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ORIGINAL RESEARCH

Evaluation of nanohardness, elastic modulus, and surface roughness of fluoride-releasing tooth colored restorative materials

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Abstract

Recently, interest in tooth-colored fluoride-releasing dental materials has increased. Although physical and mechanical properties such as surface hardness, elastic modulus and surface roughness of the restorative materials have been investigated, the effect of different immersion media on these properties is still controversial. The aim of this study was to evaluate the nanohardness, elastic modulus and surface roughness of the fluoride release of tooth-colored restorative materials after immersion in acidic beverages. Prepared samples of three restorative materials (a highly viscous glass ionomer (EQUIA Forte; GC, Tokyo, Japan), a compomer (Dyract XP; Dentsply, Weybridge, UK), and a bioactive restorative material (Activa BioACTIVE; Pulpdent, MA, USA)) were randomly divided and immersed in distilled water, a cola and an orange juice for one week. The HYSITRON T1 950 TriboIndenter device (Hysitron, USA) with the Berkovich diamond indenter tip was used for all measurements. The nanohardness and elastic modulus of the samples were measured by applying a force of 6000 μN to five different points on the sample surface. Surface roughness measurements were evaluated on random samples by scanning five random 40 $\times$ 40 μm areas. The properties were measured at the initial and one week after immersion. The values of nanohardness, elastic modulus and surface roughness were tested for significant differences using a two-way analysis of variance (ANOVA) with repeated measures ($p < 0.05$). Tukey's honest significant difference (HSD) test was used for multiple comparisons. AB (Activa BioACTIVE) had the highest initial mean values for nanohardness. After post-immersion, the highest mean value for elastic modulus was the initial AB value. The lowest mean value for roughness of 100.36 nm was obtained for the initial DX (Dyract XP) measurement. Acidic beverages had a negative effect on the nanohardness, elastic modulus and surface roughness of the restorative materials.

Keywords

Fluoride releasing restorative material; Nanohardness; Elastic modulus; Surface roughness

1. Introduction

Restorative dental treatments aim to replace tooth tissues lost due to caries or trauma with appropriate dental materials [1]. Previously, with conventional dental materials, the treatment process had only passive efficacy, such as removing caries and placing dental tissue-like dental material. Along with the newly developed materials by releasing fluoride into the dental tissues and oral environment, some of them play an active role in the treatment period [2, 3]. Fluoride released from restorative dental materials has antibacterial effects, inhibits demineralization, and increases remineralization [2]. In this way, it prevents the formation of secondary caries. Conventional glass ionomer cement (CGIC) (high viscosity glass ionomer and cermet cement), resin-modified glass ionomer cement, nano-ionomer cement, compomers and fluoride-containing composite resins are examples of restorative materials that release fluoride [3].

For preventing secondary caries, glass ionomer cement (GICs) were defined as fluoride reservoirs by releasing the fluoride ions to the tooth surface [3, 4]. However, previous studies have reported that these materials' physical and surface properties should be improved [4]. GIC with a resin coat has shown better nanohardness results and less discoloration [5]. Currently, previous studies have shown that highly viscous glass ionomer cement (HVGIC) with a resin coat has been successful in posterior restorations in terms of the relative tolerance to moisture, anti-cariogenic nature, and fluoride release [4, 5]. One of them is EQUIA Forte (EF), used with a coat. EF, which releases fluoride ions, is more abrasion-resistant than glass ionomer cement [5].

Compomer was also defined as polyacid-modified composite resins. The curing reaction of the compomer is an additional polymerization reaction as a dental composite material [6]. When the compomer contacts oral environment, water absorption begins into the material structure. Due to this process, this reaction triggers an acid-base reaction between the glass fillers and the acid groups of the functional monomer [6]. After that, fluoride is released from the glass filler to the matrix and then to the oral environment [6]. Although the compomer has better mechanical properties than CGIC, it has been reported to have less polymerization shrinkage and less fluoride release than CGIC [7]. Dyract XP (DX) is a compomer that contains strontium fluorosilicate glass filler and hydrophilic tetracarboxylic acid hydroxyethyl methacrylate ester. It has been reported that this structure can cause excessive water absorption [8].

