ACTA
STOMATOLOGICA
CROATICA
www.ascro.net

Acta Stomatol Croat. 2006;40(2):151-61.

IZVORNI ZNANSTVENI RAD
ORIGINAL SCIENTIFIC PAPER

Ante Lončar[1], Denis Vojvodić[2], Franjo Matejiček[3], Vjekoslav Jerolimov[2]

Savojna čvrstoća materijala za izradbu proteznih baza

Flexural Strength of Denture Base Materials

1 Dom zdravlja Zapad, Zagreb
Health Center "Zapad", Zagreb, Croatia
2 Zavod za stomatološku protetiku Stomatološkog fakulteta Sveučilišta u Zagrebu
Department of Prosthodontics, School of Dental Medicine, University of Zagreb, Zagreb, Croatia
3 Strojarski fakultet u Slavonskom Brodu, Sveučilište J.J. Strossmayera u Osijeku
School of Engineering in Slavonski Brod, University "Josip Juraj Strossmayer" in Osijek, Slavonski Brod, Croatia

Sažetak
Lomovi polimernoga materijala jedan su od najčešćih razloga za popravak pomičnih proteza. Zato ih se neprestance nastoji ojačati te su tako već proizvedeni i polimerni materijali visoke otpornosti na udarac. Svrha ovog istraživanja bila je metodom kratke grede odrediti čvrstoću na savijanje materijala Ivocap „Plus" High Impact, uz uporabu injekcijske metode polimerizacije te uspordba s čvrstoćom na savijanje materijala Meliodent Heat Cure, primjenom klasične tlačno-toplinske polimerizacije i to odmah nakon polimerizacije i provedenih postupaka umjetnog ostarivanja uzoraka pohranjivanjem u vodi na temperaturi od 37° C tijekom 28 dana te termocikliranja.
Istraživanje je pokazalo statistički znatno više vrijednosti čvrstoće na savijanje (130,1-150,65 MPa) materijala Ivocap „Plus" u odnosu prema materijalu Meliodent (109,2-110,3 MPa) u skupinama uzoraka ispitanima nakon polimerizacije i pohranjivanja u vodi, dok u skupini uzoraka testiranih nakon termocikliranja nije bilo statistički znatne razlike (148,75:156,25 MPa). Između uzoraka izrađenih od istog materijala nije bilo statistički znatne razlike, iako su obavljeni postupci umjetnoga ostarivanja.

Zaprimljen: 1. prosinca 2005.
Prihvaćen: 4. svibnja 2006.

Adresa za dopisivanje
Dr.sc. Ante Lončar
DZ "Zapad"
Prilaz baruna Filipovića 11
10000 Zagreb, Hrvatska
anteloncar@zg.t-com.hr

Ključne riječi
polimeri; dentalni materijali; zubna proteza

Uvod

Od početka četrdesetih godina prošlog stoljeća, kada se počeo koristiti kao materijal za izradbu pomičnih proteza, kao najpouzdaniji se pokazao metilmetakrilat. No, bez obzira na sve prednosti njegov velik nedostatak i dalje je podložnost lomu. Lomovi polimernoga materijala proteznih baza jedni su od najčešćih (64%) razloga za popravak pomičnih proteza (1-4). Teoretski, bezubi pacijent ne bi mogao slomiti potpunu protezu zbog razmjerno visoke statičke čvrstoće protezne konstrukcije i slabih okluzijskih žvačnih sila koje se postižu uporabom pomičnih proteza (5, 6). No, sve češćom uporabom

Introduction

Since the beginning of the 40s of the last century, when it was used as denture base material for the first time, methyl-metacrylate has proved to be the most reliable material. Notwithstanding the advantages, its great disadvantage is still a tendency to fracture. Fractures of the denture base polymer material are one of the most frequent (64%) reasons for denture repair (1-4). Theoretically, an edentulous patient could not be able to fracture a denture due to a reasonable high static rigidity of the denture construction and weak masticatory forces that are developed in the use of removable dentures (5,6).

