Original Article

Comparison of Elasticity Modulus and Nanohardness of Various Dental Restorative Materials

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Received 17October 2021 and Accepted 20November 2021

Abstract

Introduction: Restorative materials are under constant loadings from mastication hence, it is important to have the knowledge of structural properties of the restorative materials to have long-term success on restorations. Therefore, the aim is to compare the nanohardness and elastic modulus values of various restorative materials. **Methods:** Disc-shaped samples were prepared from a high viscosity glass ionomer - Equia Forte Fil (EFF), a compomer - Dyract (DXP), a hybrid ionomer - Geristore (GS), a giomer bulk-fill - Beautifil-Bulk (BB), two bulk-fill composites - Venus Bulk-fill (VB) and Sonic Fill 2 (SF), and a nanohybrid composite - Z250. Samples of each of the tested materials (n=9) were examined under nanoindentation to evaluate elasticity modulus (Er) and nanohardness (Hnano) scores. One of the samples had undergone through scanning electron microscopy (SEM) evaluation. Data were analyzed statistically using the Kruskal-Wallis test. **Results:** SF had the highest elasticity modulus, followed by Z250 and DXP, without any statistical differences. However, GS had the lowest elasticity modulus, followed by EFF ($P<0.001$). Among nanohardness scores, there is no significant difference between VB, EFF, DXP, Z250, and BB groups. While SF showed the highest, GS had the lowest nanohardness scores. SEM images showed the differences between filler sizes and shapes. **Conclusion:** Main structural differences between glass ionomer-based and resin-based materials determined significant differences among related parameters of the restorative materials.

Keywords: Resin composites, Glass ionomers, Hybrid materials, Nanoindentation.

OzanG, Mert ErenM, Yıldırım BılmezZ, Tugce Gurcan A, Yucel Yucel Y.Comparison of Elasticity Modulus and Nanohardness of Various Dental Restorative Materials. J Dent Mater Tech 2021; 10(4): 232-241.

Introduction

With the improvements in nanotechnology and increasing patients' expectations, novel restorative materials are manufactured and released on the market. Today, excellent esthetics, acceptable biocompatibility, and ideal mechanical properties are prerequisites for every dental material. Materials have been combined to fulfill this aim, such as hybrid ionomers, compomers (composite and glass ionomers), developed with ion-releasing properties, such as giomers, or generated with different inorganic particles by nanotechnology like bulk-fill composites. It is crucial for dental professionals to choose the ideal materials to be used in suitable cases.

Physical and mechanical measurements of *in vitro* studies offer opinions of clinical performance over time (1). For use in stress-bearing areas, parameters of elasticity modulus and nanohardness should be further investigated. However, these measurements were rarely done in newly developed materials. Hardness is the resistance of the restorative material to penetration in the chewing process that could be up to 360 N in the posterior region (2). Nano-leveled hardness is being used to predict the wear resistance against these forces. On the other hand, the elastic modulus is the relative rigidity of a

material and the ability to bend under constant forces without deformation (3). Thus, these properties should be measured to understand the clinical behavior of restorative materials under constant loadings.

In the dental market, there is a broad spectrum of restorative materials. Composite resins and glass ionomers are the most common tooth-colored materials. Structures of these materials are being improved continuously. Glass ionomers cannot be used in load-bearing areas because of their low mechanical properties, such as inferior compressive and flexural strength and the brittleness (4). High viscosity glass ionomers and compomers have been developed to improve the inadequate physical properties of conventional glass ionomers. These materials' superior mechanical properties, such as fracture toughness, hardness, and compressive strength, have been reported in many studies (5,6). Apart from this, an increased ratio of nanofillers (3) and the adhesion strength between fillers and organic matrices (7) have been shown to affect the final mechanical parameters of composite resin materials, such as toughness, rigidity, and hardness.

