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BASIC RESEARCH:

Effect of Preheating on the Physical Properties of Bis-GMA-Containing, Bis-GMA-Free and Glass Ionomer-Based Fissure Sealants

Efecto del precalentamiento sobre las propiedades físicas de selladores de fosas y fisuras que contienen Bis-GMA, libres de Bis-GMA y a base de ionómero de vidrio

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ABSTRACT: This study aimed to evaluate *in vitro* the effects of preheating on the microhardness and surface roughness of fissure sealants with different compositions. Resin-based sealants (Clinpro, Fissurit FX), Bis-GMA-free resin-based sealants (Helioseal F Plus), and glass ionomer-based sealants (Riva Protect) were tested. Fifty-six cylindrical specimens (n=7) were prepared at room temperature and at 55 ± 1°C. Preheating was performed using Ena Heat. Microhardness was measured with a Vickers tester, and surface roughness was evaluated using an atomic force microscope. Data were analyzed using one-way ANOVA, Tukey's post hoc, and t-tests. Preheating significantly increased microhardness in all groups ($p<0.05$). Surface roughness decreased in Helioseal F Plus and Fissurit FX ($p<0.05$) but remained unchanged in Clinpro and Riva Protect ($p>0.05$). These results indicate that preheating enhances the microhardness of all fissure sealants, while its effect on surface roughness depends on the material type.

KEYWORDS: Fissure sealant; Microhardness; Preheating; Surface roughness.

RESUMEN: El objetivo de este estudio fue evaluar *in vitro* los efectos del precalentamiento sobre la microdureza y la rugosidad superficial de selladores de fosas y fisuras con diferentes composiciones. Se evaluaron selladores a base de resina (Clinpro, Fissurit FX), selladores a base de resina libres de Bis-GMA (Helioseal F Plus) y selladores a base de ionómero de vidrio (Riva Protect). Se prepararon cincuenta y seis especímenes cilíndricos (n = 7) a temperatura ambiente y a 55 ± 1°C. El precalentamiento se realizó mediante el dispositivo Ena Heat. La microdureza se determinó utilizando un microdurómetro Vickers y la rugosidad superficial se evaluó mediante microscopía de fuerza atómica. Los datos se analizaron mediante ANOVA de una vía, prueba post hoc de Tukey y pruebas t. El precalentamiento incrementó significativamente la microdureza en todos los grupos ($p < 0,05$). La rugosidad superficial disminuyó en Helioseal F Plus y Fissurit FX ($p < 0,05$), mientras que no mostró cambios significativos en Clinpro y Riva Protect ($p > 0,05$). Estos resultados indican que el precalentamiento mejora la microdureza de todos los selladores de fosas y fisuras, mientras que su efecto sobre la rugosidad superficial depende del tipo de material.

PALABRAS CLAVE: Sellador de fosas y fisuras; Microdureza; Precalentamiento; Rugosidad superficial.

INTRODUCTION

Dental caries is a multifactorial disease that results from an imbalance between demineralization and remineralization processes within the bacterial biofilm (1). Approximately 50% to 90% of carious lesions occur on occlusal surfaces (2). Sealants are materials applied to the pits and fissures of caries-susceptible teeth, which harden either chemically or through visible light activation, bonding micromechanically to the tooth structure. The application of sealants on occlusal surfaces has been shown to form a physical barrier that can prevent bacterial colonization and subsequent food accumulation (3,4).

At present, fissure sealants comprising resin or glass ionomer are extensively utilised. Resin-based sealants have shown favourable retention and wear resistance (5). However, Bisphenol A Glycidyl Methacrylate (Bis-GMA), a component found in many resin-based materials, has been shown to exhibit oestrogenic activity, with the potential to disrupt hormonal balance in both children and adults. Consequently, manufacturers have introduced Bis-GMA-free fissure sealant materials to the market (6). Glass ionomer-based fissure sealants are regarded as a viable alternative to resin-based sealants. The advantages of these materials include chemical adhesion to enamel and dentin, the absence of a requirement for pretreatment of the tooth surface prior to application, and the capacity to release fluoride (7). However, the disadvantages of these materials include a reduced working time, inferior aesthetics in comparison to resin-based materials, and reduced resistance to wear and fracture (8).

