Profilometric and SEM analyses of composite surfaces after cement excess removal

Danimir P. Jevremović[1], Zorica R. Ajduković[2], Saša D. Stanković[2], Radivoje D. Radosavljević[3]

[1]*School of Dentistry, Department of Prosthetic Dentistry, Pančevo, Serbia*
[2]*University of Niš, Medical faculty, Department of Dentistry, Clinic for Prosthetic Dentistry, Niš, Serbia*
[3]*Faculty of Medicine, Department of Dentistry, Clinic for Prosthetic Dentistry, Kosovska Mitrovica, Serbia*

Abstract

Composite cements are widely used in dentistry, due to their positive characteristics (bond strength, color, low solubility etc.). However, removal of the cement presents one of the drawbacks of their use, since incomplete removal might cause bacterial adhesion, gingival irritation and subsequent inflammation. The aim of this study was to investigate surface characteristics of composite cements after different ways of excess removal, by means of profilometric and SEM analysis. Thirty leucite reinforced ceramic specimens were divided into three groups, based on the way of cement excess removal: Group 1 (polished) - excess was fully polymerized for 40 s, then removed; Group 2 (cleaned) - excess was removed with a cotton roll, after which cement was fully polymerized for 40 s; Group 3 (pre-polymerized) - excess was light cured for 5 s, after which cement excess was broken with an instrument and then fully polymerized for 40 s. Surface roughness was measured using a surface profilometer. Subsequently, the specimens were inspected by a scanning electron microscope. The data were statistically analyzed. The examination of variants of average values proved the statistically significant difference in the height of average values per group, $p < 0.0001$; the statistically significantly highest values were found for the prepolymerized group, whereas the statistically significantly lowest values were found for the polished group. The results of this study show that utmost attention has to be paid to the excess removal procedure, since surface roughness parameters directly depend on the choice of the applied technique.

Keywords: Profilometric analysis; SEM analysis; composite cement; dental materials.

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Modern prosthodontics widely uses metal free ceramic restorations, in order to achieve maximal esthetic and biocompatibility. Due to their optical, but also mechanical properties, metal free restorations have to be luted to sound teeth tissues with composite cements, by means of an adhesive cementation.

Composite cements are widely used for adhesive cementation of inlays, onlays, veneers and full crowns made of indirect composites or full ceramic materials. The marginal gap between tooth and restoration varies from 50 up to almost 500 µm, as reported in literature [1]. Such gaps reveal a sufficient area of composite cement, being exposed to oral bacteria adherence.

In the mouth, restoration is firstly covered by the salivary pellicle, containing proteins, carbohydrates and lipids. Initial colonization of oral microbes is based on electrostatic and van der Waals interactions [2]. Consequently, co-adhesion and co-aggregation result in coherent binding. The whole process depends both on surface roughness and surface free energy.

Composite cements consist of dimethacrylates, hydroxyethyl-methacrylate, and inorganic filler particles (such as barium glass fillers, ytterbium tri-fluoride and silicon-dioxide). Polymerization of monomers is initiated chemically, by light, or both, resulting in cross-linked polymer chains. Once fully polymerized, composite cement shows mechanical, chemical and optical properties superior to other cement types (zinc-phosphate, glass-ionomers etc.) [3–5]. On the other hand, hardening of the cement makes its excess removal difficult, since fully polymerized cement can be removed only with rotary instruments.

In recent years, several techniques have been adopted in order to simplify composite cement excess removal. Full polymerization refers to the usual manner of treating composite restorations, where cement excess is removed afterwards, with rotary instruments. Removal prior to polymerization (cleaning) can be used to simplify excess removal, if preparation surfaces are reachable by suitable instruments. However, unpolymerized cement is often hard to remove. Finally, the third one, called pre-polymerization or the quarter technique, considers short-time exposure to light (5 s out

Correspondence: D. Jevremović, Department of Prosthetic Dentistry, School of Dentistry, Pančevo, Žarka Zrenjanina 179, 26000 Pančevo, Serbia.
E-mail: dr.danimir@sbb.rs
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of 40 s), making excess solid but still easy to break with a scalper. Though widely accepted by clinicians, effects of the violent break on the surface texture of cement have not been fully investigated.

