Comparative Study Regarding the Surface Roughness of Highly Viscous Flowable Composites After Immersion in Acidic Drinks

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The aim of the present study was to assess, by profilometry, the effect of different immersion regimes in Coca-Cola drink on the surface roughness of two highly viscous flowable composite resins. The studied materials were Gradia Direct LoFlo (GC Corporation,Tokyo, Japan) and Micro Esthetic flow-viscous (Bisico, Germany). Fifteen cylindrical samples having the of 5 mm and the thickness of 2 mm were made from each material using plastic molds placed on glass plates. The samples were light-cured for 40s on both sides through the glass plates, to ensure complete polymerization of the material. They were divided into two groups as follows: Group I. Gradia Direct LoFlo and Group II. Micro Esthetic flow-viscous. Specimens of each group were then randomly divided into 5 subgroups. In subgroup A (control group) 4 samples were kept only in artificial saliva. In subgroups B-E (each having 4 samples) the samples were immersed in Coca-Cola drink according to 4 different protocols. The mean values of roughness parameters, Ra, were calculated. The Kolmogorov-Smirnov normality test was used to determine the distribution of data in groups. ANOVA and Tukey post hoc statistical tests were used to compare the results from the groups. For both materials, the highest surface roughness was determined for the samples that were submerged 7 times/day in Coca Cola. The assessment of surface microstructure of the samples, after immersion, revealed for both materials a statistically significant increase of Ra values.

Keywords: flowable composite, acidic drink, profilometry

Resin composites are an excellent choice for minimally invasive dental procedures and therefore they allow to maintain maximum of tooth tissues. They are considered a suitable direct posterior ûlling material showing good long-term clinical survival [1, 2].

The continuous development of resin composites has led to formulations designed to further simplify the ûlling procedure, to provide better mechanical properties, to reduce the effect of polymerization shrinkage stresses and to improve aesthetics. Marginal defects of composite ûllings are often thought to be caused by poor adaptation of the restorative material to the cavity walls [3]. To avoid these defects, particularly in posterior teeth, the use of ûowable composites has been advocated due to their ability to *wet* and adapt well to cavity margins and walls [4, 5]. However, ûowable composites have a lower ûller content and usually weaker mechanical properties than conventional composites. Therefore, flowable composites can be applied as a restoration in minimally invasive occlusal cavity preparations, as pit and fissure sealants, as minimally invasive Class II restorations, and as non-carious cervical lesions restorations [6].

Recently, a new type of highly filled ûowable composite has been developed. When comparing to conventional paste-type composites, the highly flled fowable resin contains nano-sized fllers, the surface of which has been modiûed to provide a reduced viscosity for placement but allows the composite to be used in load-bearing restorations [7, 8].

In the oral environment, the composites are subjected to the action of various physical, chemical and mechanical agents. Particular attention was paid to the influence of extrinsic acidic challenges on the surface microstructure of composite resins [9-11].

Frequent consumption of carbonated drinks, alcohol, coffee, tea or red wine can affect some physical properties of composites resins such as surface roughness and microhardness, thus undermining the quality of restorations and their resistance to dissolution and disintegration [12-14].

The effect of these acidic challenges depends on the structure of restorative materials, such as organic matrix, filler load, filler distribution and silane treatment effect on fillers [15, 16].

The aim of the present study was to assess, by profilometry, the effect of different immersion regimes in Coca-Cola drink, on the surface roughness of two highly viscous flowable composite resins.

Experimental part

The materials used in the present study were:

GRADIA DIRECT LoFlo is a light-cured, high viscosity flowable microfilled hybrid composite for simple restorations. It takes advantage of GC's new HDR (High Density Radiopaque Pre-polymerized Filler) technology. This technology allows for excellent radiopacity while providing physical properties (strength and wear) like traditional composites making it ideal as a final restorative in all primary and conservative permanent restorations. Its highly filled formula ensures exceptionally low shrinkage, this along with its optimal flow characteristics, ensures better adaptation to proximal and cervical walls and internal line angels reducing the risk of contraction stress and microleakage GRADIA DIRECT LoFlo has indication for the following: restoration of Class I, II, III, IV and V cavities (particularly for small Class I cavities/shallow Class V cavities/other small cavities), restoration of root

