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Can composition and preheating improve infiltrant characteristics and penetrability in demineralized enamel?

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Abstract: The composition of infiltrants can influence their physical properties, viscosity and depth of penetration (DP). Strategies are used to increase the DP, such as the addition of diluents or the use of heat. This study aimed to evaluate the effect of preheating and composition on physical properties and DP of infiltrants in demineralized enamel. The groups were assigned, and the following experimental formulations were made: 25%BisEMA +75%TEGDMA; 25%BisEMA +65%TEGDMA +10%ethanol; 25%BisEMA +65%TEGDMA +10%HEMA; 100%TEGDMA; 90%TEGDMA +10%ethanol; 90%TEGDMA +10%HEMA. The samples were photoactivated at two temperatures (25°C and 55°C). Degree of conversion (DC) was performed using an infrared spectrophotometer. Elastic modulus *(E)*, flexural strength (FS) and contact angle (CA) tests were also performed. The DP of an infiltrant in demineralized enamel was determined by confocal laser scanning microscopy (CLSM) using an indirect labeling technique. The data were analyzed by two-way ANOVA and Tukey's test. DC increased after preheating in all the groups; however, 90%TEGDMA+10%ethanol showed the lowest DC for both temperatures, and the lowest *E*. Preheating did not influence *E* or FS. The CA increased at 55°C for most groups, but decreased for groups containing HEMA. Temperature did not seem to influence DP, and Icon showed the lowest DP values. The 100%TEGDMA composition showed more homogeneous penetration, whereas Icon showed heterogeneous and superficial penetration. The preheating technique does not improve all properties in all the material compositions. The composition of a material can influence and improve its properties.

Keywords: Resins, Synthetic; Ethanol; Microscopy, Confocal.

Introduction

Tooth caries development is a dynamic process that evolves from an imbalance of natural demineralization-remineralization events.[1] In the incipient pre-cavitation stages, sub-superficial lesions develop, in which the mineral content of the tooth is dissolved and hollow channels are created. These channels increase the porosity of the enamel, which appears clinically as a white spot.[1] At this stage, the process is still reversible using non-invasive methods, mostly by employing fluoride-based materials, as long as the patient is willing to maintain good oral hygiene and healthy dietary habits.[2,3] If patient compliance is an issue that will ultimately compromise remineralization efforts, another alternative is to stop white spot lesion progression by infiltrating the porosities in the enamel with low-viscosity resin, a so-called infiltrant. This material functions according to capillary forces, which carry resin monomers to the pores of enamel to occlude them.[4] To this end, the progression of non-cavitated caries lesion in enamel can be avoided by reinforcing the tooth structure.[5]

Different studies have evaluated the penetrability, etching parameters, mode of use and monomeric constitution of infiltrants applied to ensure the sealing of a white spot lesion.[3,6] The monomeric constitution, surface energy and viscosity of the material can influence the infiltrant penetration depths.[6,7,8] The sealing of this lesion by the infiltrant can effectively stop caries progression, because it interrupts the supply of the substrate to bacteria.[3] However, studies that investigate the use of infiltrants to mask the visual aspect of the white spot at different depths make us reflect on the importance of filling the lesion. How it is filled may influence the aesthetic result of masking. This tells us that it is important to study how the materials perform during penetration of the lesion.[9] The optical effect that allows the altered enamel to look like the adjacent healthy enamel is related to the similarity between the infiltrating refractive index and the healthy enamel, in comparison with porous enamel, filled with air or water.[10,11]

Thus, the addition of diluents is a strategy that can be used to reduce the viscosity of the infiltrant and to increase the depth of penetration.[6] However, excess solvent, commonly ethanol or 2-hydroxyethyl methacrylate (HEMA), can jeopardize the physical properties of the infiltrant.[12,13] Icon commercial infiltrant is composed mainly of triethylene glycol dimethacrylate (TEGDMA), a low viscosity monomer of low-molecular weight. It can undergo hydrolytic degradation, as previously tested in other monomer blends.[14] Another strategy that can reduce viscosity without resorting to altering the composition is based on heating. This technique has been shown to decrease the viscosity of some restorative composite resins, but it has not been tested on infiltrants, which already have low viscosity.[15] Therefore, it is reasonable to propose preheating as a way of reducing the viscosity of the infiltrant, regardless of its formulation. Since the increase in the thermal energy of a resin material accelerates the movement of the monomers, the preheating technique could increase the reaction rate, resulting in a higher degree of conversion and improvement of the physical properties of the material.[15,16, 17]

To this end, this *in vitro* study aims to evaluate the influence of composition and preheating of infiltrants on physical properties, degree of conversion, contact angle and depth of penetration in tooth enamel. The null hypotheses tested were: a) The preheating technique would not influence the properties of degree of conversion, contact angle, elastic modulus or flexural strength. b) The composition of the infiltrants would not influence the properties of degree of conversion, contact angle, elastic modulus or flexural strength. c) Depth of penetration in demineralized enamel would not be influenced by the composition or temperature of the tested materials.

