



Surface Texture and Optical Properties of Self-Adhering Composite Materials after Toothbrush Abrasion

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ABSTRACT

This study evaluated the surface texture and optical properties of two self-adhering composites and a nanofilled composite before and after toothbrush abrasion. Vertise Flow (Kerr), Fusio Liquid (Pentron Clinical) and Filtek Z350 XT (3M ESPE) composites were divided into 3 groups ($n = 6$). Disks of 12 mm diameter and 2 mm thick were made. All materials were light-cured with a LED light source for 40 seconds. Analyses of surface roughness, gloss retention and color stability were performed 24 hours after light curing and after 20,000 brushing cycles. Data were analyzed with analysis of variance (ANOVA) and Tukey's test ($\alpha = 5\%$).

Results: Tukey's test ascertained that toothbrush abrasion resulted in rougher and matte surfaces for all composites tested. Filtek Z350 presented better gloss retention after abrasion. On surface roughness evaluation, there was no statistical difference between Fusio Liquid Dentin and Filtek Z350 resins ($p > 0.05$). Vertise Flow resin showed better color stability (ΔE), than the other two materials.

Conclusion: Nanofilled material presented better gloss retention but it did not produce the best results in aspects related to surface roughness and color stability compared to self-adhering composites.

Clinical significance: A simulation of degradation process by using toothbrush abrasion produced a rougher and matte

surface in all composites tested. The surface texture was not only related to filler's amount present in materials, but also with the organic matrix composition of them. The results suggested that the constant development of new materials, seeking for a technical simplification, seems an innovative attraction for dentist's clinical routine, even though larger studies are necessary to promote to everyone a better understanding and improvement of action and effectiveness of this new class of materials.

Keywords: Laboratory research optical properties, Self-adhesive composite, Self-adhering composites, Self-adhesive materials, Surface texture, Toothbrush abrasion.

How to cite this article: Malavasi CV, Macedo EM, da Costa Souza K, Rego GF, Schneider LFJ, Cavalcante LM. Surface Texture and Optical Properties of Self-Adhering Composite Materials after Toothbrush Abrasion. *J Contemp Dent Pract* 2015;16(10):775-782.

Source of support: FAPERJ—Jovem Cientista do Nosso Estado

Conflict of interest: None

INTRODUCTION

Since the development of bisphenol A-glycidyl methacrylate (Bis-GMA) monomer in 1956 by Bowen, dental composites have undergone diverse and progressive technological changes over the years. These changes focus on filler sizes and polymeric matrix material in order to develop systems with better mechanical and esthetic properties as well as low polymerization shrinkage.¹

In order to simplify and make restoration technique easier, a new category of material has been launched, the self-adhering composites. This new category of low-viscosity composites was developed to provide a reduced clinical time as well as a possible decrease in the sensitivity of the technique found in the conventional total-etching adhesive systems.²⁻⁴

The material's formulations were based on conventional methacrylate systems, however, they incorporated

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acidic monomers typically found in self-etching adhesives systems. Monomers, such as glycerolphosphate dimethacrylate (GPDM) and 4-methacryloxyethyl trimellitate (4-MET) are used and may be able to generate adhesion through mechanical and probably chemical interactions with tooth structure, resulting in an inter-penetrating network created between the polymerized monomers, collagen fibers and smear layer.^{5,6}

Low-viscosity composites were released in the market in the end of 90's decade. The flowability feature is attributed either to the reduction in the filler content, which reduces significantly the mechanical properties of the material and increases the polymerization shrinkage, or to adding other modifying agents, such as surfactants that improve flow and prevent a great reduction in the filler content.⁷ Mainly used as a liner or a repair material, the flowable composites can also be applicable as filling material in conservative classes I or V preparations.^{8,9}

The new developed self-adhering flowable composites may be considered as a sort of stepless system.^{2,3,10,11} The development of materials focusing on simplification of operative procedures seems to be the most logical next step for dental manufacturers.

