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IZVORNI ZNANSTVENI RAD
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Analiza parametra translucencije staklokeramike izrađene različitim tehnološkim postupcima

Analysis of Translucency Parameter of Glass-Ceramics Fabricated by Different Techniques

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Sažetak

Svrha: Analizirati parametar translucencije (TP vrijednosti) staklokeramika izrađenih različitim tehnološkim postupcima te ispitati kako na TP vrijednosti utječu korozivna sredstva. **Materijali i metode:** Izrađena su po tri uzorka IPS e.max keramike (Ivoclar Vivadent, Schaan, Lihtenštajn) u trima bojama (A2, C2 i B3) s trima različitim tehnologijama izrade (slojevanje – e.max Ceram Dentin; toplo-tlačna tehnika – e.max Press; strojno – e.max CAD). Uzorci su bili u obliku pločica dimenzija 10 mm x 12 mm x 0,8 mm. Spektrofotometrom (X-Rite DTP 20 Pulse, Neu Isenburg, Njemačka) izmjerene su CIE $L^*a^*b^*$ vrijednosti za izračun parametra tanslucencije (TP vrijednost) prije i poslije izlaganja 4-postotnoj octenoj kiselini na 80 °C tijekom 16 sati (ISO 6872). Statistički podatci obrađeni su programom IBM SPSS 22. **Rezultati:** Značajno najmanje TP vrijednosti imao je IPS e.max Ceram Dentin, a najveće IPS e.max Press u svim bojama, prije i poslije izlaganja kiselini ($p<0,001$). Razlika u TP vrijednostima između boja bila je vidljiva unutar materijala IPS e.max Ceram Dentin prije i poslije izlaganja kiselini, uz veliku snagu efekta ($p<0,001$; $\eta 2 = 0,702$ i $0,741$) te pri primjeni materijala IPS e.max Press ($p<0,001$, snaga efekta 0,547 i 0,576). Strojno izrađeni uzorci pokazali su ujednačene TP vrijednosti. Izlaganje korozivnom sredstvu nije rezultiralo statistički značajnim promjenama TP vrijednosti ni za jedan materijal. **Zaključak:** Različite staklokeramike pokazale su značajne razlike u TP vrijednostima i prema tehnološkom postupku izrade i prema različitim bojama. Izlaganje korozivnom sredstvu nije rezultiralo statistički značajnim promjenama TP vrijednosti.

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Uvod

Estetika je postala primarni kriterij uspješnosti fiksnoprotetske terapije, posebice kada je riječ o sanaciji prednjeg dijela zubnog niza. Cilj estetske stomatologije jest izrada nadomjestka koji se bojom ne razlikuje od prirodnih zuba (1, 2). Stoga su optička svojstva gradivnih materijala iznimno važna. Keramički materijali zbog svojih izvrsnih optičkih svojstava smatraju se u stomatološkoj protetici estetski superiornima (3).

Boja i izgled zuba kompleksan su fenomen koji uključuje mnogobrojne čimbenike (4 – 6). Prirodni zubi nisu jednolike građe – od cervikalnog prema incizalnom dijelu obilježavaju ih različite boje i stupnjevi translucencije. Translucenci-

Introduction

Aesthetics has become a primary criterion for successful fixed prosthodontics treatment, especially regarding restoration of the front teeth. The aim of aesthetic dentistry is to create a restoration which does not differ in colour from natural teeth (1,2). Therefore, optical properties of restorative materials are of exceptional importance. In dental prosthodontics, ceramic materials are considered superior materials to composites from the aesthetical point of view because of their excellent optical properties (3).

The colour and appearance of teeth is a complex phenomenon which includes a number of factors (4-6). Natural teeth are not of a uniform structure and are characterised ja je relativna količina propuštanja svjetlosti kroz neki objekt (1). Translucencija gradivnog materijala daje nadomjestku prirodnost i vitalnost. Stoga je za optimalne estetske rezultate, uz oponašanje boje, jednako važno oponašati i translucenciju prirodnog zuba (2).

Translucencija dentalne keramike ovisi o interakciji između keramičkog materijala i upadne svjetlosti. Miješana bijela svijetlost na površinama keramičke krunice ponaša se prema fizikalnim zakonima refleksije i refrakcije zbog različite optičke gustoće dvaju sredstava. Tada se dio svjetla reflektira, a dio lomi i prolazi kroz drugo sredstvo. Ako se manja količina svjetlosti reflektira, a veći dio propušta (lomi), nadomjestak će biti transparentan. Količina apsorbirane svjetlosti reflektira se, ili se pak prenese, ovisno o odnosu upadnih valnih dužina svjetlosti i veličini te broju čestica (7 – 10). Ako je materijal potpuno mutan (opakni), parametar translucencije (TP vrijednost) je oko nule. Samim time veća TP vrijednost znači i veću translucenciju (11, 12).

