DOI: https://doi.org/10.2298/SARH220129055P
UDC: 66.018.9

ORIGINAL ARTICLE / ОРИГИНАЛНИ РАД

Polymer characteristics and mechanical properties of bulk-fill, giomer, fiber-reinforced, and low-shrinkage composites

Dejan Perić[1], Jovana Ružić[2], Steva Lević[3], Jovana N. Stašić[4]

[1]University of Priština – Kosovska Mitrovica, School of Medicine, Dental Clinic, Kosovska Mitrovica, Serbia;
[2]University of Belgrade, Vinča Institute of Nuclear Sciences – National Institute of the Republic of Serbia, Belgrade, Serbia;
[3]University of Belgrade, Faculty of Agriculture, Belgrade, Serbia;
[4]University of Belgrade, School of Dental Medicine, DentalNet Research Group, Belgrade, Serbia

SUMMARY

Introduction/Objective The objective was to determine the degree of conversion (DC), cross-link density, percentage of leachable monomers, flexural strength (FS), and hardness (HV) of nanohybrid, nanofilled bulk-fill, giomer, fiber-reinforced, and low-shrinkage composites.

Methods Standardized specimens (n = 5/group) of Tetric EvoCeram Bulk Fill, Filtek Bulk Fill, Beautifil, EverX posterior, Kalore, Filtek Z250 (microhybrid control), and Tetric EvoCeram (nanohybrid control) were subjected to micro-Raman spectroscopy, three-point bending, and HV. Cross-linking density and leachable monomers were ascertained based on the ratio of HV and DC before and after immersion in absolute ethanol.

Results DC was in the range 50.4–70.5%, the highest for Filtek Bulk and the lowest for Kalore. The highest %DC change was in Beautifil (10.3%) and the lowest in Filtek Bulk (1.4%) and Z250 (1.28%). FS ranged between 78.9 MPa (TEC) and 126.7 MPa (Filtek Bulk). HV ranged between 58.6 (Kalore) and 113.9 (Z250) and significantly decreased post-immersion (19–55%). HV48h inversely correlated to HV% loss (r = -0.761), whilst DC positively correlated with FS (r = 0.893).

Conclusion Filtek Bulk, EverX, and Z250 showed the highest DC. The lowest DC and mechanical properties were observed for Kalore. The greatest cross-link density was shown by Filtek Bulk. There were up to 10% of leachable monomers. DC and FS positively correlated.

Keywords: composite; conversion; cross-link density; flexural strength; hardness

INTRODUCTION

It has been widely adopted that dental composites are the materials of choice for most anterior and posterior restorations [1]. An intricate balance of primarily biomechanical, but also isolation, bonding, and aesthetic demands, has led to the development of different "subclasses" of composites recommended for direct posterior restorations.

Nanohybrid/nanofilled, microhybrid, and fiber-reinforced composites have all been indicated for posterior restorations for their improved mechanical properties, either based on increased filler load or the presence of short glass fibers [2, 3]. Giomer composites are nanohybrid, fluoride release and recharge composites, also in the sculptable bulk-fill group. Low-shrinkage composites, intended to overcome polymerization shrinkage as the major shortcoming of dental composites, employ high filler load or high-molecular weight cross-linking monomers as the main strategies [4]. Despite shrinkage that may be comparable to other composite types, lower shrinkage stress was reported for some low-shrinkage than for universal or flowable composites [5, 6, 7].

Bulk-fill composites combine some of the previous approaches with increased translucency or alternative photoinitiators for increased depth of cure and decreased the number of required increments in posterior cavities [8, 9, 10]. Bulk-fill composites are a variable group of materials with mechanical properties that are comparable, at best, to universal incremental composites [2]. Exception could be the fiber-reinforced bulk-fill composite everX posterior (GC International AG, Luzern, Switzerland; previously Xenius, StikTech) with consistently higher fracture toughness compared to universal, bulk-fill, and fiber-reinforced composites [3, 11, 12].

Degree of conversion (DC) is related to material composition, temperature, as well as curing conditions [13, 14, 15].

DC is measured directly as a ratio of C = C double bonds in cured and uncured materials using micro-Raman [16] or Fourier-transform infrared spectroscopy [13, 16, 17]. Hardness (HV) and flexural strength (FS) are arguably the most frequently tested mechanical properties of dental composites [18]. The percentage of hardness loss after chemical softening in ethanol has been used as an indication of cross-link density.

Received • Примљено: January 29, 2022
Accepted • Прихваћено: June 1, 2022
Online first: June 8, 2022

Correspondence to:
Dejan PERIĆ
Anri Dinana St.
Kosovska Mitrovica 38220
Serbia
drperke@yahoo.com

The latter is important for hygroscopic and chemical stability and viscoelastic behavior of composites [19]. Higher degree of softening expressed as greater hardness loss has been related to lower cross-link density [20].