Notwithstanding, mechanical properties of newly developed materials should be assessed initially or after any type of aging procedure. It is known that the surface roughness, gloss retention and color stability are among the most important characteristics of surface texture that influence the appearance of composite resins and clinical performance.^{1,12-15}

Color stability is vital for the longevity of direct restorations in esthetically important areas.¹⁶ *In vitro* studies,¹⁷⁻²⁰ showed that resin-based composites are susceptible to staining and discoloration.

Gloss originates from the geometrical distribution of the light reflected by the surface and is an attribute of visual appearance.²¹ Differences in gloss between a restoration and the surrounding tooth structure can be detected even when colors are matched. Color stability and gloss are also influenced by surface roughness.^{18,22,23}

In oral environment, resin surfaces are exposed to erosive substances present in food and beverage and to

the abrasive effects of toothpastes and toothbrushing.²⁴ Thus, it is possible that the synergistic effect of abrasion and erosion phenomena will roughen all resin materials over the time, regardless their type and filler particles.¹⁶

The durability and quality of material surface can also be related to ideal restorative polishing and finishing.²⁵ The inadequate finishing/polishing of resin composites leads to increased plaque retention, gingival inflammation, discoloration of restorations and also leads to patient discomfort.²⁴ Jones et al²⁶ reported that a surface roughness of 0.3 µm can be detected by the tip of the patient's tongue.

Proper contour, smoothness and high gloss can produce the appearance of natural tooth structure desired by patients.²⁷ Therefore, it is of paramount importance to obtain smooth and glossy surfaces. Variables, such as the type of monomer, concentration and type of filler particles, finishing/polishing system all influence the final surface polishing of resin composites.^{24,27,28} Based on the importance of esthetic evaluation and on the need of searching the behavior analysis of this new class of materials, the aim of this study was to evaluate the surface roughness, gloss retention and color stability of two self-adhering composites, and compare them with a conventional nanofilled composite before and after toothbrush abrasion.

The null hypotheses tested were that self-adhering composites would produce: (i) rougher surfaces, (ii) less gloss retention and (iii) less color stability after toothbrush abrasion than nanofilled.

MATERIALS AND METHODS

Two self-adhering commercial composites, Vertise Flow (VF—Kerr, Orange, CA, USA) and Fusio Liquid Dentin (FL—Pentron Clinical, Orange, CA, USA) and one nanofilled composite, Filtek Z350 XT (Z350—3M ESPE, St Paul, MN, USA) were evaluated in this study (Table 1).

Specimen Preparation

Six disk specimens (10 × 2 mm) were prepared from each product. The samples were irradiated for 40 seconds from

Table 1: Materials details and manufacturers specifications

| Category | Composites | Composition | Organic filler (weight/volume) |
|-------------------------|---------------------|---|--------------------------------|
| Self-adhering composite | Vertise Flow | GPDM, HEMA, Bis-GMA, pre-polymerized fillers, nanoparticles | 70% wt |
| | | ytterbium fluoride, 1 µm of barium glass filler, colloidal nanosilica | 48% vol |
| Self-adhering composite | Fusio Liquid Dentin | UDMA, TEGDMA, HEMA, 4-MET, nanoamorphous silica, silanized | 65% wt |
| | | barium glass, minor additives, curing unit system | 52% vol |
| Nanofilled composite | Filtek Z350 XT | Bis-GMA, UDMA, TEGDMA, Bis-EMA, 20 nm (0.02 microns) of | 78.5% wt |
| | | non-agglomerated silica fillers, 4 nm (0.04 microns) to 11 nm (0.011 microns) of non-agglomerated fillers zirconia and aggregated loads of zirconia/silica [20 nm (0.02 microns) of silica and 4 to 11 nm (from 0.04 to 0.011 microns) of Zirconia] | 63.3% vol |



each surface with a light curing unit LED model Radiical (SDI) operated in standard mode, emitting 1000 mW/cm² irradiance.

