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Sensitivity of Composite Materials to Ambient Light and Clinical Working Time

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SUMMARY

Introduction The aim of this study was to determine sensitivity of composite materials to ambient light by a modified standard ISO 4049:2000 and clinical working time.

Materials and Methods The following materials were tested: nano-hybrid Herculite XRV Ultra (Kerr), micro-hybrid Herculite XRV (Kerr), Zmack (Zhermack), SuperCor (SpofaDental) and Valux Plus (3M ESPE). Five samples in each group were exposed to ambient light of 8000 lx, which comprised dental unit light and natural light. After 60 sec of exposure, each sample was visually examined for signs of inhomogeneity meaning that material did not pass the test. Clinical working time was studied by applying a custom-built, standardized indenter into composite specimens of about 2 mm thickness during the same exposure to ambient light. Completion of polymerization was tested by dissolving samples in ethanol and measuring the difference in thickness before and after the test. Results were statistically analyzed using ANOVA at the significance level of 0.05.

Results Only Herculite XRV Ultra passed the test of sensitivity to ambient light, while other materials showed signs of inhomogeneity. Herculite XRV Ultra showed significantly longer working time than other composites, the average was 250 sec ($p<0.05$). The mean values of working time for other materials ranged between 117-131 sec and there was no significant difference between them ($p>0.05$).

Conclusion Due to the lower sensitivity to ambient light and significantly longer clinical working time compared to micro-hybrid composites, Herculite XRV Ultra can be recommended for clinical procedures that require prolonged working time with material in plastic condition.

Keywords: ambient light; ISO 4049:2000; composite; clinical-time; polymerization

INTRODUCTION

Layering techniques for composite application are still recommended for cavity restoration because results indicate stronger bond with dental tissues and lower contraction stress as compared to the "bulk" technique [1, 2]. According to the manufacturer's recommendations the thickness of each layer should not exceed 2 mm, whereas the number of layers depends on the depth of cavity. Due to the constant improvements of mechanical properties of composites, their application in restorative dentistry is extended to the excessive destructions of tooth crown when they can be used for core build ups and reconstruction of loading zones [3]. It is clear that these cases require longer working time. Consequently, sensitivity to ambient light becomes very important.

Sensitivity of composite materials to ambient light is one of the properties that are tested using ISO 4049:2000 standard before new materials are registered on the market [4]. Sensitivity of material is checked visually after 60 sec of exposure to xenon light at an illuminance of 8000 lx with filters in the dark room. Loss of material homogeneity indicates that polymerisation has begun under the influence of this light source. ISO standard provides a simple criterion for evaluating materials, which is "pass/fail the test".

There are few studies available in the literature regarding the sensitivity of composite materials to ambient light. Lane et al. [5] showed that the ISO standard does not match clinical conditions and there is a significant difference in terms of working time measured during the ISO standard test and in simulated clinical conditions. In simulated clinical conditions working time of four tested composites was shorter than 60 sec after the exposure to dental unit light whereas this time was up to three times longer during the ISO standard test [5]. Despite these results, the ISO standard test to measure composite sensitivity to ambient light remained unchanged.

Dlugokinski et al. [6] investigated the degree of polymerization of three composite materials after the exposure to ambient light with and without the elimination of fluorescent light. Dionysopoulos and Watts [7] compared the sensitivity of 29 shades of 14 composite materials to dental unit light in a modified clinical protocol of ISO standard. The results of these studies indicated that ambient light can have a significant impact on the clinical working time of composites. Dentists should be familiar with this feature

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Table 1. Composite materials used in this study
Tabela 1. Kompozitni materijali korišćeni u studiji

Name Naziv	Manufacturer Proizvođač	Type Tip	Ingradients Sastav	
			Organic matrix Organski matriks	Fillers Punioci
Herculite XRV Ultra	Kerr Corporation, Orange, CA, USA	Nano-hybrid Nanohibridni	Methacrylic monomers, trimethylolpropane triacrylate, TiO_2, benzoyl peroxide, 4-methoxyphenol, initiators, pigments Metakrilni monomeri, trimetilolpropan, triakrilat, TiO_2, benzoil-peroksid, 4-metoksifenol, inicijatori, pigmenti	Ba-Al-silicate glass in the form of pre polymerized fillers, nanoparticles and hybrid fillers Ba-Al-silikatno staklo u obliku prepolimerizovanih punilaca, nanočestica i hibridnih punilaca
Herculite XRV	Kerr Corporation, Orange, CA, USA	Micro-hybrid Mikrohibridni	Methacrylic monomers, TiO_2, initiators, pigments Metakrilni monomeri, TiO_2, inicijatori, pigmenti	Ba-Al-silicate glass Ba-Al-silikatno staklo
Valux Plus	3M ESPE, St. Paul, MN, USA	Micro-hybrid Mikrohibridni	BisGMA, TEGDMA, benzotriazolyl-methylphenol, initiators, pigments BisGMA, TEGDMA, benzotriazolil-metilfenol, inicijatori, pigmenti	Silanized ceramics Silanizirana keramika
Zmack	Zhermack SpA-Badia Polesine (RO), Italy	Micro-hybrid Mikrohibridni	Bis GMA, BisEMA, TEGMA, initiators, pigments Bis-GMA, Bis-EMA, TEGDMA, inicijatori, pigmenti	Barium glass, silicon dioxide Barijumsko staklo, silicijum-dioksid
Super Cor	SpofaDental a.s., Jičin, The Czech Republic	Micro-hybrid Mikrohibridni	Methacrylic monomers, initiators, pigments Metakrilni monomeri, inicijatori, pigmenti	n/a

