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Gloss and surface analysis of various composite materials

Vanja Opačić Galić[1], Danica Popović[2], Jovana N. Stašić[1], Bojan Dželetović[1], Stefan Manojlović[3]

[1]University of Belgrade, School of Dental Medicine, Department of Restoratie Dentistry and Endododntics, Belgrade, Serbia;
[2]University of Belgrade, School of Dental Medicine, Department of Prostodontics, Belgrade, Serbia;
[3]Dental Clinic "S Dent" Subotica, Serbia

SUMMARY

Introduction To obtain the ideal aesthetics, each restorative material must imitate natural tooth in color, surface texture and be stable over time. Damage or wear of the material causes poor optical properties of the restoration, so it is necessary to repolish, repair or replace it. The aim of this study was to test the gloss and surface changes of the composite materials with filler's particles of different size, before and after material artificial mechanical ageing.
Material and Methods Four composite materials were tested, two microhybrid composites (Gradia Direct GC, Herculite XRV, Kerr) and two nanohybrid composites (Filtek Ultimate 3M; TetricEvoceram, Ivoclar). Composites' samples of 9 × 9 × 2 mm in size were polished in accordance to the standard protocol by Sof-Lex discs (2382 C, SM, F, SF) for 30 seconds. Gloss was measured after polishing and taking photos using optical microscope (400×). In the chewing simulator, ageing of the samples was conducted (100,000 cyclic kicks). After the samples' ageing, surface photos were taken and gloss measured. The obtained results were statistically processed (One-way ANOVA, t-test).
Results The best gloss after polishing was shown by Filtek Ultimate (54.00 ± 14.06), the worst by Gradia Direct (47.33 ± 7.92). There was no statistically significant difference ($p > 0.05$) in surface gloss after polishing composite. The smallest defects on the surface of composites were detected in material Gradia Direct (21363.7 μM^2) with the average value of the diameter 137 μM.
Conclusion The tested nanohybrid and microhybrid composites showed comparable gloss value before and after artificial aging. Mechanical aging caused vivid changes regarding surface defects on all tested composite materials.
Keywords: nanohybrid composite; microhybrid composite; gloss

INTRODUCTION

Many philosophers have spoken of aesthetics and beauty, and most of them agreed with Plato's saying that beauty is in the eye of the viewer and that it is subjective experience of the object of observation. Aristotle's quote that what is beautiful has to be harmonious, could apply on nowadays composite materials which in general comply with aestethic, functional and biological demands in restorative dentistry. To obtain ideal aesthetics, each restorative material must match natural tooth in color, surface texture and be stable over time [1]. The main reason for replacement of the existing restorations is mostly aesthetics [2].

One of the problems when applying composite restorative materials is the rough surface of restoration. This facilitates staining, plaque accumulation, recurrent decay, inflammation of the gingiva, and brightness reduction of the restoration. The surface roughness of Ra 0.2µm (Ra = means roughness value) is considered sufficient for plaque accumulation and discoloration [3, 4], while patient can feel roughness of 0.3 µm by the tip of the tongue [5]. O'Neill pointed out that the maximum acceptable threshold for roughness is 200 nm, after which plaque accumulation occurs [6]. Damage or wear of the material causes poor optical properties of the restoration, so it is necessary to repolish, repair or replace it.

In addition to surface roughness, gloss is another factor that plays an important role in the aesthetics of composites. Gloss is surface ability to reflect light. Human eye can easily tell the difference between the radiance of the restoration and surrounding enamel, even if the colors of these structures are the same [7]. Furthermore, glossy surface of enamel "tolerates" mechanical wear, while glossy surface of the composite, which is initially lower, tends to continue to decrease due to mechanical stresses over time. With increasing surface roughness, the degree of random light reflection increases, which eventually results in the decrease in the surface gloss [8]. Reduction of gloss and smoothness potentially leads to discoloration of the restoration [5, 11].

In the oral environment, many external and internal factors have great impact on restorations. The factors related to the material itself are primarily the structure of the matrix and the characteristics of the restoration particles, which have direct influence on the surface smoothness and susceptibility to exterior discoloration [1, 9].

Reducing the particle size of hybrid composites can enhance aesthetic properties, but with optimum physical properties, microhybrids (0.04 to 1µm) were the first one

Address for correspondence: Vanja OPAČIĆ GALIĆ, Department of Restoratie Dentistry and Endododntics, Rankeova 4, 11000 Belgrade, Serbia; *vanja.opacic.galic@gmail.com*

introduced and followed by nanotechnology with particles that are in the range of 0.1-100 nanometers. The use of nanotechnology in new composite formulations is one of the major contributions in dental materials [10, 11]. Nanocomposites are considered to be a combination of good mechanical strength of hybrid composites but with superior optical properties than micro-filled ones [4, 12]. They have high translucency, are well polished and have superior gloss with adequate mechanical properties for high stress restorations [5]. In addition to the particle size, the shape of the particles also has an impact on the resistance to occlusal wear, so Tamura et al. [13] pointed out that spherical particle composites are more resistant to simulated occlusal wear than irregularly shaped particle composites.

Resin and filler particles do not have the same wear resistance due to different degrees of hardness [9]. Thus, composite structure, refining and polishing systems have direct effect on surface (gloss and roughness), and mechanical properties (hardness and resistance to chemical degradation) [1, 14]. It has been shown that more time spent on polishing a composite, the restoration with higher and longer gloss is obtained [3].

