac{Fl^3}{4bh^3d}$$

where E denotes the flexural modulus, F denotes load (N), l stands for specimen length (20 mm), b is specimen width (mm), h is specimen thickness (mm), and d stands for deflection (mm).

Scanning electron microscope (SEM) analysis

Biaxial flexural discs were used for image analysis of the surface of the abovementioned commercial dental composites. Prior to testing, the RBC discs were stored for 24 h after curing. At the end of the 24-h period, the discs were mounted on to steel stubs with a fast-setting adhesive. The discs were then sputter-coated with palladium gold alloy (Polaron 5000, UK) for 90 s at 18 mA. The microscopic images were analysed at $\times 2000$ and $\times 100$ magnifications using a scanning electron microscope (SEM, Philip, XL-30, Netherlands) and SEM software. The voltage of the SEM was set at 20 kV.

Statistical analysis

The data was analysed using the computer software SPSS (Version 20; IBM, USA). The BFS data in relation to deformation rates as independent variables were analysed using the two-way analyses of variance (ANOVA). In addition, post-hoc Tukey and one-way ANOVA tests were applied to highlight the differences among the BFS and flexural moduli of the materials in relation to deformation rates and storage regimes. The post-hoc Tukey and one-way ANOVA tests were applied to highlight the differences among the flexural moduli of the materials in relation to storage regimes. For statistical analysis, a value of $P \leq 0.05$ was considered to be an indicator of statistical significance.

Results

The current study compared the BFS and flexural modulus of micro-hybrid and nano-filled dental restorative materials. In addition, the effects of storage medium and variable deformation rates on flexural properties were investigated.

Bi-axial flexure strength (BFS)

The average BFS of the dental composites was influenced by material type and deformation rates when tested before immersion into the medium. Using a deformation rate of 0.01 mm/min, the highest BFS was recorded for Z250 (162 \pm 19 MPa), followed by FST (154 \pm 16 MPa), Z100 (150 \pm 18 MPa), and FSB (136 \pm 18 MPa). There was no remarkable difference between the restorative materials in terms of BFS (Table 2).

Table 2: Bi-flexural strength (MPa) of various resin-based dental restoratives in dry form determined at various deformation rates.

Material	Deformation rate (mm/min)				ANOVA
	0.01	0.1	1.0	10.0	
Z100	150 \pm 18	161 \pm 20	163 \pm 21	165 \pm 21	P = 0.183
Z250	162 \pm 19	169 \pm 22	170 \pm 23	174 \pm 24	P = 0.380
FSB	136 \pm 18	150 \pm 18	158 \pm 19	160 \pm 21	P = 0.201
FST	154 \pm 16	161 \pm 19	163 \pm 20	171 \pm 21	P = 0.072
ANOVA	P = 0.058	P = 0.089	P = 0.073	P = 0.071	

Statistically significant ($P < 0.05$).

The materials exhibited a clear trend of increase in BFS with deformation rate but none exhibited a statistically significant variation (Table 2). For instance, in the case of Z100, the BFS was 150 \pm 18 MPa at a deformation rate of 0.01 mm/min, and this increased to 161 \pm 20 MPa, 163 \pm 21 MPa, and 165 \pm 21 MPa at deformation rates of 0.1, 1.0, and 10 mm/min, respectively. Similar trends were observed in the other three materials. The BFS of the materials aging under wet conditions are shown in Table 3. Regardless of deformation rates, the BFS of all the materials under wet conditions was significantly lower than that of their dry counterparts ($P < 0.05$), suggesting that ingress of water from the storage medium reduces the BFS. The BFS also declined with aging in the storage medium; the values of BFS at the end of 13 and 52 weeks were lower than that at the end of 1 week but the declines were not significant (Table 3).

At immersion duration of 1 week and deformation rate of 0.01 mm/min, the BFS was 126 \pm 18 MPa for Z100, and 124 \pm 17 MPa for Z250, which were higher than that for FSB

Table 3: Bi-flexural strength (MPa) of various resin-based dental restoratives under various storage regimes determined at various deformation rates.