Surface Gloss

The surface gloss was measured with a glossmeter (Gloss meter triangle—Zehntner) which was calibrated against a standard black glass provided by the manufacturer. Measurements were performed at 60° light incidence and reflection angles relative to the vertical axis. The measuring window was 2 × 2 mm.

Color Stability

The color of all specimens was determined with a colorimeter (Minolta CM2600d, USA) according to the CIE-L*a*b* system. The readings were taken for each specimen while placed against a white-tile ceramic background. The colorimeter was set to make color measurements (L*, a* and b*) based on average daylight (D65) illumination. All measurements were repeated twice and means for the L*, a*, and b* values were automatically calculated by the machine. The color changes, ΔE*, were calculated from the single color values L*, a* and b*, according to the following formula:

$$\Delta E^* = [(L^*_1 - L^*_2)^2 + (a^*_1 - a^*_2)^2 + (b^*_1 - b^*_2)^2]^{1/2}$$

where (L₁*, a₁*, b₁*) were the values before toothbrush abrasion and (L₂*, a₂*, b₂*) after toothbrush abrasion.

Surface Roughness

Quantitative Analysis

Portable surface roughness measurement instruments (SJ-201—Mitutoyo, Japan) were used to measure the surface roughness. The diamond stylus had a radius of 5 μm, tip angle of 90° and was traversed at a constant speed of 1.00 mm/s across the surface with a force of 6 mN. Six line scans were performed per specimen surface, three in horizontal and three in perpendicular directions. The cut-off length was 0.25 mm and the measuring length 2 mm. The amplitude parameter Ra (the arithmetic mean of the absolute departures of the roughness profile from the mean line) was measured.

Qualitative Analysis

Three-dimensional (3D) analysis of one sample per group was carried out in all specimens before and after aging procedures with the use of a profilometer, Rank Taylor-Hobson model from Talysurf Series. A 0.10 mm roughness cut-off and a 0.50 mm waviness cut-off were used to scan the surface. Data provided by topography were obtained and analyzed by the software Talymap Universal, version 3.0 (Taylor Hobson).

Aging Procedure

A toothbrush simulating machine (MEV2—Odeme, Brazil) was used to abrade the samples surfaces. The toothbrush machine had six separate 'stations' and six separate toothbrush holders which were driven by a motor. Hence six specimens were concurrently and individually subjected to an equal amount of toothbrush/dentifrice abrasion during each testing period. A toothbrush (Dr Veit Soft, Regular, Dr, Veit Produtos, Rio de Janeiro, RJ) was fixed in the toothbrush holder so that all the bristles were in contact with the specimen. The testing machine was adjusted to apply 2.5 N vertical load on the specimen during horizontal movement throughout the test. A current dentifrice (Colgate Total, Colgate-Palmolive, Manchester, UK) was used to form the slurry (2:1, water: toothpaste) according to ISO/TS 1469-1. Each 'station' of the machine was filled with 12 gm of slurry. All specimens were brushed with inverse strokes 20,000 times, as measured with an incorporated meter. The toothbrush and the slurry were replaced for each specimen.

After abrasion, specimens were removed from the machine, rinsed with tap water, cleaned in the ultrasonic bath with distilled water for 5 minutes, and gently air dried. All measurements were then repeated.

Data were submitted to analysis of variance (ANOVA) with repeated measures on time followed by Tukey's test, at a 5% global level of significance.

RESULTS

Gloss Retention (GU)

Gloss values ranged between 42.8 and 75.9 GU before and between 5.6 and 35.1 GU after toothbrush abrasion (Table 2). For all materials a statistically significant reduction in gloss was observed after toothbrush abrasion. Vertise Flow and Fusio Liquid Dentin exhibited poor gloss retention after toothbrushing.

Color Stability

ΔE* values ranged between 0.7 and 2.0 (Table 3). Vertise Flow showed significantly greater color changes than Fusio Liquid Dentin and Filtek Z350. No statistically significant differences were detected in color stability to Filtek Z350 and Fusio Liquid Dentin.

