

MECHANICAL, CHEMICAL, AND BOND ANALYSIS OF NOVEL EXPERIMENTAL AND COMMERCIAL COMPOSITES

Saad Liaquat¹, Humaira Jabeen¹

¹Department of Dental Materials, Institute of Basic Medical Sciences, Khyber Medical University, Peshawar - Pakistan

ABSTRACT

Objective: To compare and evaluate commercial grade composites with novel experimental dental composites.

Materials and methods: In this experimental study, two experimental and three commercial composites were tested and compared. The manufacturer data and available literature were used to determine the composition of commercial items. Using a four-figure balance and a concentration kit, we calculated mass and volume changes. Instron testing equipment was used to determine biaxial flexural strength (BFS) and modulus. The push-out test was used to examine composite bonding with dentine using various treatments.

Results: Mass change in the distilled water (DW) was 1.3 ± 0.1 %, and in simulated body fluid (SBF) was 1.1 ± 0.1 % for all materials. The volume changes in DW were 2.2 ± 0.2 %, and in Simulated body fluid (SBF) was 1.6 ± 0.2 % for all commercial and experimental materials. The BFS declined by 33 % for commercial, and 39 % for experimental materials in 6 months. In the case of modulus, the highest decline was associated with Vertise flow at 49 %, with the least decline of 23 % with commercial bulk filling materials. The push-out force for composites within ivory dentine suggests maximum bond strength with experimental composite C-4META.

Conclusion: The experimental composite had the potential to compete with commercial composites in mechanical, chemical, and microscopic properties, without compromising the properties.

Keywords: Commercial and control composites, Polymerization shrinkage, Volume changes.

This article may be cited as: Liaquat S, Jabeen H. Mechanical, Chemical and Bond analysis of Novel Experimental and Commercial Composites. *J Med Sci* 2022 ;30(1): 9-13

INTRODUCTION

Dental caries is a bacterial disease which cause cavitation in tooth structure ¹. It affects people of all ages. The acid produced by bacterial fermentation of carbohydrates causes dental caries, which results in the destruction of dental hard tissues ¹. If left untreated, dental caries can result in natural and physical harm. Damaged dental hard tissues are treated with various restorative materials to restore lost tooth structure and function ².

Composites consist of resin monomers and inorganic fillers. The inorganic additives can be in the form of remineralizing or antibacterial fillers. Monomers with a large molecular mass for example bisphenol A-glycidyl methacrylate (Bis-GMA) and urethane dimethacrylate (UDMA) are used to make the composite ground substance ³. Fillers on the other hand are added to increase strength, reduce polymerization shrinkage and heat gen-

eration⁴. A silane coupling agent is used for bonding these two components and to aid filler distribution. Additionally, an initiator and activator are added to control the polymerization process.

Most composites currently in the industry are bulk composites with high filler load. The composites used in this study were Gradia, and Z250. The above specific composites were chosen because of their widespread clinical use. The composites contain (Bis-GMA in Z250, and UDMA in Gradia) different bulk monomers. All these composites were supplied in cartridges that have been pre-mixed. They have an overall filler content of 80 wt % percent, making them highly viscous ^{5,6}. The material properties of these bulk filled composites have been extensively investigated. In terms of effectiveness, Z250 was the strongest commercialized product, with a strength of up to 180 MPa ^{7,8}. Gradia, on the other hand, is regarded as a weak composite with 80 MPa strength in twenty-four hours DW ⁹. Vertise flow was the self-adhesive flowable composite investigated in this study. It is the most recent addition of composites. The composition of these composites is comparable to that of bulk-filled composites, but with less filler content ¹⁰.

The other two bulk-filled composites were experimentally made. One contains the aqueous monomer with HEMA (5 wt %), while other contains the adhesive

Correspondence

Dr. Saad Liaquat

Assistant Professor

Department of Dental Materials, Institute of Basic Medical Sciences, Khyber Medical University, Peshawar

Email: saadliaquat.ibms@kmu.edu.pk

Cell: +92-333-9188098

Date Received: 03-01-2022

Date Revised: 28-02-2022

Date Accepted: 08-03-2022

and de-mineralizing monomer 4-META (5 wt %). The filler component is made entirely of large particles. The goal of these experimental composites is to compare it with commercial composites, and offer standard properties for the creation of novel composites in the future.