Material	Deformation rate (mm/min) at week 1				ANOVA
	0.01	0.1	1.0	10.0	
Z100	126 \pm 19	132 \pm 21	135 \pm 20	138 \pm 24	P = 0.101
Z250	124 \pm 17	133 \pm 22	137 \pm 21	155 \pm 25	P = 0.100
FSB	99 \pm 16	112 \pm 17	117 \pm 18	118 \pm 17	P = 0.092
FST	115 \pm 19	126 \pm 17	142 \pm 27	141 \pm 32	P = 0.093
ANOVA	P = 0.538	P = 0.060	P = 0.059	P = 0.072	
Deformation rate (mm/min) at week 13					
Z100	114 \pm 17	134 \pm 24	135 \pm 20	134 \pm 25	P = 0.081
Z250	114 \pm 16	135 \pm 14	139 \pm 23	142 \pm 25	P = 0.088
FSB	96 \pm 12	115 \pm 11	117 \pm 16	122 \pm 15	P = 0.074
FST	114 \pm 18	127 \pm 18	128 \pm 21	142 \pm 27	P = 0.059
ANOVA	P = 0.141	P = 0.079	P = 0.069	P = 0.078	
Deformation rate (mm/min) at week 52					
Z100	102 \pm 21	110 \pm 20	113 \pm 22	115 \pm 23	P = 0.121
Z250	105 \pm 17	113 \pm 19	113 \pm 21	114 \pm 22	P = 0.110
FSB	94 \pm 15	98 \pm 15	103 \pm 16	114 \pm 17	P = 0.094
FST	101 \pm 12	110 \pm 21	123 \pm 19	127 \pm 22	P = 0.109
ANOVA	P = 0.062	P = 0.078	P = 0.055	P = 0.069	

Statistically significant ($P < 0.05$).

(99 ± 16 MPa) and FST (115 ± 19) under comparable conditions. When the deformation rate was increased from 0.01 mm/min to 0.1, 1.0, and 10 mm/min, all the materials showed a slight increase in BFS but there were no significant differences among the materials in terms of BFS under variable deformation rates (Table 3). The deformation rates produced similar effects on the specimens stored for 13 and 52 weeks. The only exception was that for all the materials, the BFS values at the end of 13 and 52 weeks were lower than that at the end of 1 week at each deformation rate, but the reduction was not statistically significant. Therefore, under all conditions of deformation rate and aging time, the Z100 and Z250 specimens exhibited higher BFS compared to the FSB and FST specimens (Table 3).

Flexural modulus

A comparative analysis of the dry and wet specimens immersed for 1 week revealed a similar trend in the flexural moduli of all four RBCs. The flexural moduli of the dry specimens were significantly higher ($P < 0.001$) than all three sets of wet specimens (stored under immersion for 1, 13, and 52 weeks), whereas no significant differences ($P > 0.001$) were observed amongst the wet samples themselves (Table 4). In dry form, the flexural moduli of Z100 (18.3 ± 1.2 GPa) and Z250 (16.7 ± 0.8 GPa) were significantly higher ($P < 0.05$) than those of FSB (13.7 ± 0.6 GPa) and FST (12.7 ± 2.3 GPa). The flexural moduli of the samples under wet aging conditions were lower than under dry conditions, and this trend was observed in all the materials (Table 4).

The reduction in the flexural moduli of the different materials was as follows: Z100 (from 18.3 ± 1.2 GPa under dry conditions to 15.7 ± 0.8 GPa following immersion for 1 week), Z250 (from 16.7 ± 0.8 GPa to 13.3 ± 1.4 GPa), FSB (from 13.7 ± 0.6 GPa to 11.0 ± 2.1 GPa), and FST (from 12.7 ± 2.3 GPa to 10.4 ± 1.0 GPa). There was further reduction under wet conditions for 13 and 52 weeks, but these changes were not statistically significant ($P > 0.05$) when compared to the value obtained following immersion for 1 week.