Given that degradation of the composite in the oral environment is known, numerous *in vitro* studies have examined the effect of artificial aging on the mechanical and optical properties of these materials. Cycle loading protocols (mechanical artificial aging), immersion in media such as ethanol or water, brush simulation and light aging [2, 15, 16] are most commonly used.

The aim was to investigate gloss and surface changes of different composite materials before and after simulated aging. The null hypothesis was that there was no difference in gloss before and after the mechanical aging of different composite materials.

MATERIALS AND METHODS

Four types of composite materials were included in this investigation, two microhybrid and two nanohybrid composites (color A2) (Table 1).

For each groups of tested materials, eight samples were prepared (in silicon mold 9x9x2 mm). Each sample was polymerized across glass slide 40 s on both sides, (Woodpecker Led. H, China). Samples were polished by standard protocol aluminum oxide abrasive Sof-Lex discs (2382 C, SM, F, SF) (3M, ESPE), for 30 s each, using watercooling. After polishing the samples, (IG-331; Horiba) gloss of each sample surface of the composite material was measured by the gloss-meter. Samples were centrally set up with striking beam of 60 degrees. Polished surfaces were observed with optical microscope (x400) (Carl Zeiss Jena NU2) and photographed.

Until placed in chewing simulator, samples were kept in wet environment with constant temperature of 37°C. Samples were poured into acrylate and placed in two chambers of chewing simulator. Cyclic loading with force of 5 kg in vertical direction simulated mechanical aging. For each sample 100,000 cycles were performed. This was equivalent to one year of chewing (CS-4.2 Economyline, SD Mechanotronics, Germany). After the mechanical aging, gloss was measured for every surface. Surface of samples were photographed and saved in digital format.

The obtained results were statistically processed using One-way ANOVA and T- test.

RESULTS

The results were presented in Figures 1–6 and Table 2.

After being polished with Sof-Lex system, the highest gloss was shown in Filtek Ultimate with values 54 ± 14.08, Tetric EvoCeram with values 49.67 ± 7.03 and Herculite

Table 1. Tested materials
Tabela 1. Testirani materijali

Material Materijal	Type Tip	Fillers Punioci	Matrix Matriks	Manufacturer Proizvođač
Gradia	Microhybrid mikrohibridni	0.85 µm Silica / silika	UDMA, dimethacrylate co-monomers-dimetakrilat ko-monomer	GC Dental, Japan
Herculite XRV	Microhybrid mikrohibridni	0.3–0.6 µm Ba-Al-Silicate / Ba-Al-silikat	Bis-GMA	KerrHawe S. A., Switzerland
Filtek Ultimate	Nanohybrid nanohibridni	0.6–10 µm 4–20 nm silica, zirconia / silikat, cirkonijum	Bis-GMA, UDMA, TEGDMA, bis-EMA	3M ESPE, USA
Tetric EvoCeram	Nanohybrid nanohibridni	40–3000 nm Ba-Al-silicate, iterbiumtrifluorid, silica / Ba-Al-silikat, iterbiumfluorid, silika	UDMA, Bis-GMA	IvoclarVivadent, Liechtenstein

UDMA – urethane dimethacrylate; Bis-GMA – bisphenol glycidyl methacrylate; TEGDMA – triethylene glycol dimethacrylate; Bis-EMA – ethoxylated bisphenol-A dimethacrylate UDMA – uretan-dimetakrilat; Bis-GMA – bisfenol-glicidil metakrilat; TEGDMA – trietilen-glikol dimetakrilat; Bis-EMA – etoksilirani bisfenol-A dimetakrilat

Table 2. Composite surface defect sizes (µm) after chewing simulation
Tabela 2. Veličina defekata na površini kompozita (µm) posle simulacije žvakanja

Material Materijal	Area, µm^2 Površina, µm^2	Max diameter, µm Maksimalni prečnik, µm	Min diameter, µm Minimalni prečnik, µm	Mean diameter, µm Srednji prečnik, µm	Roundness Zaokruženo
Gradia	21363.7	226.4	103.1	137.8	1.91
Herculite XRV	268696,7	630.0	537.3	582.7	1.17
Filtek Universal	187307.0	584.2	300.8	457.2	2.34
Tetric EvoCeram	247833.3	756.3	547.4	642.4	3.61

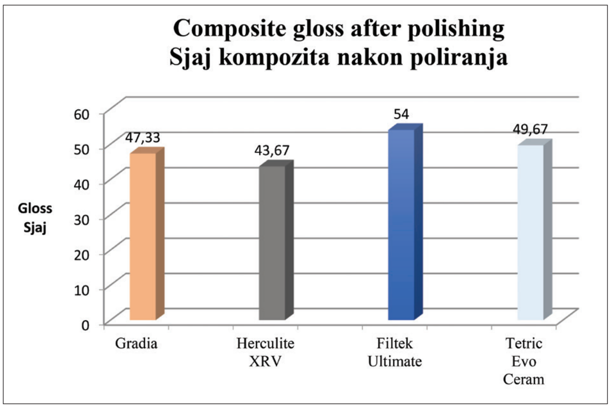


Figure 1. Gloss of composite after polishing
Slika 1. Sjaj kompozitnih materijala posle poliranja

