Effects of Hydrochloric Acid on Enamel Adjacent to Composite Restorations –an *in vitro* Study

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In this study were evaluated the chemical changes in enamel adjacent to four different composite resin restorations after their contact with hydrochloric acid. Twenty healthy molars were chosen for this study and class I cavities were prepared on occlusal surfaces. Four commercial composite resins were used for filling: Filtek Z250 (3M ESPE, USA), Filtek Ultimate (3M ESPE, USA), Herculite (SDS KERR, USA) and Point 4 (SDS KERR, USA). The samples were obtained by cutting the teeth in two halves. Some of the samples were maintained in distilled water as control group and the others were mentained for 30 min in 0,06 mol/L hydrochloric acid (pH = 1,2). The morphological aspect of the interface between enamel and composite resin was evaluated using scanning electron microscopy (SEM) and the mineral content (calcium, phosphorus, oxygen ions concentration) was assessed using EDX method. Chemical analysis showed significant changes of ions concentration both in enamel and in composite restorations. The enamel adjacent to Filtek Ultimate was less affected by the contact with hydrochloric acid when comparing to other composite resins.

Keywords: gastroesophageal reflux disease (GERD), SEM microscopy, EDX method mineral content to enamel adjacent resin composite.

The first symptoms of gastroesophageal reflux disease (GERD) may occur in the oral cavity. A significant number of patients with GERD have atypical or extraesophagael symptoms [2] such as dental erosion [1]. The relationship between the GERD and dental erosion has been first reported by G.F. Howden [3]. Dental erosion is caused by the presence of intrinsic and extrinsic acid of non-bacterial origin in the mouth, combined or separately [4]. Intrinsic causes of acid presence in the mouth are known to be regurgitation, vomiting, GERD or rumination.

Any acid with a *p*H below 5.5 can dissolve the hydroxyapatite crystals in enamel. Gastric refluxate has a *p*H of less than 2.0 [2].

There is a variety of severity scales and scoring systems to document the extent of damage resulted from erosion [5, 6]. Scales are based on degree of dentin exposed, cupping of cusp tips, loss of tooth morphology, and restoration margins raised above the level of the surrounding tooth structure.

The longevity of dental restorations depends on the durability of the material per se and its wear resistance the durability of the interface between tooth substance and restoration, the level of tooth destruction, its location and load [7, 8].

Another study investigated the chemical degradation of composite restorations after conditioning in artificial saliva and various food-simulating liquids for 1 week by measuring the change in surface hardness and the thickness of the degradation layer. Specimens were immersed either in distilled water, 0.02 N citric acid, 0.02 N lactic acid, heptane or in 75–25% ethanol–water solution. The effects of chemical media on hardness change were found to be material dependent. A significant but weak correlation was detected between change in hardness and thickness of the degradation layer [9].

The wear to three dental materials, under the action of acid *p*H, was observed in a study by M. Shabanian and L.C. Richards [10]. The three test materials were more resistant than enamel to acid, with the composite demonstrating the lowest susceptibility to acid. The resistance of the tested resin-modified glass-ionomer cement to load and acidic challenge was lower than that of the composite and higher than that of the conventional glass-ionomer cement. Studying the effect of different storage media upon the surface micromorphology of resin-based restoratives, it was found that the surface roughness of restoratives was significantly higher after a *p*H cycling regime than after storage either in distilled water or in artificial saliva [11]. There are some indications that acid–base challenges can determine some degradation of the dentine–adhesive interface [12].

The influence of different dietary solvents (0.02 M citric acid, 50% ethanol–water solution, heptane, distilled water as control) on shear punch strength was determined in another studies. The nanofilled composite resin and the ormocer have a lower strength when compared to minifilled composite, but higher than the compomer and the highly viscous glass-ionomer cement [13].