Recently, bioactive restorative materials have gained popularity. In addition to having a bioactive filler and matrix, they release fluoride and calcium ions [9]. It has been stated that it shows better aesthetic results compared to GICs. Activa BioACTIVE (AB) is described by its manufacturer as a bioactive restorative material [9]. However, it is clear that more studies about AB are needed due to the new material.

To evaluate the mechanical properties of dental materials, nanoindenter testing methods have been used [10]. This technique can be performed on the sample in minimal or non-invasive methods, with reproducible results. Nanoindentation tests can provide important information about hardness and elastic modulus values [10]. Additionally, nanoindentation with atomic force microscopy (AFM) allows for investigating topographic properties on the sample surface. The surface roughness parameters can be obtained by quantitative and topographic visually [11]. It is obvious that for the restorations to be long-term, there is a need to evaluate the current materials with current test methods.

However, it is a known fact that different beverages cause degradation in the matrix structure of resins [12]. Therefore, this study aimed to evaluate the nanohardness, elastic modulus and surface roughness of an HVGIC "EQUIA Forte", a compomer "Dyract XP" and a new bioactive material "Activa BioACTIVE" according to the immersed media by current test methods. This research hypothesizes that the tested beverages will affect the nanohardness, elastic modulus and surface roughness of restorative materials.

2. Materials and methods

In this study, a highly viscous glass ionomer cement (EQUIA Forte; GC, Tokyo, Japan), a compomer (Dyract XP; Dentsply, Weybridge, UK), and a bioactive restorative material (Activa BioACTIVE; Pulpdent, MA, USA) were used as fluoride-releasing restorative materials. The compositions of these materials are listed in Table 1.

2.1 Preparation of samples

Sectional polyethylene molds of 2 $\times$ 6 mm were used the preparation of the specimens. 24 samples were prepared for each material (n = 8). The manufacturer's recommendations were followed for curing/polymarization and preparation of the samples.

EF capsules were mixed for 10 seconds using with amalgamator device (ZoneRay, Treedental, Guangdong, China) and placed in the mold with a gun. The EF samples were coated with EQUIA Forte Coat (GC, USA). The resin containing DX and AB samples were placed in the mold in a single layer. The mylar strip and plastic mold were placed on the glass slide, respectively. All samples were polymerized for 20 s using a polywave LED (light-emitting diode) device (VALO Cordless, Ultradent Inc, South Jordan, UT, USA) in standard mode (1000 mW/cm^2). The irradiance value was measured using a radiometer (Demetron, Kerr, Sybron Dental, Orange, CA, USA). The excessive materials were carefully removed. The surfaces of the prepared samples were polished under water cooling using the FORCIPOL 2 V polishing machine (FORCIPOL 2 V, METKON, Bucharest, Romania) with 400, 800, 1200 and 2400 grit carbide paper. After finishing and polishing procedures, specimens were immersed in distilled water for 24 h and stored at room temperature (36 $\pm$ 1 °C). The restorative materials were randomly divided into subgroups.

After initial measurements, the samples were immersed in distilled water (MOS LAB, Ankara, Turkey), a cola (The Coca Cola Company, Istanbul, Turkey), and an orange juice (Cappy, Coca Cola Company, Istanbul, Turkey) for one week (n = 8). Beverages were changed daily. All measurements were repeated and recorded after immersion for a week.

2.2 Measurements of the nanohardness, elastic modulus and surface roughness

The nanoindenter testing device (HYSITRON TI950 TriboIndenter, Bruker Corp., Karlsruhe, Germany) with the Berkovich diamond indenter tip was used for all measurements. Nanohardness, elastic modulus and surface roughness measurements were obtained from each sample. Nanohardness and elastic modulus of the samples were measured by applying 6000 μN of force on five different points of the sample surface [10]. The mean was calculated from these measurements.

Surface roughness measurements were evaluated on random samples by scanning five 40 $\times$ 40 μm areas (0.05 nm/sec scan rate). For five scanning areas, the first scanning area was the center point, and the other areas were peripheral points. The surface roughness parameters were obtained numerically. The roughness values in Ra (nm) were recorded, and the mean measurements were obtained.

