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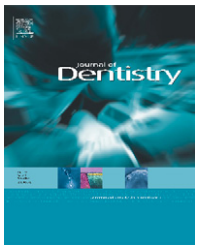

Surface roughness of etched composite resin in light of composite repair

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ABSTRACT

Objectives: In search for clinically effective composite repair protocols, the effect of various etching protocols on the surface roughness of composite resins with different filler composition were investigated.

Methods: Of two composite resins (hybrid-filled Clearfil AP-X; nano-filled Filtek Supreme XT) specimens of 3 mm thick with a diameter of 7 mm were prepared ($n = 24$). The top surface was polished with 4000-grit SiC-abrasive paper and subjected to one of eight surface treatments: ($n = 3$): negative control (NC), 37% phosphoric acid for 20 s (37PA-20 s), 3% hydrofluoric acid for 20 s (3HF-20 s), for 120 s (3HF-120 s), 9.6% hydrofluoric acid for 20 s (9.6HF-20 s), for 120 s (9.6%HF-120 s), 37PA-20 s followed by 9.6%HF for 120 s (37PA-20 s/9.6HF-120 s) and 9.6%HF for 120 s followed by 37PA-20 s (9.6HF-120 s/37PA-20 s). Roughness (S_a) was measured using a 3D noncontact optical interferometer (WYKO) and surface topography imaged by SEM. Multilevel models were used to estimate the variances within a sample and between samples in each group. Using the resulting overall variances and the means for each group, the eight groups were compared consecutively using *t*-tests ($p < 0.05$).

Results: The hybrid-filled composite resin demonstrated a significantly rougher surface than the nano-filled ($p < 0.05$). For both composites 9.6%HF-120 s, 37PA-20 s/9.6HF-120 s and 9.6%HF-120 s/37PA-20 s resulted in a large increase in roughness compared to the other groups ($p < 0.05$). For the hybrid-filled, the succeeding groups (37PA-20 s, 3HF-20 s, 3HF-120 s and 9.6HF-20 s) resulted in a statistically significant increase in surface roughness ($p < 0.02$). For the nano-filled only a statistically significant increase in roughness was found between 3HF-20 s and 3HF-120 s ($p < 0.001$) and between 9.6HF-20 s and 9.6HF-120 s ($p < 0.001$). SEM surface characterization revealed that the hybrid-filled composite resin was much more affected by etching than the nano-filled.

Significance: Composite resins should not be seen as a group of materials having identical properties when it comes to repair. The effect of etching will depend on the composition of the filler particles.

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1. Introduction

Composite restorations that are placed in anterior and posterior teeth may be prone to ageing or failure on the long term, resulting in a need for either refurbishment, repair or replacement.[1] In case of restoration repair, mostly a part of the old restoration is removed and the surface of the existing restoration acts as the substrate for bonding, as for example a fractured restoration needs to be repaired.

A successful repair requires an adequate bond between the existing restoration and restorative material, which is applied to repair the defect. Composite is most frequently used as the repairing material, thanks to its capacity to bond to enamel and dentine. Retention to the existing composite restoration can be obtained macro-mechanically by roughening with a bur and micro-mechanically by either etching with hydrofluoric acid (HF) or sandblasting. Moreover, the application of a silane coupling agent will enhance the repair bond strength.[2–7]

Sandblasting and etching with hydrofluoric acid are reliable methods to bond composite to porcelain.[8–13] However, studies on the repair strength of composite to composite show considerable differences and contradictions in their outcomes. Several studies failed to show a positive effect of hydrofluoric acid, sandblasting or roughening with a bur on the repair strength of composite,[14–15] where other studies demonstrated a beneficial effect when using such composite repair techniques.[16–22]

The variation in results may reflect the differences in composition of the composite, tested in these studies, as repair strengths may be influenced by the type and amount of filler present in the composite. The effect of different etching acids, applied with various etching times and concentration on the surface roughness of different types of composites is unknown. In order to understand repair mechanisms when adding new composite on to aged composite, knowledge of the effect of the various etching procedures on different types of composite is needed. Therefore, the aim of this study was to investigate the effect of various etching protocols on the surface roughness of composites with different filler composition.

