Influence of chemo-mechanical caries removal on the surface topography of dental composite resin and glass-ionomer materials: an in vitro study

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The aim of the present study was to investigate the influence of a chemo-mechanical caries removal system, Carisolv® gel, on the surface topography of dental filling materials. Thirty specimens of a composite resin (Spectrum™) and a compomer (Dyract® AP) and 60 specimens of a glass-ionomer (Ketac®-Fil Plus) were prepared. The surface topography was investigated with an optical interferometer before and after chemical exposure for 5, 10, or 20 min. Each specimen acted as its own control. The topographical part of the glass-ionomer materials was performed in two series with different drying procedures, since this material exhibits a higher sensitivity to dehydration than the other materials. The surface topographical investigations were complemented with contact angle measurements. After Carisolv® gel exposure the density of summits and the developed surface area ratio (3D/2D) were slightly smaller for the composite resin and the compomer. For the minimally dried glass-ionomer material, the results indicated a reduction of the height of the surface structures, as well as a surface area enlargement, after Carisolv® gel exposure. No statistically significant changes of contact angles due to Carisolv® gel exposure could be detected for any material investigated. If dental filling materials of composite resin or glass-ionomer materials are exposed to Carisolv® gel, no or only minor surface topographical changes can be expected. □ *Chemo-mechanical caries removal; dental materials; optical interferometry; surface topography*

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Minimally invasive caries removal techniques focus on keeping as much healthy tissue as possible. These techniques include laser ablation, sono-abrasion, and chemo-mechanical caries removal. The chemo-mechanical technique was introduced in the 1970s and different systems have been developed and tested since then (1–6). Carisolv® (MediTeam Dental AB, Sävedalen, Sweden) is a chemo-mechanical caries removal system that can be used for selective removal of carious dentine (7). It consists of an alkaline gel (pH = 11) with the chemically active components sodium hypochlorite (NaOCl) and amino acids. The gel is applied to the carious lesion, whereupon the tissue is softened and can be scraped off with hand instruments. The softening is assumed to be an effect of the gel's dissolution of hydrogen bonds in the carious tissue, thereby disrupting the collagen fibers (8). Some of the main indications of the method are root surface caries, deep carious lesions, and special dental care.

As a result of modern preparation techniques associated with common restorative materials, such as composite resins, compomers (i.e. polyacid-modified composite resins), and glass-ionomers, total removal of an old restoration may not always occur when treating secondary caries lesions. Carisolv® gel will therefore often come into direct contact with these materials, as well as in situations when caries is excavated from a tooth adjacent to a tooth with an earlier restoration. Several studies have reported that pH will influence degradation and erosion processes taking place at the surface or within dental materials (9–13). Most pH-related stability studies on composite resins, compomers, and glass-ionomers consider acidic solutions, although the sorption/solubility behavior of composite resins has been shown to be influenced by the alkaline pH of the solution (13). Furthermore, the effect of a solution similar to the Carisolv® gel has been reported to have a minor chemical influence on the topography of three different dental ceramics (9). However, no reports investigating the possible effect of the oxidation agent NaOCl on composite resin and glass-ionomer materials have been found in the literature.

A surface deterioration due to degradation and erosion of resin matrix and/or filler particles might result in increased surface roughness, resulting in increased plaque adhesion (14–16). Furthermore, if a restoration material surface is changed with respect to surface roughness and/or surface energy, it will probably influence the adhesion of a secondary restoration material to the old restoration (17–19).

Because pH has an influence on composite resin, compomer and glass-ionomer materials, the hypothesis of the present study is that Carisolv®, with its oxidative agent and high pH, has the ability to affect the surface

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texture of those materials. To investigate degradation and erosion processes on the surface of dental materials, an optical interferometer for topographical analysis has been shown to be a suitable analytical method (9).

The purpose of the present study was therefore to investigate the effect of Carisolv® gel on the surface topography of three commonly used restorative materials: one composite resin, one compomer, and one glass-ionomer material.