If surface roughness increases with such a procedure, it may have a negative effect on the longevity of the restoration. It has been reported that increased roughness [6–8] highly influences the adhesion of oral bacteria. Cracks, grooves and pits are responsible for initial bacterial attachment [9]. Observation of polished, cleaned surface and surface after excess breaking show clear microscopic differences [10]. Therefore, excess removal may develop a susceptible surface for oral bacteria colonization, which may result in secondary caries development, or a periodontal disease [11].

The aim of this study is to investigate surface characteristics of composite cements following different ways of excess removal, by means of a profilometric and SEM analysis.

MATERIALS AND METHODS

Specimen preparation

30 ceramic specimens were fabricated out of IPS Empress Aesthetic leucite reinforced ceramics (Ivoclar Vivadent, Schaan, Liechtenstein), according to the manufacturer's instructions. Specimens were adequately shaped, matching recommended dimensions to suit a profilometric roughness analysis. Each sample was cut into two pieces, using a diamond saw (Isomet, Buehler, IL, USA). Two parts were then cemented, using Variolink II composite cement (Ivoclar Vivadent, Schaan, Liechtenstein). Composition of Variolink II, according to the manufacturer's data, is shown in Table 1. Ceramic surfaces were initially activated (Monobond Plus, containing adhesive monomers and ethanol), after which mixed base and catalyst were applied in 1:1 ratio. Specimens were then divided into three groups, based on the way of cement excess removal:

Group 1 (polished). Excess was fully polymerized (40 s), then removed with rotary instruments and polished with the Polityp polishing system (Ivoclar Vivadent, Schaan, Liechtenstein).

Group 2 (cleaned). Excess was removed with a cotton roll, after which cement was fully polymerized (40 s).

Group 3 (pre-polymerized). Excess was light cured in 5 s, after which cement excess was broken with a standard instrument.

Polymerization was done with a LED polymerization lamp (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein).

Roughness tests

Surface roughness was measured using a surface profilometer (TR200, Micro Photonics, Allentown, USA). The tracing diamond tip was 5 μm with a tracing speed of 0.135 mm/s. Cut-off value was 0.25 mm. Each sample was measured twice. Following parameters were recorded [12,13]:

a) amplitude parameters, *R*a, *R*q, *R*z, *R*t and *R*3z; *R*a – arithmetical mean deviation of profile; *R*q – root mean square deviation of profile; *R*p – maximum depth of profile peak; *R*v – maximum depth of profile valley; *R*t – total peak-to-value height; *R*3z – third maximum peak-to-value height;

b) spacing parameters**,** *S* and *S*m; *RS* – mean spacing between adjacent local peaks of profile; *RS*m – mean spacing between profile elements.

c) hybrid parameter, *S*k; *RS*k – skewness of the profile.

Table 1. Composition of the composite cement Variolink II (mass%, source: R&D, Ivoclar Vivadent, Schaan, Liechtenstein)

Component	Base	Catalyst
Dimethacrylates	26.3	27.9
Inorganic fillers (silica, barium glass and ytterbium trifluoride)	73.4	71.2
Catalysts and Stabilizers	0.3	0.9
Pigments	< 0.1	< 0.1

SEM Analysis

Representative samples were taken from each group, dried and coated with 15–20 nm thick layer of gold with a Sputter Coater device (Polaron Range, Quorum Technologies, England). Subsequently, specimens were inspected by a scanning electron microscope (JSM – 6460LV, JEOL, Tokyo, Japan).

Statistical analysis

The data were analyzed with 1-way ANOVA and Tukey's Honestly Significant Difference (HSD) test ($p < 0.05$).

Mean values ± standard deviation (SD) and coefficient of variation (*CV*, %) of the parameters for each group were calculated. For $CV > 30\%$ differences between groups were tested for statistical significance by means of the Mediana test.

The difference between the mean values of the groups were significant if $p < 0.05$ or $p < 0.0001$. The difference between the groups was not significant if $p > 0.05$.

RESULTS

Roughness tests

The representative appearance of the profile with tested sample groups are shown in Figures 1–3.

The values of statistical significance, obtained through the ANOVA test and non-parametric Mediana and Z tests, are provided in Table 2.