Methodology

Formulation of infiltrants

Based in a previous pilot study, six experimental formulations of infiltrants and a commercial brand called Icon (DMG, Hamburg, Germany) were tested (Table 1).

The preheated groups used a thermal incubator (ThermoSmart, Astori Tecnica, Poncarale, Italy) to increase the temperature and ensure the samples remained at 55ºC at the time of photoactivation with an LED light source for 60 s (Bluephase 5i, Ivoclar Vivadent, Schaan, Liechtenstein, 1000 mW/cm^2). The procedure involved pipetting the samples into microtubes positioned in the heating device, and a digital thermometer (BAT 8; Bailey Instruments Inc, Saddle Brook, NJ) inside the samples was used to control the temperature during the experiments. Both the preparation of the experimental infiltrants and the tests were performed at a controlled temperature of 25ºC. Half of the samples were developed at ambient temperature (25◦C ± 1), and the others were made with material preheated to 55◦C.

Table 1. Composition (weight percentage) of infiltrants.

Infiltrants	Composition					
	BisEMA	TEGDMA	Ethanol	HEMA	CQ	DABE
INF	25	75	0	0	0.5	1
INF-E	25	65	10	0	0.5	1
INF-H	25	65	0	10	0.5	1
TEG	0	100	0	0	0.5	1
TEG-E	0	90	10	0	0.5	1
TEG-H	0	90	0	10	0.5	1
Icon	TEGDMA based resin matrix, initiator – additives*					

Ingredients were purchased from Sigma–Aldrich (Steinheim, Germany). TEGDMA = triethylene glycol dimethacrylate, BisEMA=ethoxylated bisphenol A glycidyl dimethacrylate, UDMA= diurethane dimethacrylate, HEMA=2-hydroxy-etilmetacrylate, CQ= camphorquinone, DABE= ethyl 4-(dimethylamino)benzoate. * Data provided by DMG, Hamburg.

Degree of conversion

The degree of conversion (DC) percentage was measured using an infrared spectrophotometer (Nexus 6700 IR Spectrometer, Thermo Scientific, Waltham, MA, USA) in transmission mode. The unpolymerized infiltrant was inserted into silicone rubber molds (n = 3; diameter = 11 mm; thickness = 0.8 mm) between two glass slides, and this set was placed on the sample holder of the spectrometer. Spectra were collected before and after photoactivation, and the methacrylate overtone peak at 6165 cm^{-1} was used to calculate the DC[18]. The samples were photoactivated using an LED device, for 60 s.

Elastic modulus and flexural strength

Ten bars (7mm x 2mm x 1 mm) were made using a metal split mold. The size of the sample was adapted to enable single-step polymerization instead of several light-activation steps at different points.[19] The samples were photoactivated using an LED device, for 60 s and then stored dry for 24 h at 37°C. The three-point bending test was performed with a universal testing machine (Instron, model 4111, Instron, Canton, MA, USA), at 0.5 mm/s speed until sample failure. The dimensions of the bars were measured using a digital caliper, and the data were imported into Bluehill software 2 (Instron, Canton, MA, USA), which calculated the elastic modulus (E) in GPa, and flexural strength (FS) in MPa.

Contact angle

The contact angles (CA) were measured between the glass slide surface and a drop of the infiltrant. Before the test, the surface was cleaned with 100% ethanol and dried for 24 h in an incubator at 37°C. Fresh surfaces were used for each test, using a goniometer equipped with a camera (Digidrop MCAT, GBX, Bourg de Peage, France). Drops of infiltrant (approximately 1 μL) were dispensed on the glass slide using a precision micro syringe. The images were analyzed by a software program (Visiodrop, GBX) that calculates CA (n=5).