The aim of this study was to compare and correlate polymer characteristics – DC and cross-link density, and mechanical properties – HV and FS, of composites from different subclasses – nanohybrid and nanofilled bulk-fill, giomer, fiber-reinforced, low-shrinkage, and universal composites. The null hypotheses were as follows: (1) there are no statistically significant differences in the DC, cross-link density, FS, and HV between tested composites and (2) there is no correlation between polymer and mechanical characteristics of the tested composites.

METHODS

Table 1 presents details on materials used in the present study. For the DC, cross-link density, and HV, standardized plastic molds 5 mm in diameter and 2 mm or 4 mm deep were used to prepare composite specimens (n = 5/group). Uncured material was placed into each mold, covered with a glass slide, and pressed to extrude excess material. Light-curing was performed for 40 seconds using a monowave LED light-curing unit (LEdition, Ivoclar Vivadent AG, Schaan, Liechtenstein) at a standardized distance of 1 mm and an intensity of ~800 mW/cm^2 (Radiometer Model 100, Kerr Corp, Orange, CA, USA). The specimens were polished in wet conditions using SiC abrasive discs (600, 1000, and 2000 grit) and polishing cloth (TexMet, Buehler, Lake Bluff, IL, USA). For FS, square beam specimens were prepared in 2 × 2 × 25 mm molds as per the ISO standard 4049:2009. Light-curing was performed through a 1 mm thick glass slide in three exposures, 40 seconds each, whilst the final light exposure was performed by moving the light tip from one end of the specimen to the other over 40 seconds. Specimens were stored in dark, light-proof containers at 37°C for 24 hours prior to measurements.

The DC was determined using micro-Raman spectroscopy (XploRA, Raman spectrometer, Horiba Jobin Yvon) using the following parameters: laser 785 nm wavelength, spectrometer grating 1200 gr/mm, acquisition time 10 s, number of acquisitions 5, objective 50 ×. Uncured composite were used as reference materials to calculate the DC according to the following formula:

$$DC = \left(1 - \frac{Rcured}{Runcured}\right) \times 100$$

where Rcured and Runcured are the ratio of spectral peaks related to aliphatic (1640 cm^{-1}) and aromatic (1610 cm^{-1}) C = C bonds in cured and uncured material, respectively. Initial measurement was done after 24 hours of storage at three random points at the bottom, unexposed surface. The measurements were repeated using the same conditions after 48 hours of specimen storage in absolute ethanol.

FS was measured using a three-point bending test in a universal testing machine (Force Gauge PCE-FM200, PCE Instruments UK Ltd, Southampton, UK) at 1 mm/minute speed until fracture. FS was determined using the equation:

$$FS = \frac{3Fl}{2bh^2}$$

where F is the maximum load measured before fracture and l is the distance between supports (20 mm), b is sample width, and h is sample height.

HV measurements were done performed using Vickers indenter in a hardness tester (Buehler Indentament 1100 series, Buehler) at 100 g over 20 seconds. Hardness measurements were performed linearly in five or nine points in 2 mm or 4 mm thick specimens, respectively. Repeated measurements were done 24 hours and 48 hours of post-immersion in ethanol.

Table 1. Materials used in the present study

Materials	Manufacturer / Type / Increment thickness	Composition	Filler content
Tetric EvoCeram Bulk Fill (Code: TEC Bulk)	Ivoclar Vivadent/ nanohybrid bulk-fill / 4 mm	BisGMA, UDMA, BisEMA, Barium aluminium silicate glass fillers, prepolymer, ytterbium trifluoride and spherical mixed oxide	79.5wt% (62.5wt% filler and 19.7% prepolymer)
Filtek™ Bulk Fill Posterior Restorative (Filtek Bulk)	3M ESPE/ nanofilled bulk-fill / 4–5 mm	Aromatic UDMA, UDMA, DDDMA, 2,2-dimethyl-4-methylene- reaction products with glycidyl methacrylate, EDMAB, zirkonia / silica and YbF3 filler, titanium dioxide	76.5wt% 58.4vol%
Beautifil Bulk Restorative (Beautifil)	SHOFU INC./ giomer / 4 mm	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, S-PRG filler based on F-B-Al-silicate glass	83.3wt%
EverX Posterior (EverX)	GC EUROPE/ fiber-reinforced bulk-fill / 4 mm	Bis-GMA, TEGDMA, PMMA, SiO2, barium glass, glass fibers 1–2 mm length	76wt% 57vol%
Kalore (Kalore)	GC EUROPE/ Nanohybrid low-shrinkage / 2 mm	UDMA, BisEMA, BHT, dimethacrylate, DX-511 co-monomers, fluoroaluminosilicate glass, pre-polymerized filler, strontium glass, SiO2	82wt% 69vol%
Tetric EvoCeram (TEC)	Ivoclar Vivadent/ nanohybrid (control) / 2 mm	BisGMA, UDMA, BisEMA, barium glass filler, ytterbium trifluoride, mixed oxide, prepolymers	82.5wt% (48.5wt% filler and 34% prepolymer)
Filtek Z250 (Z250)	3M ESPE/ microhybrid (control) / 2 mm	BisGMA, TEGDMA, UDMA, BisEMA6, EDMAB, silane treated ceramic	78wt% 60vol%

