IMPACT OF EXTENDED RADIANT EXPOSURE TIME ON POLYMERIZATION DEPTH OF FLUORIDE-CONTAINING FISSURE SEALER MATERIALS

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ABSTRACT

Physical properties such as surface hardness of dental materials are directly linked to their clinical behavior. The aim of this study was to investigate the influence of extended curing time on the polymerization depth of fluoride-containing materials used as pit and fissure sealants. Conventional and extended exposure times (20 and 60 seconds) were used to photoactivate a gold-standard pit and fissure sealant (Fluroshield, Dentsply) and a flowable composite (PermaFlo, Ultradent). Twenty square-shaped samples (n=5) were prepared using a LED device (Bluephase 16i, Ivoclar). The Knoop Hardness Number (KHN) was calculated for the top and bottom surface of each sample 24 hours after polymerization. Bottom/top hardness ratio (B/T KHN) was than calculated. Averages were analyzed by two-way ANOVA and Tukey test (α=0.05). The flowable composite had higher KHN than conventional pit and fissure sealant for all experimental conditions (p<0.05). The 60-second photoactivation time increased KHN at the bottom surface and B/T KHN only of composite specimens. The flowable composite had better physical properties than the pit and fissure sealant, and they were improved by extended curing time.

Key words: hardness, polymerization, pit and fissure sealants.

IMPACTO DO AUMENTO DO TEMPO DE FOTOATIVAÇÃO NA PROFUNDIDADE DE CURA DE MATERIAIS COM CONTEÚDO DE FLÚOR UTILIZADOS COMO SELANTES DE FÓSSULAS E FISSURAS

RESUMO

Propriedades físicas de materiais utilizados como selantes de fóssulas e fissuras estão diretamente ligadas ao seu nível de retenção in vivo. Este trabalho objetivou avaliar a dureza Knoop (KHN) de topo e de base de, além da razão entre a KHN de topo/base de materiais utilizados como selantes de fóssulas e fissuras, como influenciadas pelo tipo de material (selante de fóssulas e fissuras e resina de baixa viscosidade) e pelo tempo de fotoativação (20 e 60 segundos). Vinte corpos-de-prova foram confeccionados para os quatro grupos em análise (n=5), utilizando-se o LED Bluephase 16i. A KHN nas faces de topo e base foi obtida após 24 h de polimerização e a razão entre a KHN de base/topo foi calculada. Os valores de KHN foram comparados pelos testes ANOVA/Tukey (p < 0.05). A resina de baixa viscosidade obteve maior KHN do que o selante em todas as condições experimentais. O selante apresentou valores de base menores que os de topo em todas as condições experimentais. O tempo de 60 s aumentou os valores de KHN de base das amostras da resina, bem como na razão entre a KHN de base/topo deste material. A resina de baixa viscosidade apresentou maior dureza e maior razão dureza base/topo do que o selante testado, as quais foram aumentadas com o prolongamento do tempo de exposição radiante.

Palavras-chave: dureza, fotopolimerização, selante de fóssulas e fissurs.

INTRODUCTION

In recent years, the Minimally Invasive Dentistry approach has been highlighted in scientific and clinical dentistry worldwide.[1] In this regard, pit and fissure sealing is recognized as an effective method for preventing caries initiation and arresting caries progression by providing a physical barrier that prevents microorganisms and food particles from collecting in pits and fissures[2, 3].

Light-activated resin-based materials such as traditional sealants and flowable composites can be used as pit and fissure sealants[2, 4]. Their physical characteristics strongly influence their clinical durability and are therefore of critical importance when deciding on suitable materials[5]. One of the most important properties is the hardness of the material. Low hardness values are usually linked to poor wear resistance[6] and susceptibility to scratching[7]. Hence, it is important to investigate hardness of materials used as pit and fissure sealants, especially on the bottom surface, which is in direct contact with the enamel surface and can influence the retention of sealing materials.