A range of techniques have been developed to enhance the durability and clinical performance of restorative materials, such as preheating (9). Preheating of resin-based materials increases the mobility of monomers in the organic matrix, resulting in a higher degree of monomer conversion. This results in a reduction of residual monomers and enhancement of several physical properties, including increased flowability for better adaptation to cavity walls, elevated microhardness, and improved compressive strength (10). In a similar manner, the application of heat to glass ionomer-based materials has been shown to enhance their mechanical and physical properties, as is also the case with resin-based materials. It has been documented that there has been an increase in surface hardness, working time, and fracture resistance (11).

The present study was conducted with the objective of investigating the effects of preheating on the microhardness and surface roughness of fissure sealants with Bis-GMA-containing, Bis-GMA-free and glass ionomer-based. The hypothesis of the study is that preheating will increase the microhardness of the fissure sealant materials, but will not affect their surface roughness.

MATERIALS AND METHODS

MATERIALS USED IN THIS STUDY

In this study, the following fissure sealants were used: Riva Protect (SDI, Australia), a glass ionomer-based material; Helioseal F Plus (Ivoclar Vivadent, Liechtenstein), a Bis-GMA-free resin-based material; Fissurit FX (Voco, Cuxhaven, Germany), and Clinpro (3M ESPE, USA), both resin-based materials. The glass ionomer-based material was coated with Riva Coat. The compositions of the fissure sealants utilised in the present study are outlined in Table 1. The Ena Heat composite warmer (Micerium, S.p.A., Avegno GE, Italy) was used for preheating the materials.

SAMPLE PREPARATION

A preformed Teflon mould with a diameter of 6 mm and a thickness of 2 mm was used. The materials were divided into two groups: pre-heated and non-preheated. The non-preheated materials were used at room temperature. The materials in the preheated group were heated to 55°C and placed into the Teflon mould, whose bottom was covered with a glass slide and mylar strip, using a dispensing tip. To obtain a flat, parallel surface, another glass slide and mylar strip were placed on top. The resin-based materials were heated for 20 minutes. The capsules of the glass ionomer-based materials were modified and kept in the heating device for 60 seconds. According to the manufacturers' instructions, the resin-based materials were polymerised for 20 seconds using a LED light-curing device (Woodpecker LED C, Guangxi, China). The glass ionomer material was mixed in an amalgamator (SYG-200, Foshion, Shanghai, China) for 10 seconds, placed in the mould and left to set. It was then removed from the mould and its surface was coated with Riva Coat.

MEASUREMENT OF MICROHARDNESS

The microhardness values of the specimens prepared with four different fissure sealant materials, each divided into preheated and non-preheated groups, were measured using a Vickers hardness tester (Duroscan, EMCO-TEST Prüfmaschinen GmbH, Austria). A load of 200 grams was applied for a duration of 15 seconds at three distinct locations on each specimen, and the mean of the resulting indentations was calculated. Subsequently, the microhardness value of each specimen was meticulously documented.

MEASUREMENT OF SURFACE ROUGHNESS

The surface roughness of the specimens was measured using an atomic force microscope (Multimode 8, VEECO, USA). The surfaces were scanned in three dimensions over an area measuring 10x10 µm. Images were captured in non-contact mode at a resolution of Fwa6x256 pixels with a scan rate of 1 Hz. The mean values of surface roughness, as determined by scanning, were then calculated in order to ascertain the Ra value for each specimen.

STATISTICAL ANALYSIS

The Shapiro–Wilk test was applied to assess the normality of data distribution. One-way ANOVA followed by Tukey's post hoc test was used to determine whether there were statistically significant differences among the groups in terms of microhardness, surface roughness values. Independent samples t-test was used to evaluate the statistical differences between pre-heated and non-heated specimens within each group. $p<.05$ was considered statistically significant.

Table 1. Materials used in this study.