Penetration in artificially demineralized enamel

Preparation of the sample

The research ethics committee of (xxx) previously approved this study. One hundred human molars were used to prepare the samples, which were extracted at most 3 months prior to testing, and were kept in a 0.5% chloramine solution (pH 7). Roots of the teeth were removed with a cutting machine (Buehler LTD., Lake Bluff, USA). Afterwards, the crowns were cut from the buccal and the lingual surfaces at the mesiodistal axis, and each tooth was split into two halves. The enamel surfaces were flattened using sandpaper with grit sizes # 600 and #1200 (Buehler), under water irrigation, and polished using a felt disc and 1 μm diamond paste (Buehler).

Demineralization of enamel

Fifty-two samples were selected according to initial Knoop microhardness mean values (319.4 ± 31.9 KHN), obtained by using a microhardness machine (HMV-2000; Shimadzu Corporation, Tokyo, Japan), using a Knoop indenter, performing 5 indentations per sample, using a 25 g load for 10 s.[20] Afterwards, the surfaces were covered with nail varnish (Colorama, São Paulo, Brazil), leaving a square area of sound enamel exposed (5 mm X 5 mm). Each sample was kept in 50 mL of 0.05 M acetate solution (2.0 mL of solution/mm^2 of exposed enamel), at pH 5, and at 50% saturation in relation to the hydroxyapatite, for 10 h at 37°C.[20]

The microhardness was measured again using the same protocol used to determine the initial microhardness, and according to these new measurements. Thirty-six samples with a mean 57.5 ± 8.1 KHN were selected to determine the depth of penetration. These samples were randomly divided into 12 groups (n=3), each composed of four experimental and one commercial infiltrant plus a control group. A sound enamel sample was used as the positive control (PC) and a demineralized non-infiltered enamel sample, as the negative control (NC), at two temperatures (25°C and 55°C). The 2 experimental infiltrants containing ethanol were excluded from this test due to their unsatisfactory physical properties, according to previous analyses.

Penetration in demineralized enamel and labeling of samples by confocal laser scanning microscopy (CLSM)

The infiltrant was diffused in the samples using the indirect labeling technique.[21] The samples were etched with 15% hydrochloric acid (Icon Etch, DMG) for 120 s[4] and then washed with distilled water for 30 s. Red fluorophore was used for labeling, by keeping the available pores of the samples immersed in an ethanolic solution of 0.1% Rhodamine B Isothiocyanate (w/v) (RITC, Sigma Aldrich, Steinheim, Germany)[22] for 12 h. Afterwards, the samples were air-dried for 10 s and the infiltrant was applied on the enamel surface. The excess material was removed with cotton pellets after 5 minutes, and the sample was light-cured for 60 s. The excess Rhodamine was removed by keeping the samples in 30% hydrogen peroxide for 12 h at 37°C, and then washing them with distilled water for 60 s.

The nail varnish on the enamel surface was then removed, and the samples were cut into 0.5-mm-thick slices perpendicular to the enamel surface. Next, the sample slices were polished using sandpaper (1200, 2400, 4000; Buehler). The samples were immersed in a 0.1% (w/w) ethanolic solution of 100 µM sodium fluorescein (NaFl; Sigma Aldrich) for 3 min, and then washed with deionized water for 10 s.

CLSM images were obtained (Leica, TCS NT; Leica, Heidelberg, Germany) using an objective lens at 20X and 63X magnification, in dual-fluorescence mode, in which RITC and NaFl could be detected simultaneously (RITC: Ex 568 nm, Em 590 nm; NaFl: Ex 488 nm, Em 525/50 nm). Bi-dimensional images were obtained (xyscan), at 1024 x 1024 pixel resolution.

The infiltrant depth of penetration was measured at 5 points (both sides from the center of the lesion to the edge of the image, 50 µm away from each other), from the surface to the intersection point between the reference line and the deepest extension of red fluorescence, using ImageJ software (NIH, Bethesda, MD, USA). Next, the means were calculated.

Statistical analysis

The statistical analysis was performed using Minitab software (State College, PA, USA), considering that the data showed normal distribution and homoscedasticity. The results were analyzed with 2-way ANOVA and Tukey's test (a = 5%).