The recent material development was done by launching bulk-fill composites designed to enable clinicians to use thicker increments (4–5 mm) of composites. This has been attributed to the optical properties of bulk-fill composites. A lower filler loading and larger filler particles with accordingly smaller specific surface areas ensure less light scattering and better light transmission through the bulk of the material (8). Previous findings (9,10) that compare the structural properties of bulk-fill composites reported that the scores of elasticity modulus, Vickers hardness, and indentation modulus of these materials were between hybrid and flowable composites. In order to overcome some of the limitations, bulk-fill composites with higher viscosity were more recently produced (9). In the present study, bulk-fill composites with both viscosities are investigated.

In the view of limited research testing a wide spectrum of restorative materials and the need to predict their clinical performance, this *in vitro* study aimed to compare nanohardness (Hnano) and reduced elastic modulus (Er), which is the elasticity modulus calculated under an indenter tip, of various types of materials by using nanoindentation technique. Microstructures of the materials were also evaluated by scanning electron microscopy (SEM). The null hypotheses were that there is no difference in the values of (1) nanohardness and (2) elasticity modulus values of tested materials.

Methods and Materials

Materials and specimen preparation

Materials tested in the present study were given in Table I.

Table I. Details of the restorative materials tested in the study.

Name	Brand	Type	Filler ratio (wt.%)	Composition
Equia Forte Fil (EFF)*	GC	High viscosity glass ionomer [HV-GIC]	**	Powder: Fluoroaluminasilicate glass, polyacrylic acid, iron oxide Liquid: Polybasic carboxylic acid, water
Geristore (GS)	DenMat	Resin ionomer	50	Aromatic dimethacrylate, HEMA, Barium-fluorosilicate glass, silica, initiators, stabilizers (3.5 µm)
Dyract XP (DXP)	Dentsply Sirona	Compomer	47	TCB resin, UDMA, Strontium-fluoro-silicate glass, strontium fluoride, photoinitiator, stabilizers (0.8 µm)
Venus Bulk Fill (VB)	Kulzer	Bulk-fill composite	65	UDMA, EBPDMA, barium glass, ytterbium trifluoride, silicon dioxide

SonicFill (SF)	Kerr	Bulk-fill composite	83.5	TMSPMA, EBPADMA, TEGDMA, oxide, SiO_2
Beautiful-Bulk (BB)	Shofu	Bulk-fill composite	75	Fluoro-alumino-silicate glass, BisGMA, UDMA, TEGDMA, BisMPEPP, Reaction initiator, others
Filtek Z250 (Z250)	3M Espe	Conventional resin composite	78	Bis-GMA, Bis-EMA, UDMA, TEGDMA, zirconia, silica, (0.01 - 3.5 µm)

Abbreviations: HEMA: Hydroxyethyl methacrylate, Bis-GMA: Bisphenol A diglycidyl ether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; Bis-MPEPP: Bisphenol A polyethoxy methacrylate, TCDDA: Tricyclodecanedimethanol diacrylate; TMSPMA: 3-[trimethoxysilyl]propyl methacrylate, SiO_2: Silicon dioxide *No coatings were applied. **No fillers included.

A total of 63 samples were fabricated (n=9) by using a cylindrical metallic mold (5 mm in diameter and 2 mm thick). Each material was inserted into the mold and confined between two glass microscope slides (1 mm in thickness), and constant pressure was applied to extrude the excess materials. Except for the glass ionomer materials, all of the restorative materials were polymerized according to the manufacturers' recommended polymerization duration with a LED light-curing unit (Elipar S10, 3M ESPE) operating in standard mode. The tip of the light-curing unit was placed perpendicular to the sample's surface at a distance of 1 mm and before the beginning of each polymerization, the power of the curing unit was measured with a radiometer (Hilux Curing Light Meter). Capsules of the high viscosity glass ionomer material (EFF) were auto-mixed in the vibrating machine according to instructions and then applied to the mold. Extruded materials were removed with a #12 scalpel blade. Afterward, samples were removed from the mold and kept in distilled water at 37°C in a stove for 24 h. Aluminum-oxide discs (Sof-Lex, 3M ESPE) were used for finishing and polishing procedures of the surfaces of all samples. To reduce variability, finishing and polishing procedures were performed by the same investigator and the discs were renewed after their 3rd use. Then all of the samples were kept in distilled water at 37°C until mechanical testing.