Staklokeramika je materijal koji izvrsno oponaša zubno tkivo i ima najbolja optička svojstva među estetskim protetskim materijalima (13). Njezina prednost nad drugim gradivnim materijalima jest translucencija koja dopušta prolazak svjetlosti isto kao kod prirodnog zuba. Nastala je razvojem silikatne tj. glinične keramike postupcima kontrolirane kristalizacije stakla. Odlikuje se velikom mehaničkom otpornošću, čvrstoćom i postojanošću na temperaturne promjene (14). Tehnološki postupak izrade staklokeramičkih materijala temelji se na izradi cijelog nadomjestka od istog materijala ili izradi nadomjestka koji se sastoji od osnovne konstrukcije na koju se nanose slojevi obložne keramike. Osnovne konstrukcije su više ili manje translucentne, ali nemaju veliku sličnost s prirodnim zubom (15). U zubotehničkom laboratoriju staklokeramički protetski radovi mogu se izraditi primjenom triju osnovnih tehnoloških postupaka – slojevanjem, te toplo-tlačnom ili strojnom obradom (CAD/CAM) (16). Kvaliteta keramičkog materijala ovisi o sastavnicama – o vrsti i količini staklene matrice, o vrsti, količini, veličini i distribuciji zrna, o tehnikama izrade i obrade nadomjestka, o ciklusima i temperaturi pečenja te o hlađenju (6, 17). Budući da su svojstva svakog materijala u funkciji njegove strukture, pogreške nastale tijekom izrade ili obrade nadomjestka u zubotehničkom laboratoriju učinit će mikrostrukturu keramike nepravilnom te rezultirati neželjenim učinkom (14).

Osim svojstava gradivnog materijala, na postojanost boje nadomjestka utječe i okoliš, odnosno usna šupljina. U tom kontekstu treba istaknuti da se od gradivnog materijala, odnosno nadomjestka, očekuju dugoročno stabilna optička svojstva u usnoj šupljini. Konstantna izloženost vodenom mediju, promjeni pH vrijednosti unošenjem različitih pića i hrane te sredstvima za čišćenje usne šupljine, uz dinamička opterećenja, nastaju tribokorozijske promjene na površini nadomjestka. Korozija u ustima je, osim oblika elektrokemijske korozije i galvanizma, i korozija uvjetovana kiselim produktima mikroorganizama. Veća zastupljenost staklene matrice znači i veću podložnost korozijskim procesima i propadanju nadomjestka (18, 19).

Suvremeni dentalni materijali, na temelju europskih i američkih standarda, podliježu kontroli i ispitivanju glede

by different colour and grades of translucency from the cervical to the incisal part. Translucency is the relative amount of passage of light through an object (1). Translucency of the restorative material gives natural appearance and vitality to the restoration. Therefore, in order to achieve optimum aesthetic results, in addition to mimicking the colour of natural teeth, it is equally important to mimic their translucency (2).

Translucency of dental ceramics depends on the interaction between the ceramic material and the incident light. Mixed white light on the surfaces of a ceramic crown conducts itself in accordance with the physical laws of reflection and refraction because of different optical densities of these two media. Thus, a part of the light is reflected and a part is refracted and passes through the other medium. If a smaller amount of light is reflected, and a greater part passes through (is refracted), the restoration will be transparent. The amount of light that is absorbed, reflected or transmitted depends on the relations between incident light wave lengths and the size and the number of particles (7-10). When the material is absolutely turbid (opaque), translucency (TP value) is about zero. Consequently, the higher the TP value the greater the translucency of a material (11, 12).

Glass-ceramics is a material that mimics dental tissue to a great extent, and has the best optical properties among all prosthetic materials (13). The advantage of glass-ceramic over other restorative materials is its translucency, which allows the passage of light in the same way as in natural teeth. It was created by developing silicate ceramics by procedures of controlled glass crystallisation. It is characterised by great mechanical resistance, hardness and stability to temperature changes (14). The fabrication of glass-ceramic restorations is based on the fabrication of the entire restoration from the same material or fabrication of the core and layers of veneering ceramics. The core is more or less translucent, but does not have much similarity with natural teeth (15). Glass-ceramic restorations can be fabricated in a dental laboratory by using three fundamental techniques – layering, heat-pressing and computer-aided design and computer-aided manufacturing (CAD/CAM) (16). The quality of a ceramic material depends on its components; type and amount of glass matrix and the type, amount, size and distribution of grains, techniques of fabrication and treatment of the restoration, and cycles and temperature of firing and cooling (6, 17). Since properties of any material are in the function of its structure, errors occurring during the fabrication or treatment of the restoration in the dental laboratory will make the microstructure of ceramics irregular and will result in unwanted effects (14).

Apart from the properties of the restorative material itself, the environment, i.e. the oral cavity, also affects the stability of the restoration. In this context it should be stressed that restorative materials and restorations should possess long-term stable optical properties in the oral cavity. Continuous exposure to an aqueous medium, pH changes due to the intake of various beverages and foods and changes elicited by agents for cleaning of the oral cavity combined with dynamic loads, cause tribocorrosion changes on the surface of the restoration. Corrosion in the mouth, besides being a biokompatibilnosti. No sve te kontrole nisu apsolutni čimbenik stabilnosti materijala u ustima. O keramici se govori kao o korozijski postojanom, tj. biološki inertnom materijalu, no potpuno inertnog materijala nema (20, 21).

Svrha ovog istraživanja bila je uz pomoć spektrofotometra kvantitativno izmjeriti i usporediti optička svojstva translucencije (TP vrijednosti) triju vrsta staklokeramičkih uzoraka u tri različite boje izrađenih trima tehnološkim postupcima prije i poslije izlaganja korozivnom agensu – 4-postotnoj octenoj kiselini (ISO 6872) (22).