Table 2: Gloss values (GU) before and after toothbrushing

| Composite | GU (standard deviation) | |
|---------------------|----------------------------|--------------------------|
| | Before toothbrushing | After toothbrushing |
| Vertise Flow | 75.9 (7.8) ^{Aa} | 5.6 (0.23) ^{Bb} |
| Fusio Liquid Dentin | 42.8 (13.8) ^{Ab} | 6.5 (0.30) ^{Bb} |
| Filtek Z350 XT | 74.5 (17.58) ^{Aa} | 35.1 (1.0) ^{Ba} |

* Uppercase horizontal and lowercase vertical (p ≥ 0.005)

Table 3: ΔE^* values

| Composite | ΔE^* (standard deviation) |
|---------------------|-----------------------------------|
| Vertise Flow | 0.7 (0.111) ^B |
| Fusio Liquid Dentin | 3.1 (0.659) ^A |
| Filtek Z350 XT | 2.0 (0.05) ^A |

* Uppercase vertically

Table 4: Surface roughness values (Ra parameter) before and after toothbrushing

| Composite | Ra (standard deviation) | |
|---------------------|----------------------------|----------------------------|
| | Before toothbrushing | After toothbrushing |
| Vertise Flow | 0.04 (0.012) ^{Ba} | 0.24 (0.088) ^{Aa} |
| Fusio Liquid Dentin | 0.03 (0.022) ^{Ba} | 0.15 (0.047) ^{Ab} |
| Filtek Z350 XT | 0.02 (0.011) ^{Ba} | 0.13 (0.061) ^{Ab} |

* Uppercase horizontal and lowercase vertical ($p \geq 0.005$)

Surface Roughness

After toothbrush abrasion rougher surfaces were observed in all tested materials. It is possible to see in Taylor Robson 3D analysis, the difference between the initial and final mass after toothbrush abrasion (Figs 1 to 3). In quantitative analyzes, Ra values ranged from 0.02 to 0.04 μm before and from 0.13 to 0.24 μm after toothbrush abrasion (Table 4).

Tukey’s post hoc test revealed no significant differences among materials before abrasion. However, statistically significant mean differences in Ra values after toothbrush abrasion were detected. These differences were more prominent for the self-adhering composite Vertise Flow in comparison to Fusio Liquid Dentin and Filtek Z350 XT.

DISCUSSION

Since the characteristics of durability and quality of material’s surface (roughness, gloss and color) might be related to abrasion resistance,^{15,18,22,29} *in vivo*, they are daily influenced by general factors, such as the pressure made by patient while brushing, the toothbrush bristle’s consistence, the brushing moment and the toothpaste abrasiveness.^{24,30,31}

In order to obtain a better understanding and analysis of the behavior of the materials tested in this study, all of them were submitted to the same abrasive conditions, which consisted in a 20.000 cycle brushing aging procedure. This, according to some authors, corresponds to an average 2 years period of brushing *in vivo*.^{32,33}