Composition is based on manufacturers' technical data;
BisGMA – bisphenol-A-diglycidyl-dimethacrylate; UDMA – urethane dimethacrylate; BisEMA/BisMPEPP – ethoxylated bisphenol A dimethacrylate; DDDMA – 1,12-dodecane dimethycrylate; TEGDMA – triethyleneglycol dimethacrylate; Bis-MPEPP – bisphenol A polyethoxy methacrylat; PMMA – polymethylmethacrylate; BHT – butylated hydroxytoluene; EDMAB – ethyl 4-dimethyl aminobenzoate

Polymer cross-linking density was ascertained based on the ratio of hardness values before and after ethanol immersion. The percentage of leachable monomers was determined as the ratio of the DC before and after ethanol immersion.

Data for the DC was analyzed using general linear model for "material" and "time" factors with factor interaction included. In case of significant factor interaction, follow-up one-way analysis of variance (ANOVA) was performed for inter-material comparison at each time separately, whilst intra-material comparison of initial and 48-hour measurements was done using paired t-tests separately with the Bonferroni correction. HV and FS data were analyzed in Minitab 16 (Minitab Inc, State College, PA, USA) using one-way ANOVA with Tukey's post-hoc test. Pearson correlation and regression analyses were performed to assess the relationship between DC, cross-link density, HV and FS. The level of significance was set at 0.05.

The study was approved by the Ethics Committee of the University of Priština –Kosovska Mitrovica, Serbia.

RESULTS

Figure 1 presents DC values of the tested composites initially and after 48 h of ethanol immersion. General linear model for factors "composite" and "time" showed significant interaction ($p < 0.05$). The results of follow-up analyses are presented in Figure 1. Initially, the DC was in the range Filtek Bulk, EverX, Z250 > TEC Bulk, Beautifil, TEC > Kalore ($p < 0.05$). After immersion in ethanol, the DC was in the following order: Filtek Bulk ≥ EverX ≥ Beautifil, Z250, TEC Bulk, TEC > Kalore ($p < 0.05$). EverX, Beautifil, TEC Bulk, and TEC showed significantly higher DC after immersion than initially ($p < 0.05$), whilst no significant difference was found for Filtek Bulk, Kalore, and Z250 ($p > 0.05$). After 48 hours of ethanol storage, the %DC increase was significantly higher in Beautifil (10.3%) and TEC (8.5%), followed by TEC Bulk (4.7%) and EverX (3.5%) compared to initial DC ($p < 0.05$). The %DC change in Kalore (1.9%), Filtek Bulk (1.4%), and Z250 (1.28%) was not significant compared to initial values ($p > 0.05$), albeit Z250 showed slight decrease in DC.

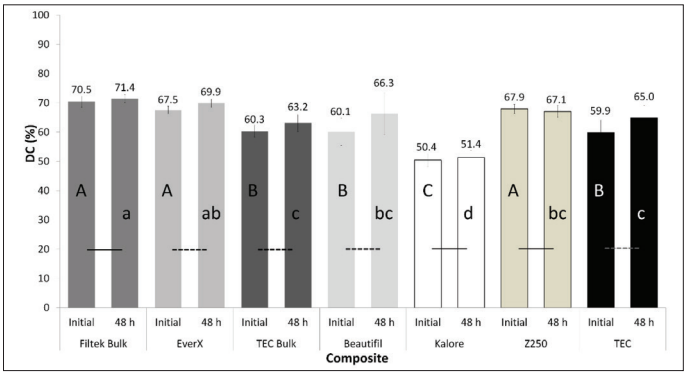


Figure 1. Degree of conversion of the tested composites initially and after 48 hours of ethanol immersion; columns represent mean and bars represent standard deviation values; different uppercase letters indicate statistically significant differences between composites initially; different lowercase letters indicate statistically significant differences between composites after immersion; dashed lines indicate statistically significant differences within each composite between different time intervals ($p < 0.05$); horizontal lines indicate no significant difference within each composite between different time intervals ($p > 0.05$)