Results

Degree of conversion

The preheating of the infiltrants increased the DC of the materials differently. In the TEG-E group, although the DC increased after preheating, it still had the lowest DC value, indicating that there was an interaction between the factors ($p < 0.001$; Table 2). Thus, except for TEG-E, the infiltrants presented a DC between 60-70% after preheating ($p < 0.001$). Icon presented the highest DC at both temperatures, although it was similar to INF-H after preheating. TEG-E presented the lowest DC at both temperatures ($p < 0.001$).

Elastic modulus and flexural strength

There was a significant interaction between the factors (temperature and composition) evaluated for *E* ($p < 0.001$) and for FS ($p = 0.004$) (Table 3). In regard to *E*, the preheating of infiltrants caused no statistical difference among the groups, except for the INF-H and TEG groups. At 25°C, the INF-H group showed the highest *E* values, whereas the formulations composed of TEGDMA with ethanol had the lowest values. At 55°C, the lowest values belonged to the TEG and TEG-E groups. The FS data indicated similar results for the *E* data, namely that the increase in temperature had no influence on the physical properties of the tested materials.

Table 2. Mean values of degree of conversion (%) of infiltrants, according to temperature.

Infiltrant	DC	
	25ºC	55ºC
Icon	41.3 (1.1) ^{Ab}	76.0 (1.7) ^{Aa}
INF	34.0 (1.1) ^{Bb}	61.3 (3.8) ^{Ca}
INF-E	28.3 (1.5) ^{Cb}	62.3 (2.5) ^{Ca}
INF-H	34.0 (2.6) ^{Bb}	73.3 (2.1) ^{Aa}
TEG	32.7 (1.2) ^{Bb}	66.7 (1.5) ^{BCa}
TEG-E	19.7 (3.1) ^{Db}	33.0 (3.5) ^{Da}
TEG-H	28.7 (2.5) ^{Cb}	69.0 (2.7) ^{Ba}

Different letters indicate a statistically significant difference ($p < 0.05$). Uppercase letters compare data in the same column, and lowercase letters compare data in the same line.

Contact angle

The TEG-E group presented an increase in the contact angle after preheating ($p < 0.001$; Table 4). Icon and TEG groups also showed a rise in the contact angle from 25ºC to 55ºC, but the most striking rise was detected in the INF group. In addition, among the experimental groups, those containing TEGDMA showed lower CA values at 25°C. However, the INF-H and TEG-H groups showed a decrease in CA after preheating. Overall, Icon showed the lowest CA, followed by TEG-H at 55°C, whereas the highest value was observed for the INF group at 55°C.

Depth of penetration analyzed by CLSM

A quantitative analysis to detect the depth of penetration of materials (Table 4) revealed no statistical differences for temperature ($p = 0.449$) or interactions of the evaluated factors ($p = 0.095$). At 55ºC, the TEG group showed greater depth of penetration compared to Icon and to the other groups. The lowest values of depth of penetration were found for Icon and TEG-H, regardless of temperature ($p < 0.05$).

The transmission mode shows non-infiltrated enamel pores as dark, and the fluorescence mode show the pores as green, owing to the presence of NaFl. The presence of infiltrant appears in red in the fluorescence mode, and the interaction between the infiltrant material and the lesion body structures can be seen in yellow, in the dual mode.

The positive control (PC) showed sound enamel with no structural alteration, just some defects in the enamel, probably caused during cutting

Table 3. Mean values of elastic modulus (GPa) and flexural strength (MPa) of infiltrants, according to temperature.

Infiltrant	E		FS	
	25ºC	55ºC	25ºC	55ºC
Icon	1.3 (0.2) ^{Ba}	1.4 (0.2) ^{Aa}	58.8 (11.4) ^{CDa}	62.4 (13.1) ^{BCa}
INF	1.3 (0.2) ^{Ba}	1.2 (0.2) ^{Aa}	67.3 (13.6) ^{Ba}	61.9 (12.9) ^{BCa}
INF-E	1.3 (0.2) ^{Ba}	1.1 (0.2) ^{Aa}	59.7 (11.1) ^{CDa}	56.2 (8.7) ^{BCa}
INF-H	2.0 (0.2) ^{Aa}	1.4 (0.2) ^{Ab}	103.2 (17.5) ^{Aa}	82.6 (10.7) ^{Ab}
TEG	1.3 (0.5) ^{Ba}	0.8 (0.2) ^{Bb}	63.5 (16.2) ^{CDa}	46.2 (14.3) ^{Ca}
TEG-E	0.7 (0.2) ^{Ca}	0.9 (0.2) ^{Ba}	37.2 (8.8) ^{Ea}	47.8 (10.2) ^{Ca}
TEG-H	1.3 (0.3) ^{Ba}	1.2 (0.2) ^{Aa}	68.0 (10.2) ^{Ba}	69.2 (7.3) ^{ABa}