Scanning electron microscope (SEM) analysis

Representative surface SEM images of the four commercial dental composites are displayed in Figure 1a. It is evident from Figure 1a and 1b that the particle size of the fillers is larger in Z250 and Z100 (micro-sized fillers) than in FSB and FST (nano-sized fillers). The SEM images of the two micro-hybrid composites reveal better filler-

monomer interaction in Z250 than in Z100, which is indicative of the higher values of strength in the former compared to the latter. On the other hand, between the two nano-filled composites, FST exhibited better filler-monomer interaction, suggesting higher values of strength, compared to FSB.

Discussion

The current study highlighted the variations in BFS among various RBC dental restorative materials as a function of aging in wet medium and variable deformation rates. However, the difference between the BFS of each material at varying deformation rates was insignificant under all storage regimes, thus validating the initial hypothesis. There is little time for viscous flow in the RBCs at higher deformation rates, which limits stress relief and affects BFS.²⁸ The BFS sensitivity of the RBCs to deformation rates under dry and wet conditions varied widely (Tables 2 and 3) and affected the interpretation of data. Therefore, the evaluation of mechanical properties using a fixed deformation rate may yield different results if the loading parameters were changed.^{28,29}

To explore the effects of filler and resin composition, three commercial RBCs (Z250, FSB, and FST) with different fillers but similar resin matrices, and two materials (Z100 and Z250) with similar fillers but different resin polymers were studied. The overall chemical structure and properties of the resin composites vary significantly depending on their constituents, chemistry, and interaction with each other. Such variables may alter the materials' susceptibility to the oral environment, leading to variations in degradation and fracture. For instance, a number of mechanisms have been reported for the failure of resin composites based on monomer type,^{30,31} degree of monomer conversion,^{32,33} fillers,³⁴ and silanisation of the filler-resin interface.³⁵

A number of studies have revealed mechanical similarities between nano-hybrid and micro-hybrid materials based on filler types, morphologies, and volume fractions.³⁶ On the other hand, researchers have also reported superior^{18,37,38} or inferior^{39,40} properties of micro-hybrid materials compared to nano-filled composites. Such variations are mainly due to the use of different characterisation techniques and parameters.⁴¹ For instance, a number of variables such as resin chemistry and photo-initiators may affect the properties of the materials.³⁶ Therefore, it is vital to determine and control experimental parameters to validate the research data.

The BFS values of all wet specimens were significantly different than those of their dry counterparts at various deformation rates. The wet environment leads to plasticisation of RBCs, thereby causing these differences at different loading rates. Overall, the wet specimens of FSB exhibited low BFS compared with other wet RBCs despite the amount of filler content being comparable among all specimens. The presence of nanoclusters in FSB make it least likely to penetrate through silane and resin and cause failure at lower stresses. In addition, the FSB had a lower depth of cure due to reflection of light by the nanoclusters, and this may have affected its BFS. The BFS values of Z250, FSB, and FST under dry and wet conditions using a fixed cross-head speed (1.0 mm/min) and variable storage protocols have previously been determined.¹⁸ Strength deterioration was greater in the specimens subjected to longer immersion

Table 4: Flexural modulus of various resin-based dental restoratives under various storage regimes.

	Flexural modulus (GPa)			
	Z100	Z250	FSB	FST
Dry	18.3 ± 1.2	16.7 ± 0.8	13.7 ± 0.6	12.7 ± 2.3
1 week wet	15.7 ± 0.8	13.3 ± 1.4	11.0 ± 2.1	10.4 ± 1.0
13 weeks wet	15.5 ± 1.0	14.0 ± 0.7	10.5 ± 0.7	10.7 ± 0.5
52 weeks wet	16.2 ± 1.0	13.2 ± 0.9	11.5 ± 0.9	10.5 ± 0.7

Statistically significant ($P < 0.05$); Deformation rate of 1.0 mm/min.