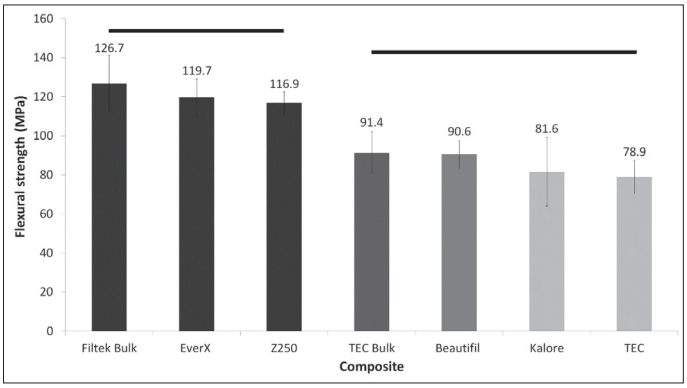


Figure 2. Flexural strength of the tested composites; columns represent mean and bars represent standard deviation values; groups connected with horizontal lines are not significantly different ($p > 0.05$)

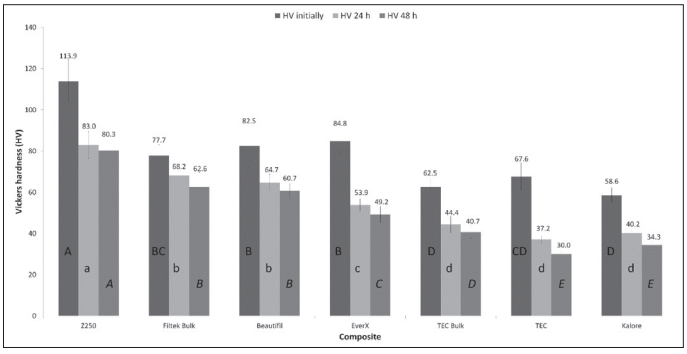


Figure 3. Vickers hardness values (mean and standard deviation) for the tested composites initially and after 24 hours and 48 hours post-immersion in ethanol; groups with different uppercase, lowercase, or italic letters are significantly different for each of the time periods ($p < 0.05$)

Filtek Bulk and EverX showed significantly higher FS than other composites, whilst the lowest values were measured for TEC ($p < 0.05$). Comparing bulk-fill composites, Filtek Bulk, and Everx had higher FS than TEC Bulk and Beautifil ($p < 0.05$) (Figure 2).

Figure 3 presents HV data for different time intervals. Z250 showed the highest HV at all time periods. Filtek Bulk and Beautifil showed consistently similar HV ($p > 0.05$), but lower than that of Z250. TEC Bulk, TEC, and Kalore exhibited lower HV than other composites ($p < 0.05$).

HV of all tested composites significantly decreased after ethanol immersion, ranging 55–19% HV loss (Figure 4). TEC showed significantly higher %HV loss (55.3%) than other materials ($p < 0.05$), followed by EverX (41.8%), Kalore (41.3%), and TEC Bulk (34.7%), whilst this decrease was the lowest in Z250 (29%), Beautifil (25.6%), and Filtek Bulk (18.8%) ($p > 0.05$).

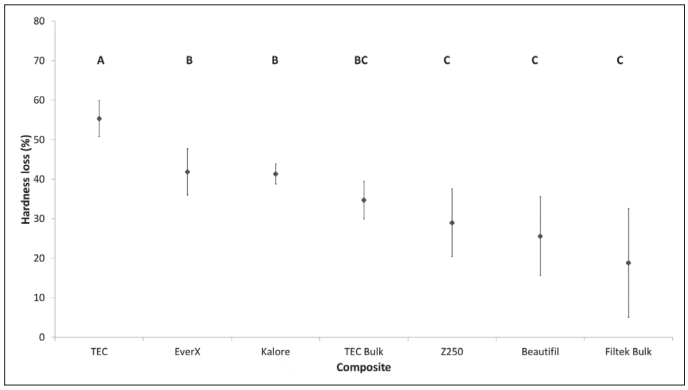


Figure 4. Percentage of hardness loss following ethanol immersion test; groups with different uppercase letters are significantly different ($p < 0.05$)

