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IZVORNI ZNANSTVENI RAD
ORIGINAL SCIENTIFIC PAPER

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Utjecaj termocikliranja na čvrstoću savijanja poliamidne baze proteze

The Influence of Thermocycling on the Flexural Strength of a Polyamide Denture Base Material

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Sažetak

Svrha: Svrha ovoga rada bila je procijeniti utjecaj termocikliranja na čvrstoću savijanja poliamidnog materijala za bazu proteze. **Materijali i metode:** Testiran je poliamidni protezni materijal (Valplast), a kao kontrola korišten je PMMA (Vertex). Trideset primjeraka svakog materijala izrađeno je za ispitivanje čvrstoće savijanja prema standardu ISO 1567. Svi su uzorci pripremljeni i čuvani 48 sati u vodi na temperaturi od 37 °C, a zatim su podijeljeni u tri jednake skupine (n = 10). Ispitivanje čvrstoće savijanja obavljeno je odmah nakon uranjanja u vodu te nakon termocikliranja (5 °C/55 °C, 2c/min.) poslije 3000 i 5000 ciklusa. Ispitivanje fleksibilnosti provedeno je na trima točkama u univerzalnom stroju za testiranje na brzini križanja od 5 mm/min. Konačna čvrstoća savijanja izračunata je formulom: FS = 3PL/2bd^2. Dvosmjerna ANOVA s Tukeyjevim post hoc testom primijenjena je na razini .05 statističke značajnosti. **Rezultati:** tatistički značajno smanjenje čvrstoće savijanja zabilježeno je nakon termocikliranja poslije 3000 ciklusa za PMMA-e i 5000 ciklusa za oba materijala. Čvrstoća savijanja PMMA-e bila je značajno veća u odnosu na poliamid za sve ispitane uvjete ($p < 0,05$). **Zaključak:** Termocikliranje je vrlo nepovoljno djelovalo na čvrstoću savijanja materijala poliamida i materijala PMMA za bazu proteze.

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Uvod

Unatoč sve većoj popularnosti dentalnih implantata za potpunu rehabilitaciju usta, opskrba bezubih pacijenata potpunom i djelomičnom protezom i dalje je korisna metoda liječenja. Pritom je pravilan odabir polimera smole za bazu proteze važan čimbenik za dugoročni uspjeh mobilnih nadomjestaka. Polimetilni metakrilat (PMMA) desetljećima je bio materijal izbora za konstrukcije baze proteza. Alternative su uključivale materijale kao što su polimeri visokog udara, polikarbonati i poliamidi koji su posljednjih desetljeća uvedeni na dentalno tržište (1 – 3).

Poliamidi [NH(CH_2)mCOn] poznati su u svakodnevnom životu kao najlon. U svojoj molekuli, veze dobivene aminskom skupinom (NH) i krajnjom karbonilnom komponentom funkcionalne skupine (COOH), reagiraju kako bi se dobila veza ugljik – dušik (amid). Aminska skupina i skupina karboksilne kiseline mogu biti ili na istome monomeru, ili se polimer može sastojati od dvaju različitih bifunkcionalnih

Introduction

Despite the increasing popularity of dental implants for full mouth rehabilitation, complete and partial dentures remain a useful treatment modality for the restoration of edentulous patients. The proper selection of the base resin denture polymer is a significant factor for the long term success of removable restorations. Polymethyl methacrylate (PMMA) has been the material of choice for denture base constructions for decades. Alternatives to PMMA include materials which have been introduced in dental market in the past decades, such as high impact polymers, poly-carbonates and polyamides (1-3).

Polyamides (NH (CH_2) mCOn) are known in everyday life as nylon. In their molecule bonds produced by an amine group (NH) and a terminal carbonyl component of a functional group (COOH) react to produce a carbon-nitrogen bond (amide). The amine group and the carboxylic acid group may either be on the same monomer, or the polymer

monomera – jedan s dvjema aminskim skupinama, a drugi s dvjema karboksilnim ili kiselinskim kloridnim skupinama (4 – 6). Najlon se sintetski proizvodi i polimerizira reakcijom kondenzacije i mora specifično uključivati ravni (alifatski) lanac monomera (5, 6).

