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The Effect of Preheating on the Water Sorption and Solubility of Fissure Sealants with Different Compositions

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ABSTRACT

Objectives: The aim of this study was to investigate how preheating influences the water sorption and solubility of fissure sealants with varying chemical compositions, including Bis-GMA-containing resin-based sealants (Clinpro and Fissurit FX), a Bis-GMA-free resin-based sealant (Helioseal F Plus), and a glass ionomer–based sealant (Riva Protect).

Materials and Methods: A total of 56 specimens (n = 7 per group) were prepared using three resin-based fissure sealants and one glass ionomer-based sealant. Among the resin-based materials, Clinpro and Fissurit FX are Bis-GMA-containing sealants, whereas Helioseal F Plus is a Bis-GMA-free resin-based sealant. In addition, Riva Protect was included as a glass ionomer-based fissure sealant. Specimens were prepared under two temperature conditions: room temperature and preheated at 55 °C. Resin-based materials were preheated for 20 minutes, while the glass ionomer-based material was heated for 60 seconds. Water sorption and solubility values were determined after 14 days of water storage following ISO 4049 guidelines. Statistical analysis included one-way ANOVA, Tukey post hoc test, and independent samples *t*-test.

Results: Riva Protect demonstrated significantly greater water sorption compared to all resin-based sealants, regardless of heating condition ($p < 0.05$). Among resin-based materials, preheating significantly increased water sorption only in Clinpro ($p < 0.05$). No significant differences were detected among the remaining resin-based sealants. Regarding solubility, Clinpro exhibited the highest values in the non-heated group, whereas Fissurit FX presented the lowest solubility overall. A statistically significant increase in solubility after preheating was observed exclusively in Riva Protect ($p < 0.05$).

Conclusions: The influence of preheating on water sorption and solubility varied according to the material composition. Despite the absence of Bis-GMA, Helioseal F Plus showed water sorption and solubility values comparable to Bis-GMA-containing resin-based sealants, suggesting its potential clinical applicability as an alternative material.

Keywords: Fissure sealants, preheating, water sorption, solubility.

Farklı İçeriklere Sahip Fissür Örtücülerin Su Emilimi ve Çözünürlüğü Üzerine Ön Isıtmanın Etkisi

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ÖZ

Amaç: Bu çalışmanın amacı, ön ısıtmanın farklı kimyasal bileşimlere sahip fissür örtücülerinin su emilimi ve çözünürlüğü üzerindeki etkisini araştırmaktır. Çalışmada Bis-GMA içeren rezin esaslı fissür örtücüler (Clinpro ve Fissurit FX), Bis-GMA içermeyen rezin esaslı bir fissür örtücü (Helioseal F Plus) ve cam iyonomer esaslı bir fissür örtücü (Riva Protect) değerlendirilmiştir.

Gereç ve Yöntemler: Bu çalışmada toplam 56 örnek (her grup için n = 7) hazırlanmıştır. Çalışmada üç rezin esaslı fissür örtücü ve bir cam iyonomer esaslı fissür örtücü değerlendirilmiştir. Rezin esaslı materyallerden Clinpro ve Fissurit FX Bis-GMA içeren fissür örtücüler iken, Helioseal F Plus Bis-GMA içermeyen rezin esaslı bir fissür örtücüdür. Ayrıca Riva Protect cam iyonomer esaslı fissür örtücü olarak çalışmaya dahil edilmiştir. Örnekler iki farklı sıcaklık koşulunda hazırlandı: oda sıcaklığı ve 55 °C'de ön ısıtma. Rezin esaslı materyaller 20 dakika süreyle ön ısıtılırken, cam iyonomer esaslı materyal 60 saniye süreyle ısıtıldı. Su emilimi ve çözünürlük değerleri, ISO 4049 standardına uygun olarak 14 günlük su depolaması sonrasında belirlendi. İstatistiksel analizlerde tek yönlü ANOVA, Tukey post hoc testi ve bağımsız örneklem *t*-testi kullanıldı.

Bulgular: Riva Protect, ısıtma koşulundan bağımsız olarak tüm rezin esaslı fissür örtücülere kıyasla anlamlı derecede daha yüksek su emilimi gösterdi ($p < 0,05$). Rezin esaslı materyaller arasında ön ısıtma, yalnızca Clinpro grubunda su emilimini anlamlı olarak artırdı ($p < 0,05$). Diğer rezin esaslı fissür örtücüler arasında anlamlı bir fark saptanmadı. Çözünürlük açısından, ısıtılmamış grupta en yüksek değer Clinpro'da gözlenirken, genel olarak en düşük çözünürlük Fissurit FX'te belirlendi. Ön ısıtma sonrasında çözünürlükteki istatistiksel olarak anlamlı artış yalnızca Riva Protect grubunda görüldü ($p < 0,05$).