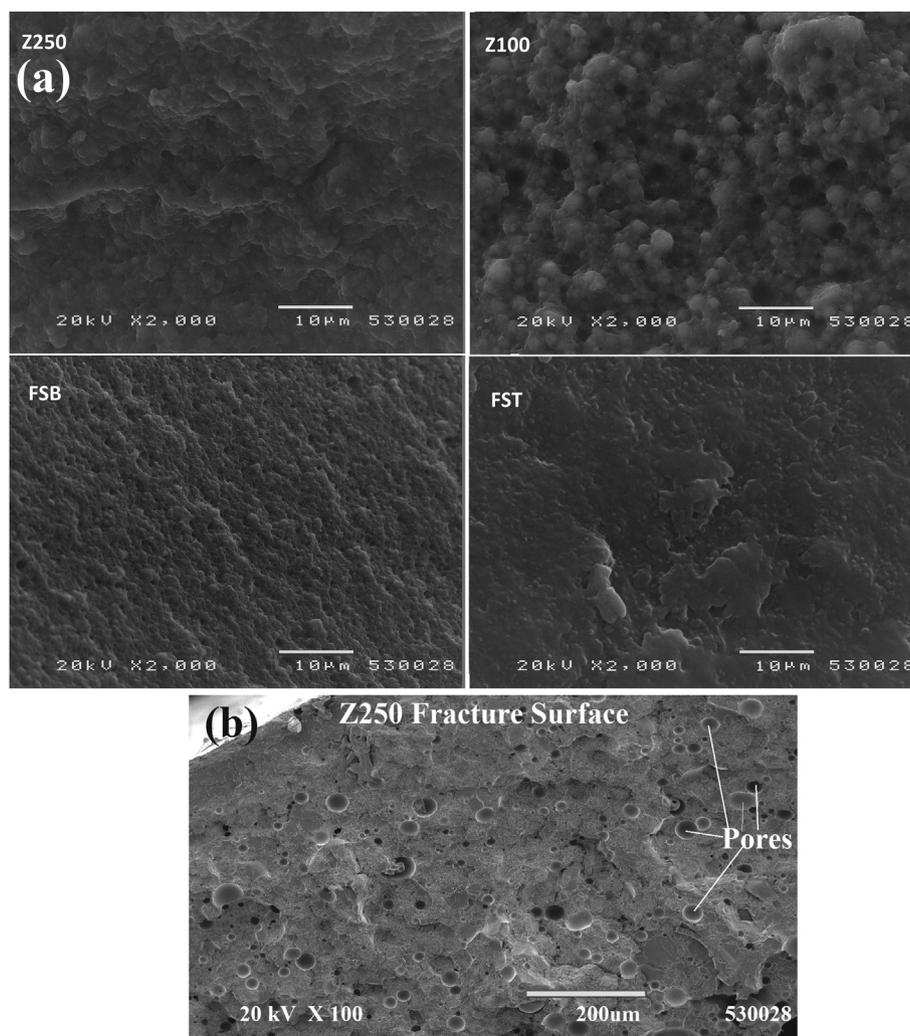


Figure 1: (a) Representative surface SEM images of four different commercial composite materials Z250, Z100, FST and FSB. Their different filler sizes along with filler-monomer interaction could provide insight into the strength and modulus values of the four commercial dental composite materials investigated. (b) SEM image of the fractured surface of Z250 after dry BFS testing. Numerous pores can be seen between the polymer network and filler, and this gives an indication of air entrapment during mixing or poor monomer-filler phase.

time (52 weeks) than in their dry counterparts. The authors also identified greater BFS degradation in RBCs containing nanoclusters, which is in agreement with the results from our study.

The dry specimens of Z100 and Z250 exhibited significantly greater flexural moduli than did those of FSB and FST. The immersed specimens of the RBCs included in this study did not demonstrate any significant variation in flexural moduli (Table 4). The decrease in flexural modulus in an aqueous medium is due to sorption and ingress of water by the specimens upon immersion. Deterioration in mechanical properties (tensile/flexural strength, fracture toughness, and flexural modulus) has been frequently associated with moisture content and aqueous degradation of the ingredients of RBCs.^{33,42,43} Two mechanisms explain the mechanical deterioration of RBCs following immersion in aqueous media: the first is the softening and swelling of the resin due to water sorption, which leads to monomer leaching,^{44,45} and the second is the impact of the failure of resins and

inorganic filler bonds on the RBCs' mechanical properties.^{34,36,46} Due to equilibration of the polymer network, the flexural modulus of RBCs does not change significantly following further immersion.⁴⁷