BisGMA – bis-phenol A-glycidyl methacrylate; TEGDMA – triethyleneglycol-di-methacrylate; Bis-EMA – bis-phenol A-glycidyl dimethacrylate; n/a – data not available
BisGMA – bisfenol-A-glicidilmetakrilat; TEGDMA – trietilenglikol-dimetakrilat; Bis-EMA – bisfenol-A-glicidildimetakrilat; n/a – nedostupni podaci

of materials they use as clinical results may be compromised in situations that require prolonged working time.

The aim of this study was to examine the sensitivity of contemporary composite materials to ambient light and their working time in simulated clinical conditions. Null hypotheses were: (1) examined composites do not meet the ISO standards requirements and (2) there is no significant difference among the composites in terms of clinical working time.

MATERIALS AND METHODS

Five types of composite materials (composition is given in Table 1) were tested. For each test five samples in each group were prepared. The experiment was conducted in the clinical operatory using dental unit light Fona A1 (Sirona Dental Systems Foshan Co., Ltd., China). The unit light was positioned perpendicularly to the countertop of the dental unit at the distance that would ensure brightness of the counter area of 8000±1000 lx in accordance to the ISO standard 4049:2000. Overall brightness was obtained from dental unit light and a small part from natural light in the room. Brightness was controlled using a luxmeter (Mastech MS8209, Dongguan Huayi Mastech Co., China).

On the countertop, in the zone of light, about 0.1 g of composite material was placed using a spherical plastic spoon on a clean glass microscope slide and exposed to dental unit light during 60 sec. Afterwards, the material was pressed by a second glass slide and shear motion was applied. The homogeneity of material was visually assessed.

The second part of the study investigated the clinical working time of composites. On the microscope slide, which served as a base, each material was applied in the manner and under the same conditions as in the first part of the experiment. The material was shaped to the thickness of 2 mm using a plastic instrument and exposed to light. For this part of the experiment, a device for standardized pressure, schematically shown in Figure 1, was made. Weight of 160 g was fixed onto this device to simulate the pressure that dentists apply to shape the composite material in the cavity. Indentor tip width was 0.5 mm. The device was placed directly above the sample and freely by its weight passed into the material. Indention was performed every 15 sec, after each time, the imprint depth was measured using a graduated probe with accuracy of 0.5 mm. For each sample three measurements were performed, with each subsequent measurement starting 5 sec

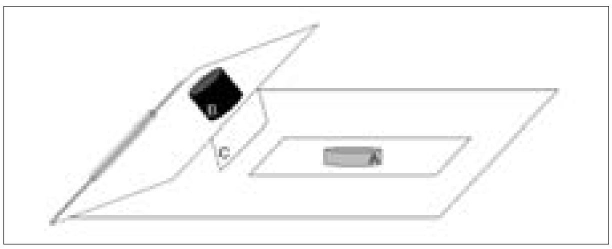


Figure 1. Sheme of the device for standardised pressure (A – composite sample; B – weight; C – indenter)
Slika 1. Shema uređaja za standardizovani pritisak (A – uzorak kompozita; B – teg; C – utiskivač)

after the previous one. The depth of the imprint less than 0.5 mm indicated the completion of polymerization time. That time point was noted as the maximum clinical working time. After that, the thickness d_1 of each sample was measured with the precision of 0.01 mm. After completion of this test, the ethanol shaking test was performed.

The ethanol shaking test or sample dissolution in ethanol is the test that measures completion of polymerization under the influence of ambient light. Verification of the test was done by placing 0.1 g (or the amount that was used for the preparation of each sample in the experimental part) of non polymerized composite in an empty clean amalgam capsule, adding 1 ml of ethanol (Sigma-Aldrich Chemie GmbH, Munich, Germany) and mixing in an amalgamator for 20 sec. This time was enough for ethanol to completely dissolve the tested portion of composite. Analogous to this procedure, each sample of composites after measuring the maximum working time was placed in the capsule, 1 ml of ethanol was added, and mixed in the amalgamator for 20 sec. After the test, the thickness of sample d_2 was measured, which in any case did not differ from the thickness d_1 for more than 0.3 mm indicating that the polymerization of each sample was complete.

Statistical analysis was performed in Minitab 16 (Minitab Inc., State College, PA, USA) using one-way analysis of variance (ANOVA) at the significance level $\alpha=0.05$. As significant difference in variance, or increased variance with higher values of measured working time was registered, data transformation 1/y was performed in order to meet the necessary conditions for ANOVA.

RESULTS

Of all tested composites, only Herculite XRV Ultra passed the test of sensitivity to ambient light, because after the exposure of 60 sec it maintained homogeneity (Figure 2a). Other composites showed signs of material inhomogeneity in the form of the loss of integrity, ridges, cracks and voids (Figure 2b-e). All these signs can be considered as indicators of polymerization.