2.3 Statistical analysis

Shapiro-Wilk test confirmed the normal distribution of nanohardness, elastic modulus and surface roughness data; therefore, parametric tests were used for inferential analysis. A paired t-test was used for intra-group comparisons of initial and after immersion in distilled water, cola and orange juice. Mauchly's test was used to examine whether the covariance matrix of the variables was spherical in repeated measurements. Nanohardness, elastic modulus, and surface roughness values were tested for significant differences ($p < 0.05$) by two-way analysis of variance

TABLE 1. Dental materials used in this study.

Materials	Lot No	Type	Composition
EQUIA Forte (EF) (GC, Tokyo, Japan)	2106012	HVGIC	Powder: 95% strontium fluoro alumino-silicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid; EQUIA Forte Coat: 40%–50% methyl methacrylate, 10%–15% colloidal silica, 0.09% camphorquinone, 30%–40% urethane methacrylate, 1%–5% phosphoric ester monomer
Dyract XP (DX) (Dentsply, Weybridge, UK)	848	Compomer	UDMA, TCB, Strontium-fluoro silicate glass (mean filler size 0.8 μm), Filler ratio: 47% wt
Activa BioACTIVE (AB) (Pulpdent, MA, USA)	210324	Bioactive Restorative Material	Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%), amorphous silica (6.7%), and sodium fluoride (0.75%). 56% by weight reactive glass particles

HVGIC: highly viscous glass ionomer cement; UDMA: Urethane dimethacrylate; TCB: tetrachlorobiohenyl.

(ANOVA) with repeated measures. Tukey's honest significant difference (HSD) test was used for multiple comparisons. This analysis was repeated six times for each solution, and all analyses comparing "distilled water × 3 brand", "cola × 3 brand", "orange juice × 3 brand", "EQUIA Forte × 3 solution", "Dyract XP × 3 solution" and "Activa BioActive × 3 solution". All statistical tests used in this study were performed at a 5% significance level, and the hypotheses were two-tailed. All statistical analyses were performed by using SPSS (Statistical Package for the Social Sciences, SPSS Inc., Chicago, IL, USA) 21.0 package program.

3. Results

3.1 Nanohardness results

Nanohardness analysis results are shown in Table 2. It was determined that brands, solutions and brand-solution interaction created statistically significant differences in hardness values at initial and after immersion in different solutions for seven days ($p < 0.001$). Both at the beginning and on the 7th day, AB samples have superior results compared to the others. The nanohardness results in all solutions statistically significantly decreased ($p < 0.05$). It was determined that the decrease in orange juice and cola was significantly greater than in distilled water ($p < 0.001$).

3.2 Elastic modulus results

Elastic modulus findings of the initial and post-immersion measurements are shown in Table 3.

Elastic modulus analysis results are shown in Table 3. It was determined that brands, solutions and brand-solution interaction created statistically significant differences in elastic modulus values before and after immersion in different solutions for seven days ($p < 0.001$). After seven days, the values of the elastic modulus of AB and DX were found to be better in all solutions compared to EF ($p < 0.05$). The decrease in elastic modulus in orange juice and cola was significantly greater than in distilled water ($p < 0.001$).

3.3 Surface roughness results

Surface roughness findings of the initial and after-immersion measurements are shown in Table 4. It was determined that the brand*solution interaction created statistically significant differences in surface roughness values before and after immersion in different solutions ($p < 0.001$). It was found that AB and DX samples were significantly superior to the EF brand in terms of surface roughness ($p < 0.05$).

A very strong statistically significant positive correlation was found between nanohardness (GPa) values and elastic modulus (GPa) values (r = 0.951; $p < 0.001$). Negative, weakly statistically significant correlations were detected between roughness (nm) values and hardness values ($r = -0.372$; $p < 0.001$) (Table 5).

4. Discussion

This *in vitro* study investigated the nanohardness, elastic modulus and surface roughness of the HVGIC, compomer, and bioactive material in different immersed media. The nanoindentation method was used for nanohardness and elastic modulus. However, the nanoindentation method with the AFM method was used for the surface roughness.