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Material	Manufacturer	Type/ BatchNo/ Shade	Organic matrix	Filler type	Filler load (wt %)
GRADIA DIRECT LoFlo	GC Corporation, Tokyo, Japan	Microfilled, hybrid/ Lot 1707141/A3	UDMA, dimethacrylate component (trade secret),	Fluoro-alumino-silicate glass filler, HDR pre-polymerized fillers	40 wt%
MICRO ESTHETIC Flow-viscous	Bisico, Germany	Nano opimized, micro hybrid/Lot 007800/ A3	UDMA, 1.4-Butandioldimethacrylate Bis-GMA free	alumino-silicate glass filler	72 wt%

Bis-GMA: Bisphenol A diglycidyl ether dimethacrylate; UDMA: Urethandimethacrylate

Table 1
DESCRIPTION OF THE RESTORATIVE MATERIALS USED IN THE STUDY

surface caries, restorations in deciduous teeth, filling tunnel shaped cavities, sealing hypersensitive areas, composite restorations repair.

MICRO ESTHETIC flow-viscous is a flowable, highly viscous, highly radiopaque (210% AL), light-curing nanohybrid composites for restoring small class I-III cavities, and class V cavities. It can be used for fissure sealings, corrections of enamel defects, blocking out of undercuts, and minor shape and color corrections to the enamel.

The high filler content of 72 wt% glass filler particles ensures the high viscosity of the flow composite. There is no running or dripping of the flow composite and it allows a highly precise application. This makes minimal invasive class V restorations easier. Excellent mechanical properties such as low polymerization shrinkage, an extremely high abrasion resistance and high flexural strength are further key properties. As this new composite does not contain bis-GMA the patient's sensitivity is reduced and its biocompatibility is increased. The description of the restorative materials used in the study is presented in table 1.

Fifteen cylindrical samples having the diameter of 5 mm and the thickness of 2 mm were made using plastic molds. The conformers were placed in tight contact with a celluloid matrix between two glass plates, in order to obtain a smooth, flat and a surface free of pores. The samples were lightcured for 40 s on both sides through glass plate to ensure complete polymerization of the material, using a light source Amarys Wireless LED (Tosi). The source emits cold radiation with a wavelength range of 430 - 485 nm, has a maximum power of 700 mW/cm^2 and provides a 3 mm polymerization depth.

After removing the samples from the molds, they were divided into two groups: Group I. Gradia Direct LoFlo and Group II. Micro Esthetic flow-viscous. Then the samples from each group were randomly divided into five subgroups. In subgroup A (control) 4 samples were kept only in artificial saliva. In B-E subgroups the samples (4 in each subgroup), were submitted to 4 different immersion protocols. Each sample of the control group was completely immersed artificial saliva (25 mL), for seven days, in a hermetically sealed container at room temperature. Artificial saliva from each recipient was daily refreshed. The chemical composition of the artificial saliva solution proposed by Brett et al., for one liter of solution, *p*H = 6.7, is shown in table 2 [17, 18].

Table 2
THE CHEMICAL COMPOSITION OF THE ARTIFICIAL SALIVA SOLUTION

Chemical compound	Weight
KCl	1.5g
$NaHCO_3$	1.5g
NaH_2PO_4	0.5g
KSCN	0.5g
Lactic acid	0.7g

The specimens from the other 4 subgroups were kept in 25 mL of Coca-Cola (S.C. Coca-Cola HBC Romania S.R.L., Voluntari, Ilfov) with a *p*H = 2.5, as follows: subgroup B- once a day, subgroup C-3 times a day, subgroup D- 5 times a day, subgroup E- 7 times a day.

The specimens were completely immersed in Coca-Cola for 5 min and the container was continuously stirred, to ensure complete contact of the samples with the immersion medium. The Coca-Cola drink was changed after each immersion. When they were not subjected to immersion, the samples from subgroups B, C, D, and E were kept in artificial saliva. Some studies have shown that the most important changes in physical properties occur within the first 7 days of exposure to acidic solutions, and for this reason the immersion protocol was performed over a 7-day period. At the end of the protocol, the samples were washed with distilled water and dried using air spray.