The resistance of composite resins to acid attack is higher when comparing to compomer and giomer materials, evidenced in a study by A.U.Yap in 2005 [14]. Analyzing these investigations, one conclusion can be drawn: under acidic conditions all dental restorative materials show degradation over time (surface roughness, decrease of surface hardness, substance loss). However, it seems that ceramic and composite materials show a good durability [15, 16].

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The appearance of new adhesive materials considerably improved minimally invasive techniques in dental erosion. In recent years, the wear resistance of posterior composite fillings has been enhanced. Therefore, the use of modern direct restorative materials can provide excellent longevity, even in load-bearing situations. Several case reports demonstrate the successful rehabilitation of (erosive) worn dentitions using adhesive techniques [17].

A. Lussi [7] recommended that the treatment of erosive tooth wear should be performed at an early stage in order to prevent the functional and aesthetic complications. Occlusal erosions typically show grooves on occlusal aspects and edges of restorations higher than the adjacent tooth surfaces. These grooves demonstrate a prolonged time of decreased *p*H values after an acidic attack, which would lead to further progression of the erosive process at that site. In such cases, minimally invasive composite fillings are able to protect the affected region. Conventional glass-ionomer cements are not recommended as permanent restorations because of their disintegration in acidic conditions [18, 19].

The advantage of direct composite restorations is that they are adaptable to the defect and the repair is easy to do. The situation is more problematic if the occlusal and vestibular erosions merge, the original tooth shape becomes hardly recognizable and the loss of vertical dimension tends to be higher than 2mm [7].

The present paper aims to analyze the interface between enamel and four different composite resins using scanning electron microscopy (SEM) and EDX method, after suspension in 0.06 mol/L hydrochloric acid (pH = 1.2) for 30 min.

Experimental part

Materials and methods

In the study were included 20 molars, extracted by periodontal reasons. The teeth were divided in four groups having five teeth each. The experimental protocol was the same as that one used in one previous study [15]. The teeth extracted were kept in 10% formaline solution and then the organic material was removed using a hand instruments. Class I cavities were prepared on occlusal surfaces. Four commercial composite resins were used to restore the cavities: Filtek Z250 (3M ESPE, USA), Filtek Ultimate (3M ESPE, USA), Herculite (SDS KERR, USA) and Point 4 (SDS KERR, USA). Restorations were made following the instructions provided by the manufacturer, using a horizontal incremental filling technique with 2 layers of 2 mm thickness. Each layer was light cured for 40 s using a LED lamp (LEDidition from Ivoclar Vivadent clinical, Austria).

The samples used in the study were obtained by cutting the teeth in two halves. Some of the samples were maintained in distilled water as control group and the others were maintained for 30 min in 0.06 mol/L hydrochloric acid (*p*H=1,2). The morphological aspect of the interface between enamel and composite resin was evaluated using scanning electron microscopy ((SEM model VEGA II LSH, TESCAN) and the mineral content (calcium, phosphorus, oxygen ions concentration) was assessed using EDX detector (QUANTAX QX2, BRUKER/ROENTEC). The data were statistically analysed using ANOVA and post-hoc Bonferroni tests (significant at a p value of less than 0.05).

Results and discussions

SEM analysis has shown significant morphological changes of both enamel and adjacent composite resins in the samples immersed in the study solution for 30 min, compared to control samples.

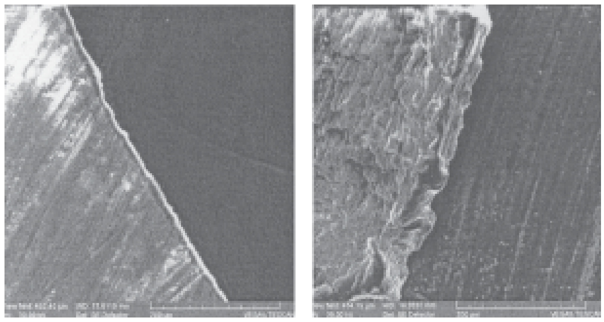

Fig. 1. SEM images at the interface between the composite resin Filtek Ultimate and enamel: a. Control, b. Study