2. Materials and methods

2.1. Specimen preparation

Two composites were selected for this study, in which the filler-type, consistency and composition were different, but with a resin matrix mainly consisting of BIS-GMA and TEGDMA. A highly filled hybrid-filled composite (Clearfil AP-X, Kuraray Co.) containing barium glass and colloidal silica fillers with a mean particle size of 3 μm and a particle distribution between 0.1 μm and 15 μm. The filler percentage is 70% in volume and 85.5% in weight. Secondly, a nano-filled composite (Filtek Supreme XT, 3 M ESPE) containing a combination of a non-agglomerated/non-aggregated 20 nm nano-silica filler and loosely bound agglomerated zirconia/silica nano-clusters, consisting of agglomerates of primary zirconia/silica particles with size of 5–20 nm fillers. The cluster particle size range is 0.6–1.4 μm. The filler percentage is 57.5% in volume and 72.5% in weight.

From each composite, two composite cylinders of 40 mm long and with a diameter of 7 mm were prepared by filling transparent plastic cylinders in 15 layers of 2.5 mm thick composite. Each layer was separately polymerized for 20 s. Finally, the bars were post-cured for 120 s, equally divided over the total length of the specimen and from all sides.

To obtain 24 identical specimens (3 mm thick with a diameter of 7 mm) per composite, the cylindrical composite bars were sectioned perpendicular to the long-axis with a diamond saw at slow speed (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) under continuous water cooling.

The top surface of the specimens was polished with a 4000-grit wet silicon carbide abrasive paper, under running water as a lubricant. Subsequently, specimens were ultrasonically cleaned for 15 min and finally dry stored at room temperature for 1 week. Specimens of each composite were randomly subjected to one of eight surface treatment techniques ($n = 3$ per group).

Group 1. No surface treatment (NC).
Group 2. Etching with 37% phosphoric acid (DMG) for 20 s (37PA-20 s).
Group 3. Etching with 3% hydrofluoric acid (Porcelock, Den-Mat) for 20 s (3HF-20 s).
Group 4. Etching with 3% hydrofluoric acid (Porcelock Den-Mat) for 120 s (3HF-120 s).
Group 5. Etching with 9.6% hydrofluoric acid (Porcelain etch, Pulpdent) for 20 s (9.6HF-20 s).
Group 6. Etching with 9.6% hydrofluoric acid (Porcelain etch, Pulpdent) for 120 s (9.6HF-120 s).
Group 7. Etching with 37% phosphoric acid for 20 s followed by etching with 9.6% hydrofluoric acid for 120 s (37PA-20 s/9.6HF-120 s).
Group 8. Etching with 9.6% hydrofluoric acid for 120 s followed by etching with 37% phosphoric acid for 20 s (9.6HF-120 s/37PA-20 s).

After each surface treatment, specimens were thoroughly rinsed with distilled water, air-dried and stored under dry conditions for 1 week.

Table 1 summarizes the product profiles, material properties and LOT-numbers of all the materials.

2.2. Roughness measurements

The surface roughness of the specimens was measured using a white-light 3D noncontact optical interferometer (WYKO, Veeco, Plainview, NY, USA). Roughness measurements of the surface were performed in vertical scanning interferometry (VSI), full resolution, back-scan of 5 μm, scan lengths of 15 μm and at a modulation threshold level of 1% with an objective of 10× with fields of view (FOV) of 1.0×. The surface roughness of each specimen was calculated using WYKO VISION 32-software (Veeco, Plainview, NY, USA).

On each specimen, scans were made at four randomly selected locations. The surfaces were studied at a magnification of 10.38× and an image size of 736 μm × 480 μm. Surface textures were flattened using the 'cylinder and tilt removal' filter function to minimize artefacts, due to the somewhat cylindrical curvature of the specimens. The actual surface

Table 1 – Product profiles, material properties and LOT-numbers of all materials used in the study.