Different letters indicate a statistically significant difference ($p < 0.05$). Uppercase letters compare data in the same column, and lowercase letters compare data in the same line.

and polishing of the slices, whereas the negative control (NC) confirmed demineralization of the enamel, as indicated by the green porous structures (Figure 1). The ICON group showed a superficial infiltration in the demineralized enamel, with shorter tags among the groups, at 25°C (Figure 2, Icon/25). At 55°C, ICON showed deeper penetration with longer tags than at 25°C. Preheated ICON

Table 4. Mean values of contact angle (o) and penetration depth (μm) of infiltrants, according to temperature.

Infiltrant	Contact angle		Depth of penetration	
	25°C	55°C	25°C	55°C
Icon	21.9 (0.4) ^{Eb}	25.8 (0.6) ^{Ca}	30.1 (4.8) ^{BCa}	22.3 (6.7) ^{Ca}
INF	28.1 (0.9) ^{Cb}	33.4 (0.8) ^{Aa}	30.2 (5.6) ^{BCa}	37.9 (6.9) ^{BCa}
INF-E	30.4 (0.5) ^{Ba}	31.3 (2.2) ^{ABa}	-	-
INF-H	34.1 (1.0) ^{Aa}	29.8 (0.9) ^{Bb}	43.1 (5.7) ^{ABa}	38.3 (2.9) ^{Ba}
TEG	26.6 (0.8) ^{Cb}	28.9 (0.7) ^{Ba}	45.5 (5.8) ^{ABa}	49.7 (11.0) ^{Aa}
TEG-E	24.1 (0.7) ^{Db}	29.3 (0.6) ^{Ba}	-	-
TEG-H	28.0 (1.6) ^{Ca}	25.4 (1.1) ^{Cb}	31.2 (6.3) ^{BCa}	40.7 (2.6) ^{Ba}

Different letters indicate statistically significant difference ($p < 0.05$). Uppercase letters compare data in the same column, and lowercase letters compare data in the same line.

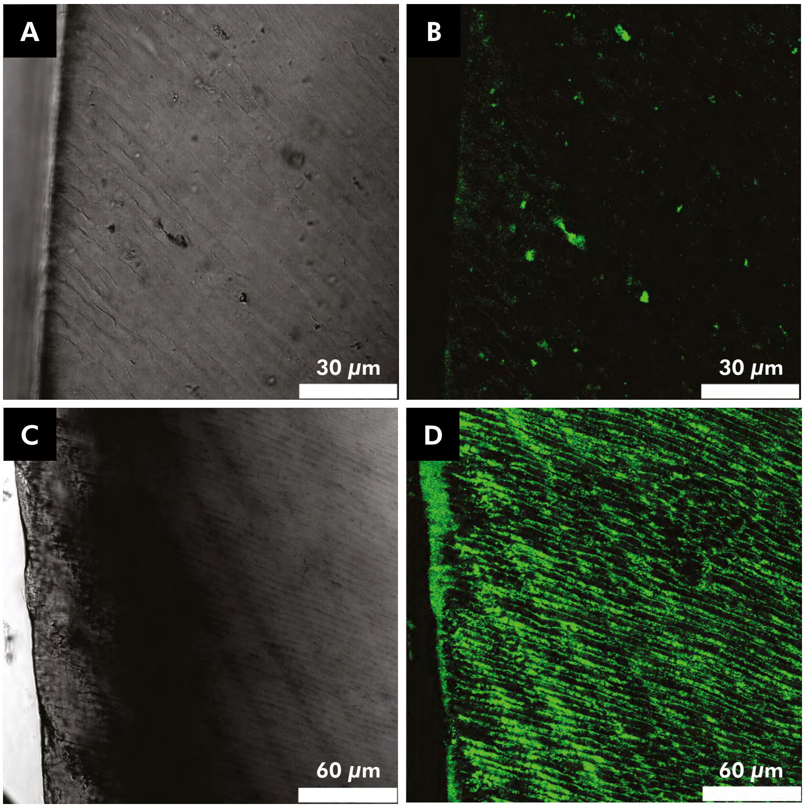

Figure 1. Representative images obtained with CLSM in combined mode of transmission and fluorescence. A. Positive control did not show any structural changes in the enamel (transmission mode). B.- There was no evidence of NaFl in the tissue (fluorescence mode). C. Negative control; porous structures of the lesion body, in black, owing to the scattering of light between the crystals (transmission mode). D. Negative control; green porous structures caused by NaFl, confirming the formation of artificial caries lesions in the enamel (fluorescence mode).