implantata, pa i za sidrenje pomičnih proteza, povećavaju se i žvačne sile kojima su izvrgnute protetske konstrukcije (7,8). Ali, presudan je utjecaj zamora materijala na čvrstoću na savijanje polimernoga materijala i upravo je on razlog za lomove proteza (9,10). Lom gornje potpune proteze najčešći je u medijalnoj liniji (11) i može se reći da se događa upravo zbog spomenutoga zamora materijala (9). Matthews i Wain (12) pronašli su da je tijekom funkcije najveći stres u materijalu na poliranoj palatinalnoj površini proteze odmah iza prednjih zuba. Zato su i zaključili da je iz navedenoga razloga baš taj palatinalni dio baze proteze, odmah iza prednjih zuba, jedna od najčešćih lokacija medijalnog loma proteze, uz već dobro poznati duboki vestibularni urez kojim se zaobilazi frenulum gornje usne (2). Za razliku od gornje totalne proteze, kod donje proteze zamor materijala nije glavni uzrok lomova, nego u oko 80% slučajeva oni nastaju zbog udarca ako padnu na tvrdu površinu (2).

Proteze od polimera nastojale su se ojačati ili modificiranjm sastava gradivnoga materijala ili, pak, ugrađivanjem ojačanja u akrilat kako bi se povećala zamorna čvrstoća na savijanje i čvrstoća na udarac (13).

Tako su se na tržištu pojavili i polimerni materijali visoke otpornosti na udarac - bili su čvršći i žilaviji od dotadašnjih polimera za izradbu proteznih baza (14, 15).

Još jedan velik nedostatak polimernih materijala jest podložnost nekontroliranim dimenzijskim promjenama zbog polimerizacijske kontrakcije. Zato se pokušavalo prevladati i taj nedostatak kompenzirajući polimerizacijsku kontrakciju materijala kako bi se postigla potpuna reprodukcija izmodeliranoga voštanog objekta i omogućilo što bolje prilijeganje izrađene pomične proteze na njezino ležište (16-18).

Kako ističe proizvođač, postupak SR-IVOCAP (Ivoclar, Schaan, Liechtenstein) u cijelosti rješava problem dimenzijskih promjena, uz povećanu čvrstoću samoga materijala (19). Zato bi taj postupak trebao biti osobito pogodan za totalne pomične proteze, djelomične pomične proteze, okluzalne splintove - nagrizne udlage, ortodontske naprave te za podlaganja pomičnih proteza.

Prednosti postupka očituju se u sljedećem:

- u kapsuli dozirani materijal mehaničkim miješanjem postaje homogen i spreman za polimerizaciju, a izbjegava se kontakt tehničara s monomerom;
- konstantno visokoj kvaliteti materijala;

With the ever-increasing use of implants, even for the anchorage of removable dentures, bite forces that are developing on the denture base are growing (7,8). But, the influence of material wear on flexural strength of the material is decisive, being the sole reason for denture fractures (9, 10). Medial line fracture of the upper denture is the most frequent (11), and it can be said that it occurs exactly because of the wear (9). Matthews and Vain (12) have reported that during function, the great stress is exhibited on the polished palatinal surface just behind the frontal teeth. They concluded that this palatinal portion of the denture base is one of the most frequent locations of medial line fracture; together with the well-known deep vestibular cut that opens the location of the upper lip frenum (2). Contrary to the upper total denture, fracture of the lower denture is not caused by wear, but occurs due to the fall on the hard surface (80% of the cases, 2).

Polymer dentures may be strengthened by modification of the material or by incorporating various enhancements into the acrylate that enhance the flexural strength and resistance to fall (13).

Subsequently, new, stronger and more resistant polymer materials for denture base appeared on the market (14, 15).

Another great disadvantage of polymer materials is a tendency to dimensional changes due to contraction during polymerization. An attempt was made for compensation in order to achieve complete reproduction of the modeled object in wax and enable better contact of the denture and its bed (16-18).

As outlined by the manufacturer, SR-IVOCAP (Ivoclar Vivadent, Schaan, Liechtenstein) procedure completely solves the problem of dimensional changes, with added hardness of the material itself (19). Therefore, this procedure should be especially applicable to totally removable dentures, partially removable dentures, occlusal splints and orthodontic appliances, as well as for relining of dentures.