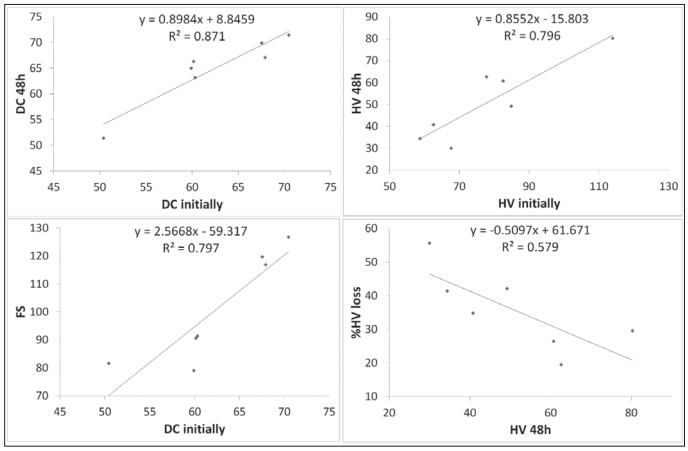


Figure 5. Regression analysis showing the relationship between tested properties with regression equations and R^2 values;
DC – degree of conversion; FS – flexural strength; HV – hardness

Figure 5 presents the results of regression analysis. Pearson's correlation showed significant positive correlation between DC initially and DC after storage as well as between HV initially and HV after storage, with Pearson's correlation coefficients r = 0.933 (p = 0.002) for DC and r = 0.892 (p = 0.007) for HV. HV after ethanol immersion was a better predictor of HV% loss than initial HV, as significant negative correlation was found between HV_{48h} and HV% loss (r = -0.761, p = 0.047) but not between $HV_{initial}$ and HV% loss (r = -0.389, p = 0.388). Significant positive correlation was found between DC and FS (r = 0.893, p = 0.007). No correlation was found between DC and HV (DC *vs.* HV initially: r = 0.669 and p = 0.100; DC *vs.* HV after storage: r = 0.545 and p = 0.206) or between FS and HV (r = 0.636, p = 0.124).

DISCUSSION

Both tested hypotheses were rejected. Tested composites showed significant differences in the DC, cross-link density, FS, and HV. Positive correlation was established between DC and FS as well as between DC and HV initially and after storage, whilst HV_{48h} and HV% loss were negatively correlated.

Sculptable nanofilled and nanohybrid bulk-fill composites – Filtek Bulk and TEC Bulk – showed similar DC to their universal counterparts – Z250 and TEC. Differences were notable between both bulk-fill as well as between universal composites. This is the case with TEC and TEC Bulk in that they have similar resin composition, prepolymer, and filler content. TEC Bulk contains Ivocerin, a benzoyl-germanium-based additional photoinitiator, a type I initiator that shows α-cleavage under formation of benzoyl and germyl radicals with no need for an amine co-initiator [8]. Ivocerin was shown to absorb light energy around 410–420 nm, unlike a similar alternative photoinitiator Lucirin TPO, making Ivocerin compatible with monowave light-curing units [8, 9]. Similar DC of TEC Bulk at full increment depth of 4 mm to TEC may be explained by increased translucency of TEC Bulk compared to TEC, allowing deeper light penetration [21]. Similarly, DC at full increment depth of TEC Bulk and TEC was seen in recent studies in different experimental setups, albeit with lower values measured [17, 21].

The present finding of higher DC of Filtek Bulk than TEC Bulk was reported previously for different curing conditions [21]. Resin composition of Filtek Bulk is considerably different from TEC Bulk and is based on a high-molecular-weight aromatic dimethacrylate monomer, addition-fragmentation monomer, UDMA, and 1,12-dodecanediol dimethacrylate, as per manufacturer's technical data. The main purpose of these monomers is the control of shrinkage and shrinkage stress of Filtek Bulk. High DC of Filtek Bulk could be related to an increased number of reactive sites and cleavage of an addition-fragmentation monomer to fragments during polymerization which may further react with other reactive sites of the developing polymer. In Z250, performing similarly to Filtek Bulk, low reactivity of BisGMA was likely compensated with TEGDMA, UDMA, and BisEMA, allowing a DC in the range of 65–70% of this universal composite similar to Filtek Bulk. A similar DC of Z250 was recently reported for a high-intensity polywave light-curing unit (~1100 mW/cm^2) and shorter curing time (20 seconds) as opposed to the presently used monowave unit (~800 mW/cm^2) and longer curing time (40 seconds), probably indicating that this is a maximum DC reachable for this material [21].

Kalore, a low-shrinkage composite based on a high-molecular-weight monomer DX-511, showed the lowest DC in the present study – around 50%. Previous studies show inconsistent results in terms of DC, with one study reporting a DC close to 45% [7]. Despite differences in experimental conditions, in terms of light-curing units, irradiance, and curing time, the material was cured according to the recommended curing times for a particular light-curing unit in all these instances. Inconsistent results indicate factors still unknown, affecting the polymerization behavior of this composite. An increase in the DC after 48 hours of storage in absolute ethanol reveals the percentage of uncured

monomers and potentially leachable small oligomeric species, trapped within the polymer network. This approach can potentially lead to elution of small oligomeric species, thus mimicking the exact percentage of uncured monomers.