Prvi pokušaji korištenja poliamida kao materijala za bazu proteze počeli su tijekom 1950-ih, ali su u tu svrhu uvelike korišteni nakon pronalaska novih generacija najlonskih materijala (4 – 6). Zbog dobrih fizičkih i mehaničkih svojstava poliamida, uključujući veliku fleksibilnost, nisku gustoću, visoku otpornost na udarce, nisku apsorpciju vode i topljivost, materijal su izbora za bazu proteza (4). To su netoksični materijali s malom mogućnošću alergijskih reakcija, a relativno im je dobra i stabilnost boje (4, 7, 8). Glavni nedostaci poliamida kao materijala za bazu proteza odnose se na njihov nizak modul elastičnosti, savijanja i vlačne čvrstoće (9, 10), nisko prianjanje na podložnu masu proteze (11), nepostojanje kemijske veze s akrilnim zubima i nemogućnost popravka (12). Iako su u mnogim studijama (7, 9, 13, 14) objavljeni podatci u vezi s čvrstoćom savijanja PMMA-e u različitim eksperimentalnim uvjetima, nedostaju dokazi o ponašanju fleksibilnosti poliamida (1, 3, 15).

Krajnja čvrstoća na savijanje materijala pokazuje svoj potencijal tako da se odupire katastrofalnom neuspjehu pod opterećenjem na savijanje. Visoka čvrstoća na savijanje osnovnog materijala za proteze nužna je za klinički uspjeh mobilnih djelomičnih nadomjestaka s obzirom na činjenicu da je alveolarna resorpcija postupan i nepravilan proces koji može rezultirati neravnim nosačem proteze na tkivu. Potrebna je visoka proporcionalna granica da bi se osnovni materijal mogao oduprijeti plastičnoj deformaciji te povećala otpornost na zamor koja je nužna da bi se podnijelo ponavljano žvačno opterećenje (16 – 18). Velika fleksibilnost poliamida prijeko je potrebna za funkciju bezmetalnih mobilnih djelomičnih proteza (PDD) (2). Učinak vode, a time i sline, te utjecaj promjene temperature na svojstva savijanja poliamida kada se konzumira vruća i hladna hrana, nije temeljito istražen.

Cilj ove studije bio je ispitati čvrstoću na savijanje poliamidnog materijala za bazu proteze u usporedbi s konvencionalnim materijalom PMMA nakon izloženosti različitom broju termičkih ciklusa. Nulta hipoteza bila je da ne postoji značajna razlika u čvrstoći savijanja prije termocikliranja i poslije toga postupka, te da broj ciklusa ne utječe značajno na čvrstoću savijanja materijala.

Materijali i metoda

Ispitivana su dva smolasta materijala za bazu proteze – PMMA (Vertex Rapid Simplified, Vertex-Dental B.V., Nizozemska) i poliamid (Valplast, Valplast Int Corp., Long Island City, NY, SAD). Uzorci od nehrđajućeg čelika upotrijebljeni su za izradu ukupno 60 šipki (30 za svaki materijal) veličine 64 mm x 10 mm x 2,5 mm, prema specifikaciji ISO 1567 (19).

may be constituted of two different bifunctional monomers, one with two amine groups and the other with two carboxylic acid or acid chloride groups (4-6). Nylon is synthetically produced and polymerized by condensation reaction and must specifically include a straight chain (aliphatic) monomer (5, 6).

The first attempts to use polyamides as denture base materials were made in the 1950s, but they have been used extensively for this purpose after the introduction of new generations of nylon materials (4-6). Physical and mechanical properties favor the use of polyamides as denture base materials since they exhibit high flexibility, low density, high impact resistance and low water sorption and solubility (4). They are non-toxic materials with a low possibility of allergic reactions. They offer relatively good color stability (4, 7, 8). One of the disadvantages of polyamides as denture base materials is their low modulus of elasticity, flexural and tensile strength (9, 10), their low adherence to denture liners (11), the absence of chemical bond with acrylic teeth and the inability of repair (12). Although a large number of studies (7, 9, 13, 14) have been published on the flexural strength of PMMA under different experimental conditions, there is a lack of evidence concerning the flexural behavior of polyamides (1, 3, 15).