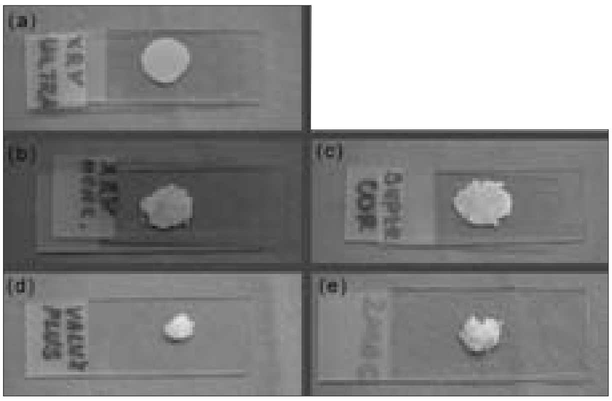
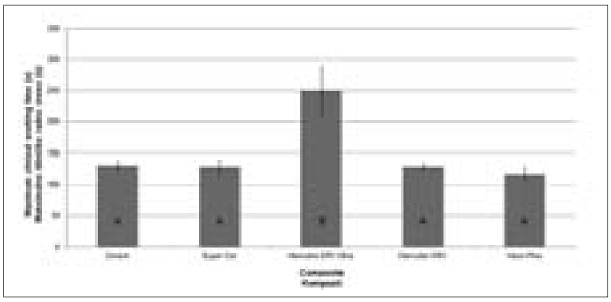


Figure 2a-e. The structure of material after the exposure of 60 sec to dental unit light: (a) Herculite XRV Ultra; (b) Herculite XRV; (c) Super Cor; (d) Valux Plus; (e) Zmack

Slika 2a-e. Struktura materijala nakon ekspozicije od 60 sekundi svetlu reflektora: (a) *Herculite XRV Ultra*; (b) *Herculite XRV*; (c) *Super Cor*; (d) *Valux Plus*; (e) *Zmack*

Graph 1 shows means and standard deviations of the maximum working time for each tested composite. Herculite XRV Ultra showed significantly longer working time than other composites, the average was 250 sec ($p<0.05$). Mean values for other materials ranged from 117 to 131 sec and there was no statistically significant difference between them ($p>0.05$).



Graph 1. Clinical working time for the tested composites (mean values and standard deviations)

Grafikon 1. Kliničko radno vreme ispitivanih kompozita (srednje vrednosti i standardne devijacije)

DISCUSSION

The first null hypothesis was accepted because composites did not pass the ISO standard test in simulated clinical conditions. The second null hypothesis was rejected, because there were significant differences in clinical working time between the tested composites.

Of all tested materials, only Herculite XRV Ultra maintained homogeneity after 60 sec of exposure to ambient light, which consisted mainly of dental unit light and less of natural daylight. Other composite demonstrated signs of inhomogeneity, which was an indicator that polymerization has started. In these cases, it was considered that material did not pass the test i.e., it showed unsatisfactory sensitivity to ambient light as required by the ISO standard 4049:2000. However, there is no data on the relation between the degree of conversion of material and signs of inhomogeneity, therefore, the minimum value of conversion after which inhomogeneity becomes apparent in material is not known.

It was not possible to compare the results of the current study with similar contemporary studies because none study has been found in the literature regarding modern composite materials. In a recent study by Kupka et al. [8] the sensitivity of experimental bioadhesive mixtures for bonding composites and metals to ambient light was tested. Experimental bioadhesives showed signs of polymerization after 2-3 min. Initiator system for these bioadhesive systems consisted of camphorquinone and co-initiator dimethylaminoethyl methacrylate, similar to the commercial adhesives and composites. Despite the differences in the composition of materials from the study of Kupke et al. [8] and the current study, the results confirmed that materials based on resins that contained camphorquinone and co-initiator showed sensitivity to ambient white light, and signs of polymerization were observed after the exposure of 1-3 min.

The results of the current study can be discussed in light of several previous studies, but with note that those are different materials considering that in the last 10-15 years composites have undergone significant changes in the composition. In the study of Dionysopoulos & Watts [7] all tested materials showed signs of inhomogeneity at different time intervals longer than 60 sec after the exposure to dental unit light of 10 klx. Although the composites mentioned in that study are no longer in use in practice, it was observed that the composite Herculite XR, a precursor of modern composite Herculite XRV, showed signs of inhomogeneity after much longer time than all other composites intended for restoration of posterior teeth. Similarly, in the current study, Herculite XRV Ultra was more resistant to ambient light than other tested composites.

The study of Dlugokinski et al. [6] measured the conversion of composites after the exposure to dental unit light for several minutes. For the Herculite XRV the conversion of 52% of maximum conversion for this material was registered after 2 min of exposure. Other composites tested in this study, Silux Plus and Prisma TPH showed considerable variability, the conversion of Silux Plus being about 30% and Prisma TPH about 60% of maximum conversion for these materials. Relatively high degrees of conversion for two of three studied composites indicate that blue light from the spectrum of dental unit light, although of less intensity than in lamps for light activation of composite, has considerable potential to initiate polymerization when materials require prolonged working time [6].