Ethanol is a potent organic solvent that allows polymer softening, swelling and elution of uncured monomers. In this study, absolute ethanol was used to extract all leachable unreacted monomers to further assess the quality of polymer networks. Up to about 10% of uncured monomers were detected from the tested materials. The highest percentage was found for giomer Beautifil and the lowest for low-shrinkage Kalore and sculptable bulk-fill Filtek Bulk. Taking into consideration the DC and %DC change, the present results suggest that around 30–35% of unreacted C = C double bonds remain in the form of pendant groups within the polymer. This relatively high percentage of unreacted C = C bonds remain due to the reduced mobility of unreacted monomers and pendant groups during diffusion-controlled propagation until the reaction stops due to polymer vitrification [14].

High values for mechanical properties – FS and HV, were not related to a specific subclass of composites, as nanofilled bulk-fill, fiber-reinforced, and universal composite were the three materials with the highest FS and HV. Further, giomer Beautifil showed similar HV to Filtek Bulk and EverX. The discrepancy could be due to differences in the range of selected materials for testing, further confirming the heterogeneity of the bulk-fill subclass. A more consistent pattern was found between the present results for the low-shrinkage Kalore and universal composites Z250 and TEC and previously reported data [18], showing that Z250 performed better whilst Kalore and TEC showed inferior results in terms of mechanical properties in both experimental setups. Mixed performance by Kalore in terms of comparable or inferior mechanical characteristics and shrinkage and lower shrinkage stress than universal or flowable composites indicates that composites from other subclasses should be preferred by clinicians over Kalore [5, 6].

The present data regarding %HV loss after ethanol softening ranged largely between the tested composites, between around 19% and 55%. This points to considerable differences in cross-link density. Absolute ethanol was used in the present study as ethanol concentration was shown to affect the outcome of softening analysis with 75% ethanol/water solution not being able to expose the differences in cross-link density of composites [20].

Filtek Bulk has shown the least %HV loss suggesting the greatest cross-link density whilst TEC showed the opposite result. Increased cross-link density of Filtek Bulk may be associated primarily with addition-fragmentation monomer and its increased ability to react with reactive sites in the growing polymer. Also, the position of aromatic groups in the high molecular weight dimethacrylate monomers in Filtek Bulk could result in differences in cross-linkage compared to BisGMA and BisEMA, the main monomers with aromatic groups in TEC.

EverX has also shown considerable %HV loss indicating lower cross-link density compared to Filtek Bulk. This could be due to the presence of PMMA, which is known to be a linear polymer. Previously, higher %HV loss for EverX (~29%) than for Filtek Bulk (~19%) was reported after 75% ethanol/water immersion [12]. In the present study, virtually the same %HV loss was detected for Filtek Bulk after storage in absolute ethanol (~19%) whilst greater %HV loss was found for EverX (~42%) compared to the previous study [12]. This further highlights the difference in ethanol concentration and the need for maximum concentration for cross-link density analysis.

Correlation analysis between filler content and mechanical properties was not done due to inconsistent data between manufacturer's technical data and literature reports [2, 19]. The filler content was not measured in the present study due to the lack of equipment. Multiple correlations using various data from the literature and manufacturers data revealed no significant differences between filler content and HV/FS. This could be due to the fact that different types of fillers (glass, prepolymerized, S-PRG glass, silica, zirconia, glass fibers) are present in the tested materials.

Positive correlation was established between the DC and FS but not between the DC and HV as was the case in previous studies [9, 21]. For the given range of composites tested in the present study, the DC was a better indicator of FS than of HV. A recent study showed that filler volume rather than filler weight percentage is a better indicator of elastic modulus and solvent sorption illustrating the complex relationship between material composition and properties [2]. HV after ethanol immersion is a good indicator of %HV loss, albeit in an inverse relationship – the higher the HV post-immersion, the lower the HV% loss and, thus, greater composite cross-link density. A positive correlation between the DC initially and DC after ethanol immersion suggests consistent presence of uncured monomers within the polymer. HV initially and HV after ethanol immersion showed consistent behavior for the tested composites and were linearly correlated. This indicates that the variation in cross-link density does not significantly affect HV of dental composites.

CONCLUSION

Based on the results of this study, the following can be concluded.

The DC of bulk-fill composites was comparable or higher than universal or low-shrinkage composites. Up to 10% of unreacted, leachable monomers and oligomeric species containing unreacted methacrylate groups were detectable within the polymer network of the tested composites.

Nanohybrid, nanofilled, fiber-reinforced, and giomer bulk-fill composites showed comparable or higher HV than nanohybrid TEC, but lower than microhybrid Z250.