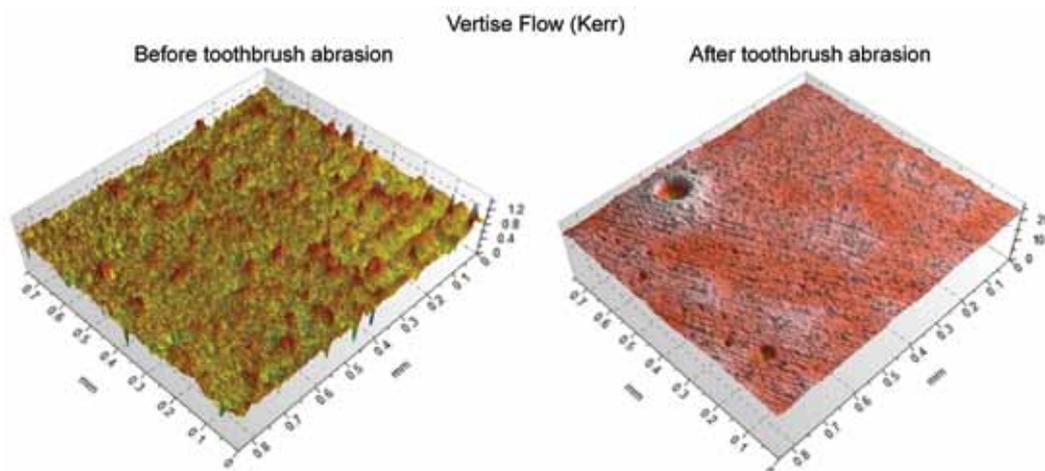


Fig. 1: Vertise Flow before and after toothbrush abrasion in 3D analysis

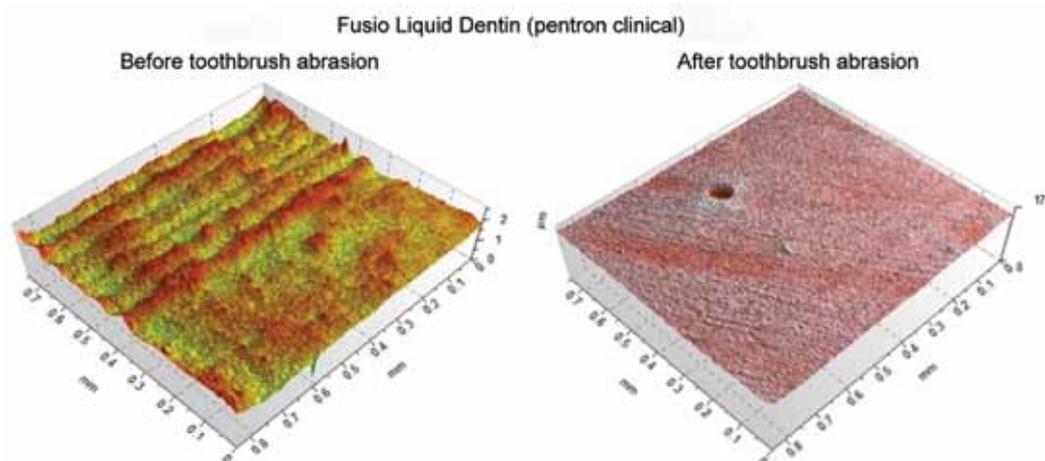


Fig. 2: Fusio Liquid Dentin before and after toothbrush abrasion in 3D analysis



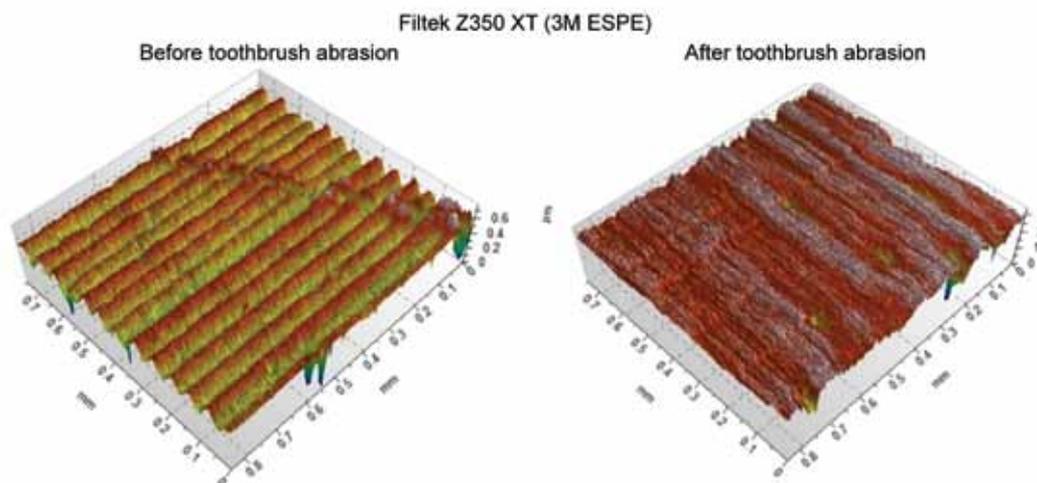


Fig. 3: Filtek Z350 XT before and after toothbrush abrasion in 3D analysis

Our results were, therefore, influenced only by specific factors related to the individual characteristics of each material, such as the inorganic composition, considering the influence of filler's morphology, size and content; organic matrix composition; quality of inorganic interface matrix/particle and the conversion degree of monomers into polymers during polymerization process.^{12,18,25,34-36}

The abrasion process usually occurs through the gradual removal of organic matrix, which ends up leaving the fillers without support and susceptible to exfoliation.³⁰ As a result, when particles of larger fillers are removed, a rougher surface is expected, hence materials with smaller fillers are more resistant to the abrasive process. This might be related to its inter-filler spacing, which grows as the filler's size grows.¹²

In this work, after the abrasive procedure, there was an increase in the superficial roughness values in all groups, which produced significant changes in the surface properties in all materials tested.

After the abrasive procedure, Vertise Flow resin had a rougher surface and showed a greater increase in Ra values (0.24 μm) compared to Fusio Liquid Dentin (0.15 μm) and Filtek Z350 (0.13 μm). This behavior might be justified, first of all, by the smaller volume of fillers (48% vol) found in this resin when compared to the other resins tested, Vertise Flow and Filtek Z350 (52 and 63.3% vol, respectively).

Secondly, we can justify this found by the organic matrix of the materials evaluated. According to some studies, a higher quantity of BisGMA offers less resistance to abrasion to the material,^{8,35} and the increase of monomers 1,6-bis (methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane (UDMA) and triethylene glycol dimethacrylate (TEGDMA) in the composition of these materials can promote more resistance to abrasion.^{35,37,38}

Therefore, the behavior of these monomers seems to follow the results obtained in this study, for Vertise