Sonuçlar: Ön ısıtmanın su emilimi ve çözünürlük üzerindeki etkisi materyalin kompozisyonuna bağlı olarak değişmektedir. Bis-GMA içermemesine rağmen Helioseal F Plus, Bis-GMA içeren rezin esaslı fissür örtücülerle benzer su emilimi ve çözünürlük değerleri göstermiştir. Bu bulgu, Helioseal F Plus'ın klinik kullanımda alternatif bir materyal olabileceğini düşündürmektedir.

Anahtar Kelimeler: Fissür örtücü, ön ısıtma, su emilimi, çözünürlük.

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Introduction

Dental decay remains one of the most prevalent chronic diseases affecting individuals of all age groups worldwide. The development of caries is a multifactorial process involving the interaction of cariogenic microorganisms, fermentable dietary carbohydrates, tooth structures, and host-related factors such as saliva. Conditions including inadequate oral hygiene, reduced salivary flow, limited fluoride exposure, and unfavorable dietary and lifestyle habits have been associated with an increased risk of caries formation.[1,2]

Pit and fissure sealants are widely accepted as an effective preventive approach for protecting occlusal surfaces, particularly in individuals with a high caries risk. These materials are applied to pits and fissures, where they adhere micromechanically following chemical or light activation, forming a physical barrier that restricts bacterial colonization and nutrient diffusion. By sealing these anatomically susceptible areas, fissure sealants contribute to the long-term prevention of occlusal caries.[3,4]

Currently, fissure sealants are mainly available as resin-based or glass ionomer-based materials. Resin-based sealants are commonly preferred due to their favorable retention and wear resistance.[4] However, many resin-based materials contain Bisphenol A Glycidyl Methacrylate (Bis-GMA), a monomer that has been reported to exhibit estrogen-like activity, raising concerns regarding potential biological effects in both pediatric and adult populations. Consequently, Bis-GMA-free resin-based fissure sealants have been introduced as alternative materials.[5]

Glass ionomer–based fissure sealants represent another preventive option and offer several advantages, including chemical adhesion to enamel and dentin, fluoride release, and reduced technique sensitivity during application.[6] Despite these benefits, their clinical performance may be limited by inferior esthetic properties, shorter working time, and lower resistance to wear and fracture when compared with resin-based sealants.[7]

Various strategies have been proposed to improve the physical properties and clinical performance of restorative materials, among which preheating has received increasing attention. Preheating resin-based materials prior to application has been reported to reduce viscosity, enhance flow characteristics, and improve adaptation to the tooth surface. In addition, elevated temperatures may promote a higher degree of monomer conversion, thereby improving cross-link density and overall material performance.[8-11]

Water sorption and solubility are critical parameters affecting the durability and longevity of dental materials. Excessive water uptake may result in volumetric expansion and plasticization of the polymer matrix, whereas increased solubility can lead to the release of unreacted components, compromising material stability over time. These properties are influenced by factors such as monomer composition, filler content, degree of polymerization, and environmental conditions.[12-14]

Despite the increasing application of preheating approaches, evidence concerning their effects on the water sorption and solubility performance of fissure sealants with different chemical structures remains insufficient. The aim of this study was to investigate the effect of preheating on the water sorption and solubility of fissure sealants with different compositions.

The null hypothesis of this study was that preheating would not significantly affect the water sorption and solubility of fissure sealants regardless of their chemical composition.

Materials and Methods

Sample Size Calculation

The required sample size was determined using G*Power software (Version 3.1.9.7, Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany). A priori power analysis was performed with an effect size (f) of 0.69, a significance level (α) of 0.05, and a statistical power of 95% (1−β = 0.95). Based on these parameters, the minimum required sample size was calculated as five specimens per group (n = 5). To enhance the statistical reliability of the study, the sample size was increased to seven specimens per group (n = 7).

