

Effect of thermal aging procedure on the microhardness and surface roughness of fluoride containing materials

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Abstract

Aim: The aim of this in vitro study was to evaluate the thermal aging effect on microhardness and surface roughness of fluoride containing restorative materials.

Material and Methods: In this study, a bioactive material (Activa Bioactive Restorative, Pulpdent, Watertown, MA, USA), a giomer (Beautiful II, Shofu, Kyoto, Japan), a nanohybrid composite (Charisma Smart, Heraeus Kulzer, Germany), a resin-modified glass ionomer (Ionoseal, VOCO, Germany), and a bulk-fill glass-hybrid material (Equia Forte, GC, Tokyo, Japan) were used. 10×2 mm discs were prepared (n=10) and then polymerized. Only for bulk-fill glass-hybrid specimens, one layer of coat was applied on top surfaces and light cured. Before and after thermal aging procedures, initial and final surface roughness and microhardness values were evaluated. One-Way ANOVA test was used for the statistical analysis (p<0.05).

Results: Thermal aging did not affect the surface roughness of restorative materials statistically (p>0.05). After thermal aging when the bottom surface of Equia Forte compared to Activa Bioactive and Beautiful II in terms of microhardness values, a statistical significant difference was observed (p<0.05).

Conclusion: Even before and after thermal aging procedures, successful results can be achieved with bulk-fill glass-hybrid material.

Keywords: Cariostatic agents; hardness tests; mechanical properties; thermal analysis; surface properties

INTRODUCTION

The current restorative materials have superior features in the clinical use, such as good bonding to enamel and dentin structure, biocompatibility, color matching, aesthetic appearance, easy application, wear resistance, ideal surface roughness and hardness (1). Despite all the positive properties of these restorative materials, there is still a need to replace the restorations. Therefore; the search for the ideal restorative material still continues (2,3). One of the most important reasons of the replacement of the restorations is the secondary caries between the tooth and the restoration surface or under the restoration (4). As a solution, fluoride ion was added to the structure of the restorative materials to prevent secondary caries formation. Also it was found that, the release of fluoride ion from the restorative material increases the resistance of the dental hard tissues from caries and also prevent early caries lesions (1,2,5,6).

The surface properties of the restorative materials affect the stability and the success of the restoration (7,8). For this reason, besides the aesthetic property of a restorative material, surface hardness and roughness features should be considered (7-9).

A restorative material with an ideal surface microhardness, increases the resistance of the material to scratching and wear. In addition to that, an ideal surface microhardness of a restorative material should resist against various chewing forces (10). On the other hand; aesthetic problems, irritation of the gums and increase of plaque retention due to surface irregularity, discoloration of the material due to absorption and adsorption of oral fluids and formation of secondary caries have negative effects on the clinical life of the restorations that are all related with the surface roughness of the material (7,8,11).

In the oral environment, the restorative materials are subjected to various factors influences such as vertical and lateral stresses, saliva, heat and pH changes (12).

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These factors influences both affect significantly the success of the restoration also the surface features of the restorative material (13). In vitro aging procedure is performed in a certain accelerated period of time, with heat, humidity and light cycles that artificially simulates the oral environment which can lead to deterioration, degradation and unwanted changes in the restorative materials. Therefore, thermal aging procedure is a very valuable technique which sheds light on the studies about restorative materials (14,15). According to ISO TR 11450 (1994) standards, the thermal aging procedure performed in 10,000 cycles simulates (16,17) an average of 1 year in vivo conditions (18).

Therefore, the aim of this in vitro study was to investigate the effect of thermal aging procedure on the microhardness and surface roughness of restorative materials containing fluoride ion. The null hypothesis tested was, thermal aging procedure had a negative effect on the microhardness and surface roughness of the restorative materials containing fluoride ion.