For surface roughness assessment, all the samples were analyzed using the Surface Roughness Measuring Tester SJ-210, Mitutoyo, Japan. Regarding the roughness standard, the evaluation was based on ISO1997 applicable standards. Ten traces were registered in different areas with a tip load of 0.75 mN, a tip diameter of 2 µm, a scanning speed of 0.5 mm/s and a cut-off length (λc) of 0.25 µm. The roughness parameters were calculated and the mean arithmetic deviation, Ra, of the assessed profile was used. The Kolmogorov-Smirnov normality test was used to determine the distribution of data in groups. ANOVA and Tukey post hoc statistical tests were used to compare the results in groups.

Results and discussions

The mean Ra values obtained by quantitative assessment of samples surface using profilometry and standard deviation (SD) are presented in figure 1.

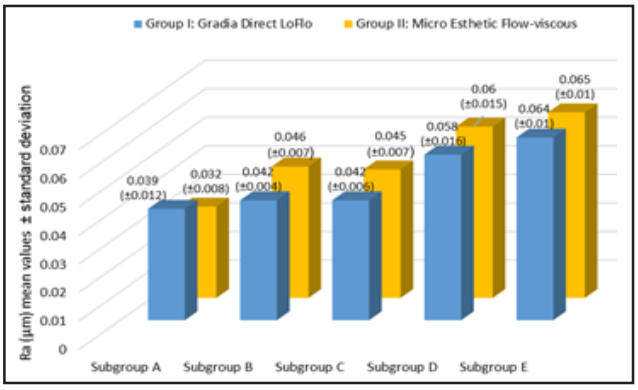

Fig. 1. Mean values of Ra parameter (±SD) in control groups and after immersion in Coca-Cola, for both tested materials

One-Sample Kolmogorov-Smirnov Test

		gradia direct loflo	micro esthetic flow viscous
N		75	75
Normal Parameters[a,b]	Mean	.04991	.04937
	Std. Deviation	.014541	.015526
Most Extreme Differences	Absolute	.152	.135
	Positive	.152	.135
	Negative	-.103	-.068
Kolmogorov-Smirnov Z		1.318	1.171
Asymp. Sig. (2-tailed)		.062	.129

a. Test distribution is Normal.

b. Calculated from data.

Table 3
THE KOLMOGOROV-SMIRNOV NORMALITY TEST RESULT

For both studied materials the lowest Ra values were obtained in subgroup A as follows Ra = 0.039 µm for Gradia Direct LoFlo and Ra = 0.032 µm for Micro Esthetic Flow-viscous. In subgroup B, increased Ra values (0.46 µm) were found only for Micro Esthetic Flow-viscous while for Gradia Direct LoFlo the Ra values (0.042) were closed to those of the control group. In subgroup C, for both materials, Ra values remained almost the same as in subgroup B. An obvious increase of Ra values was observed in subgroups D and E for both composite resins. The result of the Kolmogorov-Smirnov normality test showed that in all groups the data were normal distributed ($p > 0.05$) (table 3).

In order to compare the results in groups, ANOVA and Tukey post hoc statistical tests were used (tables 4, 5 and 6).

In group I - Gradia Direct LoFlo, the statistical analysis revealed that the values were not statistically significant when comparing subgroup A (control) to subgroup B and subgroup ($p > 0.05$). Also, when comparing subgroups B and C, respectively D and E, there were no statistically significant results. However, significant values were obtained when comparing subgroup A with subgroups D and E ($p < 0.05$).

In group II - Micro Esthetic Flow-viscous, statistically significant values were recorded when comparing subgroup A to all other subgroups. For this material the strongest statistical significance was determined when comparing subgroup A to subgroups D and E. As for the previous material, no significant values were obtained for Micro Esthetic Flow-viscous when comparing subgroup B to C and respectively subgroup D to E.