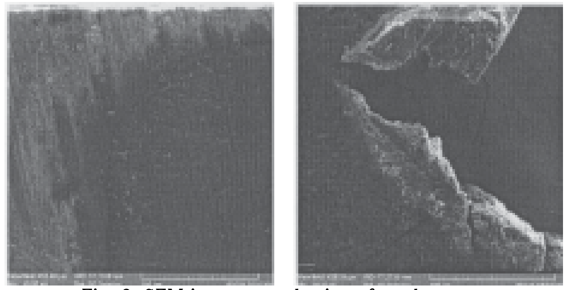

Fig. 2. SEM images at the interface between the composite resin Filtek Z 250and enamel: a.Control, b. Study

The first figure shows the degradation of the enamel structure adjacent to restoration, with alteration of enamel rods. In figure 1b. the modification of the restoration surface is evident, and a microleakage gap is present between the restoration and the tooth tissue.

In figure 2 the structure of composite Filtek Z 250 is changed and fissures are present. Several pores and fissure can be also observed in enamel.

Figure 3 shows a restoration with composite resin Herculite. Multiple porosities and fissures can be noticed.

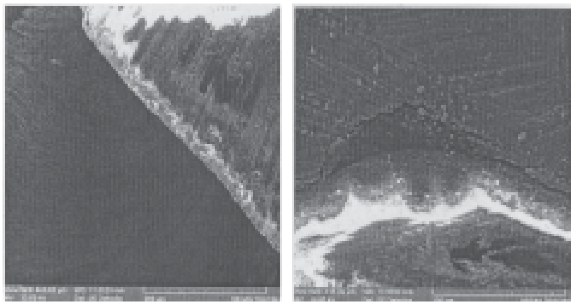

Fig.3. SEM Imgaes at the interface between the composite resin Herculite and enamel: a. Control, b. Study

In figure 4 the fissures involve the enamel adjacent to restoration with composite resin Point 4. The interface enamel-restoration has preserved the characteristics which show maintenance of the adhesion to tooth structures.

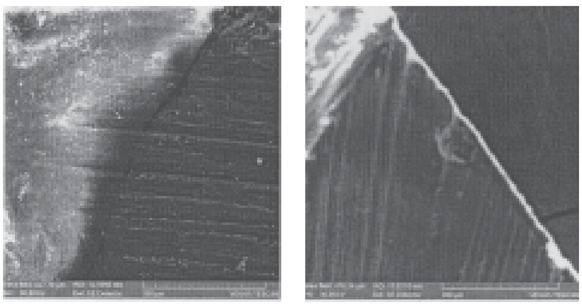

Fig. 4. Images SEM at the interface between composite resin Point 4 and enamel: a.Control, b. Study

		No samples	Mean (wt %)	Std.Dev.
Ca	Filtek Z250 control	5	47.5860	.46667
	Filtek Ultimate control	5	30.3680	.46965
	Herculite control	5	47.3080	.19563
	Point 4 control	5	48.3280	.19318
	Total	**20**	**43.3975**	**7.73448**
P	Filtek Z250 control	5	27.2020	.20142
	Filtek Ultimate control	5	18.1740	.13353
	Herculite control	5	19.0500	.37370
	Point 4 control	5	18.4660	.20231
	Total	**20**	**20.7230**	**3.85797**
O	Filtek Z250 control	5	25.2120	.05933
	Filtek Ultimate control	5	50.3900	.20199
	Herculite control	5	33.6380	.24783
	Point 4 control	5	33.2040	.35011
	Total	**20**	**35.6110**	**9.40922**

Table 1
MINERAL CONTENT IN THE ENAMEL ADJACENT TO RESTAURATIONS
(CONTROL GROUPS)

The table 1 presents the mean values of mineral content in the control group, expressed as weight percents (wt%).