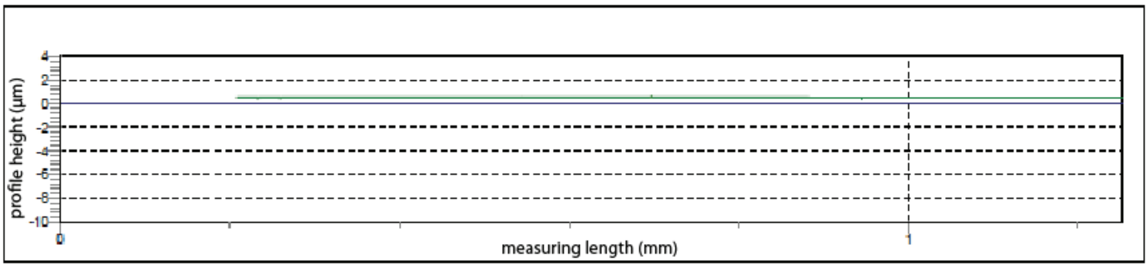

Figure 1. The surface roughness profile of the composite cement fragment after excess removal by polishing.

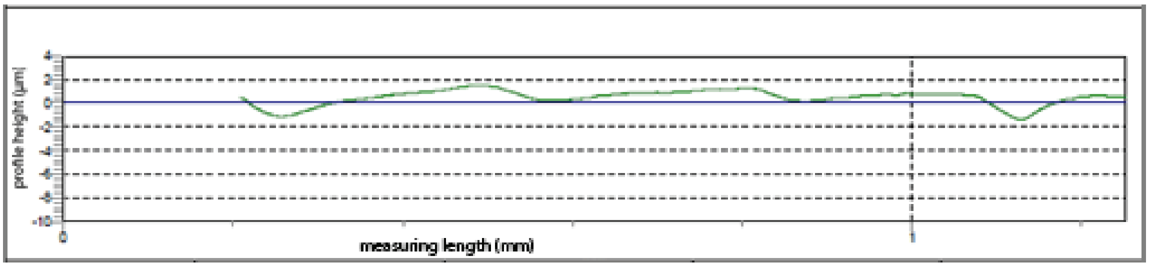

Figure 2. The surface roughness profile of the composite cement fragment after excess removal by cleaning.

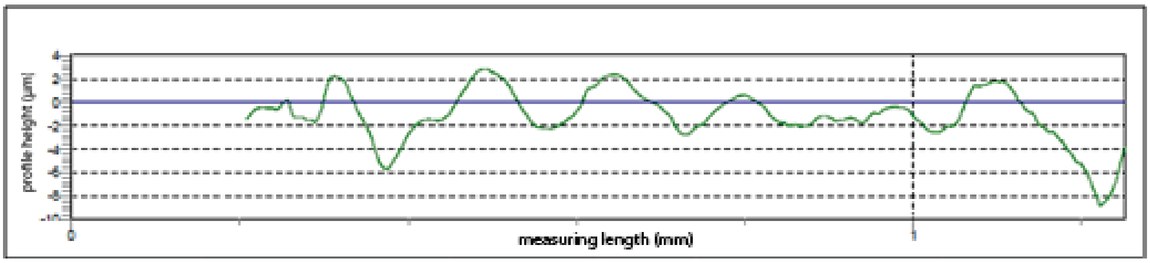

Figure 3. The surface roughness profile of the composite cement fragment after excess removal by prepolymerization.

The examination of variants of average values by means of the Ra ANOVA test proved the statistically significant difference in the height of average values per group, $p < 0.0001$; the statistically significantly highest values were in the prepolymerized group, whereas the statistically significantly lowest values were in the polished group.

An additional multiple comparison of average values by means of the Ra Tukey HSD test between groups proved the statistically significant difference in the height of average values of polished versus cleaned group, $p < 0.0001$, the statistically significantly higher values are in the cleaned group, polished versus prepolymerized group, $p < 0.0001$, the statistically significantly higher values are in the prepolymerized group, as well as in prepolymerized versus cleaned, $p < 0.0001$ the statistically significantly higher values are in the prepolymerized group (Table 3).