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
gradia direct loflo	Between Groups	.008	4	.002	16.971	.000
	Within Groups	.008	70	.000		
	Total	.016	74			
micro esthetic flow viscous	Between Groups	.010	4	.003	24.487	.000
	Within Groups	.007	70	.000		
	Total	.018	74			

Table 4
ANOVA STATISTICAL TEST RESULT

Multiple Comparisons

Dependent Variable: gradia direct loflo

	(I) category	(J) category	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	Subgroup A	Subgroup B	-.003000	.003890	.938	-.01389	.00789
		Subgroup C	-.002867	.003890	.947	-.01376	.00803
		Subgroup D	-.019200*	.003890	.000	-.03009	-.00831
		Subgroup E	-.025133*	.003890	.000	-.03603	-.01424
	Subgroup B	Subgroup A	.003000	.003890	.938	-.00789	.01389
		Subgroup C	.000133	.003890	1.000	-.01076	.01103
		Subgroup D	-.016200*	.003890	.001	-.02709	-.00531
		Subgroup E	-.022133*	.003890	.000	-.03303	-.01124
	Subgroup C	Subgroup A	.002867	.003890	.947	-.00803	.01376
		Subgroup B	-.000133	.003890	1.000	-.01103	.01076
		Subgroup D	-.016333*	.003890	.001	-.02723	-.00544
		Subgroup E	-.022267*	.003890	.000	-.03316	-.01137
	Subgroup D	Subgroup A	.019200*	.003890	.000	.00831	.03009
		Subgroup B	.016200*	.003890	.001	.00531	.02709
		Subgroup C	.016333*	.003890	.001	.00544	.02723
		Subgroup E	-.005933	.003890	.550	-.01683	.00496
	Subgroup E	Subgroup A	.025133*	.003890	.000	.01424	.03603
		Subgroup B	.022133*	.003890	.000	.01124	.03303
		Subgroup C	.022267*	.003890	.000	.01137	.03316
		Subgroup D	.005933	.003890	.550	-.00496	.01683

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

Table 5
TUKEY POST HOC STATISTICAL TEST RESULT FOR GRADIA DIRECT LOFLO

Multiple Comparisons

Dependent Variable: micro esthetic flow viscous

	(I) category	(J) category	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Tukey HSD	Subgroup A	Subgroup B	-.014000*	.003763	.004	-.02454	-.00346
		Subgroup C	-.013867*	.003763	.004	-.02440	-.00333
		Subgroup D	-.027867*	.003763	.000	-.03840	-.01733
		Subgroup E	-.033467*	.003763	.000	-.04400	-.02293
	Subgroup B	Subgroup A	.014000*	.003763	.004	.00346	.02454
		Subgroup C	.000133	.003763	1.000	-.01040	.01067
		Subgroup D	-.013867*	.003763	.004	-.02440	-.00333
		Subgroup E	-.019467*	.003763	.000	-.03000	-.00893
	Subgroup C	Subgroup A	.013867*	.003763	.004	.00333	.02440
		Subgroup B	-.000133	.003763	1.000	-.01067	.01040
		Subgroup D	-.014000*	.003763	.004	-.02454	-.00346
		Subgroup E	-.019600*	.003763	.000	-.03014	-.00906
	Subgroup D	Subgroup A	.027867*	.003763	.000	.01733	.03840
		Subgroup B	.013867*	.003763	.004	.00333	.02440
		Subgroup C	.014000*	.003763	.004	.00346	.02454
		Subgroup E	-.005600	.003763	.574	-.01614	.00494
	Subgroup E	Subgroup A	.033467*	.003763	.000	.02293	.04400
		Subgroup B	.019467*	.003763	.000	.00893	.03000
		Subgroup C	.019600*	.003763	.000	.00906	.03014
		Subgroup D	.005600	.003763	.574	-.00494	.01614

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

Table 6
TUKEY POST HOC STATISTICAL TEST RESULT FOR MICRO ESTHETIC FLOW-VISCOUS

These observations confirm that the surface roughness of the studied materials increased with the increase of the number of immersions, the highest mean value of the Ra parameter being detected after 7 immersions/day.