Testirale su se sljedeće istraživačke hipoteze:

1. TP vrijednosti staklokeramike bile su ovisne o tehologiji izrade,
2. TP vrijednosti staklokeramike bile su ovisne o boji unutar iste tehnologije izrade,
3. Korozija je utjecala na promjenu TP vrijednosti.

Materijali i postupci

Izrađeno je ukupno devet uzoraka IPS e.max staklokeramike (Ivoclar Vivadent, Schaan, Lihtenštajn) u obliku pločica dimenzija 10 mm x 12 mm x 0,8 mm ± 0,05 mm, po tri boje (A2, C2, B3). Tablica 1. prikazuje ispitivane staklokeramike. Odabrani su valjčići IPS e.max Press i IPS e.max CAD blokovi keramika visoke translucencije (high translucency-HT).

Sve uzorke pripremio je isti dentalni tehničar standardnim postupcima prema uputama proizvođača. Uzorci IPS e.max Press keramike pripremljeni su s pomoću voštanih modela koji su nakon izgaranja toplo-tlačnom tehnikom prešani u kiveti. IPS e.max Ceram Dentin uzorci napravljeni su postupkom kondenzacije, ručnim miješanjem keramičkoga praška i destilirane vode te pečenjem (Programat EP 5000, Ivoclar Vivadent, Schaan, Lihtenštajn). IPS e.max CAD uzorci izrađeni su glodalicom (Amann Girrbach Ceramill Motion 2, Koblach, Austrija) od tvorničkih blokova.

Nakon izrade su neglazirani uzorci polirerima, gumicama i silikonskim karbamidnim diskovima (Komet Dental, Gebspoliranir. Brasseler GmbH & Co. KG, Lemgo, Njemačka) ispolirani do glatke ravne površine te su oprani i očišćeni u ultrazvučnoj kupelji s destiliranom vodom 15 minuta (ISO 3696).

form of electrochemical corrosion and galvanism, also represents corrosion caused by acid products of microorganisms. The greater the share of the glass matrix, the greater the effect of corrosion processes and the deterioration of the restoration has been reported (18, 19).

Contemporary dental materials are subject to control and biocompatibility testing in accordance with European and American standards. However, all controls do not absolutely guarantee the stability of the material in the mouth. Ceramics is considered to be a material resistant to corrosion, i.e. a biologically inert material, however, no material is completely inert (20, 21).

The purpose of this study was, with the aid of a spectrophotometer, to quantitatively measure and compare optical properties of translucency (TP values) on three types of glass-ceramic specimens in three different colours fabricated by three different techniques before and after exposure to a corrosive medium, 4 % acetic acid (ISO 6872) (22).

The following research hypotheses were tested:

1. TP values of glass-ceramics depended on the fabrication technique.
2. TP values of glass-ceramics fabricated by the same technique depended on the colour.
3. Corrosion influenced changes in TP values.

Materials and procedures

Nine specimens of IPS e.max glass-ceramics (Ivoclar Vivadent, Schaan, Liechtenstein) were made in the form of plates with dimensions 10 mm x 12 mm x 0.8 mm ± 0.05 mm in three colours (A2, C2, B3). The tested glass-ceramics specimens are presented in Table 1. IPS e.max Press ingots and IPS e.max CAD ceramics blocks were fabricated in HT (high translucency).

All specimens were prepared by the same dental technician by standard procedures in accordance with the manufacturer's directions. IPS e.max Press specimens were made by using wax models, which were, after firing, pressed in moulds by the technique of heat-pressing of ingots. IPS e.max Ceram Dentin specimens were made by powder condensation technique, by manual mixing of the ceramic powder with distilled water and firing (Programat EP 5000, Ivoclar Vivadent, Schaan, Liechtenstein). IPS e.max CAD specimens were made from factory-made blocks with the use of a milling machine (Amann Girrbach Ceramill Motion 2, Koblach, Austria).

After preparation, unglazed specimens were polished to obtain a smooth and even surface by using polishers, rubbers and silicon carbamide discs (Komet Dental, Gebr. Brasseler GmbH & Co. KG, Lemgo, Germany) and were ultrasoni-

Tablica 1. Prikaz ispitivanih staklokeramika
Table 1 Layout of tested glass-ceramics

Tvornično ime • Trade mark	Vrsta staklokeramike • Glass-ceramics type	Tehnološki postupak izrade • Fabrication technique	Broj uzoraka • Number of specimens
IPS e.max Press	litij-disilikatna • lithium-disilicate	toplo-tlačna tehnika • heat-pressing technique	3
IPS e.max CAD		strojno • CAD/CAM	3
IPS e.max Ceram Dentin	nano-fluorapatitna • nano-fluorapatite	tehnika slojevanja • layering technique	3