All materials from the current study are registered on the market and are in commercial use, therefore indirectly can be concluded that they passed required ISO standard procedure. In our study, only Herculite XRV Ultra met the required criterion. A possible reason for this discrepancy may be the difference in the measuring procedure according to the ISO standard 4049:2000 and modified measurements in simulated clinical conditions, as it was concluded by Lane et al. [5]. In addition, although the packages of materials used in our study were open only for the purpose of this research, and stored in the refrigerator during the experiment, i.e., not previously used in clinical practice, the possibility of alteration of the chemical composition due to the longer or improper storage by supplier must be considered.

The current study also investigated clinical working time of these composites in order to determine when material loses its plasticity and becomes unsuitable for shaping by hand instruments. For this experiment a device (weight of 160 g) with standardized pressure was designed. This weight was chosen because the pilot study showed that the pressure while shaping dental composites does not exceed this value.

The ethanol shaking test as an indicator of the quality of polymerization was used in the current study. Similarly, Kleverlaan & De Gee [9] in their study used the acetone shaking test due to higher precision as compared to the ISO standard test that involves removal of non polymerized part by spatula or knife. Due to the lack of acetone of analytical grade, ethanol with purity ≥ 99% (Sigma-Aldrich Chemie GmbH, Munich, Germany) was used. Its ability to dissolve non polymerized composite was confirmed before the experiment. Deviation of up to 0.3 mm of the sample thickness before and after mixing was tolerated due to the solubility of the surface layer of composite. It is known that atmospheric oxygen has a negative effect on the polymerization of methacrylates. Even after polymerization using blue light the surface layer remains poorly polymerized [10].

The maximum working time for Herculite XRV Ultra exceeded 4 min during the exposure to ambient light. This time was significantly longer compared to other materials between which there was no statistically significant difference. Measured time for other four composites of about 2 min indicated that polymerization due to the exposure to ambient light and dental unit light, can complicate clinical work in situations where application and shaping require longer working time. Such situations can be expected in cases of inexperienced or less experienced therapists, such as students at undergraduate studies. Although there are numerous data in the literature regarding the trends in education in terms of posterior teeth restoration with composites [11, 12, 13], there is no information available about the time that students need to apply and shape the composite.

Technical documentation from the manufacturers of the tested materials did not list the precise amount of substances that affect reactivity and chemical stability of composites, such as initiators, inhibitors, stabilizers or catalysts. Studies have shown that small variations in the composition or concentration of these substances can have a significant effect on reactivity and conversion of composite materials [14, 15, 16]. In addition, the composition of organic matrix differs qualitatively and quantitatively in different composites. It is also known that there is a difference between acrylic monomers regarding polymerization rate due to their stereometric characteristics [17]. Therefore, it cannot be stated with certainty why Herculite XRV Ultra had longer working time than other composites. It is important to mention that all tested materials belong to the group of micro-hybrid composites, whereas Herculite XRV Ultra belong to the group of nano-hybrid composite, because it contains nano-fillers. It is well known that the addition of fillers reduces the degree of conversion of composites due to reduced mobility of growing polymer chains and limited penetration of light [18, 19]. Since the addition of nano-particles increases the weight and volume percentage of inorganic phase [20, 21], it is possible that these effects of fillers contributed to slower polymerization of nano-hybrid material Herculite XRV Ultra.

CONCLUSION

Susceptibility of four micro-hybrid and one nano-hybrid composite to ambient light, which consisted of dental unit light and natural daylight, was assessed according to the modified protocol of ISO standard 4049:2000. After 60 sec of exposure to ambient light, micro-hybrid composites showed different signs of inhomogeneity (loss of continuity, cracks, crevices and voids), which indicated initiated polymerization of material. Only nano-hybrid composite Herculite XRV Ultra maintained homogeneous structure and passed the test of sensitivity to ambient light.

In addition to lower sensitivity to ambient light, Herculite XRV Ultra also showed longer clinical working time compared to micro-hybrid composites that lost their plasticity due to polymerization that started about 2 min from the beginning of exposure to ambient light. Therefore, Herculite XRV Ultra can be recommended for clinical procedures that require prolonged working time with material in plastic condition.

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NOTE

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STATEMENT

The authors declare that they have neither any financial interest in this study, nor they are connected with the companies whose materials were used in the study.

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Osetljivost kompozitnih materijala na ambijentalnu svetlost i kliničko radno vreme

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KRATAK SADRŽAJ

Uvod Cilj rada je bio da se utvrdi osetljivost kompozita na ambijentalnu svetlost prema modifikovanom standardu ISO 4049:2000 i kliničko radno vreme kompozita.