MATERIAL and METHODS

A bioactive restorative material (Activa Bioactive Restorative, Pulpdent, Watertown, MA, USA), a giomer (Beautiful II, Shofu, Kyoto, Japan), a nanohybrid composite resin (Charisma Smart, Heraeus Kulzer, Germany), a resin modified glass ionomer material (Ionoseal, VOCO, Germany), a bulk-fill glass-hybrid restorative material (Equia Forte, GC, Tokyo, Japan) were used in this study. Ten samples were prepared from each group. The tested materials are listed in Table 1.

Specimen Preparation

For each tested material, ten cylindrical specimens (10 mm diameter×2 mm depth) were prepared using Teflon molds. The mold was first mounted on the top of a glass plate and a Mylar strip. The restorative materials were inserted into the molds overflowly according to the manufacturers' instructions. Then the teflon molds were covered with a Mylar strip and a second glass plate. Specimens were light cured with a LED Light Curing Unit (VALO Cordless,

Table 1. The tested materials

Groups	Materials	Manufacturers	Compositions	Shade	Lot Numbers
Group AB	Activa Bioactive Restorative (Bioactive restorative material)	Pulpdent, Watertown, MA, USA.	Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%), Amorphoussilica (6.7%), Sodium fluoride (0.75%)	A2	180209
Group BF	Beautiful II (Giomer)	Shofu, Kyoto, Japan	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA (88.3%), Fluoro silicate glass	A2	101711
Group CS	Charisma Smart (Nanohybrid resin composite)	Heraeus Kulzer, Germany	Bis-GMA, Barium Aluminum fluoride glass, silicon dioxide	A2	010508A
Group IS	Ionoseal (Resin modified glass ionomer material)	VOCO, Germany	Bis-GMA, UDMA, HEDMA, Fluoroaluminumsilicate	-	1749506
Group EF	Equia Forte Fill (Bulk-fill glass-hybrid restorative) + Equia Forte Coat	GC, Tokyo, Japan	Fluoro alumino silicate glass, UDMA, MMA + camphorquinone, phosphoric ester monomer	A2	1611221 1606151

Abbreviations Bis-GMA: Bisphenol A diglycidyl methacrylate; UDMA: Urethane dimethacrylate; Bis-MPEPP: Bisphenol A polyethoxy methacrylate; TEGDMA: Triethylene glycol dimethacrylate; HEDMA: 1,6-hexanediylbismethacrylate; MMA: Methyl methacrylate

Ultradent Products Inc., South Jordan, UT, USA) with an output of 1000 mW/cm² and a wavelength between 395 and 480 nanometers on the top surfaces of the materials with the light curing unit tip touching the glass.

Group AB: Activa Bioactive Restorative material was placed in a single layer in the mold and light cured for 20 sec.

Group BF: Beautifil II was placed in a single layer in the mold and light cured for 10 sec.

Group CS: Charisma Smart was placed in a single layer in the mold and light cured for 20 sec.

Group IS: Ionoseal was placed incrementally as 1 mm layers in the mold and light cured for 20 sec for each layer.

Group EF: Equia Forte capsule was placed into an amalgamator, mixed for 10 sec., placed in a single layer. Then the material was waited for the complete set.

Bottom and top surfaces of all specimens were polished using polishing discs (Sof-Lex, 3M ESPE, USA) from coarse to fine. Only for Group EF, according to the manufacturers' instruction, after polishing, one layer of Equia Forte Coat (GC, Tokyo, Japan) was applied on the top surfaces of the specimens with a microbrush and light cured for 20 sec. Then all the specimens were stored in distilled water at 37 °C for 24h. After storage, the initial surface roughness (top surfaces) and microhardness (top and bottom surfaces) measurements were performed for each specimen.

Surface Roughness

The surface roughness of the specimens (n=10) was measured using a profilometer (MarSurfM 300 C, Germany) with a tracing length of 5.6 mm and a cut off value of 0.8 mm. A reading was obtained by a diamond stylus moved at 0.5 mm/s, and then the arithmetic roughness (Ra) was recorded. This procedure was repeated at three position on the same specimen and the average was obtained from these values.