Materials	Manufacturer	Composition*	Lot No.	Filler content
Riva Protect	SDI, Australia	Fluoroaluminosilicate glass, Polyacrylic acid, Tartaric acid, Distilled water, Pigments	F22077047	
Helioseal F Plus	Ivoclar, Vivadent, Liechtenstein	HEMA, UDMA, Silicon dioxide, Titanium dioxide, Aluminium fluorosilicate glass, Camphorquinone	Z06D0G	40.5%
Fissurit FX	Voco, Cuxhaven, Germany	Bis-GMA, Bis-EMA, BHT, BDT, TEGDMA, Sodium fluoride	2402076	55%
Clinpro	3M ESPE,34 USA	TEGDMA, Bis-GMA, TBABF$_4$, Methyl dichloro-silane, Silica	10806911	0%
Riva Coat	SDI, Australia	TEG-DMA 20–30%, UDMA 60-70%	12344417	

*Hydroxyethyl methacrylate (HEMA), urethane dimethacrylate (UDMA), bisphenol A ethoxylate dimethacrylate (Bis-EMA), urethane dimethacrylate, butylated hydroxytoluene (BHT), benzotriazole derivative (BDT), triethylene glycol dimethacrylate (TEGDMA), tetrabutylammonium tetrafluoroborate (TBABF$_4$).

RESULTS

MICROHARDNESS ANALYSIS

The mean microhardness values and standard deviations of the materials in both non-heated and pre-heated conditions are presented in Table 2.

Table 2. The mean microhardness values and standard deviations of the materials.

	Pre-Heated*	Non-Heated *	P values
	Mean (Std)	Mean (Std)	
Clinpro	24.20 ± 0.73^{b}	27.71 ± 1.27^{d}	P<0.05
Helioseal F plus	21.10 ± 1.48^{b}	32.30 ± 3.75^{c}	P<0.05
Fissurit FX	37.71 ± 1.25^{a}	50.02 ± 3.25^{b}	P<0.05
Riva Protect	40.87 ± 4.05^{a}	60.42 ± 1.8^{a}	P<0.05
P values	P<0.05	P<0.05	

*Different superscript letters indicate statistically significant differences. Std: standard deviation.

Statistical analysis revealed a significant difference among the non-heated groups. A statistically significant difference was also observed among the pre-heated groups. Although the highest microhardness values in the non-heated condition were found in the Riva Protect group, the difference compared to the Fissurit FX group was not statistically significant. In the pre-heated condition, microhardness values were ranked as follows: Riva Protect (Glass ionomer based) > Fissurit FX (Bis-GMA-containing) > Helioseal F Plus (Bis-GMA-free) > Clinpro (Bis-GMA-containing).

When the effect of preheating on the microhardness values of the materials was evaluated, it was observed that the microhardness values of all groups significantly increased after heating.

SURFACE ROUGHNES ANALYSIS

In the non-heated group, statistical analysis revealed no significant difference in surface roughness among the materials. However, a statistically significant difference was observed among the materials in the pre-heated group.

Although the highest roughness value in pre-heated groups was found in the Riva Protect group (Glass ionomer based), the difference compared to the Clinpro group (Bis-GMA-containing) was not statistically significant. The surface roughness of Fissurit FX (Bis-GMA-containing) was significantly lower than that of the other materials (Table 3).

Table 3. The mean surface roughness values and standard deviations of the materials.

	Non-Heated *	Pre-heated*	P values
	Mean (Std)	Mean (Std)	
Clinpro	124.22±27.88^{a}	140.30±30.55^{bc}	P>0.05
Helioseal F plus	143.55±28.85^{a}	107.85±21.17^{b}	P<0.05
Fissurit FX	115.05±20.85^{a}	57,91±27,64^{a}	P<0.05
Riva Protect	151.13±35.94^{a}	159.52±22.09^{c}	P>0.05
P values	P>0.05	P<0.05	

*Different superscript letters indicate statistically significant differences. Std: standard deviation.

When the effect of preheating on the surface roughness of the materials was evaluated, no significant change was observed in Clinpro (Bis-GMA-containing) and Riva Protect (Glass ionomer based). In contrast, a significant reduction in surface roughness was found for Helioseal F Plus (Bis-GMA-free) and Fissurit FX (Bis-GMA-containing). Three-dimensional images obtained by atomic force microscope a 10x10 µm area of the specimen surfaces are shown in Figure 1(a–h).