The flexural modulus of Z100 was the greatest among all the RBCs examined, corresponding to its higher TEGDMA content and conversion of carbon-carbon double bonds.^{33,48} Although Z250, FSB, and FST have identical resin chemistry, FSB and FST exhibited lower flexural moduli than did Z250, and this can be attributed to the lower filler mass fraction in nano-filled materials. Additionally, the flexural moduli of micro-hybrid materials have been found to be significantly higher than those of nano-filled materials; this may be attributed to greater filler content (by weight) in micro-hybrid restorative materials than in nano-filled composites.⁴⁹ A correlation between filler content and flexural modulus has been previously reported,^{17,50} with researchers identifying higher flexural moduli associated with higher filler content. Masouras et al.⁵⁰ identified an increase

in flexural modulus with filler particle size in RBCs. Similarly, in the current study, the reason for greater flexural modulus in both the micro-hybrid RBCs than in the nano-filled RBCs could be the larger filler particle size of the former. It can be assumed that large filler particles could potentially play a significant role in sustaining greater load than small filler particles can, thereby reducing the distortion of polymer chains.

It is clear that all the RBCs, whether micro-hybrid or nano-filled, tested in this study may perform better at varying masticatory rates (loading speed). However, there are certain limitations; this is an *in vitro* study in which clinical oral environment parameters (such as cyclic loading of mastication, pH and temperature fluctuations, chewing patterns, and anatomy) were not simulated. The maximum deformation rate applied was 10 mm/min beyond which the materials could not be tested due to the inertial effects associated with the universal testing machine. The current study tested only commercial RBCs; however, similar studies on experimental RBCs could provide further insight and add to the current basic understanding of these materials.

Conclusions

The difference in the flexural strength of RBCs under varying deformation rates was not statistically significant, and this suggests that resin composite restorative materials can perform at variable masticatory rates. Nano-filled RBCs revealed lower flexural strength compared to micro-hybrid resin composites and, hence, cannot be considered for load-bearing restorations.

Conflict of interest

Authors declare no conflicts of interest.

Authors' contributions

NK and MSZ proposed the study design and literature search; NK, WD and MAK worked towards data acquisition and drafted the majority of the manuscript; ZK and MAK collected, organised, and interpreted the data and wrote a part of the Discussion section; ZK and SN participated in general discussion and critically reviewed the manuscript. All authors have critically reviewed and approved the final draft and are responsible for the content and similarity index of the manuscript.

Acknowledgements

None.