The ultimate flexure strength of a material reflects its potential to resist catastrophic failure under a flexural load. The high flexural strength of the denture base material is essential for the clinical success of removable partial restorations, given the fact that the alveolar resorption is a gradual, irregular process that may result in uneven support of tissue-borne prostheses. A high proportional limit is necessary in order for the base material to resist plastic deformation and enhanced fatigue resistance is essential to withstand repeated masticatory loading (16-18). The high flexibility of polyamides is necessary for the function of metal-free removable partial denture (RPD) clasps (2). The effect of the presence of water, and consequently saliva, and the effect of temperature changes when hot and cold foods are consumed on the flexural properties of the polyamides have not been thoroughly investigated.

The aim of the present study was to investigate the flexural strength of a polyamide denture base material in comparison to a conventional PMMA denture base material after they had been submitted to a large number of thermal cycles. The null hypothesis was that there would be no significant difference in the flexural strength before and after thermocycling and that the number of cycles would not significantly affect the flexural strength of the materials.

Materials and Method

Two denture base resin material, a PMMA (Vertex Rapid Simplified, Vertex-Dental B.V., The Netherlands) and a polyamide (Valplast, Valplast Int. Corp, Long Island City, NY, USA) were tested. Stainless steel patterns were used to fabricate a total of 60 bars (30 of each material), measuring 64 mm×10 mm×2.5 mm, according to the ISO 1567 specification (19).

Priprema uzoraka

Broj uzorka najprije je procijenjen korištenjem softvera G*Power (G*Power v.3.1.5, Franz Faul, Sveučilište Kiel, Njemačka).

Tri metalna uzorka uložena su u tikvicu s dentalnim gipsom ISO tipa III (Microstone, Whip-Mix, SAD). Prije ulaganja na svaki je postavljen vosak (3 mm u promjeru). Uzorci su uklonjeni nakon kuhanja, a šupljine kalupa napunjene odgovarajućim materijalom za izradu uzorka.

Prije ubrizgavanja u šupljine kalupa, poliamidni materijal je 11 minuta bio plastificiran u digitalnoj peći za taljenje Valplast na 280 °C. Tikvica je bila pritisnuta 3 minute u injekcijskom prešanju Valplast i zatim odložena na klupu da se ohladi prije nego što će se otvoriti.

Akrilna smola Vertex proizvedena je prema preporuci proizvođača miješanjem 1 ml tekućine (monomera) s 2,3 g praha (polimera). Nakon što je postala poput tijesta, akrilna masa umetnuta je u šupljine kalupa i polimerizirana 20 minuta na 100 °C. Nakon sušenja tikvice su 30 minuta hlađene na klupama na sobnoj temperaturi.

Svi uzorci izvađeni su iz kalupa, a višak s rubova uklonjen je Tungstenovim karbidnim svrdlima. Nakon toga uzorci su polirani do 600 granula u jednoj poliranoj jedinici (Ecomet III Buehler Ltd, Evanston, IL, SAD), a poslije toga postupka bili su 48 sati u vodi na 37 °C prema standardu ISO 1567.

Ovisno o korištenom materijalu, uzorci su podijeljeni u dvije skupine od 30 (skupina 1/Vertex, skupina 2/Valplast). Uzorci svakog materijala nakon toga podijeljeni su u tri jednake podgrupe (n = 10) (tablica 1.). Podgrupe A1 i B1 bile su podvrgnute testu savijanja odmah nakon što su 48 sati bile uronjene u destiliranu vodu na temperaturi od 37 °C. Podgrupe A2 i B2 dodatno su bile podvrgnute termocikliranju od 3000 ciklusa, a skupine A3 i B3 bile su termociklirane u 5000 ciklusa (5 °C i 55 °C, 2 ciklusa/min.) (tablica 1.). Ispitivanje savijanja na trima točkama provedeno je na univerzalnom stroju za testiranje (Tensometer 10, Monsanto, Akron, Ohio, SAD) sa silom primijenjenom pri brzini križanja od 5 mm/min. Uzorci su stavljeni u nosač s ugrađenim nosačima udaljenima 50 mm.