Different mean values of calcium, phosphorus and oxygen ion concentrations were obtained in enamel adjacent to the four composite restorations in control group. Post-hoc Bonferroni statistical test showed no significant statistical differences between the mean concentrations of calcium ions in enamel adjacent to Herculite and Filtek Z250 composite resins in control groups (table 2, $p = 1.000$, >0.05).

Significant statistical differences were obtained when compared the mean concentrations of calcium ions from Filtek Z250, Herculite, Point 4 and Filtek Ultimate ($p = 0.000 < 0, 05$). No significant statistical differences were obtained when compared the mean phosphorus ion concentration in enamel adjacent to Point 4 and Filtek Ultimate samples ($p = 0.463 > 0.05$). Significant statistically differences were obtained when compared the mean concentrations of phosphorus ion from Filtek Ultimate, Herculite, Point 4 and Filtek Z250 ($p = 0.000 < 0.05$). For oxygen ions, no significant statistically differences were obtained when compared the mean values of the ion in enamel adjacent to Herculite and Point 4 composite resins ($p = 0.066$, > 0.05). Significant statistical differences were obtained when compared the mean concentrations of oxygen ions from Filtek Ultimate, Herculite, Point 4 and Filtek Z250.($p = 0.000 < 0.05$).

We observe in the table above that average values for calcium, phosphor and oxygen are different in the four groups.

We find differences statistically significant in the four groups for each of the evaluated elements. ($p < 0.005$)

The mean values of calcium ion in the samples when Filtek Z250 was used for restoration were lower in the study group when compared to control group (study group = 40.4520; control group = 47.5860 wt%). In the samples

		Filtek Z250			Filtek Ultimate			Herculite			Point 4		
		Ca	P	O	Ca	P	O	Ca	P	O	Ca	P	O
Filtek Z250	Ca	-	-	-	0.000	-	-	1.000	-	-	0.029	-	-
	P	-	-	-	-	0.000	-	-	0.000	-	-	0.000	-
	O	-	-	-	-	-	0.000	-	-	0.000			0.000
Filtek Ultimate	Ca	0.000	-	-	-	-	-	0.000	-	-	0.000	-	-
	P	-	0.000	-	-	-	-	-	0.000	-	-	0.463	-
	O	-	-	0.000	-	-	-	-	-	0.000			0.000
Herculite	Ca	1.000	-	-	0.000	-	-	-	-	-	0.002	-	-
	P	-	0.000	-	-	0.000	-	-	-	-		0.010	
	O	-	-	0.000	-	-	0.000	-	-	-			0.066
Point 4	Ca	0.029			0.000			0.002			-	-	-
	P		0.000			0.463			0.010		-	-	-
	O			0.000			0.000			0.066	-	-	-

*. The mean difference is significant at the 0.05 level.

Table 2
BONFERRONI STATISTICAL TEST RESULTS WHEN COMPARED THE MINERAL CONTENT IN CONTROL GROUPS

		No samples	Mean (wt %)	Std.Dev.
Ca	Filtek Z250 (30 minutes)	5	40.4520	1.00403
	Filtek Ultimate (30 minutes)	5	30.3680	.30384
	Herculite (30 minutes)	5	63.9860	.31453
	Point 4 (30 minutes)	5	47.6120	.27689
	Total	**20**	**45.6045**	**12.58267**
P	Filtek Z250 (30 minutes)	5	12.0120	.11925
	Filtek Ultimate (30 minutes)	5	18.1780	.05119
	Herculite (30 minutes)	5	24.4980	.14618
	Point 4 (30 minutes)	5	25.4780	.20717
	Total	**20**	**20.0415**	**5.55893**
O	Filtek Z250 (30 minutes)	5	46.5700	.16294
	Filtek Ultimate (30 minutes)	5	50.3940	.45153
	Herculite (30 minutes)	5	10.5380	.28490
	Point 4 (30 minutes)	5	26.0100	.27632
	Total	**20**	**33.3780**	**16.54210**