Materials	Manufacturers	Composition	LOT
Clearfil AP-X	Kuraray Medical, Osaka, Japan	70 vol%, 85.5 wt% barium glass and colloidal silica filler; BIS-GMA, TEGDMA	Shade A2: 0257A, Shade A4: 449AA
Filtek Supreme XT	3M ESPE Dental products, Seefeld, Germany	59.5 vol%, 78.5 wt% aggregated zirconia/silica cluster filler and non-agglomerated/ non-aggregated silica filler; BIS-GMA, BIS-EMA, UDMA with small amounts of TEGDMA	Shade A2B: 20070926, Shade A4B: 20071129
Etching gel (medium viscosity)	DMG, Hamburg, Germany	37% phosphoric acid	591995
Porcelock Porcelain Etching Solution	DenMat, Santa Maria, CA, USA	3% hydrofluoric acid	HO95010055
Porcelain Etch Gel HF	Pulpdent Co., Watertown, USA	9.6% hydrofluoric acid	IA061130

roughness was calculated from each measurement and data were expressed as S_a (arithmetic average of the 3D roughness surface profile).

2.3. *Feg-SEM evaluation*

After the initial analysis with the stereomicroscope, specimens were processed for field-emission-gun scanning electron microscopy (Feg-SEM; Philips XL30, Eindhoven, The Netherlands) to get a more detailed view of the surface in each group. One specimen of each group was selected according to the most representative mean roughness value in each group. Accepted procedures for SEM specimen preparation were employed, as previously described by Perdigão et al.[23]

2.4. *Statistics*

One-way analysis of variance (ANOVA) and post hoc Tukey HSD multiple comparisons were used to determine statistical differences in roughness between groups ($p < 0.05$). Mann–Whitney ($p < 0.05$) tests were applied in those situations when data were not normally distributed.

3. Results

The etching protocol resulted in very large differences in roughness (S_a) on both composites when comparing groups 1–5 (NC, 37PA-20 s, 3HF-20 s, 3HF-120 s and 9.6HF-20 s) with groups 6–8 (9.6HF-120 s, 37PA-20 s/9.6HF-120 s and 9.6HF-120 s/37PA-20 s). Moreover, as the standard deviations were not identical for these two subgroups, differences between groups could not be tested using parametric tests. A non-parametric Mann–Whitney test showed a statistical significant difference between groups 1–5 and 6–8 ($p < 0.0001$). Thereafter, the two subgroups were separately analysed using one-way ANOVA and post hoc Tukey HSD.

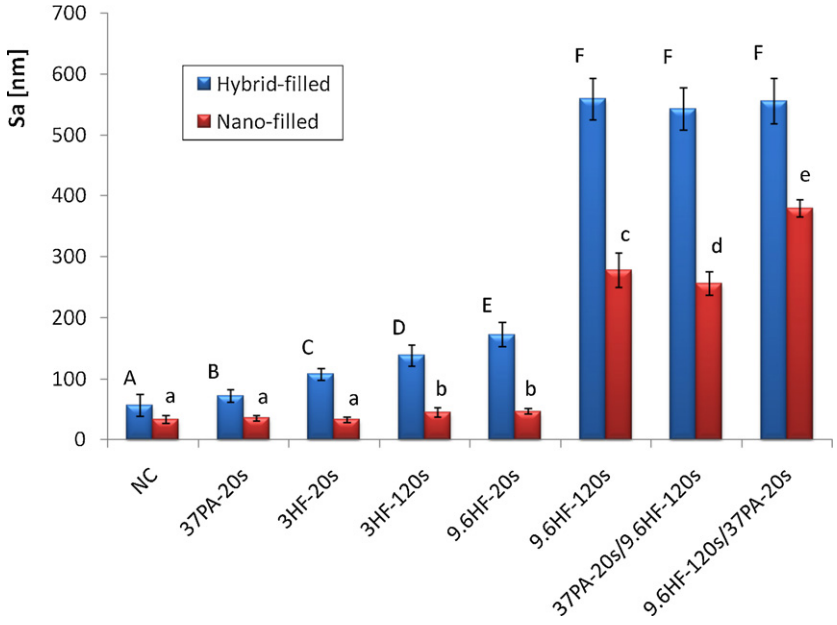

Fig. 1 – Roughness measurement (S_a) of the hybrid-filled and nano-filled composite. Differences in letters indicate statistically significant differences (ANOVA: $p < 0.05$).