Table 2. Results of testing differences of variants of average parameter values per group-ANOVA test, and results of testing central values of series per group -MEDIANA test

Parameter	Mean±SD			ANOVA F test		Mediana test
	Polished	Cleaned	Prepolymerized	F test	Sign	Sign
*R*a	0.184±0.078	1.254±0.447	3.123±0.607	231.167	$p < 0.0001$	$p < 0.0001$
*R*q	0.236±0.099	1.498±0.522	3.639±0.715	223.497	$p < 0.0001$	$p < 0.0001$
*R*p	0.451±0.168	2.036±1.042	4.364±1.117	98.427	$p < 0.0001$	$p < 0.0001$
*R*v	0.455±0.242	1.936±0.650	5.419±1.235	194.298	$p < 0.0001$	$p < 0.0001$
*R*3z	0.222±0.132	0.070±0.094	0.067±0.165	8.788	$p < 0.001$	$p < 0.0001$
*R*t	2.168±1.243	10.213±5.670	17.992±4.076	74.675	$p < 0.0001$	$p < 0.0001$
RS	0.063±0.045	0.228±0.176	0.181±0.084	10.853	$p < 0.002$	$p < 0.0001$
*RS*m	0.205±0.169	0.587±0.372	0.318±0.119	12.760	$p < 0.0001$	$p < 0.0001$
*RS*k	0.268±1.832	-1.062±2.174	-0.200±1.272	2.8131	$p = 0.068$	$p = 0.246$

Table 3. Results of testing differences of average parameter values per group – multiple comparison between groups Tukey HSD

Parameter	Polished *versus* cleaned	Polished *versus* prepolymerized	Prepolymerized *versus* cleaned
*R*a – arithmetical mean deviation of profile	0.0001	0.0001	0.0001
*R*q – root mean square deviation of profile	0.0001	0.0001	0.0001
*R*p	0.0001	0.0001	0.0001
*R*v	0.0001	0.0001	0.0001
*R*3z – third maximum peak-to-value height	0.002	0.002	0.997
*R*t – total peak-to-value height	0.0001	0.0001	0.0001
RS – mean spacing of local peaks of profile	0.0001	0.006	0.399
*RS*m – mean spacing of profile elements	0.0001	0.321	0.003
*RS*k – skewness of the profile	0.050	0.691	0.291

Similar conclusions can be obtained by comparing amplitude parameters, *R*q, *R*p, *R*v and *R*t. The lowest values of the extreme peaks of the positive profile are in the polished group, while the highest extremes are in the prepolymerized group. The lowest points (values of the valley below the medial line) are in the prepolymerized group and the highest in the polished group. The total peak to value height value is also the most expressed in the prepolymerized group.

Spacing parameter *RS* shows that the most regular profile is present in the polished group. Most profile irregularities are in the prepolymerized group. The adjacent peaks of profile values, however, do not differ significantly in groups 2 and 3. The second parameter of the horizontal roughness, *RS*m, as well shows the lowest values in the polished group. Interestingly enough, gaps between starting points of profiles does not differ between groups 1 and 3.

The values of *RS*k, the hybrid parameter of roughness, show positive values only in group 1. The lowest values (the highest negative deviations from the medial line) can be found, however, in group 2 (cleaned), considerably differing to the value in groups 1 and 3. Notwithstanding the negative values of the mean value of the *RS*k parameter in the prepolymerized group, there is no statistical difference from the polished group.

An additional testing of differences in the amount of average values of selected parameters among groups by means of the Student T test gave same results between groups as the testing of variants of average values, except in the case of *RS*k parameters (Table 3).

As KV was higher than 30% in all the selected and analyzed series for the polished and cleaned groups, an additional testing of values of selected parameters between groups was performed by means of nonparametrical tests, namely the Mediana test and the Z test, and the same results between groups were obtained as in the testing of average value variants, except in the case of *RS*k parameters.

SEM Analysis

Microscopic images of the chosen specimens can be seen in Figures 4–9. The polished group (Figures 4 and 5) shows the most homogenous surface appearance, starting already with small magnifications. With higher

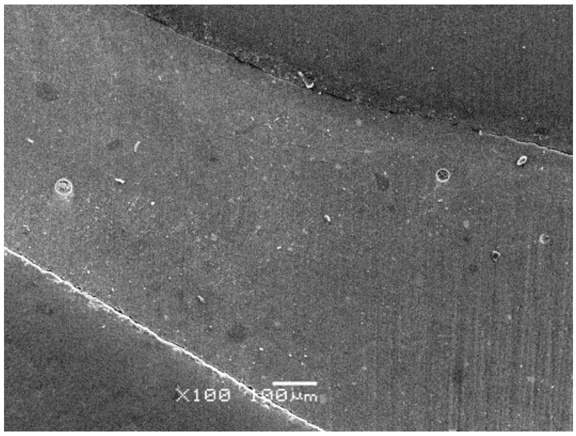

Figure 4. SEM micrograph – surface fragment of the composite cement after removal by polishing (smaller magnification, 500×).