U prvom dijelu istraživanja provedena su mjerenja CIE *L*a*b** vrijednosti staklokeramičkih uzoraka u tri različite boje (A2, C2 i B3), izrađenih trima različitim tehnološkim postupcima. *L** označava svjetlinu boje, *a** pomak prema crvenoj (pozitivno) ili zelenoj boji (negativno), a *b** pomak prema žutoj (pozitivno) ili plavoj boji (negativno). Mjerenja su provedena instrumentalno kalibriranim spektrofotometrom i kolorimetrom X-Rite DTP 20 Pulse (mjerna geometrija 45°/0°, standardni promatrač 2°, osvjetljenje D 65). Numerička vrijednost translucencije temelji se na CIE *L*a*b** trodimenzionalnom sustavu te se odnosi na razliku u boji određenog uzorka mjerenog na akromatskim podlogama. Stoga su provedene dvije serije zasebnih mjerenja (mjerenje uzoraka ispod kojih je stavljena standardna bijela podloga, te mjerenje istih uzoraka ispod kojih se nalazi standardna crna podloga). Na svakoj od devet različitih pločica obavljeno je po deset mjerenja na svakoj pozadini, tako da se mijenjao položaj mjerenog uređaja (spektrofotometra) na pet različitih mjesta s jedne strane površine uzorka pa zatim i s druge. Mjerenja su praćena s pomoću računalnog programa ColorShop X koji je integriran s X-Ritovim spektrofotometrima. Nakon izmjerenih CIE *L*a*b** vrijednosti, računala se numerička vrijednost translucencije prema jednadžbi:

$$TP= [(L^*w-L^*b)^2 + (a^*w-a^*b)^2 + (b^*w-b^*b)^2]^{1/2}$$

Svi su uzorci zatim 16 sati bili izloženi 4-postotnoj octenoj kiselini na 80 °C (ISO 6872). Nakon izlaganju korozivnom mediju ponovljena su mjerenja translucencije kako je opisano u prvom dijelu.

Statistička analiza

Normalnost distribucije provjerena je Shapiro-Wilkovim testom. Za usporedbu razlika u TP vrijednostima između vrste materijala i boja rabljene su jednofaktorske analize varijance (ANOVA) sa Student-Newman-Keulsovim post-hoc testom. Za usporedbu TP vrijednosti prije i poslije izlaganja kiselini korišteni su mješoviti tip dvofaktorske ANOVA-e s faktorima intervencija i materijala i trofaktorske ANOVA-e s faktorima intervencija, materijala i boja, te t-testovi za zavisne uzorke za procjenu razlika unutar svakog materijala i svake boje. Snage efekta kvantificirane su s pomoću η2, a za zavisne uzorke rabila se jednadžba $r = \sqrt{(t^2/t^2+df)}$. Statistički podatci obrađeni su programom IBM SPSS 22. Razina značajnosti postavljena je na $p < 0{,}05$.

Rezultati

Tablica 2. prikazuje usporedbu TP vrijednosti staklokeramika različitih boja izrađenih različitim tehnološkim postupcima prije i poslije izlaganja octenoj kiselini.

Značajno najmanje TP vrijednosti imao je IPS e.max Ceram Dentin, a najveće IPS e.max Press (u svim bojama prije i poslije izlaganja kiselini $p < 0{,}001$).

cally washed and cleaned in distilled water for 15 minutes (ISO 3696).

In the first part of the study, measurements of CIE *L*a*b** values of glass-ceramic specimens in three colours (A2, C2 and B3), fabricated by three different fabrication techniques were determined. *L** represents the lightness/darkness of a colour, *a** is a measure of redness (positive) or greenness (negative) and *b** is a measure of yellowness (positive) or blueness (negative). Measurements were conducted instrumentally by using a calibrated spectrophotometer and colorimeter X-Rite DTP 20 Pulse (45°/0°measuring geometry, 2°standard observer, illuminant D65). The numerical TP value is based on the CIE *L*a*b** three-dimensional system and represents the difference in colour of a specific specimen measured on achromatic backgrounds. Consequently, two series of measurements were conducted (measurement of specimens against standard white background, and measurement of the same specimens against standard black background). Ten measurements on each of nine different plates were made against each background, changing the position of the measuring instrument (spectrophotometer) at five different places first on one and then on the other side of the surface of the specimen. Measurements were monitored by ColorShop X software integrated into the X-Rite spectrophotometer). After measuring CIE *L*a*b** values, the numerical value of translucency parameter was calculated in accordance with the following equation:

$$TP= [(L^*w-L^*b)^2 + (a^*w-a^*b)^2 + (b^*w-b^*b)^2]^{1/2}$$

All specimens were then exposed to 4% acidic acid at 80 °C for 16 hours (ISO 6872). Following exposure to the corrosive medium, measurements of the TP values were repeated as described in the first part.

Statistical analysis

The normality of the distribution was verified by the Shapiro-Wilk test. In order to compare the differences in TP values between different types of materials and colours, one-way analyses of variance (ANOVA) with the Student-Newman-Keuls *post-hoc* test were used. In order to compare TP values before and after exposure to acid, a mixed type of two-way ANOVA with intervention and material factors and three-way ANOVAs with intervention, material and colour factors and t-tests for dependent specimens were used for the assessment of differences within each material and each colour. Effect sizes were quantified by η2, and for dependent specimens using the equation $r = \sqrt{(t^2/t^2+df)}$. Statistical data were analysed by IBM SPSS 22 software. The level of significance was set at $p < 0.05$.

Results

Table 2 shows the comparison between TP values of glass-ceramics of different colours fabricated by different fabrication techniques before and after exposure to acetic acid.

IPS e.max Ceram Dentin had significantly the lowest TP, and IPS e.max Press the highest TP values (in all colours before and after exposure to acid $p < 0.001$).