Restorative materials are exposed to intense occlusal forces in the oral cavity, and their hardness values must be high to resist these forces [13]. One of the methods that gives information about the physical properties of restorative materials is surface hardness tests. Knoop and Vickers tests are frequently used to measure the surface hardness of dental materials [13]. However, unlike these tests, which measure hardness at micro and macro levels, in nanohardness tests, the load and displacement process are recorded with higher precision and nano-sized indentations are made [10, 14]. For this reason, the nanohardness test was used to evaluate the surface hardness in this study.

The hardness value of the material is related to the material composition. The type, chemistry, morphology and size of the filler influence the hardness performance of the material [15]. Low filler content in the materials causes a low hardness value [15]. So, in our study, when the initial hardness values

TABLE 2. The nanohardness values (GPa) of tested materials.

	Distilled water		Cola		Orange juice	
	Initial	After immersion	Initial	After immersion	Initial	After immersion
EF	0.58 (0.02)^{Aa}	0.48 (0.01)^{Ca}	0.56 (0.02)^{Aa}	0.33 (0.01)^{Bb}	0.56 (0.02)^{Aa}	0.25 (0.01)^{Bb}
DX	0.66 (0.01)^{Ab}	0.51 (0.01)^{Bb}	0.67 (0.01)^{Ab}	0.29 (0.01)^{Ca}	0.66 (0.02)^{Ab}	0.23 (0.01)^{Ca}
AB	0.78 (0.03)^{Ac}	0.71 (0.01)^{Bc}	0.77 (0.03)^{Ac}	0.50 (0.01)^{Bc}	0.78 (0.02)^{Ac}	0.56 (0.02)^{Bc}

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different ($p < 0.05$). EF: EQUIA Forte; DX: Dyract XP; AB: Activa BioACTIVE.

TABLE 3. The elastic modulus values (GPa) of tested materials.

	Distilled water		Cola		Orange juice	
	Initial	After immersion	Initial	After immersion	Initial	After immersion
EF	8.70 (0.20)^{Aa}	7.28 (0.08)^{Ba}	8.71 (0.14)^{Aa}	3.33 (0.09)^{Ca}	8.58 (0.23)^{Aa}	3.07 (0.09)^{Da}
DX	9.66 (0.10)^{Ab}	7.83 (0.08)^{Db}	9.54 (0.11)^{Ab}	3.74 (0.08)^{Cb}	9.54 (0.16)^{Ab}	3.05 (0.26)^{Ba}
AB	10.69 (0.13)^{Ac}	10.16 (0.17)^{Dc}	10.65 (0.19)^{Ac}	6.42 (0.07)^{Cc}	10.55 (0.13)^{Ac}	5.19 (0.08)^{Bb}

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different ($p < 0.05$). EF: EQUIA Forte; DX: Dyract XP; AB: Activa BioACTIVE.

TABLE 4. The surface roughness values (nm) of the tested materials.

	Distilled water		Cola		Orange juice	
	Initial	After immersion	Initial	After immersion	Initial	After immersion
EF	146.89 (71.47)^{Ab}	214.76 (32.40)^{Ac}	190.92 (32.66)^{Ab}	252.04 (35.48)^{Bc}	177.04 (36.15)^{Ab}	196.11 (48.45)^{Aa}
DX	100.34 (38.05)^{Aa}	133.79 (17.92)^{Ab}	124.25 (29.76)^{Aa}	162.06 (63.60)^{Aa}	101.26 (17.51)^{Aa}	179.71 (80.12)^{Ba}
AB	122.59 (23.94)^{Ab}	118.05 (44.07)^{Aa}	134.16 (28.71)^{Aa}	214.41 (92.87)^{Aa}	155.35 (40.52)^{Aa}	162.29 (90.07)^{Aa}

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different ($p < 0.05$). EF: EQUIA Forte; DX: Dyract XP; AB: Activa BioACTIVE.

TABLE 5. The results of the Pearson correlation analysis of testing parameters.