MATERIALS AND METHODS

This was an experimental study which was conducted in University College London from 2011-2015. Three commercial composites obtained from the market and two experimental composites were assessed Table 1 table 1 shows the details of the composites.

The manufacturer data and available literature were used to determine the composition of commercial items. Samples for changes in mass and volume were measured in distilled water, and simulated body fluid for up to twelve weeks using a density test and a four-figure scale ($n=6$). Instron testing equipment was used to determine biaxial flexural strength (BFS) and modulus. BFS was carried out on specimen with a thickness of 1 mm and a circumference of 10 mm after 24 hours, one month, three months, and six months of storage in distilled water at 37 degrees centigrade ($n=6$). Dentine blocks were used for the push-out test. Every test was carried out six times in total.

RESULTS

CHEMICAL PROPERTIES

Mass Changes

Figures 1 and 2 shows mass changes in simulated body fluid (SBF) and distilled water (DW) over twelve weeks of commercial as well as experimental composites. In DW, the largest mass change was 1.3 percent, whereas in SBF it was 1.1 percent. All composites mass kept increasing for 6 weeks. It was thought that equilibrium would be established in 6-12 weeks. The bulk changes in Vertise flow composite and C-HEMA were marginally greater as opposed to Gradia, Z250 and C-4META.

Volume Changes

Figures 3 and 4 shows volume changes in DW and SBF for twelve weeks of all composites. The volume change in distilled water was 2.2 percent, while the volume change in SBF was 1.6 percent. In the initial 24 hours, there is a 0.5-1.0 percent increase in volume of all composites. C-HEMA and Vertise flow had greater final volume changes than the other bulk-filled composites. The ultimate equilibrium was thought to be achieved around six and twelve weeks. The volume changes in SBF was lower than that of DW as general.

MECHANICAL PROPERTIES

Biaxial Flexural Strength

Over the course of six months, the biaxial flexural strength of all composites was investigated in DW. Figure 5 shows the flexural strengths.

After twenty-four hours of storage in DW, the initial strengths were evaluated. C-4META, C-HEMA and Z250 all have similar initial strengths of approx. 158, 167, and 168 MPa, respectively. Gradia, and Vertise flow comprised much weaker in comparison to bulk composites. With initial strengths of 125 and 77 MPa, respectively. Z250, C-HEMA, and C-4META all showed a similar trend of strength loss over time. The strength decline at one month was more pronounced than at three and six months. Gradia and Vertise both showed an equivalent decline in strength at each time point.

Young's Modulus

The modulus of all composites was tested in DW at 37°C for six months. The moduli are given in figure 6. After twenty-four hours of storage in DW, the initial moduli were evaluated. Gradia had the lowest modulus (2.4 GPa), followed by Vertise flow (2.9 GPa), Z250 (3.7 GPa), C-HEMA (4 GPa), and C-4META (5.5 GPa).

Push out Adhesion

The ISO-required push-out test was carried out. The usage of the adhesive Ibond has shown to be the most important element in boosting bond strength in this research. Samples comprising of prior Ibond for commercial composites had adhesion strength of 33-43 MPa, in contrast to experimental composites with bond strengths of 42-50 MPa.

Higher overall adhesion strength was observed for both C-4META and flowable Vertise flow composites after acid etching and without the use of Ibond. The bond strength ranged from 26 to 35 MPa for bulk composites (Gradia, Z250, C-4META and C-HEMA) after acid treatment.

Table 1: Details of Commercial and Experimental Composite used.

Z250Filtek™	ESPE 3M, United states	Bulk Composite
Global Company Gradia Direct posterior	Global Company	Bulk Composite
Flow Vertise™	Corporation Kerr	Composite Self-Adhering
C-HEMA	Experimental Composite	Composite Bulk
C-4META	Experimental Composite	Composite Bulk

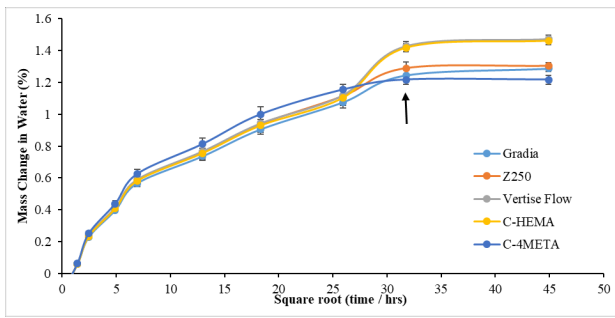


Fig 1: Mass change after storage in DW for twelve weeks, bulk change of both test and industrial composites (error bars are ninety five percent confidence intervals, n=6).