Materijal i metode rada U radu su ispitivani sledeći materijali: nanohibridni *Herculite XRV Ultra* (*Kerr*) i mikrohibridni *Herculite XRV* (*Kerr*), *Zmack* (*Zhermack*), *SuperCor* (*SpofaDental*) i *Valux Plus* (*3M ESPE*). Po pet uzoraka u svakoj grupi je izlagano ambijentalnoj svetlosti od 8000 lx, koja se sastojala od svetlosti dentalnog reflektora i prirodne svetlosti. Nakon 60 sekundi ekspozicije svaki uzorak je vizuelno ispitivan u pogledu znakova nehomogenosti, pa ukoliko ih je bilo, smatralo se da materijal nije prošao test. Kliničko radno vreme je ispitivano utiskivanjem standardizovanog utiskivača u uzorak kompozita debljine oko 2 mm tokom izlaganja istoj ambijentalnoj svetlosti. Kontrola završene polimerizacije izvršena je rastvaranjem uzoraka u etanolu i merenjem debljine uzoraka pre i posle testa. Rezultati su statistički obrađeni primenom jednofaktorske analize varijanse (ANOVA) na nivou značajnosti od 0,05.

Rezultati Samo je *Herculite XRV Ultra* prošao test osetljivosti na ambijentalnu svetlost, dok su kod ostalih kompozita uočeni znaci nehomogenosti materijala. *Herculite XRV Ultra* je pokazao i statistički značajno duže radno vreme od drugih kompozita, prosečno 250 sekundi ($p<0,05$). Srednje vrednosti radnog vremena drugih materijala bile su 117–131 sekunda, a između njih nisu postojale značajne razlike ($p>0,05$).

Zaključak Zbog manje osetljivosti na ambijentalnu svetlost i značajno dužeg kliničkog radnog vremena u poređenju sa mikrohibridnim kompozitima, *Herculite XRV Ultra* se može preporučiti za kliničke procedure koje zahtevaju produženo vreme rada s plastičnim materijalom.

Ključne reči: ambijentalna svetlost; ISO 4049:2000; kompozit; kliničko radno vreme; polimerizacija

UVOD

Slojevita tehnika primene kompozitnih materijala se i danas preporučuje za restauraciju kaviteta, jer rezultati ukazuju na jaču vezu sa zubnim tkivima i niži kontrakcioni stres u poređenju sa tzv. *bulk* tehnikom primene [1, 2]. Debljina svakog sloja ne bi trebalo da bude veća od 2 mm, u skladu s preporukama proizvođača, a broj slojeva zavisi od dubine kaviteta. Zahvaljujući stalnom unapređenju mehaničkih osobina kompozita, u savremenoj restaurativnoj stomatologiji njihova primena je proširena i na ekstremne slučajeve oštećenja krunice zuba, kada se koriste za izradu nadogradnji, kao i za rekonstrukciju zona opterećenja [3]. Jasno je da ovakvi slučajevi produžuju radno vreme, pa osetljivost kompozita na ambijentalnu svetlost dobija na značaju.

Osetljivost kompozitnih materijala na ambijentalnu svetlost je jedna od osobina koje se ispituju standardom ISO 4049:2000 pre registrovanja novih materijala na tržištu [4]. Osetljivost materijala se proverava vizuelno nakon 60 sekundi izlaganja svetlosti ksenonske lampe jačine 8000 lx s filterima u tamnoj komori. Gubitak homogenosti materijala ukazuje na to da je otpočela polimerizacija materijala pod uticajem pomenutog izvora svetlosti. ISO standard predviđa jednostavan kriterijum za ocenu materijala koji glasi „prošao/nije prošao test“.

U literaturi postoji svega nekoliko studija o osetljivosti kompozitnih materijala na ambijentalnu svetlost. Lejn (*Lane*) i saradnici [5] su naveli da ISO standard ne odgovara kliničkim uslovima, te da postoje značajne razlike u pogledu radnog vremena materijala izmerenog tokom postupka izvedenog prema ovom standardu i u simuliranim kliničkim uslovima. U ovim uslovima radno vreme četiri ispitivana kompozita bilo je kraće od 60 sekundi nakon izlaganja svetlosti reflektora, dok je ovo vreme prema proceduri ISO standarda bilo i do tri puta duže [5]. Uprkos ovim rezultatima, postupak prema ISO standardu u pogledu osetljivosti kompozita na ambijentalnu svetlost ostao je nepromenjen.

Dlugokinski (*Dlugokinski*) i saradnici [6] su ispitivali stepen polimerizacije tri kompozitna materijala nakon izlaganja ambijentalnoj svetlosti sa eliminacijom fluorescentne svetlosti i bez eliminacije. Dionisopulos (*Dionysopoulos*) i Vots (*Watts*) [7] su poredili osetljivost 29 nijansi 14 kompozitnih materijala na svetlost kliničkog reflektora u modifikovanom protokolu ISO standarda. Rezultati ovih istraživanja pokazuju da ambijentalna svetlost može značajno uticati na kliničko radno vreme kompozita. Stomatolozi bi trebalo da poznaju i ovu osobinu materijala koji koriste, jer klinički rezultati mogu biti ugroženi u situacijama koje zahtevaju produženo radno vreme.

Cilj ovog istraživanja bio je da se ispita osetljivost savremenih kompozitnih materijala na ambijentalnu svetlost i radno vreme u simuliranim kliničkim uslovima. Nulte hipoteze su glasile: (1) ispitivani kompoziti ne zadovoljavaju zahteve ISO standarda i (2) nema značajne razlike između ispitivanih kompozita u pogledu kliničkog radnog vremena.