Microhardness

Vickers microhardness (VMH) measurements were performed using a HMV Microhardness Tester (Shimadzu, Japan) at a load of 200 g for 10 sec. on both top and bottom surfaces of each sample (n=10) from 3 different area. The VMH measurements of each surface were recorded as the average of these measurements.

Thermal Aging

Following the initial measurements, all of the specimens were thermocycled in an artificial saliva (KCl, KH₂PO₄, CaCl₂.2H₂O, MgCl₂, C₈H₁₈N₂O₄S HEPES, distilled water, hydroxyapatite, octacalcium phosphate, dicalcium phosphate dihydrate) for 10,000 cycles between 5 and 55 °C±2 °C with a dwelling time of 30 sec. (Thermocycler THE-1100, SD Mechatronik, Feldkirchen-Westerham, Germany).

After thermal cycling procedure, final surface roughness (top surfaces) and microhardness (top and bottom surfaces) measurements of the same specimens were performed.

Statistical Analyses

Data were analyzed with Shapiro Wilk test for normally distribution. Parametric test was used for the data which showed normally distribution. Microhardness and surface roughness were analyzed with One-Way ANOVA test a significance level of 0.05. Multiple comparisons were made with post-hoc TUKEY HSD test

RESULTS

Surface Roughness

Thermal aging procedure did not affect the surface roughness of the restorative materials statistically (p>0.05). When the materials compared to each other, Group CS showed statistically the highest surface roughness before and after thermal aging procedure (p<0.05) (Table 2).

Table 2. Comparison of surface roughness values of test materials before and after thermal aging

Groups	Before Thermal Aging (BTA)	After Thermal Aging (ATA)	p
Group AB	0.42(0.06) Aa	0.44(0.19) Aa	0.762
Group BF	0.43(0.31) Aa	0.41(0.30) Aa	0.828
Group CS	2.69(1.74) Ba	2.52(1.44) Ba	0.804
Group IS	0.77(0.04) Aa	0.92(0.56) Aa	0.447
Group EF	0.78(0.26) Aa	0.80(0.23) Aa	0.896

*Different capital letters within the columns indicate the statistically significant differences between the groups (p<0.05)

**The same lower letters on the same line indicate no statistically significant difference between the materials before and after thermal aging. (p>0.05)

***Abbreviations: AB, Activa Bioactive Restorative; BF, Beautifil II; CS, Charisma Smart; IS, Ionoseal; EF, Equia Forte

Microhardness

After thermal aging procedure, the top surface of Group EF showed the lowest microhardness value while the bottom surface showed the highest microhardness value. After thermal aging procedure when the bottom surface of Group EF compared to Group AB and BF in terms of microhardness values, a statistically significant difference was observed (p<0.05) (Table 3).

Table 3. Comparison of the microhardness values of the top and bottom surfaces of the materials before and after thermal aging

Groups	Before Thermal Aging (BTA)			After Thermal Aging (ATA)		
	Top surface	Bottom surface	p	Top surface	Bottom surface	p
Group AB	22.95(2.35) Aa	19.27(2.3) Ab	0.001	38.88(10.00) Aa	27.05(3.32) Ab	0.006
Group BF	54.73(4.94) Ca	45.56(3.20) Bb	0.001	48.73(3.44) Ba	44.01(7.52) Ba	0.084
Group CS	53.13(6.39) BCa	49.68(7.41) Ba	0.177	60.14(6.64) Ca	53.87(9.98) BCDA	0.205
Group IS	48.73(8.07) BCDA	53.18(7.97) Bb	0.037	63.83(9.51) DCA	64.88(10.53) CDA	0.833
Group EF	42.23(6.19) Da	54.08(12.63) Bb	0.047	35.67(6.49) Aa	66.23(14.39) Db	0.000

*Different capital letters within the columns indicate the statistically significant differences between the groups ($p < 0.05$).

**The same lower letters on the same line indicate no statistically significant difference between the materials before and after thermal aging. ($p > 0.05$).

***Abbreviations: AB, Activa Bioactive Restorative; BF, Beautifill II; CS, Charisma Smart; IS, Ionoseal; EF, Equia Forte.