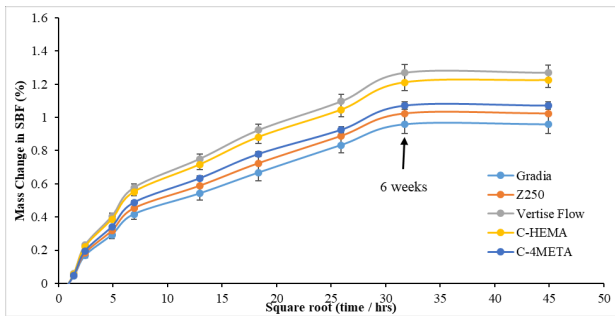


Fig 2: Mass change after storage in SBF for twelve weeks, the bulk of both research and industrial composites changed (error bars represent ninety five percent confidence intervals, n=6).

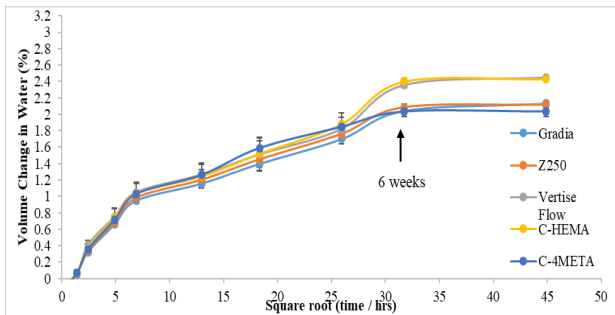


Fig 3: Volume change of both industrial composites plus test after twelve weeks of depot in DW (error bars show ninety five percent confidence intervals, n=6).

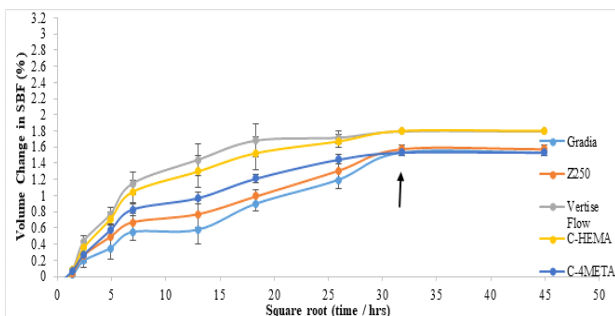


Fig 4: Volume change of both industrial composites and trial following twelve weeks in simulated body fluid (error bars represent ninety five percent confidence intervals, n=6).

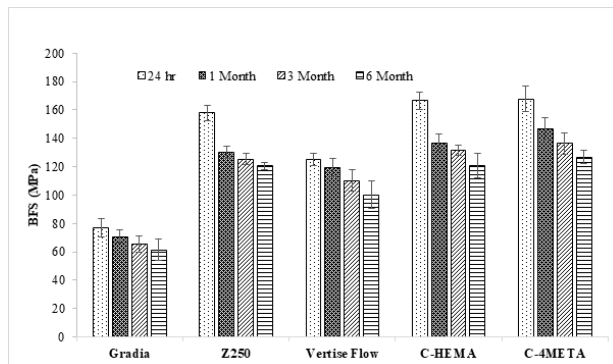


Fig 5: All composites biaxial flexural strength following twenty-four hours, one, three, and six months of storage in Distilled water. The error bars show the ninety five percent confidence interval (n=6).