References

- Puckett AD, Fitchie JG, Kirk PC, Gamblin J. Direct composite restorative materials. *Dent Clin N Am* 2007; 51: 659–675.
- Bowen RL. Use of epoxy resins in restorative materials. *J Dent Res* 1956; 35: 360–369.
- Bowen R. Synthesis of a silica-resin direct filling material: progress report. *J Dent Res* 1958; 37: 90.
- Leinfelder KF. A conservative approach to placing posterior composite resin restorations. *J Am Dent Assoc* 1996; 127: 743–748.
- Uchida H, Vaidyanathan J, Viswanadhan T, Vaidyanathan TK. Color stability of dental composites as a function of shade. *J Prosthet Dent* 1998; 79: 372–377.
- Kleverlaan CJ, Feilzer AJ. Polymerization shrinkage and contraction stress of dental resin composites. *Dent Mater* 2005; 21: 1150–1157.
- Rizvi A, Zafar M, Al-Wasifi Y, Fareed W, Khurshid Z. Role of enamel demineralization and remineralization on microtensile bond strength of resin composite. *Eur J Dent* 2016; 10: 376–380.
- Colak H, Ercan E, Hamidi MM. Shear bond strength of bulk-fill and nano-restorative materials to dentin. *Eur J Dent* 2016; 10: 40.
- Ferracane JL, Antonio RC, Matsumoto H. Variables affecting the fracture toughness of dental composites. *J Dent Res* 1987; 66: 1140–1145.
- Lu H, Stansbury JW, Nie J, Berchtold KA, Bowman CN. Development of highly reactive mono-(meth) acrylates as reactive diluents for dimethacrylate-based dental resin systems. *Biomaterials* 2005; 26: 1329–1336.
- Ikejima I, Nomoto R, McCabe JF. Shear punch strength and flexural strength of model composites with varying filler volume fraction, particle size and silanation. *Dent Mater* 2003; 19: 206–211.
- Khurshid Z, Zafar M, Qasim S, Shahab S, Naseem M, AbuReqaiba A. Advances in nanotechnology for restorative dentistry. *Materials* 2015; 8: 717–731.
- Kaur P. Nanocomposites-a step towards improved restorative dentistry. *Indian J Dent Sci* 2011; 28.
- Zafar MS, Khurshid Z, Najeeb S, Zohaib S, Rehman IU. Chapter 26-Therapeutic applications of nanotechnology in dentistry. In: Andronesu E, Grumezescu AM, editors. *Nanostructures for oral medicine*. 1st ed. Elsevier; 2017. pp. 833–862.
- Kirk RE, Othmer DF. *Encyclopedia of chemical technology*, vol. 2; 1953.
- International Standards Organisation. *ISO 4049 Dentistry-Polymer-based filling, restorative and luting materials*. Geneva, Switzerland: International Organization for Standardization; 2000.
- Beun S, Glorieux T, Devaux J, Vreven J, Leloup G. Characterization of nanofilled compared to universal and microfilled composites. *Dent Mater* 2007; 23: 51–59.
- Curtis AR, Palin WM, Fleming GJ, Shortall AC, Marquis PM. The mechanical properties of nanofilled resin-based composites: the impact of dry and wet cyclic pre-loading on bi-axial flexure strength. *Dent Mater* 2009; 25: 188–197.
- Pick B, Meira JB, Driemeier L, Braga RR. A critical view on biaxial and short-beam uniaxial flexural strength tests applied to resin composites using Weibull, fractographic and finite element analyses. *Dent Mater* 2010; 26: 83–90.
- Ban S, Anusavice KJ. Influence of test method on failure stress of brittle dental materials. *J Dent Res* 1990; 69: 1791–1799.
- Koolstra JH. Dynamics of the human masticatory system. *Crit Rev Oral Biol Med* 2002; 13: 366–376.
- Ruyter IE, Øysæd H. Compressive creep of light cured resin based restorative materials. *Acta Odontol Scand* 1982; 40: 319–324.
- Shawkat ES, Shortall AC, Addison O, Palin WM. Oxygen inhibition and incremental layer bond strengths of resin composites. *Dent Mater* 2009; 25: 1338–1346.
- Martin N, Jedyakiewicz NM, Fisher AC. Hygroscopic expansion and solubility of composite restoratives. *Dent Mater* 2003; 19: 77–86.
- Timoshenko SP, Woinowsky-Krieger S. Symmetrical bending of circular plates. In: *Theory of plates and shells*. 2nd ed. New York: McGraw-Hill; 1959.
- Greaves G, Greer A, Lakes R, Rouxel T. Poisson's ratio and modern materials. *Nat Mater* 2011; 10: 823–837.