	Elastic Modulus (GPa)	Nanohardness (GPa)
Nanohardness (GPa)		
r	0.951	
p	<0.001	
Surface Roughness (nm)		
r	−0.398	−0.372
p	<0.001	<0.001

r: Pearson correlation coefficient.

of the materials were compared, it was found that the AB material had a higher filler content and a higher hardness result than the other materials. Also, a previous study has shown that bioactive restorative material at 24 h has a statistically equal result with the composite resin. Also, it showed that AB has better hardness results than resin-modified glass ionomers [16].

A previous study reported that the EF's results with resin coated are better than the other testing materials. However, the study has not supported our results by explaining this situation with the resin-coated [5]. Another previous study that compared the relation of the hardness values of the compomer, resin-modified glass ionomer, and bioactive restorative material showed that the bioactive material has the lowest hardness value [17]. The tendencies were inconsistent with our result. This might be attributed to the nanohardness method. Also, it is clear that many studies about the nanohardness values of the bioactive materials are needed.

The material structures and the storage conditions are effective on the hardness values of restorative materials. When the resin materials were immersed in the liquid media, volumetric expansion and deformation in the resin material structure were shown. As a result of the volumetric expansion, filler particles are released from the organic matrix, and the hardness of the resin material is decreased [5, 8]. A previous study has shown that the hardness results were decreased according to storage conditions because of the immersion of restorative materials in current liquids. The negative impact is explained by the liquid absorption of the resin materials [8].

Consistent with the previous studies, all materials have shown decreased values after immersion media in our study. Glass ionomer cement and compomer have structural ions in the glass phase. Because of the structural ions' contact with the acid attack, the dissolution caused a decreased microhardness value [18]. Also, AB after immersion showed lower decreased nanohardness changes than the initials. Urethane dimethacrylate (UDMA) is one of the examples carrying higher molecular weight (MW = 470 g/mol) and low viscosity with high flexibility resulting in higher flexural strength, elastic modulus and hardness. DX includes the UDMA structure, and AB includes the blend of urethane (UDMA) with other methacrylates. This can be explained by the results of the AB immersed in media compared with the DX [19].

The study aims to evaluate the change in elastic modulus of different restorative materials, which occurs by immersing them in acidic beverages using the nanoindentation testing method. The elastic modulus is related to the amount of deformation generated by the material against the applied force [20]. A material has a high elastic modulus value; restoration with this material deforms less against the occlusal forces [20]. In our study, the initial elastic modulus values of the samples are higher than those immersed in beverages. Previous studies have shown that the water sorption of materials caused negative effects on the elastic modulus [21].

The depth-sensitive indentation technique defined with the advancement has made it possible to examine these two parameters together. Thanks to this method, we were able to obtain both nano hardness and elastic modulus values at once. Also, in the previous studies, hardness is expressed as reduced elastic modulus [14, 22]. According to our results, there is a positive relationship between hardness and elastic modulus. Similar to previous studies [21–24], a very high statistically positive correlation was found between hardness (GPa) values and elastic modulus (GPa) values [21–24].

Scanning probe microscopes and AFM have been used to evaluate surface roughness. AFM provides quantitative results with three-dimensional images [11]. In our study, to obtain quantitative results of the surface roughness, the nanoindentation with the AFM method was used for the surface roughness measurements.

The surface roughness that occurs after finishing and polishing processes significantly affects the clinical success of the restoration [25]. While composite resins with low surface roughness increase clinical success by providing the aesthetic appearance of the restoration, resins with high surface roughness cause clinical failures such as plaque accumulation, secondary caries formation and discoloration. For this reason, composite resins with low surface roughness should be preferred in restoration construction to obtain the best clinical result from composite restorations [25].

Nasim *et al*. [26] found that the coloration caused by different coloring solutions in the resin material occurred within the first week at most. However, Ortengren *et al*. [27] stated that most organic components were removed from the structure in the first seven days. In line with this information, we planned the immersion period for beverages to be seven days in the study. Orange juice and cola were chosen in this study because they are beverages that children frequently consume.