Nano-examination

Nanohardness (Hnano) and reduced elastic modulus (Er) were tested using a nanoindentor (TI 950 Triboindentor; Hysitron, USA). Prior to testing, a Berkovich diamond indenter tip was calibrated using a fused quartz reference sample. A set of indentation tests were performed on nine different locations per specimen surface to obtain more representative results, with a maximum load of 6000 µN for 2 s, under a loading/unloading rate of 1200 µN s^{-1}. After the penetration depth of the indenter tip achieved a pre-set maximum value, the normal load was reduced until the partial or complete relaxation occurred. Values for Hnano and Er have generated automatically from the software using the force (load)- displacement (depth) graph based on the OliverPharr relations(1). Load-depth curves generated from the nanoindentation scanning of one sample from each group are shown in Figure 1.

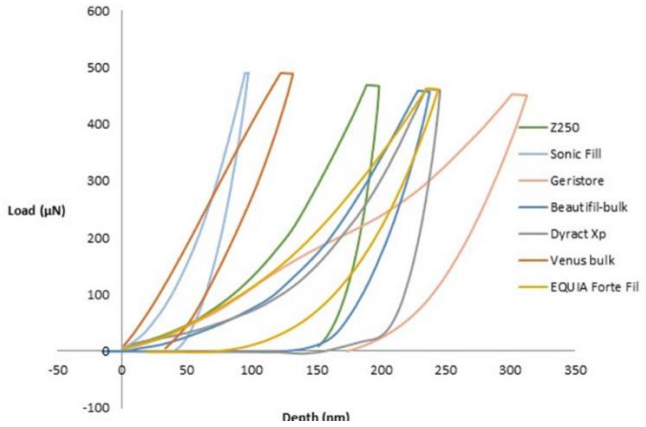


Figure 1.Load-depth curves of the one sample from each group of tested materials.

SEM examination

A novel sample was prepared from each of the groups for SEM examination. Samples were sputter-coated with gold (Polaron SC7620) and examined under the SEM (JEOL 5500 LV) at 10 kV accelerating voltage. Photographs of the representative areas of the polished surfaces were taken under ×1000 magnification by the same operator.

Statistical Analysis

The statistical analysis was done using SPSS 23.0 at a significance level of 0.05. The results were primarily analyzed using the Shapiro Wilk test to determine the existence of a normal distribution. Since the data were not normally distributed, differences observed within each material were analyzed by the Kruskal-Wallis test.

Results

Scores of the investigated properties of the tested materials are summarized in Table II. Due to the abnormal distribution of the data, statistical evaluations were calculated according to median scores.

TableII.Median, minimum-maximum values and statistical differences of nanohardness (Hnano (GPa)) elasticity modulus (Er (GPa)) of all tested materials*.

MATERIALS	n	H nano (median + min.-max.)	Er (median + min.-max.)
Equia Forte Fil (EFF)	9	0.5 (0.4 – 0.6)^{A,B}	5.6 (5 – 6.5)^{a,c}
Geristore (GS)	9	0.3 (0 – 3.6)^{A}	4.8 (1.2 – 16.6)^{a}
Dyract XP (DXP)	9	0.5 (0.1 – 0.7)^{A,B}	14.3 (9.1 – 17.1)^{b}
Venus Bulk Fill (VB)	9	0.6 (0.3 – 2.6)^{A,B}	8.5 (6.7 – 21.1)^{a,b,c}
SonicFill (SF)	9	1 (0.4 – 2.3)^{B}	17.1 (11.8 – 32.3)^{b}
Beautifil-Bulk (BB)	9	0.4 (0.1 – 1.5)^{A,B}	7.4 (3.4 – 37.8)^{a,b,c}
Filtek Z250 (Z250)	9	0.5 (0.2 – 0.9)^{A,B}	16.5 (5.2 – 22.9)^{b}

* According to Kruskal Wallis test, different uppercase letters show significant difference among restorative materials.