MATERIJAL I METODE RADA

U istraživanju je korišćeno pet vrsta kompozitnih materijala čiji je sastav naveden u tabeli 1. Za svaki test je pripremljeno po pet uzoraka u svakoj grupi. Eksperiment je urađen u kliničkoj sali, a korišćen je reflektor *Fona A1* (*Sirona Dental Systems, Foshan Co Ltd*, Kina). Reflektor je postavljen upravno na radni pult stomatološke stolice, na udaljenosti koja je obezbeđivala osvetljenost zone radnog pulta od 8000±1000 lx u skladu sa zahtevom standarda ISO 4049:2000. Ukupna osvetljenost je većim delom poticala od dentalnog reflektora, a manjim delom od dnevne svetlosti prostorije. Osvetljenost je kontrolisana luksmetrom (*Mastech MS8209, Dongguan Huayi Mastech Co*, Kina).

Na radnom pultu, u zoni osvetljenja, na čisto mikroskopsko staklo naneto je oko 0,1 g kompozita plastičnom kašičicom sfernog oblika koji je izlagan svetlu reflektora u trajanju od 60 s. Zatim je preko materijala pritisnuta druga staklena pločica i vršen pokret smicanja. Nakon toga je vizuelno ispitivana homogenost materijala.

U drugom delu istraživanja ispitivano je kliničko radno vreme kompozita. Na mikroskopsko staklo, koje je služilo kao podloga, nanet je materijal na način i pod istim uslovima kao u prethodnom delu eksperimenta. Naneta masa je zaravnjena šesticom u uzorak debljine oko 2 mm i izlagana svetlosti reflektora. Za ovaj deo eksperimenta konstruisan je uređaj za standardizovani pritisak, shematski prikazan na slici 1. Teg mase 160 g fiksiran je na utiskivač superlepkom, kako bi se simulirao pritisak stomatologa prilikom oblikovanja kompozitnog materijala u kavitetu. Širina vrha utiskivača je bila 0,5 mm. Utiskivač je postavljen neposredno iznad uzorka, a zatim pušten da se pod uticajem svoje mase utisne u materijal. Utiskivanje je vršeno na po 15 s, a nakon svakog utiskivanja proveravana je dubina utisnuća sondom sa indikatorom dubine od 0,5 mm. Za svaki uzorak su urađena po tri merenja, pri čemu je svako naredno merenje započeto sa po 5 s razlike u odnosu na prethodna. Dubina utisnuća manja od 0,5 mm ukazivala je na nemogućnost daljeg utiskivanja usled završene polimerizacije uzorka i to vreme je označavano kao maksimalno kliničko radno vreme. Nakon toga je kljunastim pomičnim merilom sa digitalnim ekranom preciznosti 0,01 mm izmerena debljina uzorka d_1, a zatim je urađen test rastvaranja uzorka u etanolu.

Testom rastvaranja u etanolu kontrolisana je polimerizacija uzoraka pod uticajem ambijentalne svetlosti. Prethodno je ovaj test potvrđen tako što je u praznu čistu kapsulu amalgama stavljeno 0,1 g nepolimerizovanog kompozita, odnosno količina koja je korišćena za pripremu svakog uzorka u eksperimentalnom delu. U kapsulu je zatim dodat 1 ml etanola (*Sigma-Aldrich Chemie GmbH*, Minhen, Nemačka), a neposredno nakon toga vršeno je mešanje u amalgamatoru u trajanju od 20 s. Ovo vreme je bilo dovoljno da etanol potpuno rastvori ispitivani uzorak kompozita. Analogno ovom postupku, svaki uzorak kompozita je nakon izmerenog maksimalnog radnog vremena stavljen u kapsulu u koju je dodat 1 ml etanola, a zatim mešan 20 s u amalgamatoru. Nakon toga je izmerena debljina uzorka d_2, koja ni u jednom slučaju nije odstupala od debljine d_1 za više od 0,3 mm, ukazujući na to da je polimerizacija svakog uzorka bila kompletna.

Statistička analiza je urađena u programu Minitab 16 (*Minitab Inc., State College, PA, USA*) primenom jednofaktorske analize varijanse (ANOVA) na nivou značajnosti $\alpha=0,05$. Kako je zabeležena razlika u varijansi, odnosno porast varijanse sa većim vrednostima izmerenog vremena, urađena je transformacija podataka po tipu 1/y, da bi bili zadovoljeni neophodni preduslovi za jednofaktorsku ANOVA.

REZULTATI

Od svih ispitivanih kompozita samo je *Herculite XRV Ultra* prošao test osetljivosti na ambijentalnu svetlost, jer je nakon ekspozicije od 60 s materijal zadržao homogenost (Slika 2a). Ostali kompoziti su pokazali znake nehomogenosti materijala u vidu gubitka integriteta, brazdi, naprslina i blazni (Slike 2b-e). Svi ovi znaci se mogu smatrati pokazateljima započete polimerizacije.