Table 2 – Roughness measurements of the hybrid-filled (Clearfil AP-X) and nano-filled composite resin (Filtek Supreme XT).

Protocol		S_a [nm] (SD)	
		Hybrid-filled	Nano-filled
1	NC	55.8 (18.0)	33.3 (6.6)
2	37PA-20s	71.7 (10.0)	35.3 (4.9)
3	3HF-20s	107.2 (9.2)	32.5 (4,0)
4	3HF-120s	138.2 (17.2)	44.4 (7.5)
5	9.6HF-20s	172.6 (19.5)	46.5 (4.0)
6	9.6HF-120s	559.0 (33.8)	277.3 (28.1)
7	37PA-20s/9.6HF-120s	542.3 (35.0)	255.9 (19.5)
8	9.6HF-120s/37PA-20s	555.7 (37.5)	378.7 (14.2)

3.1. *Roughness measurements (S_a)*

In Fig. 1 and Table 2 the results of the roughness measurements are presented, combined with an indication of statistical differences between groups. For all groups, even the control group, the hybrid-filled composite demonstrated a statistically significant rougher surface than the nano-filled composite ($p < 0.05$). For the hybrid-filled composite, etching with 3% hydrofluoric acid (3HF-20 s: 107.2 ± 9.2 nm; 3HF-120 s: 138.2 ± 16.5 nm) and 9.6% hydrofluoric acid for 20 s (9.6HF-20 s: 172.6 ± 19.9 nm) resulted in a statistically significant increase in surface roughness compared to the control group (NC: 55.8 ± 15.6 nm) ($p < 0.001$). For the nano-filled composite only 3% hydrofluoric acid for 120 s (3HF-120 s: 44.2 ± 6.8 nm) and 9.6% hydrofluoric acid for 20 s (9.6HF-20 s: 46.3 ± 3.9 nm), showed a statistically significant increase in roughness compared to the control group (NC: 33.6 ± 6.3 nm) ($p < 0.02$).

Etching with 9.6% hydrofluoric acid for 120 s (groups 6–8) resulted in a much rougher surface for both composites. For the hybrid-filled composite no statistically significant differences in roughness were found between the three groups, whereas for the nano-filled composite statistically significant differences were found ($p < 0.03$).

3.2. *Feg-SEM-evaluation*

In the control group of the hybrid-filled composite group, a polished composite surface can be seen, composed of small and large glass filler particles embedded in the resin matrix (Fig. 2a). After etching with 37% phosphoric acid for 20 s no changes are visible (Fig. 2b). Subjecting this surface to 3% hydrofluoric acid for 20 s an irregular dissolution of the glass fillers was noticed, predominantly visible on the filler surface and along the filler-resin matrix interface (Fig. 2c). After etching for 120 s a more profound etching pattern was found (Fig. 2d). A more aggressive etching pattern was found when etching with 9.6% hydrofluoric acid for 20 s (Fig. 2e). Etching with 6% hydrofluoric acid for 120 s alone or in combination with 37% phosphoric acid, similar etching patterns were found. In these three groups the majority of superficially glass fillers was totally removed (Fig. 2f–h), leaving a highly porous irregular matrix structure, which itself was not visibly affected.