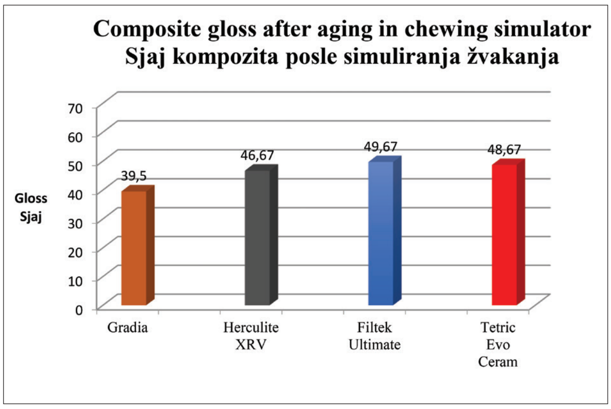


Figure 2. Gloss of composite after aging in chewing simulator
Slika 2. Sjaj kompozita posle simuliranja žvakanja

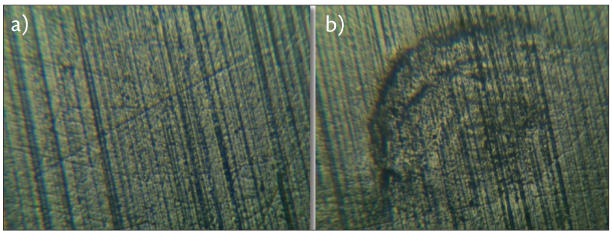


Figure 3. Composite filling surface Gradia Direct
a) after polishing (×400);
b) after aging; defect size (21.364 μm^2) (×400)
Slika 3. Kompozitna površina kompozita Gradia Direct
a) posle poliranja (×400);
b) posle starenja; veličina defekta (21,364 μm^2) (×400)

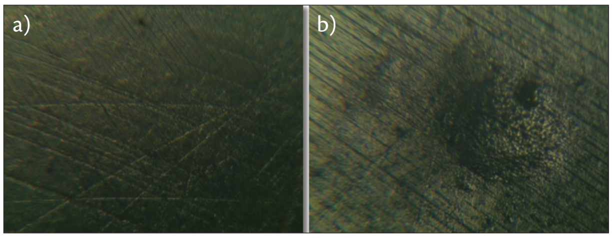


Figure 4. Composite filling surface Tetric EvoCeram
a) after polishing (×400);
b) after aging; defect size (247.883 μm^2)
Slika 4. Kompozitna površina kompozita Tetric EvoCeram
a) posle poliranja (×400);
b) posle starenja; veličina defekta (247,883 μm^2)

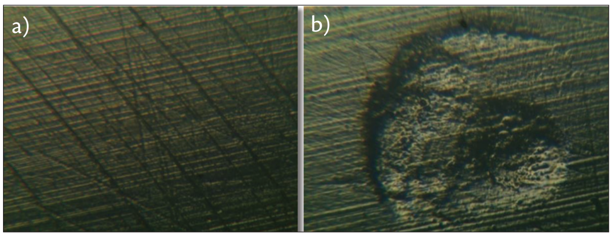
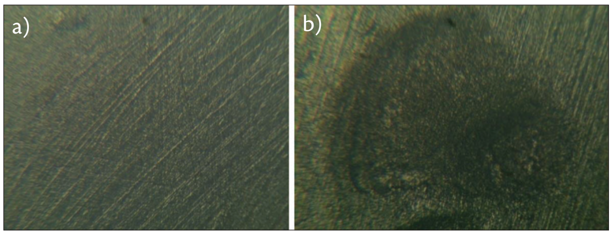


Figure 5. Composite filling surface Filtek Ultimate
a) after polishing (×400);
b) after aging; defect size (187.307 μm^2)
Slika 5. Kompozitna površina kompozita Filtek Ultimate
a) posle poliranja (×400);
b) posle starenja; veličina defekta (187,307 μm^2).



b) **Figure 6.** Composite filling surface Herculite XRV
a) after polishing (×400);
b) after aging; defect size (268.697 μm^2) (×400)
Slika 6. Kompozitna površina kompozita Herculite KSRV
a) posle poliranja (×400);
b) posle starenja; veličina defekta (268,697 μm^2) (×400)

XRV with 43.67 ± 12.14. The lowest gloss after polishing was recorded in Gradia Direct with values 47.33 ± 7.92 (Figure 1). After polishing there was no statistically significant difference in gloss between micro and nanohybrid composites.

After exposure of the composite materials to simulated mechanical aging (100.000 cycles) in chewing simulator it was observed that the highest gloss was still in Filtek Ultime with average values 49.67 ± 11.83, then Tetric EvoCeram with 48.67 ± 12.14 and Herculite XRV with 46.6 ± 11.71. After simulated mechanical aging the lowest values were found in Gradia Direct with average 39.5 ± 6.83 (Figure 2). After simulated mechanical aging there was no statistically significant difference in gloss between microhybrid and nanohybrid composite. There was no statistically significant difference in gloss values before and after aging of all tested materials.

Optical microscope revealed the smallest defects on the composite surface of Gradia Direct, with the average surface area of this defect being 21,363.7 μm^2, followed by Filtek Ultimate with 187,307.0 μm^2 and Tetric EvoCeram with 247,883.3 μm^2. The largest defect was observed on the surface of Herculite XRV with average value of 268,696.7 μm^2 (Table 2, Figures 3–6).

DISCUSSION

Polishing gives high gloss of restorations that imitate natural dental structures. Gloss plays a key role in the aesthetic experience of restorations. Many authors have found that there is a correlation between surface gloss and surface roughness, and polishing can increase gloss [1, 9, 17]. Endo T. [18] considers that if the surface roughness is less than 0.1μm, the surface of the composite will be visibly smooth. High gloss even reduces any color difference between the composite and surrounding enamel. The color of the reflected light is dominant over the color of the composite below [5, 19].