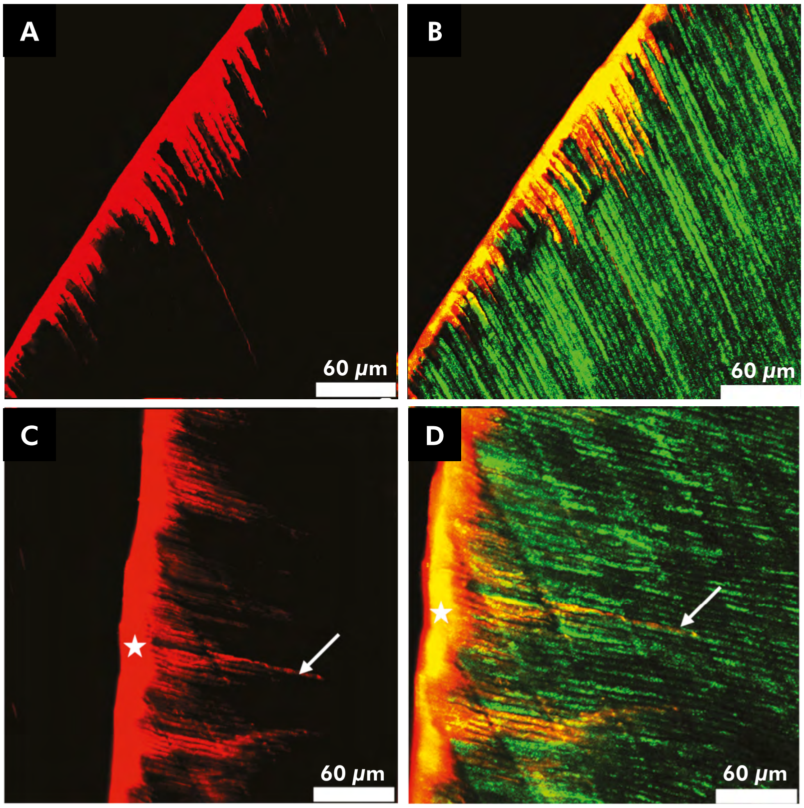

Figure 2. Representative images in dual fluorescence mode: infiltrated structures are in red (RITC), and porous structures are in green (NaFl). A. Icon at 25° C showed superficial penetration. B. Interaction between the infiltrant and the structures of the lesion body can be observed in yellow (25 ° C). C, D. Icon, at 55° C presented longer tags (white narrow), but a thick layer of material on the surface was observed, evidencing its retention on the surface of the lesion (white star).

presented longer tags, as indicated by the white arrows showing the retention of infiltrant on the surface of the lesions (Figure 2. Icon/55).

The deepest penetration was seen in the TEG group, regardless of temperature, with tags about 45 µm inside enamel pores, forming a thick layer of adhesive on the top surface (Figure 3). The preheated group tended to promote more homogeneous infiltration than the TEG group at 25°C (Figure 3/TEG55).

Discussion

The infiltrant composition and increase in temperature modified some of the properties evaluated. The DC was higher for the preheated materials, corroborating other studies on preheating of composite resins.[16] These studies showed reduced viscosity, increase in DC and improved cross-linking properties, as the major advantages of the preheating method.[23,24] Thus, the first hypothesis of the study, that preheating would not influence the physical properties of the infiltrants, was rejected. These characteristics pose an advantage, since they increase the resistance of the infiltrant material to water sorption and hydrolytic degradation, and also improve the physical properties of the material. The highest conversion value of 25ºC was attained by the TEGDMA-based commercial material (Icon) at 41.3%, whereas the experimental groups presented statistically lower values. Thus, it is reasonable to assume that this material has a higher concentration of initiators, and includes additives and other compositional trade secrets. The value found for the Icon DC was close to the values found in other studies, that is, from 50-56% DC.[25,26] By comparing the results obtained using the commercial materials with the supposedly experimental analog (100% TEGDMA)—showing