The advantages of the procedure are mirrored in the following: (a) by mechanical mixing, the material in the capsule becomes homogeneous and ready for polymerization; contact of the dental technician with the monomer is avoided; (b) high quality of the material is constant; (c) precise occlusion, i. e. there is no disturbance in the occlusion after polymerization; (d) after polymerization, the dentures are not porous, even the big ones; (e) a controlled polymerization in enabled; it starts in the frontal segment of the cuvette and slowly spreads towards the injectional canal; a constant flow of the material compen-

- preciznoj okluziji, tj. nema poremećaja u okluziji nakon polimerizacije materijala pri izradbi proteza;
- nakon polimerizacije proteze nisu porozne, čak ako su i velikih dimenzija;
- omogućena je kontrolirana polimerizacija koja počinje u prednjem segmentu kivete i postupno se širi prema injekcijskom kanalu. Stalan dotok materijala kompenzira polimerizacijsku kontrakciju i daje homogeni materijal visoke čvrstoće te dobar ležaj za proteze i dobro prisisavanje proteze na ležište;
- biokompatibilnosti materijala spram ležišta zbog homogenosti i jednoličnosti materijala;
- optimalnom poliranju i visokoj otpornosti na lom (16-19).

Materijal Ivocap, koji se koristi za zubne proteze nabavlja se pod tvorničkim nazivom Ivocap "Plus" High Impact (Ivoclar), a sastoji se od praha (polimetilmetakrilat 90,5 tež.%, ko-polimer 8,0%, benzoilperoksid 1,4%, pigment 0,1%) i tekućine (metil-metakrilat 88,4 tež.%, dimetakrilat 5,6%, ko-polimer 6,0%) (19).

Svrha istraživanja

Različita ojačanja i poboljšanja kakvoće polimera uporabom različitih tehnologija već su opisana u literaturi - kako stomatološkoj tako i tehničkoj. No, rezultati su često kontradiktorni, a upute i objašnjenja dentalnih tvrtki koje proizvode materijale za baze pomičnih proteza obično su pristrani i rijetko se slažu s rezultatima objektivnih istraživanja.

Zato se ovim istraživanjem želi ispitati vrijednosti čvrstoće na savijanje uobičajenog polimera za protezne baze tlačno-toplinskim postupkom te polimera za protezne baze injekcijskim postupkom. Kako bi se simulirao i utjecaj starenja materijala na čvrstoću na savijanje, uzorci su testirani nakon:

- polimerizacije materijala;
- 28 dana pohranjivanja uzoraka u destiliranoj vodi na temperaturi od 37° C;
- provedenog postupka s cikličkim promjenama temperature vodenog miljea u koji su uzorci potapani.

Dobiveni rezultati međusobno su uspoređeni i statistički obrađeni, kako bi se ustanovila statistička znatnost (značajnost) razlika koja bi pokazala koji je materijal bolji za kliničku uporabu.

Materijali i postupci

U ovom istraživanju uporabljeni su polimerni materijali Meliodent Heat Cure (Heraeus Kul-

sates for the polymerization contraction and yields homogeneous material of high flexural strength, as well as good fit of the denture; (f) biocompatibility of the material due to its homogeneity; (g) optimal polishing and high flexural strength (16-19).

Ivocap material that is used for dentures can be obtained by the name of Ivocap "Plus" High Impact (Ivoclar Vivadent, Schaan, Liechtenstein). It is comprised of powder (polymethyl-metacrylate 90.5 w%, co-polymer 8.0%, benzoil-peroxide 1.4% pigment 0.1%) and liquid (methyl-metacrylate 88.4 w%, dimetacrylate 5.6%, co-polymer 6.0%) (19).

Aim of study

Different strength and quality enhancers of the polymer are already described in literature, both dental and technical. The results are often contradictory, and instructions and explanations of the dental companies that produce denture base materials are usually biased and only rarely comply with the results of the objective investigations.

It is the aim of this study to assess the values of flexural strength of the common polymer used for denture bases produced by the pressure-heat procedure, and polymer produced by the injectional procedure. In order to simulate the ageing process the samples were tested after: (a) material polymerization, (b) 28 days of storage in distilled water at 37°C, and (c) cyclical changes of the temperature of the water milieu in which the samples were immersed.

The results were statistically analyzed and compared in order to obtain statistical significance that would give information about which material is better for the clinical use.

Material and Methods

Materials used in this study were Meliodent Heat Cure (Heraeus Kulzer, Hanau, Germany) and Ivocap "Plus" High Impact (Ivoclar Vivadent, Schaan, Liechtenstein). We manufactured polymer quadratic samples with smooth surfaces (18×10×3 mm).

zer, Hanau, Njemačka) i Ivocap "Plus" High Impact (Ivoclar, Schaan, Liechtenstein), kako bi se izradili polimerni uzorci glatke površine u obliku kvadra, dimenzija: dužina 18 mm, širina 10 mm, debljina 3 mm.