Radiance is an optical phenomenon that depends on the amount of light beams reflected from the surface and the degree of diffuse reflection has negative effect on surface gloss. Rodrigues Jr [7] pointed out that light reflection is influenced by the microstructural properties of the material (size, shape and refractive index of the filler particles, viscosity and refractive index of matrix, as well as the homogeneity of the matrix-filler complex). The smaller the filler particles is, smaller is the diffuse reflection [8, 17].

In our study, gloss was measured by a glossmeter with the striking angle of 60° which is close to the angle at which an average height person observes the surface, that also complies with the standard (ISO 28132014) [20]. There is no agreement at what angle gloss should be measured, whether there is better differentiation at an angle of 20° or 45° than 60° [15].

Many studies engaged in the problem of the impact of the manual skills and length of the practitioner's clinical practice [5, 7, 14]. In this study, two dentists polished the samples: one with years of experience and the other one with three years of experience. Given that within the composite groups there was no statistically significant difference, it is obvious that different practitioner skills did not affect the result. Zimmerli and Rodrigues Jr. have also found no correlation between clinical experience and polishing efficiency [7, 14]. Jung et al. [21], even pointed out that the greatest surface roughness was observed in long-term practitioners, while the student and physicians with five-year internship were similar.

There are different systems for polishing composites and almost every material manufacturer recommends their own systems, which differ in material (aluminum oxide, carbide components, diamond abrasives, silicon dioxide, zirconium oxide), abrasiveness and shape (discs, cups, cones, tapes) [22]. Polishing protocols can be multiple-step or one-step systems [4]. For the finishing and polishing systems to be effective, abrasive particles must be relatively harder than the filler particles, otherwise polishing would only remove the soft matrix while the filler particles would come through the surface [22]. However, the type and shape of the particles that make the base of the polishing agents differ from each other, and it is therefore difficult to compare polishing agents [8]. The authors pointed out that satisfactory surface and gloss roughness levels are obtained if the polishing particles are less than 13μm (1200-grit according to ISO 8486-1) [17].

The material samples in this study were polished with Sof-Lex disks, as flexible aluminum oxide disks are the most commonly used systems and leave the least rough surfaces [23]. In line with our results, Pala [1] and Rodriguez Jr. [7] pointed out that "multiple-step" systems give better gloss than "one-step" systems. The efficiency of these disks is due to even removal of both organic matrix and filler particles. Despite their smooth surface, their application is limited in the posterior region. Some authors don't like the metal center, as well as great flexibility that can leave uneven surfaces if the pressure force is higher [7].

The third factor that has an effect on polishing is the composite material itself (hardness, the shape, size, particle orientation, the degree of matrix conversion, the stability of the silanes etc. [3, 7, 9, 12, 18, 24]. Despite all these factors, polishing and surface roughness are the function of the size of the filler particles [7, 19]. Antonson et al. [5] and Tursi et al. [25] confirmed that composites with smaller particles showed higher gloss and less surface roughness. Suzuki found that polished surface of the Tetric EvoCeram nanohybrid was very smooth, with an equally abraded surface without any discontinuity between the prepolymerized fillers and surrounding matrix [11]. Although Filtek Ultimate nanohybrid showed the highest gloss as well as Tetric EvoCeram, there was no significant difference in the appearance of polished surface and surface gloss between them.

Can Say et al. [24] reported in their study that microhybrid composites had less surface roughness than nanohybrids, which can be explained by an uneven abrasion of the matrix and filler particles. It is considered that nanosize particles (20–40 nm) tend to fill the space between larger particles, thus protecting soft matrix from abrasion. The homogeneity of the material, that is, the uniform processing during polishing, ultimately gives greater gloss [1]. Lai et al. [15] based on 1990 standards, found out that good gloss is between 70 and 80 GU (gloss units), and excellent above 80 GU. Ivoclar Vivadent, however, finds that the observer cannot differentiate between 70 and 90 GU. According to the American Dental Association (ADA) in 2010, gloss of 40-60 GU is, as per most experts, desirable and acceptable gloss [1, 3]. The results of our study showed that the average gloss of the composites was between 47.33 and 54 GU, which can be considered as well polished, glossy surfaces.

Number of authors [1, 3, 13, 19] also pointed out the significant influence of filler particle shape on surface roughness, suggesting that irregular particles give rougher surface. They also argumented that spherical particles such as Filtek (3M ESPE) can also be a factor for better light reflection with irregularly shaped particles of other composites. Aesthetic properties are also enhanced when filler particles have the same refractive index as resin [15]. Composites with glass particles have been observed to have rougher surface than those with particles that are a combination of silica and zirconia, whether microhybrid or nanohybrid [5]. Although Tetric EvoCeram showed greater roughness, or less gloss than Filtek Ultimate, this was not statistically significant. Endo et al. [18] also facted that glass particles in composites influence their increased porosity and more frequent discoloration.

In addition to filler particles resin can also affect surface properties of the material. Composites with hydrophobic matrix are less stained (UDMA versus BisGMA) [9, 10, 23], while those with UDMA based matrix have rougher surface compared to those based on Bis-GMA, due to the difference in the degree of polymerization, rigidity of the molecules etc. [4, 24]. The results of our study are in agreement with previous findings, as Gradia (UDMA matrix) showed the lowest gloss after polishing with Sof-Lex discs.

After simulating aging of the composite samples, the smallest surface defects were demonstrated by Gradia Direct, a microhybrid composite, which confirmed their optimal mechanical and physical properties. However, it is important to note that in this paper there was no statistically significant difference between micro- and nanohybrid composites.