DISCUSSION

In this study, the effect of thermal aging procedure on the microhardness and surface roughness of the restorative materials which contain fluoride ion were investigated. The results showed that thermal aging procedure affected the microhardness of the fluoride containing restorative materials while it did not alter the surface roughness. Thus, the null hypothesis of this study was partially rejected.

One of the most important reasons of the replacement of the dental restorations is the formation of caries under the restoration or between the restoration-tooth interfaces due to microleakage. In order to prevent caries, fluoride-releasing restorative materials is desired. The fluoride containing materials provide the formation of fluoroapatite with less solubility which increase the remineralization process and also at the same time inhibiting the proliferation and metabolization of microorganisms in the structure of the dental plaque (19). It is accepted that all dental materials releasing fluoride ion can prevent the formation of caries by these mechanisms, and increase the remineralization on the dental hard tissues. The anticariogenic and bacteriostatic activities of the materials are related to the amount of fluoride that is released from the material (20). For this reason, fluoride ion containing materials are still popular due to long-term fluoride release capacity to the oral environment (21). Therefore, in this study, conventional and current fluoride containing restorative materials were used which are frequently preferred in the clinical practice.

Surface hardness and roughness, which give information about the mechanical properties of restorative materials, are among the most important properties affecting the clinical success of restorations. If the materials have insufficient surface properties like surface hardness and/or roughness, the longevity of the restoration will be shorten. Accordingly, the strength of the material may

decrease and the tendency to water absorption from the surface may increase which negatively affect the mechanical properties of the restoration (22). For these reasons, in this study, the first priority was to assess the surface hardness and roughness of conventional and current restorative materials containing fluoride ion.

It was stated that, the surface roughness negatively affects the marginal integrity and wear of the restorative materials resulting discoloration of the restoration, accumulation of plaque and gingival irritation. Therefore, finishing and polishing procedures of restorative materials are the most important steps during treatment for the success of restorations and also to the patients (23). Surface roughness is a two-dimensional parameter which can be measured by a special device called a profilometer. This device can provide numerical values related to surface roughness, also provides ease of use and it is the most preferred test method in the literatures (24).

Surface hardness can be described as resistance to continuous indentations. In hardness tests, the resistance of the tested material against the indentation of a tapered or spherical shaped tip is measured (25). Brinell, Rockwell, Vickers and Knoop are the most commonly used methods for microhardness measurements of dental materials. It has been reported that, Brinell and Rockwell hardness tests can be used mostly in metal alloys while Vickers and Knoop hardness tests can be used to measure the hardness of all dental materials; such as gold, porcelain, composite resins and dental cements (22,26,27). Therefore, in this study, the Vickers microhardness test was chosen.

The factors that can influence the performance of a restorative material are the type, shape, and the amount of filler particles (28). Kundie et al., mentioned that; the filler particle size and the filler content can affect the hardness of restorative material (29). Marghalani attributed the high

surface roughness to the larger size of the filler particle and the increase of irregularly shaped fillers. (30).

In the oral cavity, teeth are constantly subjected to vertical and lateral stresses, saliva, heat and pH changes (12,31). Therefore, in order to mimic the oral environment, thermal aging process is mostly used to assure in vivo conditions. Thermal aging procedure is one of the most commonly used artificial aging methods. In the literatures, mostly, distilled water was chosen as a solution during in vitro tests (32,33). However, the reason for the uniqueness of this in vitro study was that, instead of distilled water as a solution, artificial saliva was chosen to mimic the oral environment (34). In the literatures, it was indicated that 10.000 cycles correspond to a 1-year aging (16-18). Therefore, in this study, 10.000 cycles was performed in order to mimic the oral environment and to evaluate the long term mechanical properties of the tested materials.