The greatest cross-link density, as indicated indirectly by changes in HV after alcohol softening, was shown by the nanofilled bulk-fill composite Filtek Bulk, whilst the low-shrinkage Kalore and fiber-reinforced composite EverX showed the lowest cross-link density.

Conflict of interest: None declared.

REFERENCES

1. Ramkumar Y, Mukesh K. Dental restorative composite materials: A review. J Oral Biosci. 2019;61(2):78–83. [DOI: 10.1016/j.job.2019.04.001] [PMID: 31109861]
2. Gomes de Araújo-Neto V, Sebold M, Fernandes de Castro E, Feitosa VP, Giannini M. Evaluation of physico-mechanical properties and filler particles characterization of conventional, bulk-fill, and bioactive resin-based composites. J Mech Behav Biomed Mater. 2021;115:104288. [DOI: 10.1016/j.jmbbm.2020.104288] [PMID: 33383377]
3. Jafarnia S, Valanezhad A, Shahabi S, Abe S, Watanabe I. Physical and mechanical characteristics of short fiber-reinforced resin composite in comparison with bulk-fill composites. J Oral Sci. 2021;31(2):148–51. [DOI: 10.2334/josnusd.20-0436] [PMID: 33504755]
4. Miletic V. Low-Shrinkage Composites. In: Miletic V, editor. Dental Composite Materials for Direct Restorations. Cham: Springer International Publishing; 2018. p. 97–112.
5. Miletic V, Peric D, Milosevic M, Manojlovic D, Mitrovic N. Local deformation fields and marginal integrity of sculptable bulk-fill, low-shrinkage and conventional composites. Dent Mater. 2016;32(11):1441–51. [DOI: 10.1016/j.dental.2016.09.011] [PMID: 27686259]
6. Yamamoto T, Hanabusa M, Momoi Y, Sakaguchi RL. Polymerization stress of dental resin composite continues to develop 12 hours after irradiation. J Esthet Restor Dent. 2015;27(1):44–54. [DOI: 10.1111/jerd.12091] [PMID: 25855817]
7. Yamasaki LC, De Vito Moraes AG, Barros M, Lewis S, Francci C, Stansbury JW, et al. Polymerization development of "low-shrink" resin composites: Reaction kinetics, polymerization stress and quality of network. Dent Mater. 2013;29(9):e169–79. [DOI: 10.1016/j.dental.2016.01.009] [PMID: 26861171]
8. Moszner N, Fischer UK, Ganster B, Liska R, Rheinberger V. Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials. Dent Mater. 2008;24(7):901–7. [DOI: 10.1016/j.dental.2007.11.004] [PMID: 18155290]
9. Santini A, Miletic V, Swift MD, Bradley M. Degree of conversion and microhardness of TPO-containing resin-based composites cured by polywave and monowave LED units. J Dent. 2012;40(7):577–84. [DOI: 10.1016/j.jdent.2012.03.007] [PMID: 22465876]
10. Lima RBW, Troconis CCM, Moreno MBP, Murillo-Gómez F, De Goes MF. Depth of cure of bulk fill resin composites: A systematic review. J Esthet Restor Dent. 2018;30(6):492–501. [DOI: 10.1111/jerd.12394] [PMID: 30375146]
11. Lassila L, Keulemans F, Vallittu PK, Garoushi S. Characterization of restorative short-fiber reinforced dental composites. Dent Mater J. 2020;39(6):992–9. [DOI: 10.4012/dmj.2019-088] [PMID: 32779605]
12. Alshabib A, Silikas N, Watts DC. Hardness and fracture toughness of resin-composite materials with and without fibers. Dent Mater. 2019;35(8):1194–203. [DOI: 10.1016/j.dental.2019.05.017] [PMID: 31176452]
13. Wang WJ, Grymak A, Waddell JN, Choi JJE. The effect of light curing intensity on bulk-fill composite resins: heat generation and chemomechanical properties. Biomater Investig Dent. 2021;29(1):137–51. [DOI: 10.1080/26415275.2021.1979981] [PMID: 34622209]
14. Yang J, Silikas N, Watts DC. Pre-heating time and exposure duration: Effects on post-irradiation properties of a thermo-viscous resin-composite. Dent Mater. 2020;36(6):787–93. [DOI: 10.1016/j.dental.2020.03.025] [PMID: 32386715]
15. Moreira RJ, de Deus RA, Ribeiro MTH, Braga SSL, Schettini ACT, Price RB, et al. Effect of Light-curing Unit Design and Mouth Opening on the Polymerization of Bulk-fill Resin-based Composite Restorations in Molars. J Adhes Dent. 2021;23(2):121–31. [DOI: 10.1016/j.jdent.2011.05.007] [PMID: 21679742]
16. Pongprueksa P, De Munck J, Duca RC, Poels K, Covaci A, Hoet P, et al. Monomer elution in relation to degree of conversion for different types of composite. J Dent. 2015;43(12):1448–55. [DOI: 10.1016/j.jdent.2015.10.013] [PMID: 26498727]
17. Oltramare RS, Odermatt R, Burrer P, Attin T, Tauböck TT. Depth-Related Curing Potential of Ormocer- and Dimethacrylate-Based Bulk-Fill Composites. Materials (Basel). 2021;14(22):6753. [DOI: 10.3390/ma14226753] [PMID: 34832155]
18. Takahashi H, Finger WJ, Endo T, Kanehira M, Koottathape N, Komatsu M, et al. Comparative evaluation of mechanical characteristics of nanofiller containing resin composites. Am J Dent. 2011;24(5):264–70. [PMID: 22165452]
19. Alshali RZ, Salim NA, Satterthwaite JD, Silikas N. Post-irradiation hardness development, chemical softening, and thermal stability of bulk-fill and conventional resin-composites. J Dent. 2015;43(2):209–18. [DOI: 10.1016/j.jdent.2014.12.004] [PMID: 25511301]
20. Schneider LF, Moraes RR, Cavalcante LM, Sinhoreti MA, Correr-Sobrinho L, Consani S. Cross-link density evaluation through softening tests: effect of ethanol concentration. Dent Mater. 2008;24(2):199–203. [DOI: 10.1016/j.dental.2007.03.010] [PMID: 17544496]
21. Miletic V, Pongprueksa P, De Munck J, Brooks NR, Van Meerbeek B. Curing characteristics of flowable and sculptable bulk-fill composites. Clin Oral Investig. 2017;21(4):1201–12. [DOI: 10.1007/s00784-016-1894-0] [PMID: 27383375]