Tablica 2. Usporedba translucencije prije i poslije korozije između vrsta materijala unutar iste boje; t_0 = translucencija prije izlaganja korozivnom sredstvu; t_1 = translucencija poslije izlaganja korozivnom sredstvu
Table 2 Comparison of translucency before and after corrosion among materials of the same colour; t_0 = translucency before exposure to corrosive medium; t_1 = translucency after exposure to corrosive medium

			AS±SD	p*	η2
Translucencija t_0 • Translucency t_0	A2	IPS e.max Ceram Dentin	7.58±0.92[a]		
		IPS e.max Press	16.50±0.33[b]		
		IPS e.max CAD	14.17±0.85[c]	<0.001	0.966
	C2	IPS e.max Ceram Dentin	8.61±0.34[a]		
		IPS e.max Press	17.53±0.50[b]		
		IPS e.max CAD	13.87±1.17[c]	<0.001	0.963
	B3	IPS e.max Ceram Dentin	9.69±0.30[a]		
		IPS e.max Press	17.58±0.58[b]		
		IPS e.max CAD	14.27±0.50[c]	<0.001	0.981
Translucencija t_1 • Translucency t_1	A2	IPS e.max Ceram Dentin	7.50±0.69[a]		
		IPS e.max Press	16.18±0.30[b]		
		IPS e.max CAD	13.95±0.68[c]	<0.001	0.978
	C2	IPS e.max Ceram Dentin	8.62±0.41[a]		
		IPS e.max Press	17.52±0.68[b]		
		IPS e.max CAD	13.87±1.33[c]	<0.001	0.949
	B3	IPS e.max Ceram Dentin	9.62±0.48[a]		
		IPS e.max Press	17.47±0.62[b]		
		IPS e.max CAD	13.75±0.36[c]	<0.001	0.979

*ANOVA i Student-Newman-Keuls post-hoc test. • ANOVA and Student-Newman-Keuls *post-hoc* test
Vrste materijala koje imaju različita slova u eksponentu se statistički se značajno razlikuju unutar iste boje. • Types of materials which have different letters in the exponent have a statistically significant difference within the same colour.

Razlika u TP vrijednostima između boja bila je vidljiva unutar materijala IPS e.max Ceram Dentin prije i poslije izlaganja kiselini uz veliku snagu efekta ($p < 0,001$; η2 = 0,702 i 0,741) te kod materijala IPS e.max Press ($p < 0,001$ uz snagu efekta 0,547 i 0,576). IPS e.max Ceram Dentin boje A2 imao je značajno najmanje, a boje B3 najveće TP vrijednosti prije i poslije izlaganja kiselini. IPS e.max Press boje A2 prije i poslije izlaganja kiselini imao značajno manje TP vrijednosti od boje C2 i boje B3, između kojih razlika nije bila značajna. Kod materijala IPS e.max CAD razlike u TP vrijednostima između boja nisu bile statistički značajne ni prije ni poslije izlaganja kiselini (tablica 2.).

The difference in TP values among colours was evident in IPS e.max Ceram Dentin material, both before and after exposure to acid with a great effect size ($p < 0.001$; η2 = 0.702 and 0.741) and in IPS e.max Press material ($p < 0.001$ with effect size 0.547 and 0.576). The IPS e.max Ceram Dentin colour A2 had significantly the lowest and the colour B3 the highest TP values, both before and after exposure to acid. The IPS e.max Press colour A2 had significantly lower TP than the colour C2 and the colour B3, both prior and after exposure to acid. No significant difference was observed between the colour C2 and the colour B3. Using the material IPS e.max CAD the differences among colours were not statistically significant either before or after exposure to acid (Table 2).

Analiza utjecaja korozije

Dvofaktorska ANOVA nije ni u jednom uzorku detektirala značajnu interakciju testa korozije i materijala kad je riječ o TP vrijednostima. Trofaktorska ANOVA nije detektirala značajnu interakciju testa korozije, materijala i boje s obzirom na TP vrijednosti.

Kiselina je uzrokovala značajno smanjenje translucencije samo kod boje B3 IPS e.max CAD uzorka ($p = 0,006$) uz snagu efekta od 58 % (slika 1.).

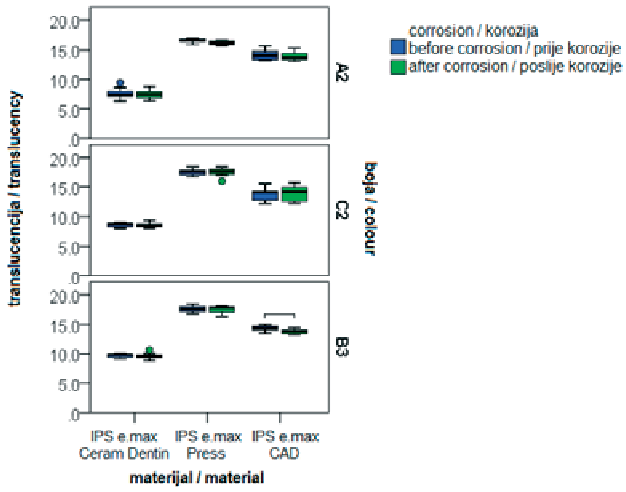
Iznos promjene TP vrijednosti zbog izlaganja kiselini, u svim je bojama bio podjednak (slika 2.).