In the present study, CS showed statistically the highest surface roughness when compared with the other tested materials before and after thermal aging procedures ($p < 0.05$). Water sorption and the solubility of a dental material may damage the polymer structure and cause negative effects on the physical and mechanical properties of a restorative material (35). The structure of the monomers in the organic matrix is one of the factors that effects water sorption of a resin material (36). Bis-GMA, which is a hydrophilic monomer, has been reported to absorb the highest amount of water than the other monomers (37,38). The manufacturer stated that, the only monomer content of CS was Bis-GMA. On the other hand, when the monomer content of BF and IS were examined, it was determined that, these materials both contain Bis-GMA and UDMA monomers. Ertaş et al., and Thakib et al., have stated that, the hydrofobic monomer, UDMA, was more resistant to discoloration than Bis-GMA due to its low water absorption and solubility characteristics (39,40). Therefore, in this study, the higher surface roughness value obtained from CS when compared with the other tested materials, can be due to the presence of the only monomer of Bis-GMA which has a feature of absorbing water. On the other hand, although the fluoride release amounts of the tested materials have not been evaluated in this study, another reason for the highest surface roughness value for CS may be related to the fluoride release to the artificial saliva used as a solution instead of distilled water. Actually different from the present study, various roughness results were obtained for CS in the literature (41,42). This could be due to experimental factors like evaluation method, storage solution type (distilled water or artificial saliva) and light curing unit (Quartz Tungsten Halogen or Light-Emitting Diode).

The structure of resin composite is directly related to the smooth surface property of the material and its susceptibility to extrinsic discoloration. Large inorganic particles present on the surface of a material can lead to increased surface roughness along with the wear and

polishing procedures (43). In a study by Hanchate et al., Charisma Smart and Z 250 were evaluated in terms of discoloration and it was found that Carisma Smart with the particle size of 0.005-10 μm , showed the highest discoloration. They have associated this result to the high particle size in Charisma Smart (44). Although the present study did not evaluate the color stability of the tested materials, the study of Hanchate and et al., sheds light to our study in terms of much discoloration of a restorative material with large inorganic particles has a correlation to the high surface roughness on the material.

In the present study, after thermal aging process, the lowest microhardness value (35.67) was calculated numerically on the coated top surface of EF while the highest microhardness value (66.23) was found on the bottom surface of EF. In a study by Faraji et al., the average microhardness value of Equia™ that applied coating material, was found 19.22 after immersing in distilled water for 6 months. On the other hand, the average microhardness value of the same specimens after immersing in distilled water for 6 months without any coating application was calculated as 32.37 (45). Based on these similar results, it can be concluded that, Equia™ reached almost twice as much microhardness value when any coating was not applied. Therefore, it can be considered that coating application can reduce the surface microhardness of the material.

When the bottom surfaces of the all tested materials were evaluated after thermal aging procedure, the statistically highest microhardness value was observed in Group EF ($p < 0.05$). This is because water is needed for continuous chemical reaction, which makes this material's bottom surface harder. In addition to that, highest microhardness value can be due to the ion exchange between EF and the artificial saliva used in thermal aging procedure. As a matter of fact, the manufacturer claims that, EF is more successful in oral environment (eg: saliva) than in vitro studies. Since artificial saliva was used in this study, surface microhardness of EF could be found statistically higher than other tested materials. As a result, it can be stated that, one of the most important mechanical properties of this material, microhardness, was not negatively affected after thermal aging process. Therefore, it can be concluded that, successful results can be achieved with prolonged clinical use of EF.

Firstly, it should be kept in mind that an in vitro study might not represent all the conditions and interactions acting on a restorative material. Therefore; limitations of the study were the ionic composition of foods/beverages, pH changes, salivary enzymes and other factors such as sliding, abrasion or wear.

CONCLUSION

Within the limitations of this study, the following conclusions can be drawn:

1-Thermal aging procedure did not effect surface roughness of the fluoride containing restorative materials.

2-When all the tested materials were evaluated in terms of surface roughness, nanohybrid composite resin material showed the highest surface roughness value.

3-Thermal aging procedure had an effect on the microhardness of the restorative materials containing fluoride ion. Successful results can be achieved with prolonged clinical use of bulk-fill glass-hybrid material.

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