This could lead to the idea that the intake of carbonated beverages, even in small quantities but with increased frequency during the day and for several consecutive days, alters the surface condition of composite resins. Our results are in agreement with other studies which showed that the exposure to acidic drinks and food intake induces the chemical dissolution of restorative materials, thus increasing the roughness of the superficial layer. Some studies have shown that a residual surface roughness may induce bacterial plaque retention [19-21]. *p*H of Coca-Cola is very low. More than that, this soft drink contains phosphoric acid which is an inorganic and strong acid. This association of a low *p*H and a strong inorganic acid could have caused a very aggressive attack on the surface of flowable composite, which led to an increase in the surface roughness. The roughness parameter Ra represents the mean arithmetic deviation of the assessed profile. The results showed that the Ra values, of both composite resins immersed in Cola-Cola drink, increased as the number of immersions increased. The higher the number of immersions the higher was the impact on the restorative materials. Our findings are in agreement with the results of other studies [22, 23].

Composites containing small filler particles are more homogeneous and their particles are less prominent on the surface, thus resulting a lower surface roughness. The filler type, size and quantity of the particles strongly influence the properties and quality of composite resins. The particles that are stripped out from the surface are very small, leaving small holes which produce an obvious increase in roughness. These findings are similar with that ones from other studies [24, 25]. Some authors suggested that relatively higher filler loading increases the stability of composite resin surface against low *p*H conditions [26-28]. However, still a not significantly increase in surface roughness of Micro Esthetic flow viscous samples when comparing to Gradia Direct LoFlo was noticed.

These changes may be due to water sorption by composite resin under acidic conditions leading to an increase in roughness, as it is composed mainly of monomers that are more susceptible to hydrolysis, like dimethacrylates [29-31]. The chemistry and the structure of polymer matrix are the most important factors influencing sorption and solubility of dental composites. The differences in water absorption of polymer network depend on monomer type (TEGDMA > Bis-GMA > UDMA > Bis-EMA) [32]. Both composites materials used in the present study contain UDMA (table 1), which is one of the most hydrophilic monomers. The organic matrix of resin composites is known to absorb a small percentage of water from the oral environment, which may alter some physical properties. Surface roughness and microhardness of dental composites has been reported to be significantly affected by water sorption and the contact time with the aqueous medium [33]. It is indeed difficult to isolate restorative materials so they can overcome all external challenges and successfully mentain their physical, chemical and mechanical properties.

The containers with Coca-Cola, in which the samples of the two materials were immersed, have been continuously stirred, to reproduce the bubbling conditions from the oral cavity. One can assume that micrometric losses occur in the superficial layer, the material exposing a new surface to the acid attack, and thus corrosion is cyclically repeated. This was also observed in other studies [22, 34]. The nature of the degradation suffered by dental materials and dental hard tissue subsequent to an erosive and cariogenic challenges is very complex. It was observed that surface roughness assessment is an appropriate method to verify small alterations in the superficial layer of composite resins after acid demineralization [35-38].

Even if the new generation of restorative materials serve to fulfill manly the esthetic demands, the effect of frequently consumed carbonated beverages on their stability and longevity needs further research. This in-vitro study thus might recommend that, in terms of resistance to degradation, flowable composite could be the material of choice for restoring teeth affected by erosion. However,

the degradation of materials is not the only factor involved in making this choice. An appropriate clinical case selection should be taken into consideration. Within the limitations of this study it can be concluded that repeated and long-term exposure to acidic beverages potentially affects the surface microstructure of esthetic dental restorative materials.

Conclusions

The surface condition of the samples from the two evaluated composite materials was affected after their immersion in the acidic medium represented by Coca-Cola drink, compared to the control subgroup. The results of the present study showed that the surface roughness values, of both restorative materials immersed in Coca-Cola, increased directly in proportion to the number of immersions, the highest value being determined for samples submerged in acidic drink 7 times a day. A greater number of immersions in the Cola drink (Coca-Cola) resulted in a higher impact on the restorative materials.

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