Tabel 3
MINERAL CONTENT IN THE ENAMEL ADJACENT TO RESTAURATIONS IN STUDY GROUPS

		Filtek Z250			**Filtek Ultimate**			**Herculite**			**Point 4**		
		Ca	**P**	**O**	**Ca**	**P**	**O**	**Ca**	**P**	**O**	**Ca**	**P**	**O**
Filtek Z250	**Ca**	**-**	**-**	**-**	**0.000**	**-**	**-**	**0.000**	**-**	**-**	**0.000**		
	P	**-**	**-**	**-**	**-**	**0.000**	**-**	**-**	**0.000**	**-**		**0.000**	
	O	**-**	**-**	**-**	**-**	**-**	**0.000**	**-**	**-**	**0.000**			**0.000**
Filtek Ultimate	**Ca**	**0.000**	**-**	**-**	**-**	**-**	**-**	**0.000**	**-**	**-**	**0.000**		
	P	**-**	**0.000**	**-**	**-**	**-**	**-**	**-**	**0.000**	**-**		**0.000**	
	O	**-**	**-**	**0.000**	**-**	**-**	**-**	**-**	**-**	**0.000**			**0.000**
Herculite	**Ca**	**0.000**	**-**	**-**	**0.000**	**-**	**-**	**-**	**-**	**-**	**0.000**		
	P	**-**	**0.000**	**-**	**-**	**0.000**	**-**	**-**	**-**	**-**		**0.000**	
	O	**-**	**-**	**0.000**	**-**	**-**	**0.000**	**-**	**-**	**-**			**0.000**
Point 4	**Ca**	**0.000**			**0.000**			**0.000**					
	P		**0.000**			**0.000**			**0.000**				
	O			**0.000**			**0.000**			**0.000**			

*. The mean difference is significant at the 0.05 level.

Table 4
BONFERRONI STATISTICAL TEST RESULTS WHEN COMPARED THE MINERAL CONTENT IN STUDY GROUPS

where Herculite composite resin was used, the mean value of calcium ion increased when compared to Filtek Z250. Regarding the phosphorus ion concentration, the lowest values were obtained when Filtek Z 250 was used for filling. The mean values of the oxigen ion in the samples when using Filtek Z 250 were higher in the study group compared to the control group, but to the values in samples when using Herculite and Point 4 were lower.

Herculite XRV™ is a microhybrid dental composite resin containing uncured methacrylate ester monomers, non-hazardous inert mineral fillers, non-hazardous activators and stabilizers, inorganic filler loading is 59% by volume with average particle size of 0.6 microns.

Point 4™ is a light-cured nanofilled composite resin that contains approximately 76% by weight (57% by volume) inorganic filler with an average particle size of 0.4 microns.

These values modified to mineral concentrations, of the samples in the study group compared to the control group, are due to the different composition and the weight to the filler of resins and composites evaluated.

D.C. Sarrett [20] observed that marginal gaps created by polymerization shrinkage do not appear to increase the risk for secondary caries, but can lead to marginal staining.

Initial marginal quality should not affect longevity, as it does not necessarily increase the risk of secondary caries. However, poor marginal quality is, in fact, likely to decrease clinical longevity due to the misdiagnosis of secondary caries.

K.J. Söderholm et al. [21, 22] observed that the longevity of composite restorations can be influenced by environmental conditions in the oral cavity. They showed that nanofilled composites are more soluble than hybrid, probably due to different sizes of particles fillers, which may cause a greater degradation than the hybrid composites nanofilled. Clinically, it seems probably that nanofilled composites would undergo higher degradation in the oral environment than hybrid composite [21, 22].

Modification of the surface can be considered as a process of degradation and erosion of the polymer matrix. The action mechanism of the acid on composite resin can be explained by the hydrolysis of the ester radicals present in the monomers dimethacrylate (Bis-GMA, Bis-EMA, UDMA, TEGDMA0 [23, 24].