Za izradbu uzoraka tlačno-toplinskim postupkom najprije su pripremljeni voštani modeli već opisane veličine uzoraka, zatim su stavljeni u Moldano (Hareus Kulzer, Dormagen, Njemačka) - tvrdom sadrom ispunjenu kivetu na udaljenost od najmanje 1 cm od njezina ruba. Kako postupak SR-IVOCAP (Ivoclar) sam po sebi već zahtijeva posebne kivete, navedeni voštani modeli postavljeni su na voštane kanale za ubrizgavanje debljine 3 mm s kojima su bili povezani plastičnim lijevkom. Zatim su također uloženi u Moldano - tvrdom sadrom ispunjenu kivetu ovog sustava na najmanje 1 cm udaljenosti od njezina ruba. Nakon stvrdnjavanja sadre rastvorene su kivete obaju sustava polimerizacije te je vosak ispran u uređaju za ispiranje (Type 5522, KaVo EWL, Biberach, Njemačka). Nakon toga su sadrene plohe u tankom sloju dva puta izolirane fluidom Ivoclar Separating (Ivoclar).

Tlačno-toplinski polimerizirajući polimer (Meliodent Heat Cure) zamiješan je prema uputama proizvođača o miješanju polimera i monomera te postavljen u impresije u sadri jednog dijela kivete. Zatim je preko njega postavljen drugi dio kivete i zatvoren te je kiveta stavljena u hidrauličnu prešu (Zlatarne Celje, Celje, Slovenija) i stlačena pod pritiskom od 200 bara. Kiveta je zatim premještena u ručni škripac te je obavljen postupak polimerizacije u polimerizacijskom uređaju (Type 5518, KaVo EWL, Biberach, Njemačka) u skladu s uputama proizvođača, tj. kiveta je stavljena u kipuću vodu - zagrijavanje je prekinuto nakon 15 minuta. Zatim se ponovno kuhala u kipućoj vodi 20 minuta te je ostavljena da se polako hladi u vodenoj kupelji.

Kod injekcijske metode priređena je kapsula Ivocap "Plus" High Impact (Ivoclar) polimernoga materijala, tako da je monomer iz bočice uliven u kapsulu s polimernim prahom i pet minuta protresen u Cap-vibratoru (Ivoclar). Obje polovice kivete oprezno su sastavljene i postavljene u zatezni okvir, umetnuta je kapsula s polimernim materijalom i priključen sustav zračnog potiska te je kapsula opterećena tlakom od 6 bara tijekom 5 minuta. Zatim je priključena kiveta 35 minuta položena u kadu za polimerizaciju (Ivoclar) u kipuću vodu. Nakon toga je premještena u hladnu vodu, ali je i dalje ostala priključena na tlak od 6 bara tijekom dvadesetominutnog hlađenja.

Wax models of these dimensions were fabricated and immersed in Moldano (Heraus Kulzer, Hanau, Germany) cuvettes filled with hard plaster, at least 1 mm from its margin. Since SR-IVOCAP (Ivoclar Vivadent, Schaan, Liechtenstein) procedure requires special cuvettes, wax models were put on wax canals for the injection (3 mm thick), to which they were connected with a plastic. Subsequently they were immersed in Moldano cuvettes filled with hard plaster, at least 1 mm from its margin. After plaster hardening, both cuvettes of the polymerization system were opened and the wax was rinsed in the rinsing machine (Type 5522, KaVo EWL, Biberach, Germany). Plaster surfaces were isolated twice in a thin layer using Ivoclar Separating fluid (Ivoclar Vivadent, Schaan, Liechtenstein).

Pressure-heat polymerization polymer (Meliodent Heat Cure) was mixed according to the manufacturer's instructions, and positioned in the plaster impressions of one part of the cuvette. The cuvette was closed and put in a hydraulic press (Zlatarne Celje, Celje, Slovenia) under 200 bars. Cuvette was subsequently moved to a manual bench vice and the polymerization was performed in a polymerization machine (Type 5518, KaVo EWL, Biberach, Germany), according to the manufacturer's instructions – the cuvette was immersed in hot water and the heating was stopped after 15 minutes. It was subsequently left in boiling water for 20 minutes and slowly cooled in a water bath.