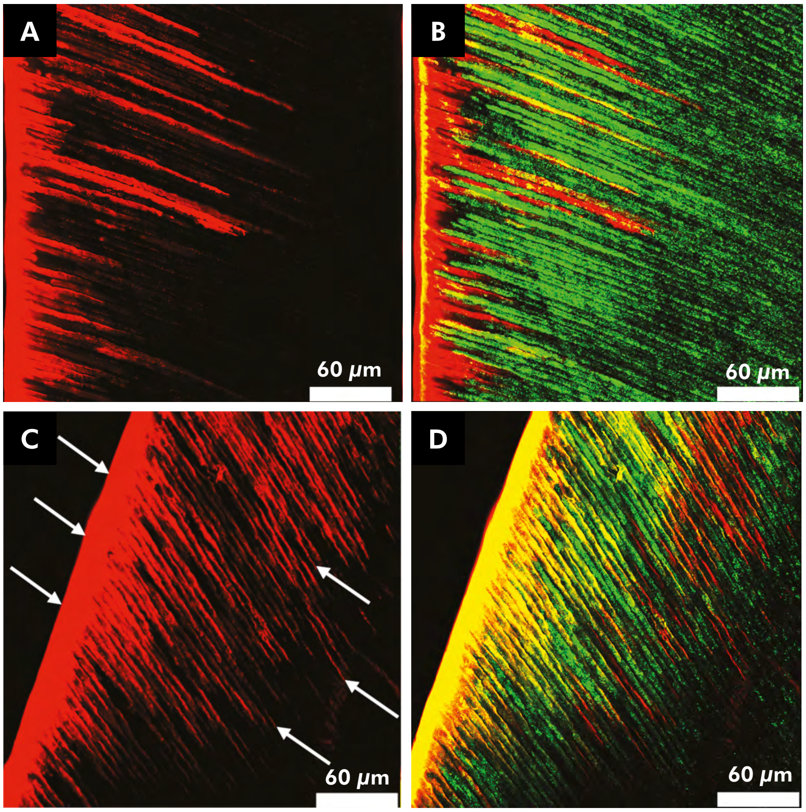

Figure 3. TEGDMA-based group showed deep infiltration into the lesion, regardless of the temperature (dual fluorescence mode). A, B. TEGDMA group at 25° C. C, D. TEGDMA group at 55° C, showing that the heated infiltrant presented more homogeneous infiltration than the resin at 25° C (white narrow).

just 32.7% of DC under the same test conditions—it is clear that the remaining experimental groups can be compared only with the experimental control. As expected, raising the temperature led to improved DC, owing to increased molecular mobility and decreased viscosity of the system.[27] In addition, the frequency of collision of unreacted monomers and radicals is greater when the curing temperature is elevated, if this temperature is below the glass transition temperature.[27] However, despite the average 30% increase in conversion for the groups polymerized at 55°C, the expected improvement in physical properties[28] was not observed. The results demonstrate that preheating did not influenced the *E* and FS of most of the groups, corroborating the results of another study on composite resin.[13] The presence of Bis-EMA and HEMA increased *E* and FS both preheated to 25°C, which could explain the better DC values shown by this group versus those containing Bis-EMA and ethanol. Ethanol can solubilize linear chains, resulting in degradation of the physical properties of the material. Thus, the second hypothesis of the study was also rejected, since the different compositions of the infiltrant influenced its properties.

Another strategy to reduce viscosity of materials is to add some sort of solvent or diluent such as ethanol and/or HEMA to the infiltrant, to improve the depth of penetration.[2,8] However, all the ethanol content cannot be evaporated clinically, nor can the HEMA be removed after penetration. This could compromise the conversion, since HEMA/ethanol favor the formation of linear chains, and the dilution of a polymeric network under formation, respectively.[29] Indeed, the results indicated that even small portions of solvents caused reduced DC and *E*. When evaluating the physical performance of infiltrants according to composition, a homogeneous behavior pattern can be observed, where the lowest *E* and FS values were observed in the TEG-E group, whereas INF-H showed the highest values. The addition of solvents to monomeric mixtures can facilitate the formation of microgels close to where the polymerization reaction begins; these microgels reduce the mobility of the radicals, thus compromising the reaction.[8] These chains show a distribution of heterogeneous mobility, where the higher the solvent concentration, the greater the formation of microgels, and, consequently, the heterogeneity.[13,29] On the other hand, the material with BisEMA/TEGDMA/HEMA in its composition had better results. The INF-H group shows a composition of 90% bi-functional monomers, which are able to form cross-links among the chains, thus reinforcing the polymer and improving its overall properties[30]; this explains the better results achieved. The addition of 25% BisEMA to TEGDMA seems to have been beneficial, since TEGDMA is a highly hydrophilic material with a greater potential for degradation than BisEMA.[3,8,14] Hence, the reduction in TEGDMA concentration, and the addition of a low viscosity hydrophobic monomer such as BisEMA, is a valid strategy for potentially creating lower viscosity materials with satisfactory polymeric properties.