Na grafikonu 1 su prikazane srednje vrednosti i standardne devijacije maksimalnog radnog vremena za svaki ispitivani kompozit. *Herculite XRV Ultra* je pokazao statistički značajno duže radno vreme od drugih kompozita, prosečno 250 s ($p<0,05$). Srednje vrednosti kod drugih materijala bile su od 117 s do 131 s i između njih nije uočena statistički značajna razlika ($p>0,05$).

DISKUSIJA

Prva nulta hipoteza je prihvaćena jer ispitivani kompoziti nisu prošli ISO standard u simuliranim kliničkim uslovima. Druga nulta hipoteza je odbačena jer su uočene značajne razlike u pogledu kliničkog radnog vremena između ispitivanih kompozita.

Od svih ispitivanih kompozita samo je *Herculite XRV Ultra* zadržao homogenost nakon 60 s izlaganja ambijentalnoj svetlosti, koja se sastojala većim delom od svetlosti reflektora i manjim delom od prirodne dnevne svetlosti. Kod ostalih kompozita uočeni su znaci nehomogenosti, koja je pokazatelj započete polimerizacije. U tim slučajevima se smatra da materijal nije prošao test, odnosno da je pokazao nezadovoljavajuću osetljivost na ambijentalnu svetlost koja je predviđena standardom ISO 4049:2000. Međutim, nema podataka o povezanosti stepena konverzije materijala i znakova nehomogenosti, pa nije poznata minimalna vrednost konverzije nakon koje nehomogenost materijala postaje uočljiva.

Nije moguće uporediti rezultate ovog istraživanja sa sličnim savremenim studijama, jer u literaturi nisu nađeni podaci za savremene kompozitne materijale. U studiji Kupke (*Kupka*) i saradnika [8] osetljivost na ambijentalnu svetlost ispitivana je kod eksperimentalnih bioadhezivnih smesa namenjenih za adheziju kompozita i metala. Eksperimentalni bioadhezivi su pokazivali znake započete polimerizacije posle 2–3 minuta. Inicijatorski sistem ovih bioadheziva činili su kamforhinon i koinicijator dimetilaminoetil-metakrilat, slično kao i kod komercijalnih adheziva i kompozita. Uprkos razlikama u sastavu materijala iz studije Kupke i saradnika [8] i našeg istraživanja, rezultati su pokazali da materijali na bazi smola koji sadrže inicijatorski sistem kamforhinona i koinicijatora ispoljavaju osetljivost na belu ambijentalnu svetlost, a znaci polimerizacije se uočavaju već nakon izlaganja svetlosti 1–3 minuta.

Rezultati naše studije se mogu diskutovati u svetlu nekoliko ranijih studija, s tim da se ima u vidu da je reč o različitim materijalima, s obzirom na to da su u poslednjih 10–15 godina kompoziti pretrpeli znatne izmene sastava. U studiji Dionisopulosa i Votsa [7] svi ispitivani materijali su pokazivali znake nehomogenosti u različitim vremenskim intervalima dužim od 60 s nakon izlaganja svetlosti reflektora od 10 klx. Iako se kompoziti iz pomenute studije više ne koriste u praksi, zapaženo je da je kompozit *Herculite XR*, preteča savremenih kompozita *Herculite XRV*, znake nehomogenosti ispoljio posle znatno dužeg vremena nego svi drugi kompoziti namenjeni restauraciji bočnih zuba. Slično tome, u našem istraživanju se *Herculite XRV Ultra* pokazao otpornijim na ambijentalnu svetlost nego drugi ispitivani kompoziti.

U istraživanju Dlugokinskog i saradnika [6] merena je konverzija kompozita nakon višeminutnog izlaganja svetlosti reflektora. Kod kompozita *Herculite XRV* utvrđena je konverzija od 52% maksimalne konverzije ovog materijala već nakon dve minute ekspozicije [6]. Drugi ispitivani kompoziti u ovoj studiji, *Silux Plus* i *Prisma TPH*, pokazali su znatnu varijabilnost rezultata, pa je tako konverzija *Silux Plus* bila oko 30%, a *Prisma TPH* oko 60% maksimalne konverzije ovih materijala. Relativno visok stepen konverzije dva od tri ispitivana kompozita ukazuje na to da plava svetlost iz spektra dentalnog reflektora, iako manjeg intenziteta nego kod lampi namenjenih za svetlosnu polimerizaciju, ima znatan potencijal da pokrene polimerizaciju kada materijal zahteva produženo radno vreme [6].

Svi materijali koje smo ispitivali registrovani su na našem tržištu i nalaze se u komercijalnoj upotrebi, pa se posredno može zaključiti da su prošli zahtevanu ISO standardnu proceduru. Ipak, samo je *Herculite XRV Ultra* ispunio traženi kriterijum. Mogući razlog za ovo odstupanje je razlika u postupku merenja po standardu ISO 4049:2000 i modifikovanog merenja u simuliranim kliničkim uslovima, kako su to zaključili Lejn i saradnici [5]. Osim toga, iako su materijali u našem istraživanju otvoreni tek za potrebe ovog istraživanja i čuvani u frižideru tokom eksperimenta (nisu prethodno korišćeni u kliničkoj praksi), u obzir se mora uzeti mogućnost alteracije hemijskog sastava zbog dugog ili neadekvatnog prethodnog čuvanja kod dobavljača.

