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Microhardness of composites cured over eugenol-contaminated dentin

Microdureza de compósitos sobre dentina contaminada com eugenol

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ABSTRACT

During fabrication of indirect esthetic restorations a temporary luting cement is necessary. This study evaluated the effect of eugenol-contaminated dentin on polymerization of composite resins. Sixty composite disks were made over previously contaminated dentin with one of five different temporary luting cements (Temp Bond, Temp Bond NE, Rely X Temp, Resin Sensitemp, Freegenol), with two different composites (Filtek Z-100 and Filtek Supreme Plus). Vickers hardness number was obtained for the light side of the sample as well as the bottom side of it. Comparisons were made under a two-way ANOVA with a Tukey-Kramer post-hoc analysis, both calculated at 0.05 significance level. The VHN of all specimens bottom side was lower than the top side in contact with the light. Reduction was in average 12% for composite Supreme Plus and 9% for composite Z-100, all within expected results. Cements such as Freegenol and Temp Bond NE produced sometimes a greater VHN reduction of the bottom side than the eugenol-containing cement. Eugenol contaminated dentin did not reduced the polymerization of composite resin cured over it.

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Keywords: Eugenol**;** Microhardness**;** Composite**;** Vickers

RESUMO

Durante a fabricação de restaurações estéticas indiretas um cimento de cimentação temporário é necessário. Este estudo avaliou o efeito da dentina contaminada com eugenol sobre a polimerização de resinas compostas. Sessenta discos de compósitos foram feitos sobre dentina previamente contaminada com um dos cinco diferentes cimento para cimentação temporários (Temp Bond, Temp Bond NE, Rely X Temp, Resin Sensitemp, Freegenol), com dois diferentes compositos (Filtek Z-100 and Filtek Supreme Plus). O número de dureza vickers foi obtida no lado da luz da amostra assim como na parte inferior do mesmo. As comparações foram feitas utilizando ANOVA com análise de postos de Tukey-Kramer, ambos calculados com significância de 0,05. O VHN de todas as espécies do lado inferior foram menores que do lado em contato com a luz. A redução média foi de 12% para o compósito Supreme Plus e 9% para o compósito Z-100, todos os resultados dentro do esperado. Os cimentos com Freegenol e Temp Bond NE produziram algumas vezes uma redução maior de VHN no lado inferior que do lado do cimento contendo eugenol. Dentina contaminada por eugenol não reduziu a polimerização da resina composta sobre ela.

Palavras-chave: Eugenol; Microdureza; Compósito; Vickers.

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INTRODUCTION

It is true that eugenol inhibits polymerization[1], it interferes with the free radicals associated with the composite's polymerization slowing it, reducing the degree of polymerization[2]. The OH group, present in the eugenol molecule, produces a positive charge in these free radicals, blocking their reactivity[3]. Peters and Col[4] found that marginal adaptation of composite resins decreased if an endodontic sealer containing eugenol was used prior to the bonding. Sarac and col[5] found that a gross removal of eugenol-containing temporary cement did lower the bond strength when compared to other more active cleaning techniques such as the use of a rotary instrument or air abrasion[6].

The ability of eugenol to penetrate dentin tubules and contaminate, depends on the concentration, it happens with pure eugenol, but not as much with the eugenol in a zinc oxide-eugenol based cement[7] because liberation of eugenol molecules from the cement is by hydrolysis[8-11]. Hume[12]

also said that in a cavity well sealed the only water that can cause the eugenol to be released, comes from the dentin tubules, but if there is filtration along the margins of the restoration, the amount of water that can cause the liberation of the eugenol towards the dentin is greater. Unreacted eugenol from temporary cements can penetrate dentin. Hume in 1998[12] found that the diffusion rate of eugenol released from zinc oxide-eugenol cements increased to a peak after 1 day of about 0.3 nmol/min and then decreased slowly to 0.08 nmol/min after 14 days. For this study a 7 day storage time under water was determined to be enough to contaminate dentine.

Many studies showed that zinc oxide-eugenol cements do not reduce the bond strength to dentin[13-19] while others showed that it is affected[20,21].