Materials

Among the resin-based sealants, Clinpro (3M ESPE, USA) and Fissurit FX (Voco, Cuxhaven, Germany) contain Bis-GMA, whereas Helioseal F Plus (Ivoclar Vivadent, Liechtenstein) is a Bis-GMA-free resin-based sealant. The fourth material was Riva Protect (SDI, Australia), a glass ionomer-based fissure sealant. The surface of the glass ionomer-based sealant was protected using Riva Coat. Detailed information regarding the chemical composition of the tested fissure sealants is presented in Table 1. Preheating of the materials was performed using the Ena Heat composite warming device (Micerium, S.p.A., Avegno GE, Italy).

Sample Preparation

Cylindrical specimens were produced using a Teflon mold with dimensions of 6 mm in diameter and 2 mm in height. From four different fissure sealant materials, seven specimens were prepared at room temperature and seven specimens under preheating conditions at 55 ± 1 °C for each material, resulting in a total of 56 specimens (n = 7 per group). The materials were allocated into two experimental conditions: preheated and non-preheated. Sealants in the non-preheated group were applied at ambient room temperature. For the preheated group, materials were warmed to 55°C prior to placement into the mould, which was positioned on a glass slide covered with a Mylar strip. The materials were dispensed into the mould using a dispensing tip, and a second glass slide and Mylar strip were placed on top to ensure smooth and parallel specimen surfaces.

Resin-based sealants were maintained in the heating device for 20 minutes before application. In contrast, the

capsules of the glass ionomer-based material were adapted for preheating and conditioned in the warming unit for 60 seconds. Polymerisation of the resin-based specimens was carried out using an LED light-curing unit (Woodpecker, Guangxi, China) with a light intensity of 1000 mW/cm² for 20 seconds, according to the manufacturer's instructions. The glass ionomer material was mechanically mixed for 10 seconds in an amalgamator (SYG-200, Foshion, Shanghai, China), placed into the mould, and allowed to set. After removal from the mould, the specimen surface was coated with Riva Coat. All procedures were performed by a single operator under standardized laboratory conditions to minimize variability.

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 dichlorosilane, 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 ($TBABF_4$)

Measurement of Water Sorption and Solubility

The specimens were individually identified and transferred to a desiccator containing silica gel (Desi Kimya, Istanbul, Turkey). They were initially kept in an incubator at 37±1°C for 22 hours and subsequently maintained at room temperature for an additional 2 hours. Each specimen was weighed using an analytical balance accurate to 0.0001 g. This drying and weighing procedure was repeated until the change in mass over a 24-hour period was less than 0.1 mg, and the stabilized mass was defined as m_1. The diameter and thickness of each specimen were measured at three different locations with a digital caliper, and the mean specimen volume (V) was calculated using the equation $\pi \cdot r^2 \cdot h$.

The dried specimens were placed separately into individual containers and submerged in distilled water maintained at 37°C ±1°C, with a standardized volume of 5 mL assigned to each specimen. After 14 days of immersion, the samples were removed from the water, carefully blotted with absorbent paper to eliminate surface moisture, and gently air-dried for 15 seconds. Each specimen was weighed exactly one minute after removal from the water using an analytical balance, and the obtained values were recorded as m_2. To re-establish a constant dry mass, the drying protocol was repeated, and the final mass values were designated as m_3.

Water sorption and solubility for each specimen were calculated in µg/mm³ using the equations specified in ISO 4049. The corresponding values at the conclusion of the 14-day storage period were determined accordingly.

$\mathbf{W_{ws}} = (m_2 - m_3) / V$

W_{ws}: Water sorption

m_2: Mass of the specimen after 14 days of immersion in distilled water

m_3: Mass of the specimen after the second drying procedure

V: Volume of the specimen

$\mathbf{W_{sl}} = (m_1 - m_3) / V$

W_{sl}: Solubility

m_1: Initial mass of the specimen measured after drying in the incubator

m_3: Mass of the specimen after the second drying procedure

V: Volume of the specimen

Statistical Analysis

All statistical analyses were performed using SPSS statistical software (Version 21.0, IBM Corp., USA). Data distribution was evaluated for normality using the Shapiro-Wilk test. Differences among the experimental groups regarding water sorption and solubility were analysed using one-way analysis of variance, followed by Tukey's post hoc multiple comparison test. Comparisons between preheated and non-preheated specimens within each material group were performed using the independent samples *t*-test. A significance level of $p < 0.05$ was adopted for all statistical analyses.

Results

Water Sorption Analysis

In the non-heated group, statistical analysis revealed that the water sorption of Riva Protect was significantly higher than that of the other materials. Similarly, in the preheated group, Riva Protect exhibited the highest water sorption value (Table 2). When the effect of preheating on water sorption was evaluated, a statistically significant increase was observed only in the Clinpro group.