The injectional method was performed in a way that the Ivocap "Plus" High Impact (Ivoclar Vivadent, Schaan, Liechtenstein) capsule was prepared. The monomer from the bottle was poured in the capsule that contained the polymer powder. It was shaken for 5 minutes in Cap vibrator (Ivoclar Vivadent, Schaan, Liechtenstein). Both parts of the cuvette were joined and the system of pressurized air was connected at 6 bar pressure during 5 minutes. The cuvette was immersed in boiling water in a polymerization bath for 35 minutes, moved to cold water, still connected to 6 bar pressure during 20 minutes of cooling.

After polymerization and cooling the cuvettes of both systems were opened and the samples were manually cleaned of plaster. Polymer excess was removed by a carbide bur (Ivomill, Ivoclar Vivadent, Schaan, Liechtenstein). The margins were polished with sandpaper (Sianor 7/0B, Frauenfeld, Switzerland). The samples of the stated dimensions were controlled with a caliper (Dentarium 042-751, Dentarium, Ispringen, Germany), with allowed deviation of 0.05 mm.

Nakon polimerizacije i završetka hlađenja kivete obaju sustava su rastvorene, uzorci ručno očišćeni od sadre, a suvišak polimera uklonjen je s uzoraka karbidnim glodalom – frezom (Ivomill, Ivoclar). Obrađivanje rubova uzoraka dovršeno je brusnim papirom (Sianor 7/0 B, Frauenfeld, Švicarska). Dobiveni uzorci već navedenih dimenzija kontrolirali su se pomičnim mjerilom (Dentarium 042-751, Ispringen, Njemačka), uz dopušteno odstupanje od 0,05 mm.

Opisanim postupcima izrađeno je po 15 uzoraka za svaki polimerizacijski postupak, a oni su, pak, podijeljeni u tri brojčano jednake skupine od po 5 uzoraka u svakoj, te su ispitani metodom kratke grede (20) kako bi se odredila čvrstoća na savijanje, i to:

1. skupina uzoraka testirana je nakon polimerizacije;
2. skupina uzoraka nakon polimerizacije potopljena je u destiliranu vodu na temperaturi od 37 ° C i pohranjena 28 dana u termostatu (Btuj, Poznan, Poljska), a zatim je testirana;
3. skupina uzoraka nakon polimerizacije podvrgnuta je postupku termocikliranja, tj. naizmjeničnom uranjanju u toplu i hladnu vodu i pohranjivanju na hladnom zraku. Za postupak termocikliranja koristila se Hanssonova metoda (21) prema kojoj se uzorci potapaju u kipuću vodu 25 minuta, a zatim 5 minuta u vodu temperature 10° C. Taj se postupak ponavlja 10 puta. Nakon toga uzorci se ostavljaju jedan sat na zraku na temperaturi od -22 ° C i ponovno se 5 minuta potapaju u kipuću vodu. Nakon provedenog termocikliranja uzorci su testirani.

Svi uzorci ispitani su uporabom univerzalne kidalice za ispitivanje materijala (Amsler, Schafhausen, Švicarska) koja ima umjernicu za sva područja rada, pa tako i za područje sile od 0 do 3000 N, a u tom je rasponu i obavljeno ispitivanje.

Uzorci su bili opterećeni prema shemi prikazanoj na Slici 1, s brzinom spuštanja pritiskivača od 1,5 mm/s.

Mjerena je sila koja je uzrokovala lom uzoraka te je izračunata čvrstoća na savijanje prema formuli (22):

$$\sigma_{maks} = \frac{F_{maks} \cdot l}{4} \frac{6}{b \cdot h^2}, \quad \frac{N}{mm^2}$$

u kojoj je:

F_{maks} – sila pritiskivača, N,
l – raspon oslonaca, mm (ovdje uvijek 15 mm),
b – širina uzorka, mm (ovdje uvijek 10 mm),
h – visina uzorka, mm (ovdje uvijek 3 mm).

Described procedures were used for obtaining 15 samples for each polymerization procedure. The samples were divided in three groups containing 5 samples that were tested by the short bar system (20) in order to determine flexural strength: (1) sample group tested after polymerization, (2) sample group that was immersed in thermostat (Btuj, Poznan, Poland) filled with distilled water (37°C), left there for 28 days and tested afterwards, (3) sample group that was thermocycled after polymerization (alternating immersion in warm and cold water, storage in cold air). Thermocycling procedure was performed according to Hansson's method (21). The method uses immersion in boiling water for 25 minutes and immersion in cold (10°C) water for 5 minutes. This procedure is repeated 10 times. Afterwards, the samples are left to dry in air at -22°C and afterwards immersed in boiling water for 5 minutes. The samples were tested after thermocycling.