Curing exposure time and distance between the light tip and the surface of the resinous material may modify hardness[8]. Aguiar *et al.*[9] showed the importance of overexposure (three times longer than the curing time recommended by the manufacturer) in providing greater top and bottom surface hardness of composite materials photoactivated from a distance, since light irradiance decreases in such situations. However, there is little information on the impact of extended photoactivation time on the polymerization depth of resinous materials used as pit and fissure sealants. Resinous materials used as pit and fissure sealants are applied on the occlusal surface more thinly than a resin composite in a cavity, facilitating light transmittance and allowing satisfactory conversion of monomers at the bottom. On the other hand, the tip of the light device cannot be placed directly on the top of the sealant surface due to the morphology of the fissure and cusps, which decreases the light irradiance reaching the material and may impair the efficiency of polymerization. This highlights the importance of investigating whether an extended curing time to photoactivate the pit and fissure sealant and flowable composite might improve their physical properties.

Thus, the aim of this work was to evaluate the polymerization efficacy of a pit and fissure sealant and a flowable composite photoactivated according to conventional manufacturer's recommended exposure time and to extended exposure time, assessed by top and bottom Knoop Hardness Number (KHN) and bottom/top hardness ratio (B/T KHN). The following null hypotheses were tested: (1) there will be no statistically significant difference between the materials, curing times and sample surface for KHN; (2) there will be no statistically significant difference between the materials and curing times for B/T KHN.

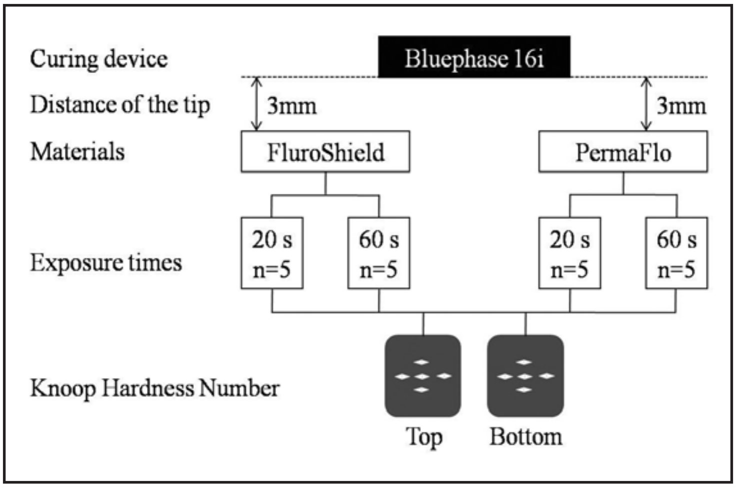

Fig. 1: Experimental design of this study.

METHODS

Experimental design and sample confection

The experimental design is shown in Fig. 1. For the analysis of KHN, we tested (1) *material* at two levels, (2) *curing time* at two levels and (3) *surface* at two levels. For the assessment of B/T KHN, only (1) *materials* and (2) *curing times* were tested. The materials used in this *in vitro* study, their composition and manufacturer's recommended curing times are listed in Table 1. Twenty square-shaped samples were made from a pit and fissure sealant (FluroShield) (FS) and a flowable com-

Table 1: Materials tested in this study, classification, manufacturer's recommended curing time (MRCT) and lot number.

Materials	Classification	Composition	MRCT	Lot
FluroShield – FS (Opaque White Shade) Dentsply Indústria e Comércio, Petrópolis, RJ, Brazil	Pit and fissure sealant	UED-BisGMA[A] (<40%); Resins (<10%); PENTA Phosphate (<5%); Bis-GMA[B] (<5%); Glass filler (<30%); Silica amorphous (<2%); TiO_2 (<3%); NaF (<5%);	20 s	#127474B
PermaFlo PF (A2 shade) Ultradent Products, South Jordan, UT, USA	Flowable composite	Bis-GMA[B] (8.5%); TEGDMA[C] (20%); Sodium Monoflurophosphate (0.3%); Zirconium filler (68%)	20s	#S069

[A] Urethane modified Bis-GMA dimethacrylate; [B] Bisphenol A-Glycidyl Methacrylate; [C] Triethylene Glycol Dimethacrylate.

posite (PermaFlo) (PF) using rectangular silicon molds with a 9mm^2 area and 1mm thickness. The materials were inserted into the molds and light-activated using a Light Emitting Diode (LED) (Bluephase 16i, Vivandent, Bürs, Austria – 1400mw/cm^2). The tip of the device was placed at 3mm from the top surface of the materials by means of a digital caliper, as described by Aguiar *et al*[9]. Before curing, a mylar strip was placed over the mold to provide a smooth top surface similar to the bottom one.