Materials and methods

The materials used were a composite resin (Spectrum™), a compomer (Dyract® AP), and a glass-ionomer (Ketac®-Fil Plus) (Table 1).

Material preparation

Thirty specimens of the composite resin and the compomer materials and 60 specimens of the glass-ionomer were prepared. For preparation of the specimens, a Teflon mold (diameter 15 ± 1 mm and 0.5 ± 0.1 mm thick) was used. The manufacturer's instructions were followed meticulously. The composite and the compomer were filled into the mold with surplus. In order to smoothen the specimen surfaces in a standardized manner, a sheet of polyethylene film was positioned on top of the material and excess material was removed by pressing a glass plate against the film. The glass plate was removed and the specimens were irradiated by an external light source (Visilux 2, Dental Products/3M Company, USA) in 5 overlapping sections for the recommended exposure time of 20 s/section. After irradiation, the polyethylene film was immediately removed and the specimens were immersed in deionized water. The upper and lower surfaces were left untouched, i.e. fingerprints on the specimen surfaces were avoided. Preparation of the glass-ionomer specimens followed the same procedure, except for the light curing part. This material was chemically cured and the specimens were cured for the time given by the manufacturer. Since previous studies (20–23) have shown that water has an effect on composite resin and glass-ionomer materials, all specimens were stored for at least 7 days in 37 ± 2°C before further treatment in order to minimize the influence of the water on the topographical results.

Topographical analysis

The surface topography of each specimen was evaluated with an optical interferometer (MicroXam™, PhaseShift, Tucson, USA) before and after Carisolv® gel exposure. In order to prevent water droplets from interfering with the measurement, the specimens were left for approximately 30 min to spontaneously dry in ambient temperature before analysis. An area of each composite resin and compomer specimen (300×400 μm) was investigated. The area measured before exposure was relocated to the second analysis, i.e. the topography before and after chemical exposure could be compared, which means that each specimen acted as its own control.

The topographical measurements of the glass-ionomer specimens were undertaken slightly differently. Because of a lower glossiness of the glass-ionomer surface, the optical reflectivity was not as high as for the composite and the compomer. In general, the reflectivity can be increased by reducing the measuring area; an area of 200×260 μm was therefore chosen for the glass-ionomer. Furthermore, the topographical analysis of the glass-ionomer specimens was performed in two series. In one series the same drying procedures as those used for the composite resin and the compomer materials were employed. In the second series, minimized drying times were used, i.e. the specimens were

Table 1. Description of materials examined

Dental material	Principal type	Composition according to the manufacturer	LOT	Manufacturer
Spectrum™	Composite resin	Bis-GMA adduct Bis-EMA TEGDMA Photo initiators Stabilizers Ba-Al-borosilicate (particle size <1 μm) Silicon dioxide (particle size 0.04 μm)	9910001292	Dentsply DeTrey GmbH, Konstanz, Germany
Dyract® AP	Compomer	Polymerizable resins TCB resin Strontium-fluoro-silicate glass Strontium fluoride Photo initiators Stabilizers	9910000639	Dentsply DeTrey GmbH, Konstanz, Germany
Ketac®-Fil Plus	Glass-ionomer	Sr-Al-La-fluorosilicate glass Pigments Copolymer acid Tartaric acid Water	015/010	ESPE Dental AG, Seefeld, Germany

left to dry spontaneously only until excess water had vanished, since this type of material is sensitive to dehydration. For all three kinds of material a digital Gaussian filter, with a size set to 200 × 200 μm, was used to remove deviations caused by form. The following topographical parameters describing the surface roughness were calculated:

S_a [μm]: arithmetic average height deviation from a mean plane.
S_q [μm]: root-mean square height deviation from a mean plane, standard deviation for the height distribution.
S_{sk} (unitless): skewness, the symmetry of surface about the average height.
S_t [μm]: maximum peak to valley height of the surface.
S_{ds} [mm^{-2}]: number of summits of a unit sampling area.
S_{dr} [%]: developed surface area ratio (3D/2D).