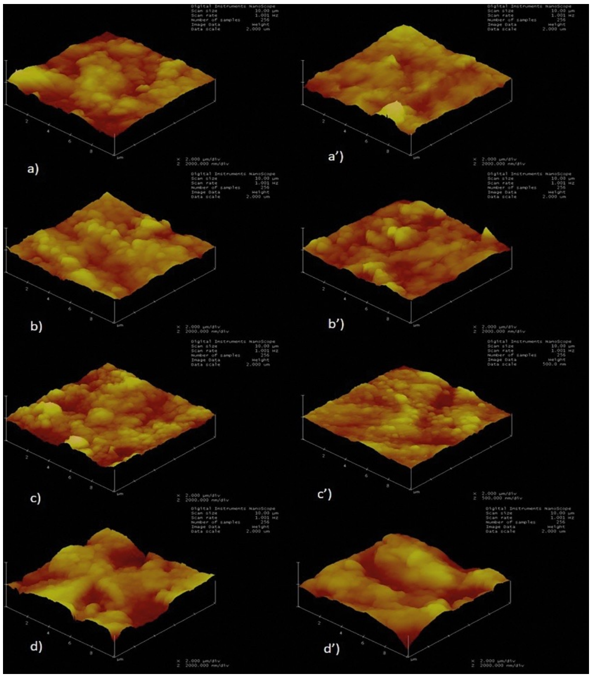

Figure 1. Three-dimensional images of specimen surfaces obtained by scanning a 10×10 µm area: unheated (a, b, c,d) and heated (a, b, c, d). a: Clinpro, b: Helioseal, c: Fissurit FX, d: Riva Protect.

DISCUSSION

Various techniques have been developed to enhance the durability and clinical performance of restorative materials. One of these techniques is preheating, which was initially designed for composite materials and later applied to other types of restorative materials (9). A comprehensive review of the literature reveals that studies involving the use of pre-heated fissure sealants are limited in number. In a study conducted by Skrinjaric *et al*. (12), glass ionomer-based fissure sealants were subjected to preheating and monitored over a one-year period. The retention rates of the pre-heated fissure sealants were found to be lower compared to those that were not pre-heated. In another study, Hristov *et al*. (13), investigated the penetration of fissure sealants into various fissure types using micro-CT by applying both heat and vibration, and reported that this approach increased the penetration depth of the fissure sealants. A review of the literature indicates that no study has yet compared pre-heated glass ionomer-based fissure sealants with resin-based fissure sealants.

It is considered that a temperature increase of 5.5 °C in the pulp may cause damage to the pulpal tissue (14). In a study, it was observed that the pre-heated material caused a temperature change in the pulp; however, due to the material rapidly losing heat after being removed from the heating device, the risk of pulpal damage was deemed low (15). Additionally, fissure sealants are used too far from the pulp. Therefore, the temperature values used in our study were set as room temperature and 55 °C.

Another important factor related to preheating is determining the appropriate heating duration required to improve the properties of restorative materials. In previous studies, the heating time for resin-based materials has been reported to range between 40 seconds and 24 hours, while for glass ionomer-based materials, this duration ranges between 40 and 90 seconds (9). Accordingly, in our study, resin-based materials were pre-heated for 20 minutes and the glass ionomer-based material for 60 seconds.

One of the mechanical properties affecting the clinical success of materials is surface microhardness. A high degree of monomer conversion increases the microhardness of the materials. As this value increases, the resistance to masticatory forces improves and the wear decreases (16). In our study, among the unheated groups, the highest microhardness value was found in the glass ionomer-based Riva Protect group; however, no significant difference was observed when compared to the resin-based Fissurit FX material. The microhardness values of Clinpro (a filler-free material) and Helioseal F Plus (a Bis-GMA-free material) were statistically lower compared to both Riva Protect and Fissurit FX. It is considered that an increase in the filler content of resin-based materials raises the molecular weight, which in turn improves microhardness (17-18). In a study by Kusgoz, *et al*. (19), a nano-filled resin-based fissure sealant was compared with a glass ionomer and a filler-free sealant. Consistent with our findings, the highest microhardness was observed in the nano-filled material, while the lowest was found in the filler-free Clinpro. The microhardness of the glass ionomer-based material was lower than the nano-filled material but higher than the filler-free one. In our study, the Bis-GMA-free fissure sealant consistently showed lower microhardness values compared to both the Bis-GMA-containing Fissurit FX and the glass ionomer-based Riva Protect, regardless of heat application. This is attributed to the high molecular weight of the Bis-GMA monomer, which forms denser cross-links, enhancing surface microhardness (20). Although Clinpro contains Bis-GMA, it exhibited lower microhardness values than Helioseal F Plus due to its lack of fillers.