The contact angle reflects the interaction of a non-polymerized liquid on a solid surface. This is an important property that should be considered for penetration of resin materials in dental surfaces. Other factors that also influence resin infiltration are surface energy, viscosity of the material, and capillarity forces of the enamel.[6] The present study sought to evaluate the effect of preheating and different monomeric compositions on the CA of infiltrants. Considering the influence of the surface energy of the object on the wettability of the material, a glass surface was used instead of dental enamel for the purpose of standardization of the test, and thus exclusively evaluating the effect of preheating and composition. In view of the results, it can be seen that both preheating and composition influenced the CA.

Preheating caused only a slight increase in CA in the materials containing only TEGDMA (100% TEGDMA and Icon), whereas the increase in CA after preheating in the INF group, was more expressive. This may be attributed to the higher viscosity of this group, which had BisEMA in its formulation. Although the viscosity was not tested properly in this study, because the composition of the tested groups was known, it may be inferred that the viscosity of the monomers influenced their properties somewhat. In addition, it can be speculated that raising the temperature to 55°C may have promoted a structural molecular change in the non-solvent groups, making the interaction of the material with the surface more difficult, and increasing the CA.

In regard to the materials that contained some type of solvent, an increase in CA could be observed after preheating the TEG-E group. This can be explained by the possible evaporation of ethanol when heated. On the other hand, only mixtures containing HEMA showed lower CA on the solid surface after preheating, thereby presenting the best combinations for this property. Preheating did not volatilize HEMA; therefore, its presence made the group less viscous, owing to its low molecular weight.[12] This factor, combined with molecular agitation due to heating, may have favored greater dispersion.

Moreover, although the samples were kept at 55°C, a slight drop in temperature may have occurred when the drop was applied to the glass slide, thus influencing the results. In the other tests, a digital thermometer with a probe was used to ensure that the temperature of the samples would be maintained immediately before photoactivation. However, our methodology made this resource inadvisable, since a probe would interfere the in the spread of the material on the glass slide, and thus affect the CA.

As for depth of penetration, the groups containing ethanol were excluded due to the unsatisfactory physical results. Some studies have shown that even though there are advantages to having ethanol in the composition, resins free of solvents are the preferred indication.[2,7] Regardless of the differences found for CA, the temperature did not influence the results of the depth of penetration, since this property was measured on the glass slide. Most likely, the change in viscosity caused by the increase in temperature was not enough to alter the pattern of penetration. In relation to composition, the divergence in performance between the Icon and TEGDMA groups at 55°C has already been pointed out. The manufacturer of Icon states that the composition of the infiltrant is basically TEGDMA. However, this group presented a different pattern of diffusion, in comparison with the equivalent experimental group that showed the greatest depth of penetration among the preheated compositions. In contrast, Icon had one of the lowest depths of penetration. This leads us to speculate that Icon's composition of 89.1% tetraethylene glycol dimethacrylate solvated in ethanol (9.9%)[31] probably led the ethanol to cause a difference. That is, ethanol would be responsible for a heterogeneous pattern of penetration in demineralized enamel. Thus, the third hypothesis of the study was partially rejected, since the different compositions of the infiltrant had an effect on the DP, but the preheating did not influence this property. Future investigations must be conducted to define the best composition and to improve the preheating technique of infiltrants. Moreover, the viability of clinical application of this technique should be evaluated, since it did not present any advantages in all compositions or improve all the tested properties.

Conclusions

The preheating of the tested materials influenced most of the properties evaluated, except for depth of penetration. However, the preheating technique did not always improve these properties (*E*/FS and CA), as seen in relation to the composition of the infiltrant, which influenced all the properties tested. Thus, the preheating technique for infiltrants still cannot be indicated for any specific type of composition.

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