Yap and col[1] did not find any effect in the bond strength of composite when IRM was used. They found that when the cement was used in concentrations of 10gr/2ml, the bond strength was lower, but statistically the same to the control group that used a polycarboxylate cement. On a similar research by Ferreto and col[22], no significant reduction on the bond strength of six dentin boding agents when bonded to dentin contaminated with temporary cements. The results were statistically the same to those groups that used non-eugenol temporary cement, but some groups with non-eugenol temporary cement produced lower bond strength to some bonding agents, compared to the group with the eugenol temporary cement. Similar results have been published[13,23,24]. Abo-Mahar and col[25] also found the bond strength to dentin was not affected by the use of temporary cements containing eugenol that was removed with an excavator before bonding a resin luting cement. Wooly and David[19] suggest that this could be due to contamination of the temporary cement of the cut dentin, rather than due to a negative effect of the eugenol. Terata[26] stated that although macroscopically the dentin surface may appear clean, there are still remnants of the cement microscopically. The use of just pumice and water to clean dentin after the application of a provisional luting cement was described by Mojon and col[27]; and Grasso and col[28].

As stated above, many studies have evaluated bond strength as a mean to measure the effect of the eugenol on the polymerization of the bonding agent or the composite resin. The use of superficial Microhardness testing in the evaluation of polymerization of composites has been used for many years. Ferracane[29] found that there is a correlation between the superficial hardness and the degree of conversion of the double bonds, but this correlation is not as exact at higher depths of cure[30]. Wilson and col[31] stated that surface Microhardness cannot be use to directly compare the degree of conversion of composites, but the use of top-bottom comparisons does have a linear relationship between degree of conversion and hardness. The easiest way to start evaluating the effect of the eugenol on polymerization would be to start evaluating the surface Microhardness of composites cured over eugenol-contaminated dentin, if some effects are found, then we could move to test it on bonding agents, or to include steps like acid-etching to the process, as a mean of cleaning the dentin instead of just doing a gross cleaning of it.

The purpose of this study was to evaluate the surface microhardness of composites cured over dentin previously contaminated with various temporary cements.

MATERIAL and METHODS

Sixty, recently extracted molars, caries and restoration free, were cut to expose deep dentin using a low speed diamond saw under water (Isomet Low Speed Saw, Bueheler, Lake Bluff, IL, USA). The root portion of each tooth was cut and the remaining was placed under acrylic resin just to make manipulation easier. All specimens were polished under water with a SiC paper of 600 grit to produce a smooth surface and an uniform smear layer. Specimens were divided in twelve groups (n=5) to receive five different temporary cements, each will have one of two composite resins placed over it after cleaned.

Ten of the twelve groups were contaminated with crown and bridge provisional luting agents: Rely X Temp (3m ESPE, St Paul MN), Resin Sensi Temp (Sultan Healthcare, Hackensack NJ), Temp Bond NE (Kerr, Orange CA), Freegenol (GC America, Alsip IL) all non-eugenol cements, and Temp Bond (Kerr, Orange CA) an eugenol containing cement (Table 1); two groups per cement. Equal portions of base and catalyst were mixed and placed over the exposed dentin, trying to produced a layer

of approximately 1 mm thick, allow to set undisturbed and then placed under water. Two groups left the dentin untreated to serve as controls.

Specimens were stored under water at 37º C for seven days. Then the provisional cement was removed manually and the dentin surface was cleaned using a fine grain pumice powder under medium pressure with a low speed handpiece for 30 seconds. Teflon mold, 3 mm thick, and 10 mm diameter, was placed over the exposed dentin, filled with composites Filtek Supreme XT and Z-100, in one increment, covered with a mylar strip, flattened with a glass slab of 1 mm thick. All samples were cured for 20 seconds (HiLux LED, 800 mW/cm2). Then removed from the mold, placed in a dark envelope to avoid further exposure to the light.

Table 1. Materials used in study.