In this study, it was observed that preheating caused an increase in the microhardness of the glass ionomer fissure sealant material as well as other resin-based fissure sealants. This can be attributed to the fact that heating glass ionomer-based materials prolongs the stabilization of chemical bonds and accelerates matrix formation, thereby improving mechanical properties like microhardness (21-22). Similar to our study, Lopes *et al*. (23), investigated the effect of preheating on the microhardness of glass ionomer cements. They pre-heated capsule-form glass ionomer cements in a composite heater set at 54 °C for 30 seconds before mixing in the amalgamator and found that preheating increased the microhardness of the cements. In our study, heat application to the resin-based fissure sealants—Fissurit FX, Helioseal F Plus, and Clinpro—led to increased microhardness. This is likely due to the elevated kinetic energy of monomers in the resin matrix caused by heat, which promotes a higher degree of polymerization (10) . In a study by Lucey *et al*. (24), preheating resin-based materials to 60°C significantly increased microhardness compared to room-temperature materials.

When examining surface roughness values in the unheated group, no statistically significant difference was observed among the materials. In line with our findings, Carvalho *et al*. (25), reported no significant difference in surface roughness among different glass ionomer and resin-based materials when left untreated. Studies examining the surface roughness of fissure sealants, such as that by Ilısulu *et al*. (26), found no significant difference in roughness among unprocessed Clinpro, Fissurit FX, and other resin-based materials, even when exposed to acidic conditions. Similarly, Yousry *et al*. (27), compared the surface roughness of Bis-GMA-containing and Bis-GMA-free resin-based materials and found comparable results. In our study, although Riva Protect had the highest surface roughness in the pre-heated group, the difference with Clinpro was not statistically significant. This may be attributed to the larger glass particles in glass ionomer materials compared to resin-based materials, which reduces filler-matrix homogeneity and increases surface roughness (28-29). Heat application in resin-based materials reduces viscosity and increases flow, but this may also lead to bubble entrapment during placement (30). Clinpro, being filler-free and having the lowest viscosity, demonstrated relatively higher surface roughness. In our study, heat application led to a statistically significant reduction in surface roughness for the Bis-GMA-free resin Helioseal F Plus and the filler-containing Fissurit FX, whereas no significant change was observed in the other materials. It is believed that heat modifies viscosity, thereby affecting surface roughness (31). Structural differences among materials may lead to varying effects of heat on roughness. In a study by Bodrumlu and Arslan (32), no significant difference was found in the surface roughness of a compomer at temperatures between 23 °C and 55 °C. A review of the literature revealed no studies investigating the surface roughness of pre-heated fissure sealants.

CONCLUSION

In conclusion, the application of preheating to fissure sealants increased the microhardness values of all tested materials, while its effects on surface roughness varied depending on the material composition. Although Helioseal F Plus does not contain Bis-GMA, it demonstrated comparable results to Bis-GMA-containing materials, indicating its potential to serve as a viable alternative in clinical applications.

CONFLICT OF INTEREST: The authors declare no conflict of interest related to this study.

AUTHOR CONTRIBUTION STATEMENT: Conceptualization and design: B.Y. and B.B.; Literature review: B.Y.; Methodology and Validation, B.Y. and B.B.; Formal analysis: B.Y.; Investigation and data collection: B.Y.; Resources: B.Y. and B.B.; Data analysis and interpretation: B.Y.; Writing-original draft preparation: B.Y. and B.B.; Writing-review & editing: B.Y. and B.B.; Supervision: B.B.; Project administration: B.B.

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