Mathematical descriptions of the parameters can be found in the literature (24).

Carisolv® gel exposure

In order to avoid dilution of the Carisolv® gel, the specimens were left for 5 min to spontaneously dry in ambient temperature before exposure. In the second series of glass-ionomer specimens, with minimized drying times, the specimens were exposed to Carisolv® gel immediately after the first topographical analysis. The exposure was performed in a standardized manner and Carisolv® gel uncolored (batch no. 01–009) was mixed just before abundant application on each specimen surface.

For all materials, three different exposure times were used, 5 ($n = 10$), 10 ($n = 10$), and 20 min ($n = 10$). During exposure, the specimens were kept at +32°C and after exposure they were meticulously rinsed with deionized water. Before the second topographical investigation the specimens were left to dry in the same manner as before the first topographical analysis. Until the contact angle analysis was performed the samples were kept in deionized water at room temperature in order to avoid changes that might take place on the surface because of dehydration.

Contact angle analysis

The contact angle indicates the surface energy of the surface, but surface roughness also has an impact on the contact angle (25); the topographical analysis was therefore complemented with contact angle measurements. Four samples from each material and exposure time were randomly chosen after at least 7 days of water storage, as described above. Because the analysis has to be performed with dry samples, the samples were carefully dried with a paper napkin until all visible moisture was removed. Great care was taken not to contaminate the surface before analysis. Immediately after the drying procedure, the contact angle measurements were performed with water in a DAT 1100 (Fibro Systems, Sweden). The equipment was calibrated before analysis. The size of water drop was 5.0 μl, and the instrument was set on stroke 8 and time out 0.2 s. The contact angle was measured 1 s after the drop had come into contact with the surface. Each surface was analyzed twice and the mean angle was recorded. All measurements were performed at +20°C.

Statistical analysis

To study differences in surface roughness between the different dental materials, mean and standard deviation values were calculated of the parameters achieved from the topography measurements. Each specimen acted as its own control, since measurements were performed before and after chemical exposure. Topographical differences before and after chemical exposure were therefore calculated for each specimen. Normal distribution was assumed and checked with histograms, which were also used to detect possible outliers. For all materials, confidence intervals with 0.95 significance were calculated of the average difference for the different exposure times. Besides statistical significance, the topographical parameter changes were interpreted with respect to trends among the different exposure times, but also with respect to the relative magnitude between the parameter value and the mean and standard deviation of the difference before and after exposure.

Contact angle differences within each group of material with increased exposure time were statistically studied with Scheffe's F-test at a significance level of 0.05. Statistical analyses in this study were performed with SPSS (SPSS, Chicago, IL, USA).

Results

Topographical analysis

The topographical parameter values are presented in Table 2. The composite resin material and the compomer exhibited similar surface roughness, as shown by the parameter values. The amplitude surface structures (S_a, S_q, and S_t) and the developed surface area ratio (S_{dr}) of the glass-ionomer seemed to be larger, as well as the surface topography variation among different specimens within this group.

The histograms of the topographical differences before and after chemical exposure confirmed the normality assumption. A few measurements were indicated as outliers. However, the outlier indications were evenly spread over different measurements and these measurements most often were indicated as outliers only with respect to one parameter and not to the rest. If strictly considering statistically significant pairwise differences, the density of summits (S_{ds}) of the composite resin and the skewness (S_{sk}) of the glass-ionomer material decreased after 20 min chemical exposure (Table 3). Furthermore, a statistically significant decrease of the root-mean square height deviation (S_q) of the minimally dried glass-ionomer

Table 2. Surface topography parameters for the materials investigated with the optical interferometer. Figures presented are mean values, standard deviations within parentheses