Finally, it is important to note that in our study, a simulation of accelerated aging of materials under laboratory conditions was performed. The presence of water, temperature changes, pH levels, as well as the abrasive nature of food or tooth brushing are all factors that influence the properties of composite restorations and their surfaces. Also, the samples were flat, for the sake of uniformity of the experiment, but clinically, the composite restorations have irregular geometric structures with concave and convex surfaces.

CONCLUSION

With the limitations of this study, it can be concluded that investigated nanohybrid and microhybrid composites showed comparable gloss values before and after artificial aging. The null hypothesis was accepted. Filtek Ultimate showed the highest gloss before and after aging, while Gradia Direct had the lowest. The one-year chewing simulation caused the least surface defects with Gradia Direct, while the largest defects were observed with Herculite XRV composite materials.

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Analiza sjaja i površine različitih kompozitnih materijala

Vanja Opačić Galić[1], Danica Popović[2], Jovana N. Stašić[1], Bojan Dželetović[1], Stefan Manojlović[3]

[1]Univerzitet u Beogradu, Stomatološki fakultet, Klinika za bolesti zuba, Beograd, Srbija;
[2]Univerzitet u Beogradu, Klinika za protetiku, Stomatološki fakultet, Beograd, Srbija;
[3]Stomatološka ordinacija „S Dent" Subotica, Beograd

KRATAK SADRŽAJ

Uvod Da bi se dobila idealna estetika, svaki restaurativni materijal mora oponašati prirodni zub po boji i teksturi površine i mora biti stabilan u vremenu. Oštećenje ili habanje materijala uzrokuje loša optička svojstva ispuna, pa ga je neophodno prepolirati, reparirati ili zameniti. Cilj rada je bio da se ispitaju sjaj i površinske promene kompozita sa različitom veličinom čestica punilaca, pre i posle artificijalnog mehaničkog starenja.

Materijal i metode Testirana su četiri kompozita, dva mikrohibridna (Gradia Direct GC; Herculite XRV, Kerr) i dva nanohibridna (Filtek Ultimate 3M; Tetric Evoceram, Ivoclar). Uzorci kompozita (9 × 9 × 2 mm) polirani su po standardnom protokolu diskovima Sof-Lex 30 sek. Posle poliranja izmeren je sjaj, a uzorci su posmatrani pod optičkim mikroskopom (×400). U simulatoru žvakanja uzorci su izloženi artificijalnom starenju sa 100.000 cikličnih udaraca. Posle mehaničkog starenja ponovo je izmeren sjaj, a površine su fotografisane i sačuvane u digitalnom formatu. Dobijeni rezultati su statistički obrađeni (One-way ANOVA, t-test).

Rezultati Najveći sjaj posle poliranja pokazao je Filtek Ultimate (54,00 ± 14,06), a najmanji Gradia Direct (47,33 ± 7,92). Nije bilo statistički značajne razlike sjaja između testiranih materijala posle poliranja ($p > 0,05$). Najmanji defekti na površini kompozita uočeni su kod materijala Gradia Direct (21.364 µm), sa srednjom vrednošću prečnika 137 µm.

Zaključak Ispitivani nanohibridni i mikrohibridni kompoziti su pokazali komparabilne vrednosti sjaja pre i posle artificijalnog starenja. Mehaničko starenje je izazvalo vidljive promene u vidu površinskih defekata na svim ispitivanim kompozitima.

Ključne reči: nanohibridni kompoziti; mikrohibridni kompoziti; sjaj

UVOD

Mnogi filozofi govorili su o estetici i lepoti, i većina njih se složila sa Platonovom izrekom da je lepota u oku posmatrača i da predstavlja subjektivan doživljaj predmeta posmatranja. Mišljenje Aristotela da je lepo samo ono što je skladno moglo bi se primeniti na današnje kompozitne materijale, koji uglavnom zadovoljavaju estetske, funkcionalne i biološke zahteve u restaurativnoj stomatologiji.

Da bi se ostvarila idealna estetika, svaki restaurativni materijal mora odgovarati prirodnom zubu po boji i teksturi površine i mora biti stabilan u vremenu [1]. Glavni razlog za zamenu restauracija na zubima je uglavnom estetske prirode [2].

Jedan od problema pri primeni kompozitnih restaurativnih materijala je hrapava površina ispuna. Ovo utiče na prebojavanje, akumulaciju plaka, sekundarni karijes, zapaljenje gingive i smanjenje sjaja restauracije. Hrapavost površine čija je Ra vrednost 0,2 µm (Ra = *mean roughness value*) smatra se dovoljnom za akumulaciju plaka i prebojavanje [3, 4], a hrapavost od 0,3 µm (Ra) pacijent može osetiti vrhom jezika [5]. *O'Neill* čak ističe da je maksimalno prihvatljiv prag za hrapavost 200 nm (Ra), posle čega dolazi do akumulacije plaka [6]. Oštećenje ili habanje materijala uzrokuje loša optička svojstva ispuna, pa ga je neophodno prepolirati, reparirati ili zameniti.

Osim hrapavosti, sjaj površine je drugi faktor koji ima važnu ulogu u estetici kompozita. Sjaj je svojstvo površine da reflektuje svetlost. Ljudsko oko lako uočava razliku između sjaja ispuna i okolne gleđi, iako su boje ovih struktura iste [7]. Takođe, sjajna površina gleđi „podnosi" mehaničko habanje, dok sjajna površina kompozita, koja je inicijalno niža, ima tendenciju da i dalje opada zbog mehaničkih opterećenja tokom vremena. Sa povećanjem hrapavosti površine povećava se stepen nasumične refleksije svetlosti, što na kraju rezultira smanjenjem sjaja površine [8]. Smanjenje sjaja i glatkoće može dovesti do diskoloracije ispuna [1, 5].