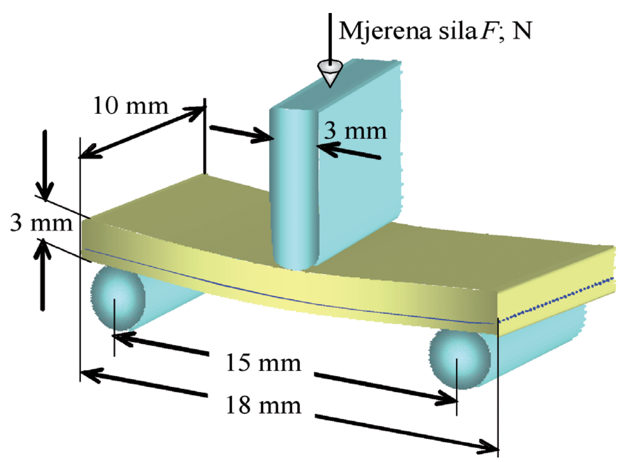
All samples were tested by a universal breaker for material testing (Amsler, Schaffhausen, Switzerland) that has a scale for all areas of work, even for the workforce from 0 to 3000 N. It was in this range that the test was performed.

The samples were loaded according to the scheme (Figure 1) and the speed of the loader was 1.5 mm/s.

The force that caused the fracture was measured, as well as flexural strength, according to the formula (22):

$$\sigma_{maks} = \frac{F_{maks} \cdot l}{4} \frac{6}{b \cdot h^2}, \quad \frac{N}{mm^2}$$

F_{max} – force of the loader (N),
l – distance between posts (15 mm),
b – width of the sample (10 mm),
h – height of the sample (3 mm).



Slika1. Dimenzija i shema opterećivanja uzoraka
Figure 1 Dimensions and scheme of specimens loading

Brojčani rezultati vrijednosti čvrstoće na savijanje dobiveni u istraživanju obrađeni su računalom Intel Pentium 4, u programu SPSS (Chicago, Illinois, SAD). U statističkoj obradi podataka obavljena je deskriptivna analiza istraživanih varijabli i jednosmjerna analiza varijance, a za određivanje statističke razlike između čvrstoća na savijanje uzoraka izrađenih iz dvaju različitih polimernih materijala primijenjen je Studentov t-test za nezavisne uzorke.

Rezultati

Sve vrijednosti čvrstoće na savijanje uzoraka izražene su u N/mm^2, tj. u megapaskalima (MPa).

Izrađena je Tablica 1 u kojoj su aritmetičke sredine, standardne devijacije, standardne pogreške, donja i gornja granica intervala pouzdanosti (95%) te izmjerene minimalne i maksimalne vrijednosti čvrstoće na savijanje za svaku skupinu i podskupinu uzoraka.

Numeric results of the flexural strength have been processed by a computer, using a statistical package SPSS (SPSS Inc., Chicago, USA). Statistical analysis was performed using descriptive statistics and one-way analysis of variance. The statistically significant difference between flexural strengths of samples was performed using Student's t-test for independent samples.

Results

All values of flexural strength were in N/mm^2, i.e. megaPascals (MPa). Table 1 represents mean values, standard deviations, standard errors, upper and lower values of the confidence interval (95%), as well as minimum and maximum values of flexural strength for each group and subgroup of samples.

Tablica 1. Deskriptivna statistika vrijednosti čvrstoće na savijanje ispitivanih uzoraka; MAT. = materijal; P = postupci ostarivanja uzoraka - uzorci testirani: 1 = nakon polimerizacije, 2 = nakon 28 dana pohranjivanja u vodi na temperaturi od 37^o C, 3 = nakon termocikliranja; N = broj uzoraka; x = aritmetička sredina (MPa); SD = standardna devijacija; SE = standardna pogreška; min.-maks. = raspon minimalne i maksimalne vrijednosti čvrstoće na savijanje.
Table 1 Descriptive statistics of flexural strength values of investigated specimens; MAT. = material; P = specimen ageing procedures - specimens tested: 1 = after polymerisation, 2 = after 28 days immersion in water temperature 37^{o}C, 3 = after thermocycling, N = number of specimens; x = arithmetic mean (MPa); SD = standard deviation; SE = standard error; Min.-Max.= range of minimal and maximal values of flexural strength.