Images of the nano-filled composite show a different appearance than the hybrid-filled material. A polished surface can be seen, composed of nano-clusters of different sizes, embedded in the resin matrix (Fig. 3a). Etching with 37% phosphoric acid for 20 s (Fig. 3b), 3% hydrofluoric acid for 20 s (Fig. 3c) as well as etching with 3% hydrofluoric acid for 120 s

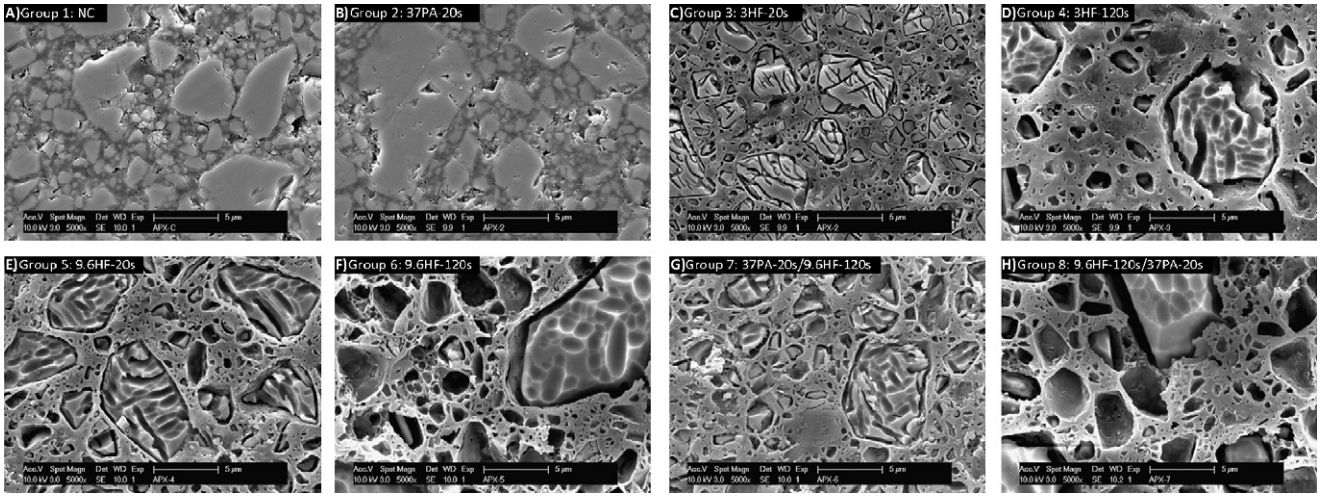

Fig. 2 – Representative SEM images of the hybrid-filled composite (Clearfil AP-X), when exposed to the different etching protocols: (A) a polished hybrid-filled composite surface, composed of small and large glass filler particles embedded in the resin matrix; (B) etching with 37% phosphoric acid for 20 s had no visual effect on the composite surface; (C) after etching with 3% hydrofluoric acid for 20 s an irregular dissolution of the glass fillers was noticed, predominantly visible on the filler surface and along the filler-resin matrix interface; (D) etching with 3% hydrofluoric acid for 120 s a more profound etching pattern was found; (E) a more aggressive etching pattern was found when etching with 9.6% hydrofluoric acid for 20 s; (F–H) etching with 6% hydrofluoric acid for 120 s alone or in combination with 37% phosphoric acid, similar etching patterns were found. In these three groups the majority of superficially glass fillers was totally removed, leaving a highly porous irregular matrix structure, which itself was not visibly affected.