After polymerization, each specimen was removed from its mold and stored dry in a black receptacle at 37ºC for 24 hours.

Knoop Hardness Number (KHN) assessment

After storage, all the specimens were taken to a microhardness device (HMV 2T, Shimadzu, Tokyo, Japan), to measure top and bottom KHN. Five indentations were taken: one central (defined by the location of light application) and the other four at a distance of approximately 100 micrometers from the central location[10] under a 50gf load for 15 seconds[11, 12]. Bottom/top hardness ratio was calculated for each specimen.

Statistical analysis

The statistical analysis was performed using SAS (Statistical Analysis System 8.2) software. Subdivided parcels analysis of variance (ANOVA) (Split Plot) test ($p<0.05$) and a Tukey test at the 5% significance level were used to compare top and bottom hardness of specimens. Two-way ANOVA was performed to analyze bottom/top hardness ratio ($p=0.05$).

RESULTS

KHN

The ANOVA showed statistically significant differences between materials ($p<0.01$), between curing times ($p=0.03$), between surfaces ($p<0.01$) and in the interactions of material versus surface ($p<0.01$). Comparison of KHN among the groups is shown in the Table 2.

The Tukey test showed that PF had a higher KHN than FS ($p<0.05$) for all experimental conditions. Only PF showed differences between the curing times tested, in view of the fact that samples photoactivated for 20s had lower KHN than those cured for 60s ($p<0.05$). FS showed similar KHN for the two curing times evaluated ($p>0.05$). Moreover, only FS showed differences among top and bottom KHN, given that bottom surface had lower KHN than the top one ($p<0.05$).

B/T KHN

The ANOVA showed statistically significant differences between materials ($p<0.01$). The Tukey test showed that each material had similar ratios at both curing times. On the other hand, sealant B/T KHN was lower than that of the flowable composite at 60 s curing time (Table 1).

Table 2: Koop Hardness Number means (standard deviations) according to the factors under study.

Surfaces	Curing times	Materials	
		PF	FS
Bottom	20s	44.60 (3.68)Ab	*21.36 (1.25)Ba
	60s	48.16 (1.35)Aa	*20.00 (0.87)Ba
Top	20s	44.81 (2.03)Ab	23.07 (1.59)Ba
	60s	50.19 (0.84)Aa	23.81 (1.22)Ba

*Different from the top by variance analysis (p<0.05).
Mean values with the same letter (lower letter for vertical case and upper letter for horizontal one) were not statistically different by Tukey test (p>0.05).

Table 3: Bottom/top hardness ratio (standard deviations) according to the factors under study.

Materials	Curing times	
	20 s	60 s
FS	0.9976 (0.6)Aa	0.9593 (0.7)Aa
PF	0.9289 (0.1)Aa	0.0820 (0.1)Ab

Mean values with the same letter (lower letter for vertical case and upper letter for horizontal one) were not statistically different by Tukey's test (p>0.05).

DISCUSSION

Under all experimental conditions, PF had higher KHN than FS and extended exposure time increased KHN only for PF regardless the surface analyzed, so that the first null hypothesis tested was partially validated. The hardness of resin-based materials is influenced by several factors such as organic matrix composition, type and amount of filler particles, and degree of conversion[13]. Thus, differences in KHN between PF and FS may be explained by differences in filler type and content as well as organic matrix composition and degree of conversion of the two materials.