Snaga loma (F) zabilježena je u njutnima (N), a čvrstoća savijanja (FS) izračunata je u megapascalima (MPa) prema formuli $FS = 3PL/2bd^2$ (P = maksimalno opterećenje, L = duljina uzorka, b = širina uzorka i d = debljina uzorka). Vrijednosti učestalosti opterećenja (N) Valplasta izvedene su iz krivulje *stres – naprezanje* na mjestu proporcionalne granice.

Kako bi se testirao učinak materijala, broj toplinskih ciklusa i njihove interakcije na čvrstoću savijanja učinjeni su

Specimen preparation

The specimen number was estimated before testing. For this purpose, G*Power software (G*Power v.3.1.5, Franz Faul, University of Kiel, Germany) were used.

Three metal patterns were invested in a flask with ISO type III dental stone (Microstone, Whip-Mix, USA). Before investing, a wax sprue (3 mm in diameter) was positioned on every pattern. The patterns were removed after boil-out and the mold cavities were filled with the respective material for specimen fabrication.

Before injection into the mold cavities, the polyamide material was plasticized in a digital melting Valplast furnace at 280°C for 11 min. The flask was pressed for 3min in a Valplast injection press and then allowed to bench cool before opening.

The Vertex acrylic resin was fabricated according to the manufacturer's recommendation by mixing of 1ml of liquid (monomer) to 2.3 g of powder (polymer). When it reached the dough stage, the acrylic mass was inserted in the mold cavities and polymerized at 100°C for 20 min. After curing, the flasks were bench-cooled at room temperature for 30 min.

All specimens were removed from the molds and the excess margins were trimmed with tungsten carbide burs. Subsequently, the specimens were polished up to 600 grits in a polishing unit (Ecomet III Buehler Ltd, Evanston, Ill, USA). The specimens were then stored in water at 37°C for 48 hours, according to ISO 1567.

According to the material used, the specimens were divided in two groups of 30 (Group 1/Vertex, Group 2/Valplast). The specimens of each material were further divided into three equal subgroups (n=10) (Table 1). Subgroups A1 and B1 were submitted to flexural test immediately after storage in distilled water for 48 hours at 37°C. Subgroups A2 and B2 were further submitted to thermocycling for 3000 cycles, while groups A3 and B3 were submitted to thermocycling for 5000 cycles (5°C and 55°C, 2 cycles/min) (Table 1). The three point flexural testing was accomplished in a universal testing machine (Tensometer 10; Monsanto, Akron, Ohio) at a force applied with a crosshead speed of 5 mm/min. Specimens were placed in a rig with incorporated supports distanced at 50 mm.

The fracture force (F) was recorded in Newtons (N) and the flexural strength (F_S) was calculated in MPa following the formula $F_S = 3\ PL/2\ bd^2$ (P=maximum load, L= specimen length, b=specimen width, and d=specimen thickness). The Valplast failure load values (N) were derived from the stress-strain curve at the point of proportional limit.

Tablica 1. Grupe i podgrupe testiranih materijala
Table 1 Groups and subgroups of tested materials

	Podgrupe • Subgroups		
	A	**B**	**C**
	48 sati • 48 hours (voda 37° C • water 37°C)	3000 ciklusa • 3000 cycles (TC*)	5000 ciklusa • 5000 cycles (TC*)
Grupe • Groups 1 (Vertex)	A1	B1	C1
2 (Valplast)	A2	B2	C2

*TC = Termocikliranje • Thermocycling

prema rangiranim podatcima, dvosmjerna ANOVA s pomoću Tukeyjeva post hoc testa. Svi su testovi imali razinu statističke značajnosti .05. Za analizu podataka korišten je statistički softver (Sigma Plot, Verzija 12.0, SSI, Jandel CA, SAD).

Rezultati

Rezultati ispitivanja čvrstoće savijanja pokazali su statistički značajnu razliku između ispitivanih materijala u jednakim eksperimentalnim uvjetima ($p < 0,05$). Postojala je statistički značajna interakcija između materijala i termocikliranja ($p < 0,05$). Općenito, oba materijala pokazala su smanjenje vrijednosti čvrstoće savijanja kada je povećan broj ciklusa. Statistička procjena među svim uvjetima hidro-termocikliranja bila je značajna za sve podskupine i za oba materijala, osim za podskupinu od 48 sati i 3000 ciklusa za Valplast. Vrijednosti čvrstoće savijanja Vertexa, nakon što je 48 sati bio uronjen u vodu, bile su gotovo dvostruke u usporedbi s vrijednostima Valplasta (119,13 do 60,31 MPa).