Material	Carisolv® exposure (min)	n	Type of topographical parameter: Amplitude				Spatial	Hybrid
			S_a (μm)	S_q (μm)	S_{sk}	S_t (μm)	S_{ds} (mm^{-2})	S_{dr} (%)
Spectrum™	0	30	0.47 (0.19)	0.80 (0.33)	−0.40 (0.85)	9.87 (2.51)	0.016 (0.008)	0.44 (0.29)
	5	10	0.47 (0.21)	0.82 (0.38)	−0.44 (0.56)	11.04 (3.96)	0.014 (0.007)	0.55 (0.37)
	10	10	0.34 (0.18)	0.58 (0.30)	−1.52 (1.99)	9.05 (2.12)	0.014 (0.007)	0.45 (0.27)
	20	10	0.49 (0.24)	0.84 (0.40)	−0.15 (0.21)	9.74 (3.51)	0.012 (0.005)	0.31 (0.19)
Dyract® AP	0	30	0.40 (0.26)	0.69 (0.49)	−0.90 (3.17)	9.73 (5.14)	0.022 (0.012)	0.81 (0.79)
	5	10	0.34 (0.34)	0.58 (0.67)	−0.93 (0.98)	9.79 (5.47)	0.022 (0.014)	0.88 (0.95)
	10	10	0.45 (0.17)	0.78 (0.33)	−0.69 (1.12)	10.02 (4.15)	0.022 (0.009)	0.69 (0.58)
	20	10	0.30 (0.10)	0.52 (0.16)	−2.26 (4.87)	9.85 (3.99)	0.016 (0.009)	0.56 (0.57)
Ketac®-Fil Plus	0	30	1.73 (0.46)	2.29 (0.59)	−0.46 (0.51)	32.66 (7.24)	0.086 (0.006)	149.1 (69.4)
	5	10	2.13 (0.49)	2.85 (0.67)	−0.98 (0.51)	47.46 (14.94)	0.089 (0.006)	238.5 (116.4)
	10	10	1.59 (0.51)	2.10 (0.65)	−0.67 (0.42)	28.94 (7.42)	0.084 (0.008)	140.5 (108.1)
	20	10	1.62 (0.57)	2.14 (0.70)	−0.58 (0.59)	31.77 (7.90)	0.088 (0.008)	126.6 (65.2)
Ketac®-Fil Plus, minimally dried	0	30	1.21 (0.42)	1.62 (0.53)	−0.58 (2.26)	20.43 (8.49)	0.030 (0.019)	9.03 (10.84)
	5	10	0.94 (0.26)	1.28 (0.33)	−0.80 (1.14)	19.63 (8.26)	0.029 (0.016)	9.89 (7.93)
	10	10	1.07 (0.34)	1.62 (0.90)	−2.10 (3.87)	28.72 (28.35)	0.027 (0.021)	15.56 (24.44)
	20	10	1.11 (0.46)	1.49 (0.62)	−1.53 (1.77)	21.64 (8.58)	0.028 (0.015)	11.06 (10.75)

material was recorded after 5 min exposure. If considering trends among the different exposure times for the pairwise differences (Table 3) and the absolute topographical parameter values (Table 2), the parameters of the composite resin and the compomer describing the density of summits (S_{ds}) and the developed surface area ratio (S_{dr}) were slightly smaller after Carisolv® gel exposure than before. S_{dr} of the minimally dried glass-ionomer was slightly increased after exposure, although the pairwise differences of the height describing parameters (S_a and S_{sk}) indicated larger parameter values before exposure than after.

Contact angle analysis

Although changes of the contact angle with increased time of exposure to Carisolv® was recorded (Table 4), no significant difference was found for any of the materials tested ($P > 0.05$, Scheffe's F-test). The composite resin material displayed a slight decrease in contact angle, while the compomer and the glass-ionomer displayed an increase of the contact angle with increased exposure time.