U oralnom okruženju na ispun deluju brojni spoljašnji i unutrašnji faktori. Faktori vezani za sam materijal su pre svega struktura matriksa i karakteristike čestica punilaca, koje imaju direktan uticaj na glatkoću površine i prijemčivost za spoljašnja prebojavanja [9, 1].

Potreba za usavršavanjem kompozitnih materijala dovela je do razvijanja hibridnih kompozita. Redukovanjem veličine čestica hibridnih kompozita radi povećanja estetskih osobina, ali sa optimalnim fizičkim osobinama, prvo nastaju mikrohibridni (0,04 do 1 µm), a zatim, uvođenjem nanotehnologije, nanohibridni kompozitni materijali, čije su čestice u opsegu od 0,1 do 100 nanometara. Upotreba nanotehnologije u novim formulacijama kompozita je jedan od najvećih doprinosa dentalnim materijalima [10, 11]. Za nanokompozite se smatra da su kombinacija dobre mehaničke snage hibridnih kompozita, a da imaju superiornije optičke karakteristike od mikropunjenih [12, 4]. Oni imaju visoku translucenciju, dobro se poliraju, imaju superioran sjaj uz odgovarajuća mehanička svojstva za restauracije pod visokim stresom [5]. Osim veličine čestica, na otpornost na okluzalno „habanje" utiče i oblik čestica, pa *Tamura* i sar. [13] ističu da su kompoziti sa sferičnim česticama otporniji na simulirana okluzalna trošenja od kompozita sa česticama iregularnog oblika. Smola i čestice punioca se ne abradiraju na isti način, zato što su različitog stepena tvrdoće [9]. Stoga, sastav kompozita i sistemi za finiranje i poliranje imaju direktan uticaj na svojstva površine (sjaj i hrapavost) i na mehanička svojstva (tvrdoća i otpornost na hemijsku degradaciju) [1, 14]. Dokazano je da duže vreme poliranja kompozita rezultira ispunom sa višim i dugotrajnijim sjajem [3].

S obzirom na to da dolazi do degradacije kompozita u oralnoj sredini, brojne studije *in vitro* su ispitivale uticaj arteficijalnog starenja na mehanička i optička svojstva ovih materijala. Najčešće se koriste protokoli cikličnog opterećenja (mehaničko arteficijalno starenje), potapanje u medije kako što su etanol ili voda, simulacija četkanja i svetlosno starenje [15, 16, 2].

Cilj rada je bio da se ispitaju sjaj i površinske promene kod različitih kompozitnih materijala, pre i posle artifijalnog starenja. Postavljena je nulta hipoteza da ne postoji razlika u sjaju pre i posle mehaničkog starenja različitih kompozitnih materijala.

MATERIJAL I METODOLOGIJA

U ovo istraživanje su uključene četiri vrste komercijalnih kompozitnih materijala, dva mikrohibridna i dva nanohibridna kompozita (boja A2) (Tabela1).

Za svaku grupu testiranih materijala napravljeno je po osam uzoraka (u silikonskom kalupu 9 × 9 × 2 mm). Svaki uzorak je polimerizovan preko staklenih pločica 40 sek. sa obe strane (Woodpecker Led. H, China). Uzorci su ispolirani po standardnom protokolu aluminijum-oksid abrazivnim diskovima Sof-Lex (2382 C, SM, F, SF) (3M, ESPE), 30 sek. svaki, uz vodeno hlađenje. Posle poliranja uzoraka, meračem sjaja (IG-331; Horiba) izmeren je sjaj površine za svaki uzorak testiranih kompozitnih materijala. Uzorci su centralno postavljani, sa upadnim zrakom od 60 stepeni. Ispolirane površine su posmatrane pod optičkim mikroskopom (×400) (Carl Zeiss Jena NU2) i fotografisane.

Do postavljanja u simulator žvakanja, uzorci su čuvani u vlažnoj sredini na konstantnoj temperaturi od 37 stepeni. Ulivani su u akrilat i postavljani u dve komorice simulatora žvakanja. Mehaničko starenje je izvođeno cikličnim opterećenjem silom od 5 kg u vertikalnom smeru. Izvedeno je 100.000 cikličnih udaraca za svaki uzorak, što predstavlja ekvivalent jednogodišnjem žvakanju (CS-4.2 Economyline, SD Mechanotronics, Germany). Posle mehaničkog starenja uzoraka izmeren je sjaj za sve površine, i uzorci, odnosno površine su ponovo fotografisane i sačuvane u digitalnom formatu.

Svi dobijeni rezultati su statistički obrađeni jednosmernom analizom varijance (One-way, ANOVA) i t-test-om.

REZULTATI

Rezultati ovih istraživanja predstavljeni su na grafikonima 1 i 2, u Tabeli 2 i na slikama 1–4.

Posle poliranja sistemom Sof-Lex, najveći sjaj pokazao je Filtek Ultimate sa vrednostima (SV ± SD) od 54 ± 14,08, zatim sledi Tetric EvoCeram sa vrednostima sjaja od 49,67 ± 7,03, potom Herculite XRV sa 43,67 ± 12,14, a najmanji sjaj posle poliranja pokazala je Gradia Direct sa vrednostima od 47,33 ± 7,92 (Grafikon 1). Nije bilo statistički značajne razlike sjaja između mikrohibridnih i nanohibridnih kompozita posle poliranja.