In all samples analyzed in this study the scanning electron microscopy revealed the presence of mineral particles which protrude on the surface, which can suggest the alteration in organic matrix.

S. Wongkhantee et al. (2006) [25] noted a lower resistance to acid attack in composites microfille comparing to universal hybrid composites, explained by the higher ratio of organic matrix in microfille composites.

In Filtek Z250, the majority of TEGDMA has been replaced with a blend of UDMA (urethane dimethacrylate) and Bis-EMA (Bisphenol A polyethylene glycol diether dimethacrylate). Both of these resins have higher molecular weight and therefore have fewer double bonds per unit of weight. The high molecular weights also influence the measurable viscosity and results in less shrinkage, reduced aging and a slightly softer resin matrix. Additionally these resins exhibit a higher hydrophobicity and are less sensitive to changes in atmospheric moisture. In Filtek Ultimate Universal Restorative the resin system is slightly modified from the original Filtek Z250 and Filtek Supreme resin.

In a previous study, I. Nica et al. (2012) [26] observed excellent chemical and stable results for Filtek Ultimate in comparison with Filtek Supreme XT and Filtek Z250. The superficial content of oxygen is significantly increasing with the polymerization time for Filtek Supreme XT and Filtek Z250, and consequently this process favors the formation of oxygen layer [26].

The resin from surface was imperfectly polymerized, which could affect the properties of the surface [27]. Under ideal conditions the adhesive interface is a continuous distribution area of the stress between filler and matrix, which requires a coupling agent with intermediate properties between filler and matrix. When this occurs, the area will be more resistant to degradation.

Another important role of the coupling agent is that, at least to a certain degree, it protects the filler from degradation by hydrolysis. Any of the matrix resin which is insufficiently polymerized can be dissolved in alcohol and the acidic solution so that the mineral particles can be removed quickly. Another possible cause of the degradation of composite surface is the weak bond between the fillers and the matrix. This correlates with insufficient treatment of filler surface with silane, which results in filler erosion [28].

Previous studies have demonstrated that the resistance of the composite resin to the acidic attack is higher in those who have a higher loading with mineral particles [29, 30].

In addition to the content of mineral particles, the properties of the filler, its distribution and treatment with silane, are also important factors in determining the resistance of composite materials to erosion.

The degree of impairment of composite resins surface is correlated with the level of acidity of the solution in which materials are submerged.

The interface enamel and Filtek Ultimate was the most stable, maintaining the average values of ion concentrations after immersion in acid solution. The mineral structures of enamel adjacent to the others composite resins, underwent changes of ion concentrations after the immersion in acid solution. These results can be explained by the stability of the evaluated composite resins to acidic challenge.

In a study about the corrosion effect of several solution on Filtek Supreme Universal, I Nica et al. (2014) [31] observed an increase in the percentage of oxygen, as a consequence of oxides formation on the surface of samples. The concentration of calcium ions has increased as a result of the products of chemical reaction.

In a study by C. Arnauteanu et al. in 2013 [32] about the impact of various acidic drinks on enamel, SEM analysis revealed severe erosion in the enamel directly exposed to test solutions without artificial saliva protection. Also, the drops of calcium and phosphorus ions in enamel after immersion in acidic solutions were more important than those registrated for the enamel protected with artificial saliva.

Conclusions

The mineral concentration (calcium and phosphorus ions) in the enamel adjacent to Filtek Z 250 composite resin recorded the highest changes in values after immersion in hydrochloric acid.

The chemical composition of enamel adjacent to nanofille composites Filtek Ultimate, was less affected comparing to others composites.

SEM analysis showed significant changes in both enamel and the composite restorations.

Dentists must be aware of the differences concerning the materials behaviour in acidic environment when they decide to use composite resins for the treatment of erosions in patients with GERD.

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