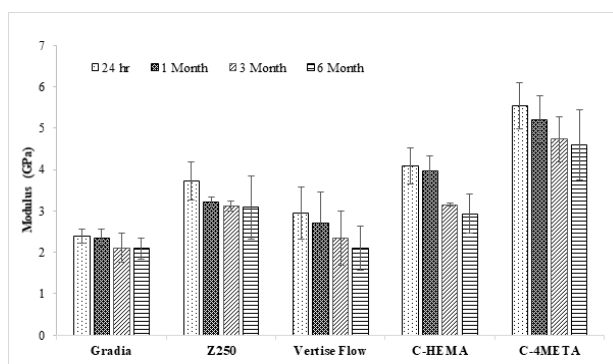


Fig 6: After twenty-four hours, one, three-, and six-months storage in DW, the Young's Modulus of all composites was evaluated. The error bars show the ninety five percent confidence interval (n=6).

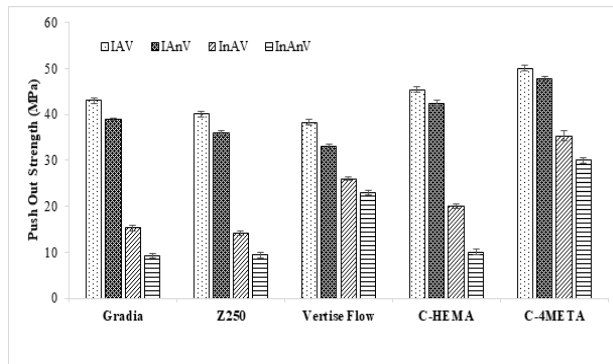


Fig 7: Use of various dentine treatment to assess the adhesion of each composite to dentine: (Only Ibond applied) IAnV, (Only thirty five percent phosphoric acid etching) InAV, (Acid cleaning accompanied by Ibond adhesive material) IAV, (No dentine prior cleaning) InAnV. The error bars show the ninety five percent confidence interval (n=6).

The experimental self-adhesive C-4META and Vertise flow composite adhesion strength was in between 23 to 30 MPa without any dentine prior treatment. The rest of bulk filled composites had adhesion strength of 9 to 10 MPa.

DISCUSSION

Three commercial composites of which 2 were mass occupied and one was flow able composite and two experimental mass occupied composites were evaluated in this study. In addition the C4 Meta composite also possessed self-adhesive properties. The majority monomers shows least wetting as well as self-adhesive properties¹¹. Water sorption by composites causes a variety of mechanical, biological, and chemical changes in the material. Water sorption elicits unreacted radicals which are produced from compounds, which should be of concern. These unreacted monomers have the potential to leak into the dental environment and cause cytotoxicity¹². Furthermore, residual monomers cause the polymer matrix to plasticize and facilitate water sorption. This leads to hydrolytic breakdown, resulting in lower mechanical properties^{13, 14}.

The mechanical characteristics of dental composites should be strong to handle masticatory load¹⁵. During mastication upon composite various stresses are incorporated, which includes compressive, shear and tensile stresses. All three stresses are combined in the form of flexural stress¹⁶. As a result bending strength may be the most accurate approach for assessing the technical execution of experimental composite when compared with the commercially available composites. The biaxial flexural strength values suggests that filler loading as an important factor in the stability of the material to behave in long term. The addition of self-adhesive components to the experimental composite showed added benefit in the bonding, without compromising the strength of the material when compared with the commercially available ones¹⁷.

The adherence of material to dentine was checked using a push out test. At first dentine blocks were made and was checked against all composites with varying dentine prior treatments. For class I cavities, the push out test was a good model¹⁸. The values obtained with Ibond use on dentine were in agreement with literature using un-etched human dentine¹⁹. With water present, the anhydride group in the 4-META within Ibond is hydrolysed to provide two carboxylic acid groups. It is suggested that these may partially de-mineralise the dentine to allow some micro-mechanical interlocking, but in addition enable a chemical bond with calcium in remaining hydroxyapatite. Hence improving the bond strength of the material.

Our study concludes that the experimental composite had the potential to compete with commercial composites in mechanical, chemical, and microscopic properties, without compromising the properties.

ACKNOWLEDGEMENT

The author would like to acknowledge Higher Education Commission Pakistan for funding.