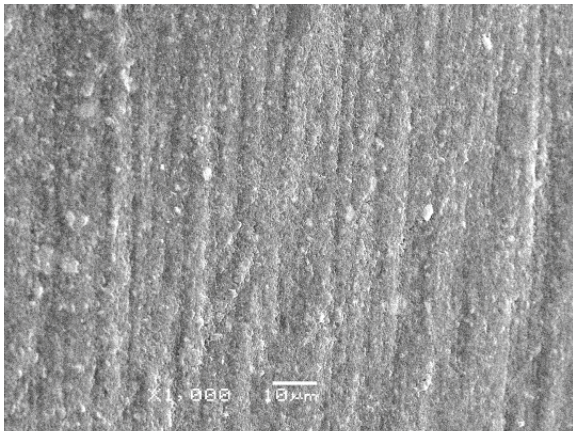

Figure 5. SEM micrograph – surface fragment of the composite cement after removal by polishing (higher magnification, 1000×).

magnifications, traces of polishing instruments can be seen, corresponding to the pathway of the instrument.

The cleaned group exhibits rough areas as well, within the median line, corresponding to the path of the cotton roll, used for cleaning purposes. However, other areas show visible valleys where too much cement has been removed (Figures 6 and 7).

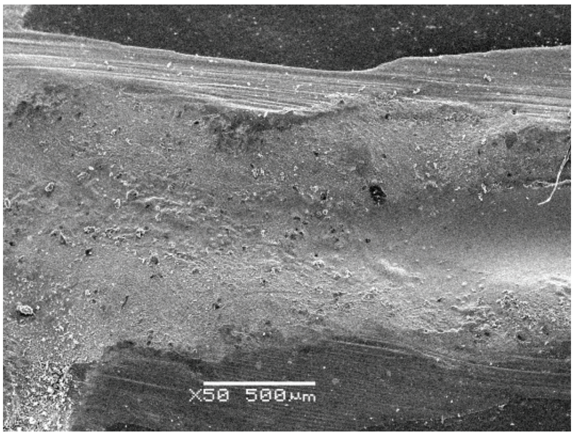

Figure 6. SEM micrograph – surface fragment of the composite cement after removal by cleaning (smaller magnification, 500×).

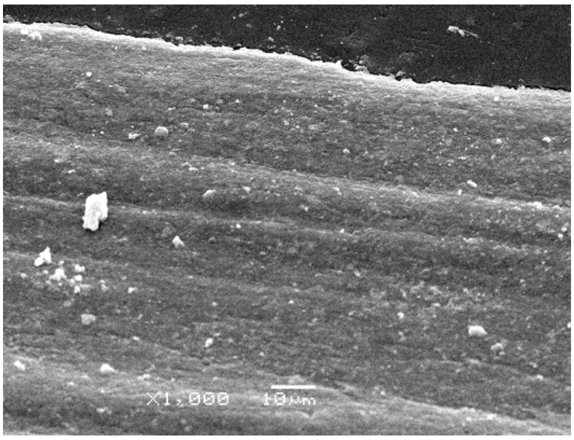

Figure 7. SEM micrograph – surface fragment of the composite cement after removal by cleaning (higher magnification, 1000×).

The roughest area is presented in the prepolymerized group. With small magnifications (Figure 8), an explicit area of a broken cement surface can be detected. High magnifications (1000×, Figure 9) exhibit rough surfaces, with many high peaks and deep valleys visible.

DISCUSSION

Clinical studies have shown that roughness has a very important role in both supra and sub gingival plaque formation. Though sub and supra gingival plaques differ in variety of parameters, increased roughness *in vivo* is commonly related to the increased risk of caries and periodontal disease [14]. Initially, surface irregularities provide increased colonization area and good protection against detachment forces (such as chewing, oral fluids, desquamation, or oral hygiene). This provides both shift to irreversible attachment and subsequent plaque growth, since its maturation depends mainly on multiplication of present bacteria, and not on the adherence of new ones, present in the oral fluids [2,7].

Figure 8. SEM micrograph – surface fragment of the composite cement after removal by prepolymerization (smaller magnification, 500×).