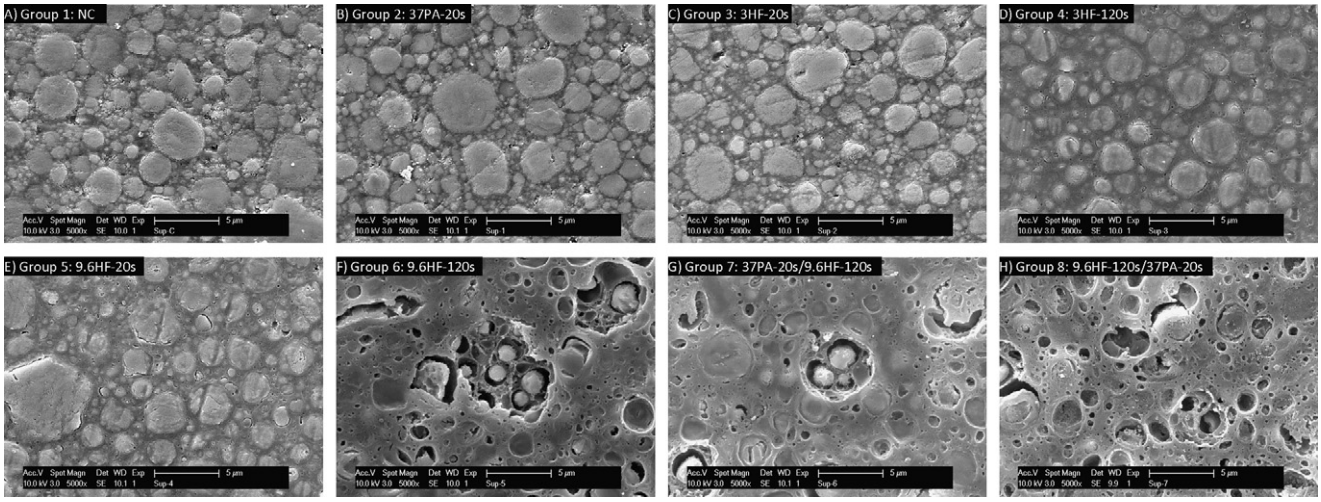

Fig. 3 – Representative SEM images of the nano-filled composite (Filtek Supreme XT), when exposed to the different etching protocols: (A) a polished nano-filled composite surface, composed of aggregated/agglomerated zirconia–silica clusters embedded in the resin matrix; (B–D) after etching with 37% phosphoric acid for 20 s, or with 3% hydrofluoric acid for 20 s and 120 s no visual effect on the composite surface was seen; (E) etching with 9.6% hydrofluoric acid for 20 s resulted in a superficial etching pattern becoming visible mainly around the nano-clusters; (F) when etching with 9.6% hydrofluoric acid for 120 s alone or in combination with 37% phosphoric acid it was found that nano-clusters were disintegrated and were removed, resulting in a porous structure mainly composed of resin matrix.

(Fig. 3d) hardly showed any effect. Etching with 9.6% hydrofluoric acid for 20 s resulted in a superficial etching pattern becoming visible mainly around the nano-clusters (Fig. 3e). However, when etching with 9.6% hydrofluoric acid for 120 s alone or in combination with 37% phosphoric acid it was found that nano-clusters were disintegrated and were removed, resulting in a porous structure mainly composed of resin matrix (Fig. 3f–h).

4. Discussion

When measuring surface roughness, normally the R_a value is presented.[21,22,24,25] This is the roughness parameter which is calculated from a 2D profile. For these techniques usually a probe is used to trace a relatively flat surface along a straight line (i.e. profilometer). When roughness is measured in 3D, the roughness value can be presented as the S_a (arithmetic average of the 3D roughness profile). Besides the S_a value, some other parameters can be measured, such as the S_q (root-mean-square roughness), S_z (average maximum profile of the ten greatest peak-to-valley separations in the evaluated area), S_{sk} (measure of the asymmetry of the profile about the mean plane) and the S_{ku} (measure of the distribution of spikes above and below the mean line). For this study, only the S_a has been used, as this parameter presents the main outcome to determine relevant differences of roughness values between the etching techniques, whilst the other variables (S_q, S_z, S_{sk} and S_{ku}) only present additional information about the specific roughness profile.[26,27]

The results of this study showed that composites can respond differently on certain etching procedures. As expected, besides the cleaning effect of the surface, phosphoric acid alone had no effect on surface roughness, whilst hydrofluoric acid with a higher concentration and a longer etching time dissolved glass filler particles in both composites, resulting in an increased surface roughness. Nevertheless, surface changes were more apparent on the hybrid-filled than on the nano-filled composite, which might be explained by the difference in composition of the filler particles. The filler of the hybrid-filled composite Clearfil AP-X is a barium-glass, whilst the nano-fillers of Filtek Supreme XT are composed of zirconia and silica. The zirconia part of the nano-filler in Supreme XT resisted the etching by hydrofluoric acid much better than the barium-glass filler. However, the zirconia nano-filler part is surrounded by a silica phase and once this phase is dissolved by etching the zirconia filler part will be dislodged from the resin matrix (Fig. 3f–h).