Discussion

In the present study, the chemical influence of Carisolv® gel on the surface topography of three different dental filling materials was examined. The topographical investigation was complemented with contact angle analysis. Some surface alterations due to the chemical exposure could be detected, although these were small.

Table 3. 95% confidence intervals of pairwise differences for the dental materials before/after Carisolv® gel exposure. Confidence intervals that do not include the value 0 are given in italics

Material	Carisolv® exposure (min)	n	Type of topographical parameter: Amplitude				Spatial	Hybrid
			ΔS_a (μm)	ΔS_q (μm)	ΔS_{sk}	ΔS_t (μm)	ΔS_{ds} (mm^{-2})	ΔS_{dr} (%)
Spectrum™	5	10	−0.03 ± 0.16	−0.04 ± 0.30	−0.40 ± 1.08	−1.14 ± 1.46	−0.001 ± 0.005	−0.12 ± 0.14
	10	10	0.11 ± 0.20	0.20 ± 0.37	1.17 ± 1.31	0.59 ± 2.39	0.002 ± 0.009	−0.06 ± 0.21
	20	10	0.03 ± 0.18	0.01 ± 0.30	−0.11 ± 0.34	0.69 ± 3.10	*0.008 ± 0.006*	0.20 ± 0.23
Dyract® AP	5	10	0.10 ± 0.14	0.17 ± 0.24	−0.77 ± 3.37	−0.06 ± 4.98	0.095 ± 0.142	0.02 ± 0.50
	10	10	*0.07 ± 0.07*	0.10 ± 0.11	0.31 ± 0.49	0.38 ± 1.46	0.004 ± 0.005	0.40 ± 0.70
	20	10	−0.04 ± 0.10	−0.07 ± 0.18	1.63 ± 5.51	−0.79 ± 2.87	−0.039 ± 0.098	−0.11 ± 0.26
Ketac®-Fil Plus	5	10	−0.08 ± 0.15	−0.13 ± 0.22	0.16 ± 0.44	−12.82 ± 13.65	−0.002 ± 0.006	−29.11 ± 39.58
	10	10	0.05 ± 0.21	0.04 ± 0.27	0.17 ± 0.42	5.26 ± 8.48	0.001 ± 0.005	−17.12 ± 64.56
	20	10	−0.11 ± 0.21	−0.12 ± 0.25	*0.54 ± 0.50*	−2.64 ± 5.54	−0.001 ± 0.007	−12.08 ± 28.12
Ketac®-Fil Plus, minimally dried	5	10	*0.34 ± 0.34*	*0.51 ± 0.42*	−0.38 ± 2.39	5.07 ± 7.88	0.004 ± 0.010	1.57 ± 3.79
	10	10	0.015 ± 0.16	−0.20 ± 0.45	1.77 ± 3.18	−10.68 ± 18.45	0.000 ± 0.022	−8.51 ± 17.13
	20	10	0.15 ± 0.22	0.17 ± 0.27	*1.30 ± 1.30*	−3.09 ± 5.59	−0.001 ± 0.006	−2.49 ± 4.08

When analyzing the topography with optical instruments, reflectivity of light from the sample surface is a requirement. The composite resin material and the compomer both reflected the light very well. The glass-ionomer showed a lower optical reflectivity, probably due to the higher surface roughness. Surface structures scatter incident light so that less light can be detected by the instrument. In general, it is possible to obtain a higher reflectivity when focusing on a smaller area, and therefore a smaller measurement area was chosen for the glass-ionomer measurements. When increasing the measurement area, larger wavelengths describing the form of the surface are included in the measurement. However, considering the preparation process, covering the surface with a polyethylene film, the specimen surfaces were expected to be smooth and to have little form variation. Furthermore, a Gaussian filter was used to separate form from roughness, which means that the different measurement areas should influence the results only to a minor extent.