27. Junior R, Adalberto S, Zanchi CH, Carvalho RVD, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. **Braz Oral Res** 2007; 21: 16–21.
28. Musanje L, Darvell B. Effects of strain rate and temperature on the mechanical properties of resin composites. **Dent Mater** 2004; 20: 750–765.
29. Kumar N. Inconsistency in the strength testing of dental resin-based composites among researchers. **Pak J Med Sci** 2013; 29: 205–210.
30. Rüttermann S, Dluževskaya I, Großsteinbeck C, Raab WH, Janda R. Impact of replacing Bis-GMA and TEGDMA by other commercially available monomers on the properties of resin-based composites. **Dent Mater** 2010; 26: 353–359.
31. Gajewski VE, Pfeifer CS, Frôes-Salgado NR, Boaro LC, Braga RR. Monomers used in resin composites: degree of conversion, mechanical properties and water sorption/solubility. **Braz Dent J** 2012; 23: 508–514.
32. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. **Clin Oral Investig** 2013; 17: 227–235.
33. Aljabo A, Xia W, Liaqat S, Khan M, Knowles J, Ashley P, et al. Conversion, shrinkage, water sorption, flexural strength and modulus of re-mineralizing dental composites. **Dent Mater** 2015; 31: 1279–1289.
34. Ferracane JL. Resin composite—state of the art. **Dent Mater** 2011; 27: 29–38.
35. Halvorson RH, Erickson RL, Davidson CL. The effect of filler and silane content on conversion of resin-based composite. **Dent Mater** 2003; 19: 327–333.
36. Ilie N, Hickel R. Resin composite restorative materials. **Aust Dent J** 2011; 56: 59–66.
37. Najeeb S, Khurshid Z, Zafar MS, Khan AS, Zohaib S, Martí JMN, et al. Modifications in glass ionomer cements: nano-sized fillers and bioactive nanoceramics. **Int J Mol Sci** 2016; 17: 1134.
38. Najeeb S, Khurshid Z, Agwan AS, Zafar MS, Alrahabi M, Qasim SB, et al. Dental applications of nanodiamonds. **Sci Adv Mater** 2016; 8: 2064–2070.
39. Shah M, Ferracane J, Kruzic J. Mechanistic aspects of fatigue crack growth behavior in resin based dental restorative composites. **Dent Mater** 2009; 25: 909–916.
40. Shah M, Ferracane J, Kruzic J. R-curve behavior and toughening mechanisms of resin-based dental composites: effects of hydration and post-cure heat treatment. **Dent Mater** 2009; 25: 760–770.
41. Ilie N, Rencz A, Hickel R. Investigations towards nano-hybrid resin-based composites. **Clin Oral Investig** 2013; 17: 185–193.
42. Sideridou I, Tserki V, Papanastasiou G. Study of water sorption, solubility and modulus of elasticity of light-cured dimethacrylate-based dental resins. **Biomaterials** 2003; 24: 655–665.
43. Costella A, Trochmann J, Oliveira W. Water sorption and diffusion coefficient through an experimental dental resin. **J Mater Sci Mater Med** 2010; 21: 67–72.
44. Örtengren U, Wellendorf H, Karlsson S, Ruyter I. Water sorption and solubility of dental composites and identification of monomers released in an aqueous environment. **J Oral Rehabil** 2001; 28: 1106–1115.
45. Anttila EJ, Krintilä OH, Laurila TK, Lassila LV, Vallittu PK, Hernberg RG. Evaluation of polymerization shrinkage and hygroscopic expansion of fiber-reinforced biocomposites using optical fiber Bragg grating sensors. **Dent Mater** 2008; 24: 1720–1727.
46. Carvalho RM, Manso AP, Geraldeli S, Tay FR, Pashley DH. Durability of bonds and clinical success of adhesive restorations. **Dent Mater** 2012; 28: 72–86.
47. Wei Y, Silikas N, Zhang Z, Watts DC. Hygroscopic dimensional changes of self-adhering and new resin-matrix composites during water sorption/desorption cycles. **Dent Mater** 2011; 27: 259–266.
48. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Degree of conversion and mechanical properties of a BisGMA: TEGDMA composite as a function of the applied radiant exposure. **J Biomed Mater Res B Appl Biomater** 2008; 84: 503–509.
49. Boaro LCC, Gonçalves F, Guimarães TC, Ferracane JL, Versluis A, Braga RR. Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. **Dent Mater** 2010; 26: 1144–1150.
50. Masouras K, Silikas N, Watts DC. Correlation of filler content and elastic properties of resin-composites. **Dent Mater** 2008; 24: 932–939.

How to cite this article: Kumar N, Zafar MS, Dahri WM, Khan MA, Khurshid Z, Najeeb S. Effects of deformation rate variation on biaxial flexural properties of dental resin composites. *J Taibah Univ Med Sc* 2018;13(4):319–326.