According to Beun *et al.*[14], the mechanical properties of dental composites depend greatly on the concentration and size of the filler particles. In this regard, the surface hardness of resin-based materials can be increased with a greater amount of filler particles[15, 16]. PF has a higher filler load than FS, so that KHN was higher for PF than for FS. Chen *et al.*[17] showed a linear correlation between degree of conversion (DC) and KHN. A previous study also showed that PF has higher DC than FS[18], which also influenced the KHN for the materials tested in this study.

Physical characteristics are very important for the choice of appropriate materials[19]. The hardness of a sealant may determine its resistance to abrasion[20] compromising its clinical behavior and leading to failures. It has been demonstrated that a flowable composite (Flow-It!) has a markedly higher retention rate than that of the conventional pit and fissure sealant Fluroshield[21]. The flowable composite used in conjunction with an adhesive system prior to its placement could result in enhanced retention compared to the conventional resin sealant. Additionally, there may be improved polymerization of flowable composites compared to pit and fissure sealants, which increases resistance to wear, decreases susceptibility to scratching and enhances the retention rate of the sealing material.

Since PF is a fluoride-containing flowable composite that can be employed as a pit and fissure sealant, its fluoride release should be evaluated in comparison with FS. Moreover, clinical trials should be performed in order to confirm the efficacy of PF as a secondary preventive agent, given that Borges *et al.*[3] demonstrated that sealing non-cavitated occlusal caries in dentin with FS can arrest their progression since the sealant remains bonded to the enamel. Thus, the retention of PF may be higher due to its superior physical properties compared to FS, as shown in this study and elsewhere[18].

Musanje *et al*[22] showed that higher initiator concentrations can increase the KHN of experimental composites. PF might have a higher amount of polymerization initiator than FS, so that light activation for three times longer than the manufacturer's recommended curing time provided superior B/T KHN due to higher monomer conversion. The 20-second light exposure might have been enough to excite all the photoinitiator molecules in FS, so that increasing manufacturer's recommended curing time did not provide higher conversion[18] and KHN to FS in comparison to PF. However, further research is needed to confirm the amount of photoinitiator system present in the two materials.

Regarding the shade of the composite, it has been demonstrated that a more translucent material allows better light transmission from the light polymerizer, which results in a higher degree of conversion and consequently greater hardness[23]. The differences between the hardness of bottom and top surfaces are known to be smaller in more translucent materials than in less translucent ones[24]. In addition to the different composition of the two materials tested in this study, the fact that PF (A2) is more translucent than FS, which is opaque white, may have facilitated light transmittance to the bottom surface of the flowable resin, improving its bottom polymerization and hardness distinctly in comparison to FS. This might be the reason why the bottom and top KHN of PF did not show statistically significant differences, unlike the opaque FS.

Bottom/top hardness ratio results obtained in the present investigation were higher than 80%. Although this value (bottom/top hardness ratio ≥ 0.8) is used as criterion for adequate curing of a composite[25], an optimal bottom/top hardness ratio for sealant material is not defined in the literature. Since fissure sealants are applied more thinly than composites, a more accurate curing depth should be expected. The 60-second photoactivation time provided highest B/T KHN in this work. Thus, the second null hypothesis tested was partially validated.

B/T KHN of the composite photoactivated for 20 seconds was not statistically different from the one obtained for the fissure sealant, although bottom KHN showed statistically significant differences between both materials at 20-seconds photoactivation time. Differences in the KHN parameter may have been overcome during ratio process with top KHN. This finding shows the importance of performing top and bottom hardness tests in addition to B/T KHN analysis.

PF is a fluoride-containing flowable composite that showed higher KHN values than FS, especially on the bottom surface, when it was photoactivated for three times longer than the manufacturer's recommended curing time in this *in vitro* study. Thus, clinical trials should be conducted to confirm the benefits of this curing mode on the longevity of PF.

The flowable composite Permaflo had higher KHN than the pit and fissure sealant FluroShield. Overexposure to light increased the hardness of the composite, and the KHN for the bottom surface was comparable to the value for the top surface only for this material. Bottom/top hardness ratio was similar for both materials tested when photoactivated for 20 seconds. However, a 60-second curing time increased this ratio for the flowable composite.

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