Nakon što je svaki kompozitni materijal bio izložen simuliranom mehaničkom starenju (100.000 cikličnih udara) u simulatoru žvakanja, uočeno je da je i dalje najveći sjaj zadržao Filtek Ultimate sa prosečnom ocenom 49,67 ± 11,83, zatim slede Tetric EvoCeram sa 48,67 ± 12,14 i Herculite XRV sa 46,67 ± 11,71, a najmanji sjaj i posle starenja imala je Gradia Direct sa prosekom od 39,5 ± 6,83 (Grafikon 2). Pokazalo se da i posle simuliranog mehaničkog starenja mikrohibridni u odnosu na nanohibridne kompozite nisu pokazali statistički značajnu razliku u pogledu sjaja površine.

Promene u vrednostima izmerenog sjaja re i posle starenja materijala nisu ukazale na statistički značajnu razliku.

Optičkim mikroskopom uočeni su najmanji defekti na površini kompozita kod materijala Gradia Direct; prosečna površina ovog defekta iznosila je 21363,7 μm^2, zatim slede Filtek Ultimate sa 187307,0 μm^2, Tetric EvoCeram sa 247883,3 μm^2, a najveći defekt je uočen na površini Herculite XRV, čija je srednja vrednost iznosila 268696,7 μm^2 (Tabela 2, slike 1–4).

DISKUSIJA

Poliranjem se dobija visok sjaj restauracija koje imitiraju prirodne zubne strukture. Sjaj ima važnu ulogu u estetskom doživljaju estetskih restauracija. Mnogi autori su utvrdili da postoji korelacija između sjaja površine i hrapavosti površine, kao i da se sjaj konstantno povećava poliranjem [17, 1, 9]. *Endo T.* [18] smatra da ako je hrapavost površine manja od 0,1 μm, površina kompozita će biti vidljivo glatka. Visok sjaj čak i smanjuje eventualnu razliku u boji kompozita i okolne gleđi. Boja odbijene svetlosti je dominantna u odnosu na boju kompozita ispod [19, 5].

Sjaj je optički fenomen koji zavisi od količine svetlosnih zraka koji se reflektuju od površine, a stepen difuzne refleksije negativno utiče na sjaj. *Rodrigues Jr.* [7] ističe da na refleksiju svetlosti utiču mikrostrukturna svojstva materijala, pre svega veličina, oblik i indeks refrakcije čestica punilaca, viskoznost i indeks refrakcije matriksa, kao i homogenost kompleksa matriks-punilac. Što su manje čestice punilaca, manja je difuzna refleksija [17, 8].

U ovom istraživanju sjaj je meren meračem sjaja sa upadnim uglom od 60°, što je najbliže uglu pod kojim osoba prosečne visine posmatra površinu, a što se slaže i sa standardom (ISO 2813-2014) [20]. Ne postoji usaglašenost pod kojim uglom treba meriti sjaj, da li je bolja diferencijacija pod uglom od 20° ili 45° u odnosu na 60° [15].

Mnoge studije su se bavile problemom uticaja manuelne spretnosti i dužine kliničke prakse izvođača [14, 7, 5]. U ovom istraživanju uzorke su polirala dva izvođača sa višegodišnjim i trogodišnjim iskustvom. S obzirom na to da unutar grupa kompozita nije utvrđena statistički značajna razlika, može se zaključiti da njihovo iskustvo ili različit pritisak primenjen pri poliranju nije uticao na rezultat. *Zimmerli* i *Rodrigues Jr.* takođe nisu pronašli korelaciju između kliničkog iskustva i efikasnosti poliranja [14, 7]. *Jung* i sar. [21] čak ističu da su najveću hrapavost površine uočili kod dugogodišnjih praktičara, dok su student i lekari sa petogodišnjim stažom bili izjednačeni.

Postoje različiti sistemi za poliranje kompozita i gotovo svaki proizvođač materijala preporučuje i svoje odgovarajuće sisteme, koji se razlikuju po materijalu (aluminijum-oksid, karbidne komponente, dijamantski abrazivi, silicon-dioksid, cirkonijum-oksid), abrazivnosti u obliku (diskovi, kupe, konusi, trake) [22]. Protokoli za poliranje mogu biti sistemi *multiple-step* ili *one-step* [4]. Da bi sistemi za finiranje i poliranje bili efikasni, abrazivne čestice moraju biti relativno tvrđe od čestica filera, jer bi u suprotnom prilikom poliranja došlo do uklanjanja samo mekanog matriksa, dok bi čestice punilaca prominirale iz površine [22]. Ipak, vrsta i oblik čestica koje čine bazu sredstava za poliranje međusobno se razlikuju, pa je zato veoma teško upoređivati sredstva za poliranje [8]. Autori ističu da se zadovoljavajući nivo hrapavosti površine i sjaja dobija ako su čestice za poliranje manje od 13μm (1200 grita po ISO 8486-1) [17].

Uzorci materijala u ovom istraživanju su polirani diskovima Sof-Lex, jer su fleksibilni aluminijum-oksidni diskovi najčešće korišćeni sistemi i ostavljaju najmanje hrapave površine [23]. U skladu sa našim rezultatima, *Pala* [1] i *Rodriguez Jr.* [7] ističu da sistemi *multiple-step* daju bolji sjaj od sistema *one-step*. Efikasnost ovih diskova je posledica ravnomernog uklanjanja i organskog matriksa i čestica punioca. Uprkos glatkoj površini, njihova primena je ograničena u bočnoj regiji. Neki autori im zameraju i metalni centar, kao i veliku fleksibilnost, koja može ostavljati neujednačene površine ako je sila pritiska veća [7].