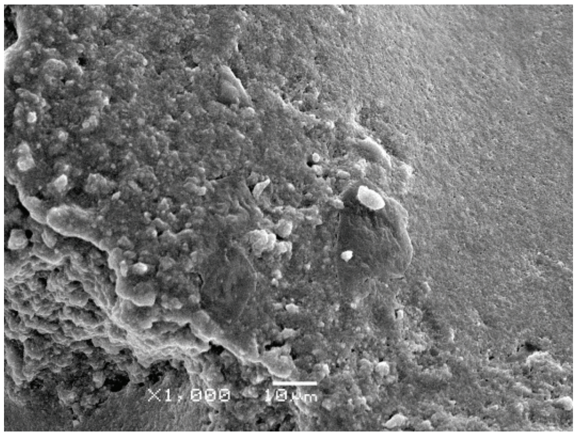

Figure 9. SEM micrograph – surface fragment of the composite cement after removal by prepolymerization (higher magnification, 1000×).

A surface roughness analysis, presented in this study, has clearly shown that the majority of the parameters have significant differences among tested groups. *R*a, shows the highest values in pre-polymerized samples (group 3). It is the most common parameter used in dental material research [15-18], especially when using tactile profilometry. *R*a is the arithmetic mean of the departures of the roughness profile from the mean line, however, it calculates valleys in an inverted form, being therefore insensitive to differentiating rough and undulant surfaces. Surface texture analysis therefore needs further comments from other relevant parameters.

In contrast to *R*a, *R*q is sensitive to deviations above and below the median line. The reading accounts for extreme peaks and valleys. *R*q values also show significant differences among all tested groups. *R*p and *R*v values are also statistically significant. *R*p parameter has the highest value within group 3. Prominently high peaks above the median line can cause soft tissue irritation. On the contrary, high values of *R*v confirm existence of deep pockets, which are unable to reach with a brush. High *R*v values in group 3 predict this group to be prone to bacterial segregation. *R*t is an extreme value, showing large scatter [18]. Highest *R*t values, shown in pre-polymerized group, correspond to the high rough profile of those specimens. It should be, however, noted that contact profilometry tends to undersize extreme values, due to the incapability of reaching top points of the narrow profiles [19].

Amplitude parameters are generally considered as the most important property of the surface [20]. The second group, spacing parameters, describes texture, randomness and periodicity of the surface [21].

RS calculates horizontal spacing between adjacent peaks whereas *RS*m is mean spacing between profile peaks. It can be seen from the results that the polished group has different *RS* values, compared to the other two groups. This can correlate to the efficiency of the polishing process, where untreated groups show more random distribution. Interestingly, *RS*m values show that peaks in the polished and pre-polymerized group exhibit similar repeatability.

Wietnam and Eames [22] reported that plaque accumulation occurs on composite specimens with *R*a of 0.7 to 1.44 μm. Those values correspond to cleaned and pre-polymerized group results. Polishing can, however, improve surface roughness, and a highly polished surface should be obtained for a plaque-free environment [23]. Clinically, polishing is not always possible to perform, since many areas are hard to reach with rotary instruments. Another question is, however, whether roughness in group 2 and 3 can be decreased further if the margin position allows polishing to be conducted.

Efficiency of the polishing procedure depends on many factors, including, of course, the composite cement filler content and type [24]. Another issue is the accessibility of the exposed surface to the polisher. *R*a alone cannot predict the efficiency of such a procedure, since it does not distinguish peaks and valleys, and only the former can be polished away. Hybrid parameters depend both on amplitude and spacing properties. *RS*k, or skewness, measures symmetry, showing positive values for the roughness above and negative values for the roughness below the median line. *RS*k values have interesting implications, since, for polishing efficiency, the abrasive material has to be harder than the polishing one [25]. The fact is, however, that ceramic restoration exhibits much higher hardness values, making valley surfaces much harder to polish, being out of reach of the instrument. Only polishers with high elasticity can perhaps be able to reach such surfaces. It can be therefore concluded that the surface roughness of specimens with negative *RS*k values can hardly be improved further, as seen especially in the "cleaned" group.

Negative *RS*k values in group 2 can be due to the nature of the cleaning process. Excess removal with a cotton roll can often over-remove cement, especially with larger gaps. In the mentioned case, applied pressure can create a big depression, resulting in the mentioned negativity of the *RS*k parameter. Many practitioners have stopped using super-floss in this case, since its structure collects too much of the uncured cement, commonly creating marginal gaps.