U studiji je ispitivano i kliničko radno vreme kompozita, da bi se utvrdilo kada materijal gubi plastičnost i postaje nemoguće oblikovati ga ručnim instrumentima. Za ovaj eksperiment konstruisan je uređaj za standardizovani pritisak sa tegom od 160 g, jer je tokom pilot-studije pre početka eksperimenta utvrđeno da pritisak stomatologa tokom modelovanja kompozita ne prelazi ovu vrednost.

U radu je primenjen test mešanja u etanolu kao indikator kvaliteta polimerizacije. U radu Kleverlana (*Kleverlaan*) i De Gea (*de Gee*) [9] korišćen je sličan test mešanja u acetonu zbog veće preciznosti rezultata u odnosu na test iz ISO standarda koji podrazumeva uklanjanje nepolimerizovanog dela špatulom ili skalpelom. Usled nedostatka acetona analitičke čistoće, korišćen je etanol čistoće ≥99% (*Sigma-Aldrich Chemie GmbH*, Minhen, Nemačka), čija je sposobnost rastvaranja nepolimerizovanog kompozita potvrđena pre početka eksperimenta. Tolerisano je odstupanje do 0,3 mm u debljini uzorka pre i posle mešanja zbog rastvorljivosti površinskog sloja kompozita. Poznat je loš uticaj atmosferskog kiseonika na polimerizaciju metakrilata, a i nakon svetlosne polimerizacije plavom svetlošću površinski sloj ostaje slabije polimerizovan [10].

Maksimalno radno vreme za *Herculite XRV Ultra* premašivalo je četiri minuta tokom izlaganja ambijentalnoj svetlosti. Ovo vreme je bilo značajno duže u odnosu na ostale materijale, između kojih nije uočena statistički značajna razlika. Izmerena vremena za preostala četiri kompozita od oko dva minuta ukazuju na to da polimerizacija usled izlaganja ambijentalnom svetlu, odnosno svetlu reflektora, može otežati klinički rad kada primena kompozita i modelovanje iziskuju duže vreme. Takve situacije se mogu očekivati u radu neiskusnih ili manje iskusnih terapeuta, poput studenata osnovnih studija. Iako u literaturi postoje brojni podaci o trendovima u nastavi u pogledu restauracije bočnih zuba kompozitima [11, 12, 13], nema dostupnih informacija o vremenu koje je potrebno studentima za primenu i modelovanje kompozita.

U tehničkoj dokumentaciji proizvođača ispitanih kompozita nije navedena tačna količina supstanci koje utiču na reaktivnost ili hemijsku stabilnost kompozita, poput inicijatora, inhibitora, stabilizatora ili katalizatora. Studije pokazuju da male varijacije u sastavu ili koncentraciji ovih supstanci mogu značajno uticati na reaktivnost i konverziju kompozitnih materijala [14, 15, 16]. Osim toga, i sastav organskog matriksa se razlikuje kvalitativno i kvantitativno, a poznato je da između monomera postoji razlika u brzini umrežavanja zbog stereometrijskih osobina metakrilnih monomera [17]. Zbog toga se ne može sa sigurnošću tvrditi zbog čega je *Herculite XRV Ultra* imao duže radno vreme od drugih kompozita. Važno je pomenuti i da svi ispitani kompoziti pripadaju grupi mikrohibridnih, a da je *Herculite XRV Ultra* nanohibridni kompozit, jer sadrži i nanočestične punioce. Poznato je da dodavanje punilaca snižava stepen konverzije kompozita zbog smanjene mobilnosti rastućih polimernih lanaca i ograničenog prodora svetla [18, 19]. S obzirom na to da sa dodatkom nanočestica raste i maseni i zapreminski procenat neorganske faze [20, 21], moguće je da pomenuti efekti punilaca doprinose sporijoj polimerizaciji nanohibridnog materijala *Herculite XRV Ultra*.

ZAKLJUČAK

Ispitivanje osetljivosti četiri mikrohibridna i jednog nanohibridnog kompozita na ambijentalnu svetlost, koja se sastojala od svetlosti dentalnog reflektora i prirodne dnevne svetlosti, urađeno je po modifikovanom protokolu standarda ISO 4049:2000. Nakon 60 sekundi izlaganja ambijentalnoj svetlosti, mikrohibridni kompoziti su ispoljili različite znake nehomogenosti (gubitak kontinuiteta, brazde, blazne i pukotine), koji su ukazivali na započetu polimerizaciju materijala. Samo je nanohibridni kompozit *Herculite XRV Ultra* zadržao homogenu strukturu i prošao test osetljivosti na ambijentalnu svetlost.

Osim manje osetljivosti na ambijentalnu svetlost, *Herculite XRV Ultra* je imao i dvostruko duže kliničko radno vreme u poređenju s mikrohibridnim kompozitima kod kojih je potpuni gubitak plastičnosti usled polimerizacije nastupao oko dva minuta od početka izlaganja ambijentalnoj svetlosti. Zbog toga se *Herculite XRV Ultra* može preporučiti za kliničke postupke koji zahtevaju produženo vreme rada s plastičnim materijalom.

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