Rezultati ispitivanja čvrstoće na savijanje nalaze se u tablici 2. Statistički značajno smanjenje čvrstoće savijanja materijala PMMA (Vertex) zabilježeno je nakon 3000 i 5000 termalnih ciklusa (119,13 MPa u usporedbi s 99,82 MPa i 94,80 MPa) ($p < 0,05$). Nije zabilježena statistički značajna razlika u otpornosti na savijanje poslije 3000 termičkih ciklusa kada je ispitan poliamidni materijal (Valplast) (60,31 MPa u usporedbi s 56,34 MPa). Ali, termocikliranje od 5000 ciklusa rezultiralo je statistički značajnim smanjenjem vrijednosti čvrstoće na savijanja za Valplast (60,31 MPa u usporedbi s 35,39 MPa).

A two-way ANOVA to the ranked data was done to test the effect of material, number of thermal cycles and their interactions on the flexural strength using the post hoc Tukey's test. All tests used a .05 level of statistical significance. Statistical software (Sigma Plot, Version 12.0, SSI, Jandel CA) was used for data analysis.

Results

The results of the flexural strength test revealed a statistically significant difference between the tested materials under the same experimental conditions ($p<0.05$). There was a statistically significant interaction between materials and thermocycling ($p<0.05$). Generally, both materials showed a decrease in flexural strength values when the number of cycles was increased. The statistical estimation among all hydrothermocycling conditions was significant for all subgroups for both materials except for the subgroup of 48 hours to 3000 cycles for Valplast. Vertex flexural strength values after 48 hours storage in water were almost double compared to the ones of Valplast (119.13 to 60.31 MPa).

The results of the flexural strength testing are presented in Table 2. A statistically significant decrease in the flexural strength of the PMMA material (Vertex) was recorded both after 3000 and 5000 thermal cycles (119.13 MPa compared to 99.82 MPa and 94.80 MPa respectively) ($p<0.05$). No statistically significant difference in flexural strength was recorded for 3000 thermal cycles when the polyamide material (Valplast) was examined (60.31 MPa compared to 56.34 MPa). However, thermocycling for 5000 cycles resulted in a statistically significant decrease in flexural strength values for Valplast (60.31 MPa compared to 35.39 MPa).

Tablica 2. Srednje vrijednosti i standardne devijacije svih podgrupa u MPa
Table 2 Mean values and standard deviations of all subgroups in MPa

A1	A2	B1	B2	C1	C2
119.13 (±7,28)[a]	**60.31** (±2,34)[b]	**99.82** (±14,02)[c]	**56.34** (±3,1)[b]	**94.80** (±13,06)[d]	**35.39** (±2,67)[e]

Jednako potencirana slova znače nepostojanje statističke značajnosti. • Same superscript letters mean no statistical differences.

Vrijednosti čvrstoće na savijanje dvaju testiranih materijala pokazale su statistički značajnu razliku za sve testirane eksperimentalne uvjete: nakon 48 sati skladištenja u vodi vrijednosti Vertexa bile su gotovo dvaput veće od vrijednosti Valplasta (119,13 do 60,31 MPa). Statistički značajna razlika između dvaju materijala pronađena je nakon što su bili 3000 puta izloženi termalnim ciklusima. Nakon 5000 termalnih ciklusa zabilježena je statistički značajna razlika u vrijednostima čvrstoće na savijanje između dvaju materijala.

Rasprava

Nulta hipoteza nije potvrđena jer su među testiranim skupinama zabilježene značajne razlike u čvrstoći savijanja.

Iz rezultata ove studije može se zaključiti da promjene temperature i uranjanje u vodu tijekom termocikliranja smanjuju čvrstoću na savijanje i PMMA-e i poliamidnih materijala za bazu proteze.

Flexural strength values of the two materials tested revealed a statistically significant difference for all the experimental conditions tested: after 48 hours of water storage the Vertex values were almost double than the ones of Valplast (119.13 to 60.31 MPa). A statistically significant difference between the two materials was found after submission to 3000 thermal cycles. After 5000 thermal cycles a statistically significant difference in flexural strength values between the two materials was also recorded.