	Material	Manufacturer	Code
Provisional Luting Agents	Rely X Temp	3M ESPE	RX…
	Resin Sensi Temp	Sultan Healthcare	ST…
	Temp Bond NE	Kerr	TN…
	Temp Bond	Kerr	T…
	Freegenol	GC America	F…
Composite Resins	Filtek Supreme Plus	3M ESPE	…S
	Filtek Valux Plus	3M ESPE	…Z

Specimens were evaluated for VHN (Vickers hardness) on a micro-hardness tester (Micromed 2001, Buehler) with a load of 200 gr. for 10 seconds. Three measurements were made on each surface, the light side and the bottom side (dentin side); an average of those three was used as the VHN for that surface.

A two-way ANOVA was run at 0.05 significance level to establish differences among provisional luting agents and between sides. Since VHN of both composites evaluated is different due to their own characteristics, no attempt was made to try to compare them. Means were compared using a Tukey-Kramer analysis, also calculated at a 0.05 significance level.

RESULTS

Composites Filtek Supreme Plus and Filtek Z-100 showed different superficial hardness between them, Supreme Plus had a hardness of 90 VHN and Z-100 of 117 VHN on average. These results are expected when we take a deeper look at their filling particles composition and size. It was not part of the objectives of this study to compare superficial hardness between composites; therefore the results are given separate for each composite resin.

Both sides of each sample were measured, the "top side" which was the side closest to the curing unit's tip, and the "bottom side", which is the side of the composite opposite to the curing unit's tip and in contact to the contaminated dentin. All samples were 3 mm thick, so the curing light had to go through that thickness of composite to cure the bottom. VHN of all bottom measurements in all groups were lower to that of the top side, including groups cured over dentin contaminated with a eugenol containing provisional cement, groups with non-eugenol cements and temporary resin cements, except groups RXZ and FZ who had the same superficial hardness top and bottom.

Filtek Supreme XT

VHN for this composite measured from 79 VHN for group FS up to 104 VHN for group TNS. Average hardness for this composite on the top side was 90 VHN.

The lowest VHN of the bottom side was group RXS with only 75 VHN. Group TS was the highest (See table 2) with 83 VHN, this is the group cured over the eugenol-contaminated dentin. The difference between both groups was only 8 VHN and was not statistically significant. Three groups evaluated in this study were non-eugenol cements: Temp-Bond NE, Freegenol and Rely X Temp, and no effect on the composite polymerization were found.

The VHN reduction between top and bottom sides of all groups was expected and within range of acceptability, according to ISO 4049-2000. The eugenol contaminated side reduction was statistically significant compared to the top side, but only a 15% reduction at 3 mm of depth. The greatest reduction in hardness between top and bottom measurements was on group TNS with 25% reduction (See table 3), and the lowest with groups FS and STS with only a 3% reduction. On average, the VHN of the bottom side dropped 12% when compared to the top side.

Table 2. Vickers hardness (VHN) for composite Filtek Supreme Plus; top and bottom measurements, and percentage of change between them.

Temp Luting Agent	Top side	Bottom side	% of change
Tempbond (TS)	98	83	15.3
Tempbond NE (TNS)	104	78	25
RelyX Temp (RXS	84	75	10.7
Freegenol (FS)	79	76	3.8
Resin Sensitemp (STS)	84	82	2.4
Control	92	77	16.3
Average	90	78	12

Filtek Z-100

The superficial hardness for this composite on the top side was from 112 VHN for FZ and 126 VHN for group TZ. The average microhardness for this composite was 117 VHN. On the bottom side of the samples, the highest value was with group RXZ with 118 VHN and the lowest with group TNZ at 97 VHN (Table 3). Both luting agents are eugenol-free cements. Group TZ had a VHN of 112. The variation of values for the bottom surface of composite Z-100 was only 11 VHN. There was no significant difference among groups on this surface.

Table 3. Vickers hardness (VHN) for composite Filtek Z-100; top and bottom measurements, and percentage of change between them.