Due to statistical analysis, considerably diverse nanohardness and elasticity modulus (Er) values were gained among materials P<0.05). SF showed the highest nanohardness score, which was not significantly different from VB, DXP, EFF, BB, and Z250 (P>0.05). However, a marked difference in nanohardness values was obtained between SF and GS groups in which GS showed the lowest score among all. SF also had the highest Er score among all groups, followed by Z250 and DXP with no significant difference (P>0.05). GS had predominantly the lowest Er value among all groups, followed by EFF. Er scores of VB and BB were not significantly different than other tested materials (P>0.05).

The comparison of the Hnano and Er scores of all groups can be seen in Figures 2 and 3.

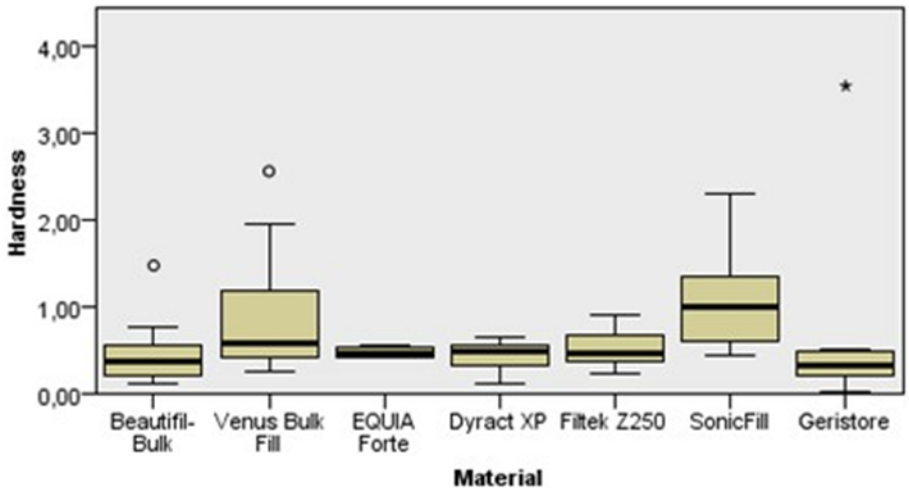


Figure 2. Box plot graphic of Hnano scores of all tested materials.

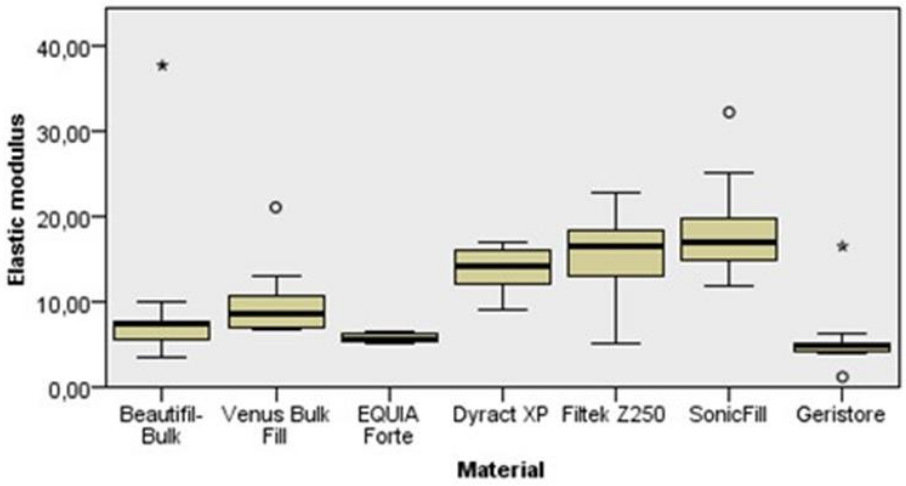
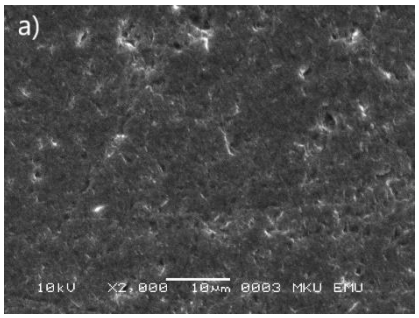


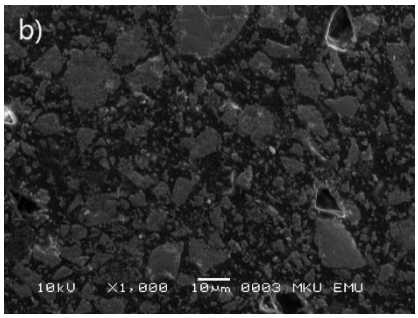
Figure 3. Box plot graphic of Er scores of all tested materials.