Besides bacterial adhesion, another disadvantage of rough surfaces is staining susceptibility. Rough surfaces, in combination with a dietary intake, favor pigment retention. Staining is often seen in older ceramic restorations, at their margins [1]. Staining is, however, not related to roughness alone, but also depends on the type of the resin phase of the composites [26]. Urethane dimethacrylate seems to be more resistant than *bis*-GMA. Chemical formulation depends on the type of the used composite cement. Clinical relevance has, however, to be determined via further *in vivo* trials.

CONCLUSION

Surface roughness is commonly seen as a risk factor for the increased bacterial adherence. Based on the profilometric and SEM data acquired in this study, it can be concluded that different ways of excess removal significantly influence surface characteristics of the used composite cement. Prepolymerization, as a commonly used procedure, reveals the roughest surface among all the procedures tested. Polishing should be used whenever possible, in order to minimize risk of creating rough, irregular surfaces.

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IZVOD

PROFILOMETRIJSKE I SKENIRAJUĆE ELEKTRONSKO MIKROSKOPSKE ANALIZE KOMPOZITNIH POVRŠINA NAKON UKLANJANJA VIŠKA CEMENTA

Danimir P. Jevremović[1], Zorica R. Ajduković[2], Saša D. Stanković[2], Radivoje D. Radosavljević[3]

[1]*Stomatološki fakultet, Klinika za stomatološku protetiku, Pančevo, Srbija*
[2]*Univetzitet u Nišu, Medicinski fakultet, Klinika za stomatologiju, Odeljenje za stomatološku protetiku, Niš, Srbija*
[3]*Medicinski fakultet, Klinika za stomatološku protetiku, Odsek za stomatologiju, Kosovska Mitrovica, Srbija*

(Naučni rad)

Bezmetalne keramičke nadoknade neophodno je cementirati na pripremljena zubna tkiva kompozitnim cementom. Kada je kompletno polimerizovan, kompozitni cement pokazuje mnogo bolje mehaničke, hemijske i estetske osobine u odnosu na konvencionalne cemente. Sa druge strane, nakon njegovog vezivanja uklanjanje viška cementa je otežano. Nekoliko tehnika uklanjanja viška cementa usvojeno je u kliničkoj praksi. Međutim, pukotine, kanali i udubljenja, koji ostaju na površini kompozitnog cementa, mogu biti odgovorni za povećanu prijemčivost bakterija. Iz ovoga se može zaključiti, da uklanjanje viška cementa može stvoriti podložnu sredinu za razvoj i naseljavanje bakterija, koji mogu dovesti do sekundarnog karijesa ili periodontalnog oboljenja. Cilj ove studije je da se profilometrijskom i SEM analizom ispita karakteristika površine kompozitnih cemenata nakon različitih načina uklanjanja viška cemenata posle vezivanja. Trideset keramičkih fiksnih nadoknada (leucitna ojačana keramika) podeljeno je u tri grupe, na osnovu načina uklanjanja viška cementa nakon vezivanja: grupa 1 (poliranje), višak se kompletno polimerizuje (40 s), a onda uklanja; grupa 2 (čišćenje), višak se uklanja vaterolnom, a onda polimerizuje (40 s); grupa 3 (prepolimerizacija), višak se polimerizuje 5 s, nakon čega se uklanja standardnim instrumentom, a zatim polimerizuje u potpunosti (40 s). Hrapavost površine je merena uz pomoć površinskog profilometra, a uzorci su potom ispitivani skening elektronskim mikroskopom. Rezultati su statistički obrađeni. Dokazana je statistički značajna razlika u visini prosečnih vrednosti prema grupama, $p < 0,0001$; statistički značajno najviše vrednosti su u grupi prepolimerizacijom, a statistički značajno najniže vrednosti su u grupi poliranjem. Dobijeni rezultati ukazuju na neophodnost obraćanja pažnje na postupak odstranjenja viška cementa. Naime, iako se tehnika prepolimerizacije čini jednostavnom i prihvatljivom sa aspekta praktične primene, karakteristike površine nakon ovog postupka su slabije, u poređenju sa drugim metodama. S druge strane, ove metode je teže, a u nekim slučajevima i nemoguće primeniti, što zahteva dalja istraživanja, s ciljem pronalaženja jednostavnog a efikasnog načina uklanjanja viška kompozitnog cementa.

Ključne reči: Profilometrijska analiza • SEM analiza • Kompozitni cement • Dentalni materijali