Temp Luting Agent	Top side	Bottom side	% of change
Tempbond (TZ)	126	112	11.1
Tempbond NE (TNZ)	113	97	14.2
RelyX Temp (RXZ	118	118	0
Freegenol (FZ)	112	111	0.9
Resin Sensitemp (STZ)	117	100	14.5
Control	114	101	11.4
Average	117	106	9

As an interesting result, groups RXZ and FZ had the same superficial hardness on the bottom side as the top side. This means that the light intensity on this composite went unchanged through the whole composite sample. Groups with the greatest reduction were TNZ and STZ with approximately 14% reduction between top and bottom side. There was no significant difference between the superficial hardness of top and bottom sides

DSICUSSION

Polymerization of composite resins occurs when double bonds on the Bis-GMA molecule open. This break of the double bonds is possible by the action of camphorquinone molecules, which are activated by light energy. The amount of energy absorbed by the composite determines, up to a point, of the degree of polymerization. The relationship between superficial hardness and the degree of double bond conversion, was studied many years ago by Ferracane[29] and Wilson and col[31]. This was made by means of infrared spectroscopy, to measure the amount of double bond conversion.

The amount of light energy that can reach the camphorquinone molecules lowers as it goes through the composite due to light dispersion and refraction of the filler particles, the absorption of light by the same camphorquinone molecules and the pigments inside the composite, and due to the composite own opacity. All of this produces a lower degree of polymerization and therefore smaller microhardness. ISO 049-2000 takes this into account when depth of cure has to be calculated for each composite. The superficial hardness of the bottom side has to be close to that of the top side to be considered acceptable cured. The composites used in this study had a recommended depth of cure of 2 mm, but in order to seek a greater impact of the dentin contamination over which it was cured, we decided to use 3 mm thick sample. Yet the polymerization was more than acceptable for both composites. Composite Z-100 also had a recommended curing time of 40 seconds and it was only cured for 20 seconds to keep this variable the same for all groups in the study. The superficial hardness of the bottom side of composite Z-100 was statistically the same to the top side.

The superficial hardness of the bottom side of composite Supreme Plus and its top side was statistically different, but only samples cured over dentin contaminated with Temp Bond NE had a 25% reduction, the highest recorded in this study. This result was unexpected since this cement does not have in its composition anything that can interfere with the polymerization process. Wooly and David[19] stated that contamination of the cement itself have more effect in the degree of polymerization than the eugenol present in it.

Z-100 had a greater superficial hardness than Supreme Plus; this result may be explained in its filler. Its particles are greater in size and of irregular shape,

and may produce a harder surface than the nanofilled Supreme Plus.

The hardness reduction of the bottom side, on both composites cured oven the eugenol-contaminated dentin was on average only 13%. If we take in consideration that all samples were 50% thicker than manufacturer's recommendations, we can state that the polymerization was clinically acceptable, and that the reduction is due to the decrease in the energy density of the curing light at that depth rather than a negative effect of the eugenol remnants. The percentage of change between top-bottom hardness found in this study was similar or lower than the obtained by Tsai and col[32] and by Rueggerberg and others[33].

The manufacturer states that Z-100 should be cured at 2 mm curing depth and for 40 seconds, this depth and curing time resulted unnecessary and to much, because this composite cured adequately for just 20 seconds and at 3 mm of depth (average VHN reduction from the top side was only 9%), it does not mean that the clinician should use such big increments, because they will have greater polymerization shrinkage and an unfavorable C-factor.

The results of this study are similar to what Wooly and David[19] found, when he stated that contamination from the temporary cement, regardless if it has eugenol or not, is more to blame for a decrease in bond strength. Erkut and col[34]. Also found that the use of provisional luting agents decreased the bond strength to dentin regarless if it had eugenol or not. In this study the same statement can be made as to contaminants from the temporary cement is more to blame for differences in the degree of polymerization, differences that were not statistically significant.

In this study, the composite was cured over the contaminated dentin, this is not a normal clinical procedure and one of the limitations of this study, yet, the author believes that even with new technology, such as the ability to measure nano-hardness, to measure the hardness of the hybrid layer or the resin tags inside the tubules, we will find again that the remnants of eugenol are not enough to negatively affect the polymerization of the adhesive resin.

CONCLUSION

Eugenol contaminated dentin did not reduced the polymerization of composite resin cured over it.

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