SEM evaluation

SEM images of all tested materials are seen in Figure 4.

Various types and shapes of the inorganic phase of tested dental materials were distinctly followed in SEM figures. The irregular glass filler, nano-, and elliptic-filler shapes are obvious in Figure 4 (4b, c, d, f, g). Some missing fillers (Figure 4a, f, g) that may be protruding from the material surfaces, scratches, and voids (Figure 4a, e, g), possibly created after the finishing procedures, are also seen.





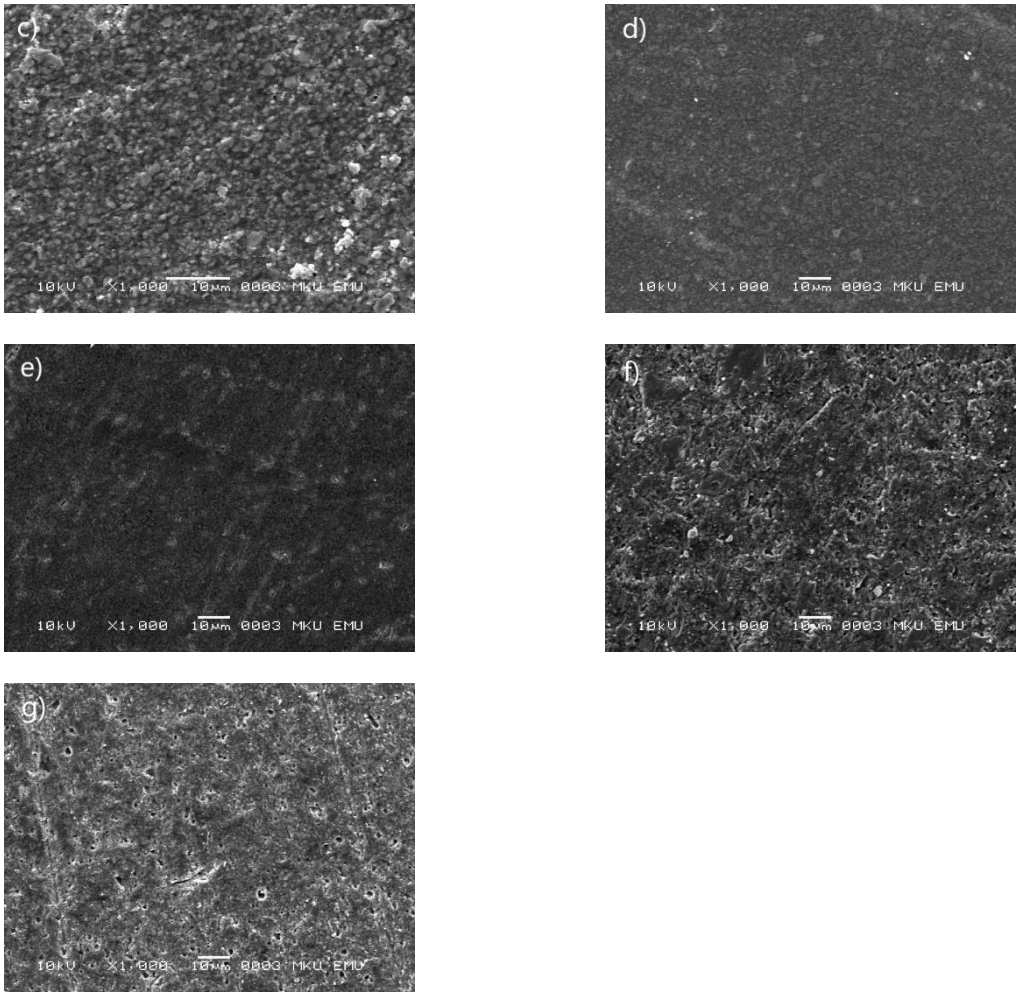


Figure 4. Scanning electron micrographs of the surfaces of the tested restorative materials at ×1000 magnification.*
* Images represent a) EFF, b) GS, c) DXP, d) VB, e) SF, f) BB, g) Z250.