Besides the degree of glossiness, glass-ionomer materials differ from composite resins and compomers with respect to the hydrated silica gel, which surrounds unreacted glass particles (26). When the water evaporates from the glass-ionomer the material alters and changes in the surface texture will take place (26). Thus, in order to treat the specimens in a manner as closely as possible to the clinical handling, the analysis of the glass-ionomer specimens was repeated with minimized dehydration times. The exposure times, the rinsing, and the temperature used were also chosen with respect to clinical relevance, considering that the mean ($\pm s$) time for caries removal with Carisolv® has been reported to be 10.4 ± 6.1 min (5). It is possible that the drying and rinsing procedures had an impact on the topographical values, but the same procedures were used for all specimens within each group, which also made it possible to use the exposure time as an extra control.

Contact angle analysis can give an indication of the wettability of a material, which can have clinical applications when a material is to be repaired. Different liquids have been used for contact angle measurements on polymers (27). Water is commonly used for contact angle measurements and is relevant from a clinical point of view. In the present study, the contact angle measurements were therefore performed with water.

Table 4. Contact angles measured. The figures represent mean values, standard deviations within parentheses

Material	Carisolv® exposure (min)	*n*	Contact angle (°)
Spectrum™	0	2	55.5 (7.2)
	5	4	58.4 (4.2)
	10	4	56.7 (3.8)
	20	4	55.1 (6.9)
Dyract® AP	0	2	46.0 (4.5)
	5	4	45.5 (10.3)
	10	4	50.6 (1.4)
	20	4	56.8 (5.8)
Ketac®-Fil Plus	0	–	–
	5	4	31.8 (11.7)
	10	4	38.1 (11.1)
	20	4	37.5 (22.2)

Three different types of restorative material were employed. On the whole, composite resins consist of inert glass powder and a resinous monomer that polymerizes during the curing process. A coupling agent (usually silane) is used to provide a bond between the filler particles and the resin. Glass-ionomers consist of acid decomposable glass powder and a water-soluble acid. When these two parts are mixed, ions from the glass leach into the aqueous surrounding. The material sets as the carboxylic groups in the acid form hydrated cross-links with Ca^{2+} and Al^{3+} from the glass particles. Compomers contain polymerizable acids and involve features of both composite resins and glass-ionomers.

As described in the review by Örtengren (28), degradation of composite resin materials is a complex process, since the different phases can react individually. Polymers and residual monomers can degrade by oxidation and hydrolysis (29), resulting in polymer chain scission. Furthermore, unreacted monomers from incomplete polymerization and elements from the filler particles may be dissolved from the material (21, 29). Biodegradation of glass-ionomer cements has been described as a complex process of absorption of water, surface disintegration, and leakage of ions (29). It is reasonable that the hydrated cross-linking between the particles and the matrix, in glass-ionomers, could be affected (i.e. hydrolyzed) by an alkaline surrounding environment. In the present study, the additional analysis of the contact angle indicates such behavior. If there is a loss of filler particles, the surface will consist of more organic matrix resulting in an increased contact angle. Both the compomer and the glass-ionomer tested in this study indicated a tendency of increasing contact angle with increasing exposure time to Carisolv® gel. A smoothening of a surface could also be expected to result in an increased contact angle. This was the case for the compomer, but not for the composite resin that showed a minor decrease of the contact angle despite slight smoothening of the surface. On the contrary, a surface roughening after exposure of the minimally dried glass-ionomers was indicated in terms of a surface area enlargement, even though the height describing parameters indicated a smoothening, explained by a denser surface structure.

Both topographical and contact angle changes due to Carisolv® gel exposure are small in this study, and more particularized clinical conclusions drawn from such in vitro findings would be speculative. Furthermore, it is not possible to exclude that other composite resin and glass-ionomer materials exhibit different behavior at contact with Carisolv® gel than the materials investigated (13). In conclusion, the results of this study indicate that if Carisolv® gel comes into contact with an earlier restoration of a composite resin or glass-ionomer material, there will be little or no effect on the surface topography.

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