Discussion

The null hypothesis was not verified because significant differences in flexural strength were recorded among the tested groups.

From the results of the present study it may be concluded that temperature fluctuations and immersion in water during thermocycling decrease the flexural strength of both PMMA

U ovom radu PMMA je pokazala znatno veću čvrstoću na savijanje u odnosu na poliamid. Velik problem bio je utvrđivanje točke neuspjeha fleksibilnih poliamidnih materijala tijekom ispitivanja savijanja. U ovoj studiji pretpostavlja se da je točka neuspjeha vrijednost koja odgovara proporcionalnoj granici materijala u krivulji *stres – naprezanje*. Kliničko značenje čvrstoće na savijanje u proporcionalnom ograničenju jest da odražava otpornost materijala na plastičnu deformaciju.

U nizu studija (3, 9, 15, 20 – 22) ispitivala se čvrstoća na savijanje poliamidnih materijala u odnosu na PMMA-u u različitim eksperimentalnim uvjetima. Vrijednosti čvrstoće na savijanje poliamida dobivene u ovoj studiji u skladu su s rezultatima drugih autora koji su testirali slične materijale nakon jednakog izračuna točke neuspjeha (10, 15). Razlike u vrijednostima čvrstoće savijanja poliamida koje su prijavili drugi autori mogu se pripisati različitoj mikrostrukturi ispitivanih specifičnih zaštićenih materijala (engl. *trademark*) ili razlikama u izračunu točke neuspjeha (3, 9). Visoke vrijednosti čvrstoće na savijanje (163,62 MPa) koje je objavio Abhay sa suradnicima (1) vjerojatno mogu biti posljedica različitog načina izračuna.

Zbog njihove velike fleksibilnosti, poliamidni materijali za baze proteza ne lome se u kliničkim situacijama čak ni u slučaju ekstremnih žvačnih sila. Oni se ne mogu smatrati klinički učinkovitima ako plastična deformacija prekorači proporcionalnu granicu jer rezultiraju dimenzijskim nepravilnostima nadomjestaka (23). U tom slučaju, materijali baze proteza mogu rezultirati slabijim žvačnim svojstvom i alveolarnom resorpcijom. Žvačne sile odraslih muškaraca s kompletnom denticijom kreću se od 60 do 305 N, s prosječnom vrijednošću od 137 N i 150 N (24, 25). Srednja vrijednost proporcionalne točke Valplasta u ovoj studiji nakon 3000 hidro-termocikliranja iznosila je oko 220 N, što je vidljivo na slici 1. To znači da potpuna proteza izrađena od poliamidnog materijala Valplast uspješno izdržava uobičajene žvačne sile, ali trajna deformacija nastaje kod ekstremnih opterećenja. U svim slučajevima zabilježene vrijednosti testiranih materijala zadovoljavaju predloženu minimalnu prihvatljivu silu (55 N) ISO 1567.

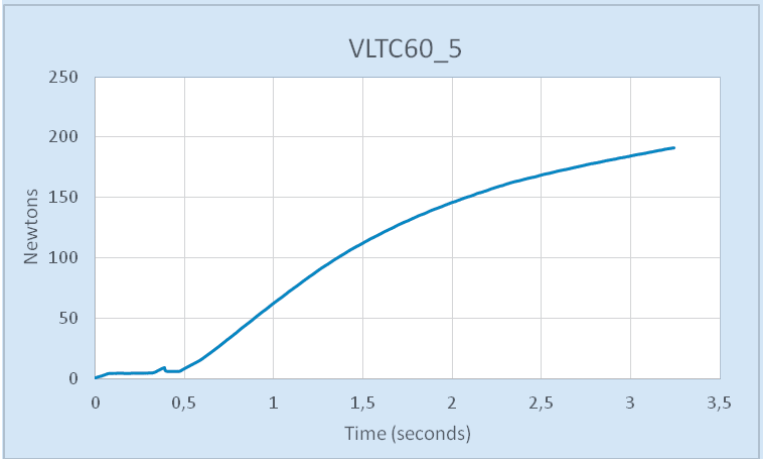
Mnogi istraživači (26 – 29) izvijestili su da apsorpcija vode i toplinske promjene (22, 30 – 32) smanjuju čvrstoću na

and polyamide denture base materials.