Treći faktor koji ima uticaja na poliranje je sam kompozitni materijal, odnosno njegova tvrdoća, oblik, veličina, orijentacija čestica, stepen konverzije matriksa, stabilnost silana i sl. [12, 9, 7, 24, 3, 18]. Uprkos svim ovim faktorima, poliranje i hrapavost površine su u funkciji veličine čestica punilaca [24, 7]. *Tursi* i sar. [25] istakli su da kompoziti sa manjim česticama pokazuju veći sjaj i manju hrapavost površine, što potvrđuje i *Antonson* sa saradnicima [5]. *Suzuki* [11] nalazi da je polirana površina nanohibridnog kompozita Tetric EvoCeram veoma glatka, sa jednako abradiranom površinom bez ikakvog diskontinuiteta između prepolimerizovanih punilaca i okolnog matriksa. Iako je nanohibridni Filtek Ultimate pokazao najveći sjaj, a potom i Tetric EvoCeram, razlika nije bila značajna ni u izgledu ispolirane površine, ni u sjaju.

Can Say i sar. [24] čak ističu da su u njihovim istraživanjima mikrohibridni kompoziti imali manju hrapavost površine u odnosu na nanohibridne, što objašnjavaju neravnomernom abrazijom matriksa i čestica punioca. Smatra se da čestice nanoveličine [20–40 nm] imaju tendenciju da popunjavaju prostor između većih čestica, čime štite mekani matriks od abrazije. Homogenost materijala, odnosno ravnomerno obrađivanje prilikom poliranja daje na kraju veći sjaj [1]. *Lai* i sar. [15] na osnovu standarda iz 1990. God. ističu da je dobar sjaj između 70 i 80 GU, a izvrstan iznad 80 GU. *Ivoclar Vivadent* pak ističe da posmatrač ne razlikuje sjaj između 70 i 90 GU. Prema Američkoj dentalnoj asocijaciji (ADA) iz 2010. godine, sjaj od 40 do 60 GU (gloss units) po mišljenju većine eksperata je željen i prihvatljiv sjaj [3, 1]. Rezultati ove studije su pokazali da je prosečan sjaj kompozita bio između 47,33 i 54 GU, što se može smatrati dobro ispoliranim i sjajnim površinama.

Brojni autori [19, 3, 1, 13], takođe, ističu značajan uticaj oblika čestica punilaca na hrapavost površine, sugerišući da čestice iregularnog oblika daju hrapaviju površinu. Takođe, ističu da sferične čestice kakve su u Filteku (3M ESPE) takođe mogu biti faktor za bolju refleksiju svetlosti u odnosu na kompozite sa česticama iregularnog oblika kod drugih kompozita. Do unapređenja estetskih svojstava dolazi i kad čestice punioca imaju isti refraktivni indeks kao i smola [15]. Uočeno je da kompoziti sa česticama stakla imaju hrapaviju površinu od onih sa kombinacijom silike i cirkonije, bez obzira na to da li su mikrohibridni ili nanohibridni [5]. Iako je Tetric EvoCeram pokazao veću hrapavost, odnosno manji sjaj od Filtek Ultimate, to nije imalo statistički značaj. *Endo* i sar. [18] takođe ističu da čestice stakla u kompozitima utiču na njihovu povećanu poroznost i češću diskoloraciju.

Osim čestica punilaca, i smola može da utiče na svojstva površine materijala. Kompoziti sa hidrofobnim matriksom manje se prebojavaju (UDMA u odnosu na BisGMA) [9, 23, 10], dok oni sa matriksom na bazi UDMA imaju hrapaviju površinu u poređenju sa onima na bazi Bis-GMA, zbog razlike u stepenu polimerizacije, krutosti molekula i sl. [24, 4]. Rezultati ovog istraživanja su u saglasnosti sa prethodnim zaključcima, jer je Gradia (matriks UDMA) pokazala najmanji sjaj posle poliranja diskovima Sof-Lex.

Posle simulacije starenja kompozitnih uzoraka, najmanji defekt površine pokazala je Gradia Direct, mikrohibridni kompozit, koji je potvrdio njihova optimalna mehanička i fizička svojstva. Ipak, važno je istaći da u ovom radu nije bilo statistički značajne razlike, između mikrohibridnih i nanohibridnih kompozita.

Na kraju, bitno je istaći da je u ovoj studiji izvedena simulacija ubrzanog starenja materijala u laboratorijskim uslovima. Prisustvo vode, temperaturne promene, nivo pH vrednosti, kao i abrazivna priroda hrane ili četkanje zuba su svakako faktori koji utiču na svojstva kompozitnih restauracija, odnosno na njihove površine. Takođe, uzorci su bili ravni, radi uniformnosti eksperimenta, ali klinički, kompozitne restauracije su iregularne geometrijske strukture sa konkavnim i konveksnim površinama.

ZAKLJUČAK

Uz ograničenja ove studije može se zaključiti da su ispitivani nanohibridni i mikrohibridni kompoziti pokazali komparabilne vrednosti sjaja pre i posle arteficijalnog starenja. Nulta hipoteza je potvrđena. Najveći sjaj pre i posle starenja pokazao je Filtek Ultimate, a najmanji Gradia Direct. Simulacija jednogodišnjeg žvakanja izazvala je najmanje defekte površine kod kompozita Gradia Direct, dok su najveći defekti uočeni kod kompozitnih materijala Herculite XRV.