Analysis of the effect of corrosion

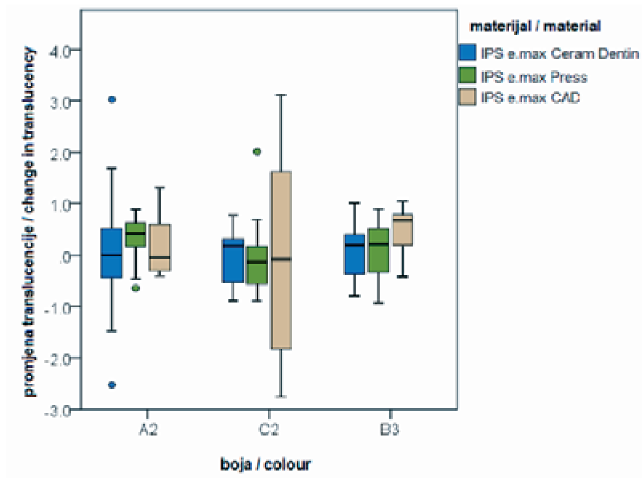
The two-way ANOVA did not show a significant interaction of the corrosion test and the material tested with respect to TP values. The three-way ANOVA did not detect a significant interaction of the corrosion test, the material used and the colour with respect to TP values.

Acid caused a significant reduction in TP values only in the IPS e.max CAD specimen colour B3 ($p = 0.006$) with the effect size of 58% (Figure 1).

The amount of change of TP values due to the exposure in acid was almost equal in all colours (Figure 2).



Slika 1. Usporedba translucencije prije i poslije izlaganja kiselini s obzirom na materijal i boju
Figure 1 Comparison of translucency before and after exposure to acid with respect to material and colour



Slika 2. Usporedba iznosa promjene translucencije nakon izlaganja kiselini između materijala i boja
Figure 2 Comparison of the amount of change in translucency due to exposure to acid between materials and colours

Rasprava

Prve dvije istraživačke hipoteze su prihvaćene. Pregledom relevantne literature nije pronađen veći broj radova o translucenciji keramičkih materijala kao vrlo važnom svojstvu estetskih materijala. Ako se znaju optička svojstva određenih restaurativnih materijala, njihova primjena u kliničkoj praksi postaje jednostavnija. Stoga je svrha ovoga rada bila pridonijeti razjašnjenju toga kompleksnog pitanja. Ispitivane su keramike koje *pokrivaju* indikacije od izrade tankih ljuskica do mostnih konstrukcija.

Tehnološka trajnost i dugoročno stabilna optička svojstva nadomjestka ovise o mehaničkim svojstvima materijala određenih sastavom i mikrostrukturom, tijeku izrade u zubotehničkom laboratoriju, kvaliteti obrade površine nadomjestka, vezivnom sredstvu i kvaliteti cijelog postupka izrade (3, 23, 24). Smatra se da tehnika slojevanja podliježe najvećim pogreškama zbog ljudskog čimbenika i mogućih manualnih propusta.

U ovom istraživanju zabilježene su statistički značajne razlike među staklokeramikama izrađenima različitim tehnološkim postupcima, stoga se prihvaća prva istraživačka hipoteza. Najveće TP vrijednosti imao je uzorak izrađen toplo-tlačnom tehnikom (IPS e.max Press), što je u skladu s istraživanjima Bagisa i suradnika (12). No isti autori navode da su uzorci dobiveni strojno (IPS e.max CAD) najmanje translucentni, a u ovom su istraživanju najmanje TP vrijednosti izmjerene u uzorcima izrađenima tehnikom slojevanja (IPS e.max Ceram Dentin). To se može objasniti činjenicom da su odabrani valjčići e.max Press i e.max CAD blokovi keramika visoke translucencije (high translucency -HT), a e.max Ceram Dentin je optički relativno gust. Ipak, uočava se sličnost – strojno obrađena staklokeramika pokazala je manji stupanj translucencije od one izrađene toplo-tlačnom tehnikom.

Analizirajući u ovom istraživanju translucenciju različitih boja unutar jedne tehnike izrade uočene su značajne ra-

Discussion

Research hypotheses have been accepted. By reviewing the relevant literature, translucency of ceramic materials, which is a very important property for the aesthetic appearance of materials, has not been thoroughly investigated. The knowledge of optical properties of specific restorative materials makes their use in the clinical practice easier. Thus, the aim of this paper was to contribute to the clarification of this complex issue. Ceramics covering the indications from fabrication of thin veneers to fixed partial dentures were tested. Technological durability and long-term stable optical properties of the restoration depend on the mechanical properties of the material which are determined by its composition and microstructure, the course of fabrication in a dental laboratory, the quality of the finishing treatment of the restoration, the connective agent and the quality of the entire fabrication procedure (3, 23, 24). It is considered that the layering technique is liable to greatest errors because of the human factor and possible oversights in manual procedures.