In the present study, PMMA presented significantly higher flexural strength in relation to polyamide. Determining the failure point of the flexible polyamide materials during flexural testing has been a substantial problem. In the present study, the value corresponding to the proportional limit of the material in the stress–strain curve was presumed to be the failure point. The clinical importance of the flexural strength at the proportional limit is that it reflects the resistance of a material to plastic deformation.

In a number of studies (3, 9, 15, 20-22), the flexural strength of polyamide materials in comparison to PMMA under a variety of experimental conditions has been examined. The values of the flexural strength of polyamide reported in the present study are in agreement with the results of other authors, who tested similar materials following the same calculation of the failure point (10, 15). The differences in the polyamide flexural strength values reported by other authors may be attributed to different microstructures of specific trademark materials tested or to the differences in the calculation of the failure point (3, 9). The high flexural strength values (163.62 MPa) recorded by Abhay et al. (1) may probably be due to different ways of calculating.

Due to their high flexibility, polyamide denture base materials do not fracture in clinical situations even under extreme bite forces. They cannot be considered clinically efficient if plastic deformation exceeds the proportional limit, due to the resulting dimensional inaccuracies in the restoration (23). In this case, denture base materials may lead to inferior masticatory ability and alveolar resorption. Masticatory bite forces which are exerted by adult men with full dentition range between 60 N and 305 N, with a mean value of 137 N and 150 N (24, 25). The mean value of the proportional point of Valplast in the present study was about 220 N after 3000 hydro-thermocycling as it can be seen in Figure 1, which means that a full denture constructed by Valplast polyamide material can withstand the usual masticatory forces successfully. Nevertheless, there will be a permanent deformation in extreme loads. In all cases, the recorded values of tested materials exceeded the minimum accepted force values (55 N) proposed by ISO 1567.



Slika 1. Dijagramska ilustracija krivulje savijanja Valplasta nakon 3000 hidro-termocikliranja
Figure 1 Diagrammatical illustration of bending curve of Valplast after 3000 hydro-thermocycling.

savijanje materijala za bazu proteze. U većini ispitivanja materijali PMMA pokazuju veće vrijednosti apsorpcije vode od poliamidnih. Kada je PMMA uronjena u vodenu otopinu, otapala i druge topljive komponente mogu se dulje izlijevati, a voda ili slina se apsorbiraju. Apsorbirana voda štetno utječe na fizikalna i mehanička svojstva smolastoga materijala za bazu proteze (33). Tijekom vremena, uglavnom zbog polarnih svojstava molekula smole, voda može omekšati akrilnu smolu koja djeluje kao otapalo akrilata i smanjuje čvrstoću materijala (34).

S druge strane, niska apsorpcija vode i topljivost poliamida pripisuju se amidnim skupinama (28) – što je viša koncentracija amidne skupine, to je veća apsorpcija vode. Predloženo je da se koncentracija amidne skupine poliamidnih materijala za baze proteze postavi na nisku razinu kao kod najčešće korištenih industrijskih materijala, poput najlona 6 ili 66 (27).

Rezultati ovog istraživanja u skladu su sa zaključcima Machada i suradnika (30) koji su istaknuli značajno smanjenje čvrstoće na savijanje PMMA-e termociklirane u 5000 ciklusa između 5 i 55 °C. Autori su to pripisali porastu temperature, što je uzrokovalo bržu difuziju molekula vode između polimernih lanaca, djelujući kao otapalo i omogućujući da se lanci lakše sklope jedni na druge pod opterećenjem. U drugoj studiji su Takahashi i suradnici (22) pripisali smanjenje otpornosti poliamida na savijanje termičkim promjenama koje uzrokuju kontinuiranu ekspanziju i kontrakcije te rezultiraju statičkim zamorom materijala. Može se zaključiti da je u ovom istraživanju produljeno uranjanje u vodu tijekom termocikliranja značajno smanjilo čvrstoću na savijanje materijala PMMA, a smanjenje čvrstoće na savijanje poliamida uglavnom je posljedica temperaturnih promjena.