Discussion

Over many years, there have been various changes in the organic and inorganic phases of these materials to improve the materials' response to immense masticatory forces and durability. The materials' advantageous parts and desirable structures were brought together, such as hybrid ionomers, and launched for clinical use. However, there are still commonly mentioned clinical issues relating to dental restorative materials, such as bulk fractures, wear, and marginal degradation, which could be related to material shortcomings (12,13,14). In the present study, two of the mechanical properties directly affecting the wear and degradation of the biomaterials were investigated: nanohardness and elasticity modulus.

Nanoindentation is a widespread method for measuring the mechanical properties of dental materials and is used in many types of research (15-17). The advantage of using nanoindentation techniques compared to conventional testing methods is not to damage the Material's microstructure (18). Besides, nanoindentation is used in a static approach which was reported to show a reliable correlation between indentation modulus and the elastic modulus for composite materials (10, 19, 20). Therefore, nanoindentation was used for the nanomechanical evaluation of various tested materials in the present study.

Differences in both organic and inorganic matrices of resin composites may affect the response of restorative materials to oral forces. In a study evaluating the structural properties of composite resins, it was reported that the structure of the organic matrix, types, and the number of fillers were the most crucial aspects of determining the mechanical properties of dental composites (21, 22). Bulk-fill composites are a material category of resin composites altered by their incremental application of 4- or 5- mm's. These materials represented a lower total filler-matrix interface than conventional composite resins with lower filler size, resulting in increased light transmittance throughout the composite

bulk and the enhanced depth of cure (23). Therefore, with the improved curing of the material, the nanohardness values were expected to be higher in this category of resin composites (24). The results of the nanohardness values of the present study are partly per the related finding that SF, which is a bulk-fill composite, showed the highest nanohardness score among all groups significantly. However, other groups had no significant difference from each other. Nevertheless, the other tested bulk-fill composites (VB) showed superior results over other composite groups; thus, the first hypothesis of the present study is rejected.

Due to the nanohardness results, GS, termed as "resin ionomer" by the manufacturer, had the most extensive lowest score. It is a hybrid material containing fluorosilicate glass similar to the matrix of glass ionomer materials. Monomers (HEMA and dimethacrylates) and inorganic fillers (silica) of GS are similar to conventional resin composites. Irregularly shaped fillers with miscellaneous dimensions were seen at SEM images (Figure 4b). As this material has many restorative indications, including both the cervical and coronal parts of the teeth, it should resist both the destructive forces, temperature changes, and staining probabilities of the oral environment. However, in both parameters, GS demonstrated lower scores than other tested materials. It could be attributed to GS's main organic monomer, which is a highly hydrophilic HEMA monomer. When HEMA-based material is exposed to acidity or an aqueous environment, ester groups in the structure hydrolyze quickly (25). In the present study design, tested materials were kept in distilled water for more than 24 hours. Thus, this process may affect the resistance of the organic matrix and accelerate the degree of hydrolysis. Another factor to GS's inferior nanohardness values may be caused by the lower filler ratio of the material (50%) compared to other groups (Figure 4).