Flow contains Bis-GMA but does not contain UDMA and TEGDMA monomers, found in Fusion Liquid Dentin and Z350. Although there was a difference in the amount of filler particles (%vol), after the abrasive procedure there was no statistical difference in roughness values of Fusio Liquid Dentin and Filtek Z350.

When exposed to brushing, most nanohybrids and microhybrids compounds maintain superficial roughness lower than 0.2 μm , which, according to some studies, might be considered a threshold for plaque retention.^{14,22,24} Moreover, it has already been reported that a superficial roughness rate between 0.25 and 0.5 μm can be detected by the patient's tongue.^{26,30} Among the results obtained in this study, only Vertise Flow resin presented inferior results, with a roughness value on the sideline of this threshold for plaque retention.

Previous studies have already revealed a strong correlation between surface roughness and gloss.^{24,29} Since gloss is a visual feature produced by specular and selective reflection of incident light on a material's surface, it is influenced by the material's refraction index and by the angle of light incidence.^{39,40} The lighting angle affects the quantity of light reflected by the material.

According to Silkas et al¹³ in the 60° incident angle, light is considered more trustful to a clinical point of view, for it is closer to the observer's perception of the teeth's color. Higher gloss values indicate smoother surfaces.¹⁴

According to some authors,^{14,41} by increasing the roughness it is possible to increase the reflex of random light, decreasing the gloss, which indicates an inverse relation between roughness and gloss in compound resin fillings. Even though, a study done by Takanashi³⁰ did not demonstrate an association between these two factors. According to the author, gloss is also influenced by other additional factors besides surface roughness, such as the differences of refraction indexes of resin's matrix, the differences of efficiency of matrix/filler interface bond,²⁵

and the difference of intrinsic factors of the material itself (type, shape, size, hardness and filler distribution).^{12,30}

In all materials tested in this study, surface became statistically less glossy after the abrasive procedure. A reduction in gloss retention of 92% was observed for Vertise Flow resin, 84% for Fusio Liquid Dentin and 53% for Filtek Z350. Although Fusio Liquid Dentin presented results for roughness statistically similar to Filtek Z350, in the gloss retention analysis the results were different.