Zaključci

Uključujući ograničenja ove studije *in vitro*, može se zaključiti sljedeće: termocikliranje je nepovoljno utjecalo na čvrstoću na savijanje poliamidnih i PMMA materijala za bazu proteze; termocikliranje u 3000 ciklusa značajno smanjuje čvrstoću savijanja materijala PMMA; termocikliranje u 5000 ciklusa značajno smanjuje čvrstoću na savijanje i PMMA-e i poliamidnog materijala za baze proteze.

Poliamidni materijal pokazao je znatno niže vrijednosti čvrstoće na savijanje od PMMA-e u svim ispitanim eksperimentalnim uvjetima; oba materijala imaju vrijednosti čvrstoće na savijanje unutar specifikacija ISO-a.

Sukob interesa

Nema sukoba interesa.

Many researchers (26-29) reported that water sorption and thermal changes (22, 30-32) decrease the flexural strength of denture base materials. In most of the studies, PMMA materials showed higher water sorption values than polyamide denture base materials. When PMMA is immersed in water solutions plasticizers and other soluble components may leach out over extended periods, while water or saliva is being absorbed. Absorbed water has a detrimental effect on the physical and mechanical properties of the resin denture base material (33). Over a period of time, primarily because of the polar properties of the resin molecules, water can soften an acrylic resin by acting as a plasticizer of acrylates and reducing the strength of the material (34).

On the other hand, the low water sorption and solubility of polyamides is attributed to the amide groups (28): the higher the amide group concentration, the greater the water sorption. It has been suggested that the amide group concentration of the polyamide denture base materials should be adjusted to a level as low as that of commonly used industrial materials such as nylon 6 or 66 (27).

The results of the present study are in agreement with the findings of Machado et al. (30), who reported a significant decrease in the flexural strength of PMMA thermocycled for 5000 cycles between 5 and 55°C. The authors attributed this to the increase of temperature, which caused water molecules to diffuse more rapidly between the polymer chains, acting as plasticizers and allowing the chains to slip over each other more easily under load. In another study, Takahashi et al. (22) attributed the decrease of polyamide flexural strength to the ongoing thermal changes that cause continuous expansions and contractions and lead to static fatigue of the material. It may be concluded that in the present study the prolonged immersion in water during thermocycling led to significant reduction of the flexural strength of PMMA material, while the decrease of flexural strength of polyamide was mostly due to temperature changes.

Conclusions

Within the limitations of this *in vitro* study, the following conclusions may be drawn:

Thermocycling adversely affected the flexural strength of polyamide and PMMA denture base materials. Thermocycling at 3000 cycles significantly reduced the flexural strength of PMMA denture base material. Thermocycling at 5000 cycles significantly reduced the flexural strength of both PMMA and polyamide denture base material.

The polyamide denture base material exhibited significantly lower flexural strength values than PMMA for all the experimental conditions tested.

Both materials presented flexural strength values within the relative ISO 1567 specification.

Conflict of interest

None declared

Abstract

Objective: The aim of the present study was to evaluate the influence of thermocycling on the flexural strength of a polyamide base denture material. **Materials and methods:** A polyamide denture base material (Valplast) was tested, whereas a PMMA material (Vertex) was used as a control. Thirty specimens of each material were fabricated for flexural strength testing according to ISO 1567. They were prepared and stored in water at 37ºC for 48 hours. The specimens of each material were divided into three equal groups (n=10). Flexural strength testing was performed immediately after water storage and after thermocycling (5ºC / 55ºC, 2 c/min) for 3000 and 5000 cycles. A three point flexural test was performed on a universal testing machine at a crosshead speed of 5 mm/min. The final flexural strength was calculated using the formula: $F_S = 3\ PL/2\ bd^2$. A two-way ANOVA with post-hoc analysis using Tukey's procedure was applied at .05 level of statistical significance. **Results:** A statistically significant reduction in flexural strength was recorded after thermocycling at 3000 cycles for PMMA and at 5000 cycles for both materials. The flexural strength of PMMA was significantly higher compared to polyamide for all the conditions tested ($p<0.05$). **Conclusion:** Thermocycling had a significant adverse effect on the flexural strength of polyamide and PMMA denture base materials.

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Key words
Denture bases; Flexural Strength; Resins, Synthetic; Polyamide; Nylons; Materials Testing

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