The less aggressive etching procedures (3HF-20 s, 3HF-120 s or 9.6HF-20 s), resulted in a relatively small increase of roughness, where the increase in roughness was statistically significantly larger for the hybrid-filled composite than for the nano-filled composite. However, longer etching with 9.6% hydrofluoric acid significantly increased roughness on both composites. Amongst these more aggressive procedures (groups 6–8), no differences were found in roughness for the hybrid-filled composite, whereas statistically significant differences were found for the nano-filled composite. When the nano-filled composite was etched for 120 s with 9.6% hydrofluoric acid and followed by etching with phosphoric acid a significantly higher surface roughness was found compared to etching with hydrofluoric acid alone. This effect could not be demonstrated for the hybrid-filled composite. The SEM images showed that in the hybrid-filled composite the less aggressive etching procedures already resulted in gross dissolution of the filler particles, whilst in the more aggressive groups almost no glass particles were left on the surface. Additional etching with phosphoric acid did not result in a significant increase in

roughness, as almost all glass fillers were already removed by hydrofluoric acid alone. For the nano-filled composite, the less aggressive procedures did not show much effect on the surface roughness. An explanation might be that etching for 120 s with 9.6% hydrofluoric acid (group 6) dissolved the silica phase of the zirconia particles, that were, however, not yet removed by rinsing. Possibly, the dissolution products hold some of the particles in place. The additional etching with phosphoric acid could have removed the previously loosened zirconia particles from the resin matrix.

The finding that the obtained surface roughness after etching with hydrofluoric acid largely depends on the composition of the composite, might also explain the differences found in literature regarding the effect of etching with hydrofluoric acid on the repair bond strength. For example, in studies were zirconia/silica clusters were used, etching with hydrofluoric acid failed to show a positive effect on micro-tensile bond strength,[21] whereas in a study were barium–glass containing composites were used, a positive effect on micro-tensile bond strength was found.[17] Unfortunately, no studies were found in which a direct comparison was made between a hybrid-filled composite, composed of barium–glass filler particles, and a nano-filled, composite composed of zirconia/silica nano-clusters.

In most repair procedures the production of a micro-mechanical surface is followed by silanization in order to establish a chemical bond between the glass particles and the fresh resin.[7,19,28,29] When silane agents are applied, covalent bond develops between the monomers in the adhesive system and the inorganic filler particles in the composite. Moreover, the silane also increases the surface wettability of the adhesive system, facilitating a better infiltration of the adhesive resin into the irregularities. However, the prerequisite to obtain this chemical bond is the presence of glass filler particles on the surface of the composite. When due to aggressive etching with hydrofluoric acid the glass fillers are dissolved or completely removed, the benefit of silanization is questionable. Moreover, when after etching the surface is composed of resin only, this structure may be too fragile to give adequate support to the new repair material. Probably, there should be an equilibrium between a certain amount of surface roughness and the amount of fillers present at the surface, and still tightly connected to the resin matrix. However, this study showed that different composites result in different surface reactions to the applied techniques, by which adhesive composite-repair protocols should be further investigated.

Furthermore, the actual clinical relevance of the increased roughness after acid etching and the eventual repair bond is still unknown. It is questionable if a uniform repair protocol for composites can be made considering the different manner the two composites investigated in this study reacted. Moreover, it may well be that not the most rough materials, but those materials with the best balance between surface roughness and remaining filler particles enable the best repair bond strength.

5. Conclusions

Based on the results of this study, it can be concluded that composites should not be seen as a group of material having identical properties when it comes to repair. The effect of etching will largely depend on the composition of the filler particles.

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