Another tested material containing a lower filler ratio was DXP (47%), which presented an average nanohardness value with a similarly reduced elasticity modulus to Z250 and SF. Microscopically, fillers distribute the incoming force into smaller components, prevent the crack from growing, and are directly affected by the elasticity modulus of the material (10,26,27). In theory, if filler contents were increased with decreased particle sizes and inter-filler spacing, the elasticity modulus of the material may increase considerably with the fatigue and stress limit (28,29). A similar image of the related structure can also be seen in the SEM images (Figure 4c) of the DXP group. To explain more, the filler ratio, its distribution, and the size of the filler affect the mechanical properties of particular resin composites. The reason may be explained by the larger surface-area-to-volume ratio of the fillers present in restorative materials that also tends to accelerate the water uptake and results in the decomposition of the filler/matrix interface, lowering the mechanical properties (10). That is also why nanohybrid resin composites are showing lower hardness values compared to microhybrid composites in general. As a microhybrid composite, Z250 showed significantly higher Er values than other groups and similar nanohardness scores to other bulk-fill composites. Thus, the second hypothesis of the present study is rejected as well. One of the tested nanohybrid composite, VB, had significantly reduced nanomechanical properties compared to Z250 which may be caused by the lower viscosity level of VB. As it is a flowable composite, VB is indicated in the small-/medium-size cavities of permanent teeth and in all-sized cavities of deciduous teeth. According to the results gained in the present study and compared to other tested composite resins, it may be beneficial to use VB as an underlying composite in the stress-bearing areas of large cavities in permanent teeth. A similar outcome was mentioned in related parameters of previous studies as well (30, 31).

Evaluating the flowable bulk-fill composites, a lower elasticity modulus may allow stress dissipation during the curing process, allowing a larger incremental usage (32, 33). BB, a giomer bulk-fill with high viscosity (sculptable), showed lower Hnano and Er results; however, SF, which is termed as both flowable and a sculptable bulk-fill composite, had the highest scores significantly. SF is used with a handpiece that modifies its level of flowability up to 87%. However, when the sonic energy is eliminated, the composite returns to a more viscous situation (34). The higher scores of SF, which were in accordance with previous studies (34,35), could be attributed to its increased filler ratio (83.5%) and its high polymer density, or both. The densed structure of SF may be followed from the part of its surface character (Figure 4e).

Considering there is no significant difference among nanohardness scores of materials except for the Z250 material, using only Er scores is not adequate to reach a general outcome in structural properties of the tested materials. The Er values describe the stiffness/resilience of the materials under forces (3). It is desirable to use restorative materials that are resilient enough to act with the tooth structure as a unit and stiff enough to withstand masticatory forces. Nevertheless, the differences in the structure of ion releasing may affect the response of restorative materials. EFF, as a glass ionomer material, is expected to have the highest fluoride release due to its thicker glass-hydrogel matrix (36). Besides, GS, BB, and

DXP contain fluoro-silicate matrices with various other ions. DXP had a higher nanohardness score than GS and BB, which could be attributed to its lower fluoride release as a more composite-like structure (37). It is evident that DXP's curing reaction is initiated by light polymerization. However, after water uptake utilizing its highly hydrophilic TCB monomer (38), an acid-based reaction occurs, and fluoride ions are released. Conversely, the acid-based reaction in BB, a giomer composite, which occurred in S-PRG fillers during their manufacturing process, resulted in a surface-modified layer that protected the glass matrix from the damaging effects of water absorption (27). However, BB's related cross-linked polymer matrix results in higher nanomechanical properties than the materials with the gel network formed by an acid-based reaction. Numerically lower scores of BB than DXP were obtained, and those scores are compatible with Yap et al.'s study (39).

Finally, it should be noted that the present study is fulfilled for *in vitro* conditions. There were not any time-dependent or oral environment-related parameters that could alter the results, e.g., saliva. Samples were only kept in distilled water during the experimental process; thus, the effect of temperature or Ph fluid changes that could affect the ion releasing properties of the materials are not investigated.

Conclusion

Within the limitations of the present *in vitro* study, the main structural differences between glass ionomer-based and resin-based materials determined significant differences among tests. Given the lower nanohardness and elasticity modulus properties of glass ionomer-based materials, hybrid materials that could be more closely related to composite resins could be manufactured to eliminate the concerns regarding mechanical behavior to occlusal loadings. Further *in vitro* studies should be carried out to address more tests to predict the clinical performance of various restorative materials over time.

Conflict of interest

Authors declare that there is no conflict of interests.

Acknowledgements

None.

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