Statistically significant differences among glass-ceramics fabricated by different techniques were recorded in this study; therefore the first research hypothesis is accepted. The specimen made by heat-pressing technique (IPS e.max Press) had the greatest TP values, which is in accordance with a research conducted by Bagis et al. (12). However, those authors stated that specimens fabricated by computer-aided manufacturing (IPS e.max CAD) had the lowest TP values, whereas in this study the lowest TP values were measured in the specimen fabricated by the layering technique (IPS e.max Ceram Dentin). This can be explained by the fact that e.max Press ingots and e.max CAD ceramics blocks were selected in the high translucency (HT), whereas e.max Ceram Dentin was optically relatively thick. However, a similarity is observed in the fact that glass-ceramics fabricated by computer-aided manufacturing showed lower TP values than glass-ceramics fabricated by heat-pressing technique.

zlike, stoga se prihvaća druga istraživačka hipoteza. Utvrđeno je da su uzorci izrađeni tehnikom slojevanja i toplo-tlačnom tehnikom pokazali odstupanja u translucenciji, za razliku od strojno izrađenih uzoraka kod kojih nije bilo razlike, kako prije tako ni poslije korozije. Najmanje TP vrijednosti u svim bojama pokazali su uzorci izrađeni tehnikom slojevanja. Najmanje TP vrijednosti imali su uzorci boje A2, a najveću B3. Kod prešanih uzoraka najmanje TP vrijednosti ponovno ima boja A2, a iste vrijednosti pokazuju boje C2 i B3. Uzorci izrađeni strojno nisu pokazali razlike u TP vrijednostima među bojama.

Tehnika slojevanja podliježe najvećim varijacijama jer se ručno miješa keramički prašak s tekućinom. Konačna kondenzacija uvjetovana je veličinom keramičkih zrnaca, količinom tekućine unesene tijekom modelacije i tehnike kondenzacije. Tako da su, od tehničara do tehničara, moguće velike razlike u proizvodu. Postupci kondenzacije uvelike utječu na optička i mehanička svojstva keramičkog materijala. Chu i suradnici ističu da precizan omjer keramičkog praška i tekućine prema uputi proizvođača ne osigurava uvijek pravilnu gustoću te time translucenciju ili opacitet budućeg nadomjestka (25). Ističe se i važnost kemijske strukture sastavnica te veličina kristala uloženih u staklenu matricu (8).

Za razliku od tehnike slojevanja, polugotovi tvornički proizvodi (keramički valjčići IPS e.max Press ili blokovi za glodanje IPS e.max CAD) strukturno su postojaniji. U njima su keramičke čestice u tvornici sabijene pod visokom temperaturom i tlakom te se tako dobiva vrlo gust materijal s homogenom mikrostrukturnom slikom (12).

Wang je ustanovio da na translucenciju staklokeramičkog nadomjestka znatno utječe vrsta i debljina obložne zubne keramike. Translucencija zubnih keramika eksponencijalno se povećava sa smanjenjem debljine (7). Lim je prikazao usporedne vrijednosti translucencije izmjerene spektrofotometrom (veće vrijednosti) i spektroradiometrom (26). Ostali autori dovode u korelaciju translucenciju zubnih keramika s osvjetljenjem (27, 28), utjecajem glazirane/neglazirane površine staklokeramike (29) te s utjecajem cementa i boje prepariranog zuba (30).

Umjetnim izlaganjem uzoraka korozivnom sredstvu dobiva se uvid u postojanost keramike i postojanost svojstva translucencije u korozijski kompromitiranoj usnoj šupljini znatno brže i jednostavnije nego u ispitivanjima *in vivo*. Uporaba octene kiseline opravdana je iz više razloga – često se rabi u kućanstvu, pH (2,4) je vrlo sličan nekim pićima, a takav pH nalazi se i ispod plaka. Ujedno je u ustima takav niži pH čest i kod pacijenata s bolestima želuca.

Poznato je da se zbog djelovanja korozivnog agensa na keramički nadomjestak narušava veza u osnovnoj gradivnoj jedinici stakla – silicijevim tetraedrima, što znači da veća količina staklene matrice u materijalu, uz isti korozivni učinak okoliša, rezultira većom korozijom. Blokovi za strojnu obradu sadržavaju znatno manje ili ništa staklene matrice od keramike za tehniku slojevanja te su zato kemijski postojaniji (31, 32, 33). No utjecaj kiseline na translucenciju staklokeramika nije bio statistički značajan u ovom istraživanju ni za jednu tehnologiju izrade, pa se odbacuje treća istraživačka hipoteza. Rezultati su u skladu s rezultatima drugih au-

Significant differences were observed in this study by analysing the TP values of specimens of different colours fabricated by the same technique; therefore the second research hypothesis is accepted. It was established that specimens fabricated by the layering technique and heat-pressing technique showed deviations in TP values, in contrast to the specimens fabricated by computer-aided manufacturing which showed no difference, both before and after exposure to corrosion. Specimens fabricated by the layering technique showed the lowest TP values in all colours. Specimens of the colour A2 had the lowest TP values, whereas TP values of the colour B3 were the greatest. Among specimens fabricated by the heat-pressing technique, the colour A2 again showed the lowest TP values, whereas the colours C2 and B3 showed the same values. Specimens fabricated by computer-aided manufacturing did not show difference of TP values of different colours.

The layering technique is liable to greatest variations because ceramic powder is manually mixed with the liquid. The final condensation is conditioned by the size of the ceramic grain, the amount of the liquid introduced during modelling and the condensation technique itself. Thus, great differences in the final product from one technician to another may occur. Condensation procedures have a significant effect on optical and mechanical properties of the ceramic material. Chu et al. emphasised that the precise ceramic powder/liquid ratio in accordance with the manufacturer's directions does not always ensure proper thickness and porosity and consequently the translucency or opacity of the restoration (25). The importance of the chemical structure of the components and the size of crystals inserted in the glass matrix is also emphasised (8).