Also, beverages with low pH can cause solubility in the restorative materials. This solubility results in surface erosion and dissolution, affecting the surface roughness and hardness of the materials [28]. In our study, when all samples were examined, samples immersed in cola and orange juice showed higher surface roughness values than distilled water.

According to a systematic review and meta-analysis study, the authors concluded that resin-modified GIC/GICs showed higher surface roughness compared to resin composites in all follow-ups of clinical studies [29]. In our study, when the surface roughness of all the materials was examined, glass ionomer base material with resin coat showed higher surface roughness than resin-containing materials. In a study by Savas S *et al*. [30], evaluating the surface roughness of glass ionomer-containing materials after immersion in cola, HVGICs (EF and GCP Glass Fill) showed marked deterioration at the end of the immersion period. In our study, in the same way, EF samples immersed in cola showed the highest surface roughness. Kazak M *et al*. [31], examined the surface roughness of fluoride-releasing materials, and there was no statistically significant difference in surface roughness when they compared AB and EF samples, which were kept in distilled water for 24 h before thermal aging. In our study, the surface roughness of AB and EF samples kept in distilled water for 24 h, EF samples showed higher surface roughness than AB.

Bayrak GD *et al*. [32], examined the surface roughness of restorative materials, and there was no statistically significant difference in surface roughness when they compared AB and DX samples, which were kept in distilled water for 24 h. In our study, the surface roughness of AB and DX samples kept in distilled water for 24 h, DX samples showed higher surface roughness than AB samples. This result can be attributed to the higher resin content in AB.

This study showed that cola and orange juice beverages negatively affected the nanohardness, elastic modulus and surface roughness of these materials. Our hypothesis was accepted. However, the simulation of the oral environment could not be recreated. This study's limitations include the ionic composition of food/beverage, pH changes, salivary enzymes, wear and abrasion. It should be noted that an *in vitro* study may not be representative of all conditions and interactions that affect restorative materials. Therefore, the physiochemical reactions between the materials and the beverages should be analyzed in future experiments.

5. Conclusions

The fluoride releasing materials examined in this study can be classified as HVGIC (EQUIA Forte), compomer (Dyract XP) and bioactive restorative material (Activa BioActive) according to the different immersed media. Based on the results of this study,

1. According to the nanohardness results, Activa BioActive had a higher nanohardness results than the EQUIA Forte and Dyract XP. After immersion, beverages negatively affected the nanohardness of the all of the materials.

2. For the elastic modulus, Activa BioActive and Dyract XP have better results compared to EQUIA Forte. Beverages negatively affected the elastic modulus of the fluoride releasing materials.

3. According to the surface roughness results, Activa BioActive and Dyract XP have better results compared to EQUIA Forte. After immersion, the roughness values of the materials have increased.

4. The nanohardness, elastic modulus and surface roughness of bioactive restorative material may be comparable or even superior to HVGIC and compomer.

5. The present findings demonstrate that nanoindentation techniques can potentially be used to evaluate both nanohardness, elastic modulus and surface roughness of dental materials. Thanks to the nano-sized indentations, this test technique gives the objective results for the dental materials having nano-sized composition. It can be considered as a routine method of dental materials.

Further *in vitro* studies should be carried out to investigate the other restorative material to improve the understanding of mechanical properties and clinical performance.

ABBREVIATIONS

AB, Activa Bioactive; AFM, atomic force microscopy; CGIC, conventional glass ionomer cement; DX, Dyract XP; EF, EQUIA Forte; GIC, glass ionomer cement; HVGIC, highly viscous glass ionomer cement; TCB, tetrachlorobiohenyl; UDMA, urethane dimethacrylate.

AVAILABILITY OF DATA AND MATERIALS

The data presented in this study are available on reasonable request from the corresponding author.

AUTHOR CONTRIBUTIONS

ZG—designed the research study; performed the research. HDK—analyzed the data. ZG and HDK—wrote the manuscript; revised and edited the manuscript. All authors read and approved the final manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

This study was approved by the Hatay Mustafa Kemal University Non-interventional Clinical Research Ethics Committee (17/06/2021/35) and complied with the principles of the Declaration of Helsinki.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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