Карактеристике полимера и механичке карактеристике *bulk-fill*, гиомера, влакном ојачаних и нискоконтракционих композита

Дејан Перић[1], Јована Ружић[2], Стева Левић[3], Јована Н. Сташић[4]

[1]Универзитет у Приштини – Косовска Митровица, Медицински факултет, Одсек стоматологије, Косовска Митровица, Србија;
[2]Универзитет у Београду, Институт за нуклеарне науке „Винча", Београд, Србија;
[3]Универзитет у Београду, Пољопривредни факултет, Београд, Србија;
[4]Универзитет у Београду, Стоматолошки факултет, Истраживачка група *DentalNet*, Београд, Србија

САЖЕТАК

Увод/Циљ Циљ овог рада је испитати степен конверзије, густину полимерне мреже, проценат излужених мономера, савојну чврстоћу и тврдоћу нанохибридних *bulk-fill*, гиомера, влакном ојачаних и нискоконтракционих композита.

Методе Стандардизовани узорци (*n* = 5/ група) *Tetric EvoCeram Bulk Fill*, *Filtek Bulk Fill*, *Beautifil*, *EverX posterior*, *Kalore*, *Filtek Z*250 (микрохибридна контрола) и *Tetric EvoCeram* (нанохибридна контрола) испитивани су применом микрораманске спектроскопије, тестом савијања у три тачке и анализом тврдоће по Викерсу. Густина полимерне мреже и излуженост мономера добијени су на основу односа тврдоће и степена конверзије пре и после потапања узорака у апсолутни етанол.

Резултати Вредности степена конверзије биле су у опсегу 50,4–70,5%, највеће за *Filtek Bulk* и најмање за *Kalore*. Највећа промена процента степена конверзије добијена је за *Beautifil* (10,3%), а најмања за *Filtek Bulk* (1,4%) и *Z*250 (1,28%). Вредности савојне чврстоће биле су између 78,9 *MPa* (*TEC*) и 126,7 *MPa* (*Filtek Bulk*). Вредности тврдоће кретале су се од 58,6 (*Kalore*) до 113,9 (*Z*250) са значајним смањењем после потапања (19–55%). *HV*48*h* је показао обрнуту корелацију са процентом смањења тврдоће (r = -0,761), док је степен конверзије имао позитивну корелацију са савојном чврстоћом (r = 0,893).

Закључак *Filtek Bulk*, *EverX* и *Z*250 показали су највеће вредности степена конверзије. Најмање вредности степена конверзије и механичких карактеристика уочене су за *Kalore*. Највећа промена густине полимерне мреже уочена је код композита *Filtek Bulk*. Излуживање мономера било је и до 10%. Вредности степена конверзије и савојне чврстоће показују позитивну корелацију.

Кључне речи: композит; конверзија; густина полимерне мреже; савојна чврстоћа; тврдоћа