In contrast to the layering technique, factory-made semi-finished products (IPS e.max Press ceramic ingots or IPS e.max CAD blocks for milling) are structurally more stable. Their ceramic particles were pressed in the factory under high temperature and pressure which results in a very thick material with a homogeneous microstructural picture (12).

Wang found out that the translucency of the glass-ceramics restoration is significantly affected by the type and thickness of the veneering dental ceramics. Translucency of dental ceramics increases exponentially with reduced thickness (7). Lim showed comparable values of translucency measured by a spectrophotometer (higher TP values) and by spectroradiometer (26). Other authors bring into correlation the translucency of dental ceramics with illumination (27, 28), the impact of glazed/unglazed surface of glass-ceramics (29) and the impact of the cement and the colour of the prepared tooth (30).

By artificial exposure of specimens to a corrosive medium an insight is gained into the stability of glass-ceramics and the stability of the translucency property in the oral cavity compromised by corrosion in a significantly more rapid and simpler manner than in *in vivo* studies. The use of acetic acid is justified for more reasons; it is often used in households, its pH (2.4) is very similar to that of some beverages, and such pH is also present in the areas below plaque. Such lower pH is also often present in the mouth of patients suffering from stomach diseases.

tora (22, 12). Može se zaključiti da su keramički materijali kemijski postojani. Pojedina istraživanja povezuju nastanak hrapavosti na površini nadomjeska zbog dugotrajne izloženosti kiselom agensu, s posljedično mogućim promjenama optičkih svojstava (21, 28). No o toj kompleksnoj temi potrebna su dodatna istraživanja.

Zaključak

Uzimajući u obzir ograničenja ovog istraživanja, zaključuje se da su različite staklokeramike pokazale značajne razlike u TP vrijednostima, kako prema tehnološkom postupku izrade, tako i prema različitim bojama. Izlaganje korozivnom sredstvu nije statistički značajno promijenilo TP vrijednosti. Stoga su staklokeramike, bez obzira na to kojim su tehnološkim postupkom izrađene, pokazale odličnu kemijsku stabilnost.

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Izjava

Autori ističu da nisu bili ni u kakvom sukobu interesa.

It is known that, because of the effect of the corrosive medium on the ceramic restoration there is a disruption of bonds in the fundamental structural units of glass, silica tetrahedrons, which means that a greater amount of glass matrix in the material with the same corrosive effect of the environment results in greater corrosion. Blocks for computer-aided manufacturing contain significantly much less glass matrix than ceramics used in the layering technique and are therefore chemically more stable (31, 32, 33). However, the effect of the acid on translucency of glass-ceramics was not statistically significant in this study for any of the fabrication techniques; therefore the third research hypothesis is rejected. The results are in accordance with the results obtained by other authors (22, 12). A conclusion can be made that ceramic materials are chemically stable. Some studies bring into relation the appearance of roughness on the surface of the restoration resulting from long-term exposure to acid medium and consequent changes in the optical properties (21, 28). However, additional research on this complex topic is needed.

Conclusion

Under the limitation of this study, it was concluded that different types of glass-ceramics showed significant differences in TP values, both with respect to the fabrication technique and with respect to colour. Exposure to a corrosive medium did not result in a statistically significant change in TP values. Thus, glass-ceramics fabricated by all fabrication techniques showed excellent chemical stability.

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Conflict of interest

None to declare.

Abstract

The purpose of the study was to analyse translucency parameter (TP values) of glass-ceramics fabricated by different techniques and investigate the effect of the corrosive medium on TP values. **Materials and methods:** Three specimens of each type of IPS e.max ceramics (Ivoclar Vivadent, Schaan, Liechtenstein) were made in three colours (A2, C2 and B3) by three fabrication techniques (layering – e.max Ceram Dentin; heat-pressing – e.max Press; CAD/CAM – e.max CAD). Specimens were made in the form of plates (10 mm x 12 mm x 0.8 mm). CIE $L^*a^*b^*$ values were measured by a spectrophotometer (X-Rite DTP 20 Pulse, Neu Isenburg, Germany) before and after exposure to 4% acetic acid at 80 °C for 16 hours (ISO 6872) to calculate translucency parameter (TP values). Statistical data were analysed using the IBM SPSS 22 software. **Results:** IPS e.max Ceram Dentin had significantly the lowest TP values, and IPS e.max Press the highest TP values of all colours (A2, C2, B3), both prior and after exposure to acid ($p<0.001$). The difference in TP values among colours was evident in the IPS e.max Ceram Dentin material, both before and after exposure to acid with a great effect size ($p<0.001$; $\eta2 = 0.702$ and 0.741), and in the IPS e.max Press material ($p<0.001$, effect size 0.547 and 0.576). CAD/CAD specimens showed uniform TP values between three colours. Further, exposure to a corrosive medium did not result in a statistically significant change of TP values in any of the materials tested. **Conclusions**: Different types of glass-ceramics showed significant difference in TP values both with respect to the fabrication technique and colour. Exposure to a corrosive medium did not result in a statistically significant change of TP values.

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Glass ceramic: Spectrophotometry; Optical phenomena; Corrosion; Acetic Acid; translucency

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