Solubility Analysis

According to the analysis results, in the non-heated group, the highest solubility value was observed in the Clinpro material. No statistically significant difference was found between Helioseal F Plus and Riva Protect, and between Fissurit FX and Riva Protect. In the preheated group, Fissurit FX exhibited a significantly lower solubility

compared to the other materials (Table 3). When the effect of preheating was evaluated, no significant change was observed in the other materials, while a statistically significant increase was found in Riva Protect.

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

	Non-heated *	Heated*	p values
	Mean (Std)	Mean (Std)	
Clinpro	23.42 ± 1.64[a]	26.33 ± 3.09[a]	*p < 0.05*
Helioseal F plus	17.44 ± 2.90[a]	17.95 ± 1.86[a]	*p > 0.05*
Fissurit FX	13.09 ± 4.02[a]	14.72 ± 1.86[a]	*p > 0.05*
Riva Protect	171.46 ± 17.31[b]	177.19 ± 20.76[b]	*p > 0.05*
p values	*p < 0.05*	*p < 0.05*	

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

Table 3. Mean solubility values and standard deviations of the materials

	Non-heated *	Heated*	p values
	Mean ± Std	Mean ± Std	
Clinpro	-11.62 ± 3.21[c]	-11.07±2.95[b]	*p > 0.05*
Helioseal F plus	-6.65 ± 1.05[b]	-7.27 ± 1.91[b]	*p > 0.05*
Fissurit FX	-1.17 ± 1.39[a]	-1.44 ± 1.82[a]	*p > 0.05*
Riva Protect	-3.64 ± 3.43[ab]	-10.82 ± 4.95[b]	*p < 0.05*
p values	*p < 0.05*	*p < 0.05*	

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

Discussion

The null hypothesis of the present study was that preheating would not significantly affect the water sorption and solubility of fissure sealants regardless of their chemical composition. However, the findings partially rejected this hypothesis. Various techniques have been developed to enhance the durability and clinical performance of restorative materials. One of these techniques is preheating. Initially introduced to improve the handling and properties of composite resins, this approach has subsequently been applied to other restorative materials.[10] An evaluation of the existing literature indicates that research focusing on the use of preheated fissure sealants remains scarce. Skrinjaric et al.[15] assessed glass ionomer-based fissure sealants following preheating and evaluated their clinical performance over a one-year follow-up period, reporting lower retention rates in the preheated group compared with non-preheated sealants. More recently, Hristov et al.[16] examined the penetration behavior of fissure sealants in different fissure morphologies using micro-computed tomography, demonstrating that the combined application of heat and vibration enhanced sealant penetration depth. Despite these findings, the literature lacks studies directly comparing preheated glass ionomer-based fissure sealants with resin-based fissure sealants.

An increase in pulpal temperature of approximately 5.5 °C has been reported to pose a risk of pulpal injury.[17] Previous investigations have shown that although preheated restorative materials can induce a transient rise in pulpal temperature, the rapid heat dissipation following removal from the heating unit substantially reduces the likelihood of pulpal damage.[18] Moreover, fissure sealants are applied at a considerable distance from the pulp tissue. Based on these considerations, the temperatures selected for the present study were room temperature and 55 °C.

Another critical aspect of the preheating process is the determination of an adequate heating duration to enhance the physical properties of restorative materials. Earlier studies have documented heating times ranging from 40 seconds to 24 hours for resin-based materials, whereas glass ionomer–based materials have typically been heated for periods between 40 and 90 seconds.[10] Yang et al.[19] reported that varying preheating durations did not significantly affect the degree of conversion, maximum polymerization rate, or polymerization shrinkage of the composite material; however, preheating resulted in higher surface microhardness values. In the present study, a 20-minute preheating duration was selected as a moderate and standardized value representing the average of previously reported times, in order to ensure thermal equilibration and a homogeneous temperature distribution within the material.[20] In line with these considerations, resin-based materials were preheated for 20 minutes, while the glass ionomer–based material was preheated for 60 seconds. However, it should be noted that this duration may not fully reflect routine clinical practice.