REFERENCES

1. Selwitz RH, Ismail AI, Pitts NB. Dental caries. *The Lancet*. 2007;369(9555):51-9.
2. Pitts NB, Zero DT, Marsh PD, Ekstrand K, Weintraub JA, Ramos-Gomez F, et al. Dental caries. *Nature reviews Disease primers*. 2017;3(1):1-16.
3. Tsi trou E, Kelogrigoris S, Koulaouzidou E, Antoniad es-Halvatjoglou M, Koliniotou-Koumpia E, van Noort R. Effect of Extraction Media and Storage Time on the Elution of Monomers from Four Contemporary Resin Composite Materials. *Toxicology International*. 2014;21(1):89-95.
4. Schneider LFJ, Cavalcante LM, Silikas N. Shrinkage Stresses Generated during Resin-Composite Applications: A Review. *Journal of Dental Biomechanics*. 2010;1(1).
5. Watanabe H, Khera SC, Vargas MA, Qian F. Fracture toughness comparison of six resin composites. *Dental materials*. 2008;24(3):418-25.
6. Uhl A, Völpe l A, Sigusch BW. Influence of heat from light curing units and dental composite polymerization on cells in vitro. *Journal of dentistry*. 2006;34(4):298-306.
7. Lien W, Vandewalle KS. Physical properties of a new silorane-based restorative system. *Dental materials*. 2010;26(4):337-44.
8. Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. *The Journal of the American Dental Association*. 2003;134(10):1382-90.
9. Blackham J, Vandewalle K, Lien W. Properties of hybrid resin composite systems containing prepolymerized filler particles. *Operative Dentistry*. 2009;34(6):697-702.
10. Ferracane JL. Resin composite—state of the art. *Dental materials*. 2011;27(1):29-38.
11. Poitevin A, De Munck J, Van Ende A, Suyama Y, Mine A, Peumans M, et al. Bonding effectiveness of self-adhesive composites to dentin and enamel. *Dental Materials*. 2013;29(2):221-30.
12. Ak AT, Alpoz AR, Bayraktar O, Ertugrul F. Monomer Release from Resin Based Dental Materials Cured With LED and Halogen Lights. *European Journal of Dentistry*. 2010;4(1):34-40.
13. Liu Y, Tjäderhane L, Breschi L, Mazzoni A, Li N, Mao J, et al. Limitations in Bonding to Dentin and Experimental Strategies to Prevent Bond Degradation. *Journal of Dental Research*. 2011;90(8):953-68.
14. Park J-G, Ye Q, Topp EM, Misra A, Spencer P. Water sorption and dynamic mechanical properties of dentin adhesives with a urethane-based multifunctional methacrylate monomer. *Dental materials : official publication of the Academy of Dental Materials*. 2009;25(12):1569-75.
15. Zakir M, Al Kheraif AAA, Asif M, Wong FSL, Rehman IU.

A comparison of the mechanical properties of a modified silorane based dental composite with those of commercially available composite material. *Dental Materials*. 2013;29(4):e53-e9.

16. Junior SAR, Ferracane JL, Della Bona AJDm. Flexural strength and Weibull analysis of a microhybrid and a nanofill composite evaluated by 3-and 4-point bending tests. *2008;24(3):426-31*.
17. Pinna R, Bortone A, Sotgiu G, Dore S, Usai P, Milia E. Clinical evaluation of the efficacy of one self-adhesive composite in dental hypersensitivity. *Clinical oral investigations*. 2015;19(7):1663-72.
18. Kececi AD, Kaya BU, Adanir N. Micro push-out bond strengths of four fiber-reinforced composite post systems and 2 luting materials. *Oral Surgery, Oral Medicine, Oral Pathology, Oral Radiology, and Endodontology*. 2008;105(1):121-8.
19. Krifka S, Börzsönyi A, Koch A, Hiller K-A, Schmalz G, Friedl K-H. Bond strength of adhesive systems to dentin and enamel—Human vs. bovine primary teeth in vitro. *Dental materials*. 2008;24(7):888-94.

Author Declaration: The article is based on PhD (Dental Materials) research thesis of **Dr. Saad Liaqat**, University College London.

CONFLICT OF INTEREST: Authors declare no conflict of interest

GRANT SUPPORT AND FINANCIAL DISCLOSURE:
HEC

AUTHOR'S CONTRIBUTION

Following authors have made substantial contributions to the manuscript as under

Liaqat S: Conceiving and designing the study, analysis, interpretation of data

Jabeen H: Writing of manuscript, Bibliography

Authors agree to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.