Filtek Z350 resin presented higher values of gloss retention in GU, and statistically there was no difference between the final values obtained for self-adhering resins Fusio Liquid Dentin and Vertise Flow. The best performance of Filtek Z350 resin can be justified by the higher quantity of fillers (%vol) and also by the shape and size of its fillers. Small particles cause a decrease of diffuse reflex, resulting in a glossy outlook,²⁹ and the spherical shape of the particles, like the ones found in Filtek Z350, can offer a high light reflex, improving the gloss of these materials.²²

Although gloss is considered a more sensible characteristic for surface quality evaluation after brushing,¹ in this study no direct relation between surface roughness and gloss of these materials was observed. This seems to agree with Takanashi's³⁰ study.

Concerning color stability, in this study the evaluation was made by comparing each material's ΔE . It has been demonstrated in literature that ΔE values above one are perceptible by the naked eye,⁴² and ΔE values similar or superior to 3,3 are considered clinically unacceptable.^{43,44}

After the abrasive procedure performed in this study, Vertise Flow resin presented a smaller variation of ΔE (0.7). Superior results were found in resins Fusio Liquid Dentin (ΔE 3.1) and Filtek Z350 (ΔE 2.0), without statistical difference between the last two ones.

It is known that color variation may be caused by material's intrinsic and extrinsic factors.^{16,17,45,46} Since in this study, the specimens were not exposed to any coloring agent, the changes in optic properties resulted only from physic-chemical reaction occurred internally in the materials tested.

It has been reported in literature that the conversion degree of each material may be strongly related to the optic changes of themselves,^{18,47} once the presence of double carbon bonds that did not reacted during to polymerization process, it makes the resins more susceptible to degradation, which may create a variation of refraction index of these materials. This results in opacity changes, promoting color alteration and influencing the ΔE values over the time.^{38,48}

A study done by Eliades⁴⁹ analyzed some features concerning the resins Vertise Flow and Fusio Liquid Dentin, and compared them to other fluid resins. The

observed result was that the conversion degree of self-adhering resins was extremely high compared to others, 76.1% (VF) and 59.2% (FS). Possible explanations to the high conversion rate are related to the small and flexible monomers employed in the composition of these materials; the presence of HEMA, which, for being very reactive quickly interacts with residual C = C bonds; the effects of molecular orientation of hydrophilic monomers and acid monomers, which bring C = C bonds to a position that promotes the conversions,⁵⁰ and finally, the higher quantity of catalysts and co-catalysts used in photopolymerizable systems with acidic monomers.⁵¹

The high value observed for conversion degree of self-adhering resin Vertise Flow may justify the superior color stability found in this study. Besides that, the interior color stability found in Fusio Liquid Dentin and Z350, may be also justified by the presence of TEGDMA monomer in the organic matrix of such materials. Due to the presence of three etoxi groups in its main chain, this monomer has a higher propensity to water absorption.^{16,38,48}

CONCLUSION

The surface texture not only related to filler's amount present in the materials, but also with the organic matrix composition.

Nanofilled material presented better gloss retention but it did not produce the best results in aspects related to surface roughness and color stability compared to self-adhering composites.

CLINICAL SIGNIFICANCE

A simulation of degradation process by using toothbrush abrasion produced a rougher and matte surface in all composites tested. The results suggested that the constant development of new materials, seeking for a technical simplification, seems an innovative attraction for dentist's clinical routine, even though larger studies are necessary to promote to everyone a better understanding and improvement of action and effectiveness of this new class of materials.

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