In a study by Dinakaran,[13] resin-based materials, resin-modified glass ionomers, and conventional cements were evaluated in various beverages, and glass ionomer cements were found to have higher sorption and solubility values than resin-based materials. In a study conducted by Toledano et al.,[12] the water sorption and solubility of restorative materials with different compositions were compared, and the lowest values were observed in compomer materials, whereas the highest values were reported for glass ionomer–based materials. In our study, water sorption values were Riva Protect> Clinpro> Helioseal F plus> Fissurit FX in both non-heated and heated groups. However, no statistically significant difference was found among the resin-based materials in terms of water sorption, which may be due to differences

in filler and monomer content. The TEGDMA molecule is more hydrophilic than Bis-GMA, UDMA, and Bis-EMA, while Bis-EMA and UDMA-Bis-EMA-based resins are more hydrophobic than Bis-GMA. The presence of hydrophilic monomers increases water sorption, while hydroxyl groups in the molecular structure also contribute.[21,22]

In the present study, although differences were observed among the water sorption values of the resin-based materials, these differences were not statistically significant. The observed variations may be attributed to differences in the filler and monomer compositions of the materials. Similarly, Berger et al.[22] reported that resin-based materials with different filler contents exhibited comparable water sorption values, whereas filler size was found to influence the solubility of the materials.

Regarding solubility, in this study, the highest value was observed in the filler-free Clinpro, while the lowest was recorded in the filler-containing Fissurit FX; however, the difference between Fissurit FX and Riva Protect was not statistically significant. Clinpro's high solubility may be attributed to its TEGDMA and Bis-GMA content. Despite containing these monomers, Fissurit FX's lower solubility can be linked to its hydrophobic Bis-EMA component and high filler content. Helioseal F Plus, which does not contain Bis-GMA but includes the hydrophilic HEMA monomer, showed higher solubility, likely due to increased water interaction.[23] Differences in solubility among resin-based fissure sealants may also depend on polymerization rate and degree.[24] Muntean et al.[25] compared two resin sealants with different filler loads and found that the highly filled Fissurit FX exhibited lower water sorption and solubility.

When the effect of heat application on water sorption was evaluated, a statistically significant increase was observed only in the Clinpro material. This finding may be associated with the increased viscosity of the filler-free material after heating, which may lead to the formation of microporosities within the material and consequently greater water uptake.[26] However, in contrast to the findings of the present study, some studies conducted with composite resins have reported that heat application may reduce water sorption and solubility. [27] Castro et al.[28] evaluated the water sorption and solubility of composite materials preheated at different temperatures over a 7-day period and reported that increasing temperature resulted in decreased sorption and solubility values. Similarly, Lima et al.[29] reported that preheating resin cements at 54 °C reduced their water sorption and solubility values compared with room temperature. These discrepancies may be attributed to differences in material composition, heating protocols, and storage conditions among the studies.

In the present study, Fissurit FX exhibited the lowest solubility value in the preheated group, which may be attributed to its chemical structure and hydrophobic monomer content.[25] A statistically significant increase in solubility after heat application was observed only in the glass ionomer–based material, likely due to its hydrophilic nature and the presence of water-soluble components.[30]

This study has several strengths, including the evaluation of fissure sealants with different chemical compositions under standardized experimental conditions and the use of the ISO 4049 protocol for assessing water sorption and solubility, which increases the reliability of the findings. However, the use of different preheating protocols for resin-based and glass ionomer-based materials represents an important methodological limitation, as these approaches differ in both duration and application mechanism and are not directly comparable. Therefore, comparisons between these material groups should be interpreted with caution. In addition, this study was conducted under in vitro conditions and included a limited number of materials; thus, the results cannot fully reflect the complex oral environment or be generalized to all fissure sealant formulations. Future studies should incorporate standardized heating protocols, long-term aging procedures, thermal cycling, and clinical evaluations. From a clinical perspective, preheating may improve the flowability and penetration of fissure sealants into pits and fissures; however, the present findings suggest that its effect on water sorption and solubility largely depends on the chemical composition of the material.

Preheating may improve the flowability and penetration of fissure sealants, enhancing marginal adaptation. However, its effect on water sorption and solubility appears to be material-dependent. Therefore, it may be recommended for resin-based sealants, although potential changes in physicochemical properties should be considered. Additionally, the 20-minute preheating duration may not be clinically practical, and shorter protocols should be investigated.

Conclusions

In conclusion, the effect of preheating on water sorption and solubility varied according to the material composition. While preheating influenced these properties differently among the tested fissure sealants, Helioseal F Plus-despite being Bis-GMA-free-exhibited water sorption and solubility values comparable to those of Bis-GMA-containing materials, suggesting that it may be a suitable alternative for clinical use.

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Conflicts of Interest Statement

The authors declare no conflict of interest related to this study.

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