Mat.	P	N	x	SD	SE	95% interval pouzdanosti • 95% interval reliability		Min.- Max.	
						Donja granica • Lower limit	Gornja granica • Upper limit		
Melio-dent	1	5	109,2	14,1459	6,3262	91,6356	126,764	92,5	124,25
	2	5	110,3	46,9938	21,0163	51,9495	168,651	28,75	144
	3	5	148,75	10,5327	4,7104	135,672	161,828	132,5	160
Ivocap	1	5	130,1	30,468	13,6257	92,269	167,931	85	160
	2	5	150,65	6,056	2,7083	143,131	158,17	144	158,75
	3	5	156,25	8,3385	3,7291	145,896	166,604	145	166,25

Kako bi se odredila normalnost distribucije vrijednosti čvrstoće na savijanje ispitivanih uzoraka, obavljen je Kolmogorov-Smirnov test koji je pokazao normalnu distribuciju (P>0,05).

Jednosmjerna analiza varijance vrijednosti aritmetičkih sredina čvrstoće na savijanje ispitivanih uzoraka, pokazala je kako ne postoji statistički znatna razlika (P>0,05) između pojedinih podskupina uzoraka testiranih nakon polimerizacije i provedenih postupaka umjetnoga ostarivanja, a izrađenih od istoga polimernoga (Tablica 2).

Studentov t-test upozorio je na sljedeće razlike (Tablica 3). Ivocap Plus materijal imao je statistički znatno (P<0,05) više vrijednosti čvrstoće na sa-

In order to determine the normality of distribution of flexural strength values, Kolmogorov-Smirnov test was performed, showing normal distribution (p>0.05).

One-way analysis of variance of mean values of flexural strength showed no statistically significant difference (p>0.05) between subgroups of the same material that were tested after polymerization and after artificial ageing procedures (Table 2).

Student's t-test showed following differences (Table 3). Ivocap "Plus" had significantly (p<0.05) higher values of flexural strength when compared to Meliodent Heat Cure in samples tested immediately after polymerization, and after 28 days in distilled vijanje od materijala Meliodent Heat Cure, i to kod uzoraka testiranih odmah nakon polimerizacije i nakon što su uzorci bili 28 dana pohranjeni u destiliranoj vodi na temperaturi od 37° C. Između Ivocap Plus i Meliodent Heat Cure uzoraka ispitanih nakon provedenog postupka termocikliranja nije bilo statistički znatne razlike (P>0,05) u vrijednostima čvrstoće na savijanje.

water. There was no significant difference in materials after thermocycling (p>0.05) regarding flexural strength.

Tablica 2. Jednosmjerna analiza varijance između skupina uzoraka; MATER.= materijal; DF = stupnjevi slobode; F = F-vrijednost; P = vjerojatnost, *= statistički znatno na razini pouzdanosti od 95%, NS = nije statistički znatno na razini pouzdanosti od 95%

Table 2 One way analysis of variance between specimen groups; MATER.=material; DF=degrees of freedom; F=F-value; P=probability, *=significant at the level of 95%, NS=not significant at the level of 95%

Mater.	Analiza • Analysis	Suma kvadrata • Sum of squares	DF	Kvad. arit. sred. • Square of mean	F	P
Melio-dent	Između skupina • Between groups	5073,03	2	2536,51	3,02	0,087 NS
	Unutar skupina • Within groups	10077,9	12	839,821		
	Ukupno • Total	15150,9	14			
Ivocap	Između skupina • Between groups	1895,81	2	947,904	2,749	0,104 NS
	Unutar skupina • Within groups	4138,03	12	344,835		
	Ukupno • Total	6033,83	14			

Tablica 3. Prikaz vrijednosti T-testa između podskupina uzoraka; P = postupci ostarivanja uzoraka - uzorci testirani: 1 = nakon polimerizacije, 2 = nakon 28 dana pohranjivanja u vodi na temperaturi od 37° C, 3 = nakon termocikliranja; (x) = aritmetička sredina u MPa, * = statistički znatna razlika na razini vjerojatnosti od 95%.

Table 3 Account of t-test values between specimens subgroups; P = specimen ageing procedures - specimens tested: 1 = after polymerisation, 2 = after 28 days immersion in water temperature 37°C, 3 = after thermocycling, (x) = arithmetic mean in MPa, *= statistically significant difference on the level of 95%

Skupine	Podskupine (P)	1	2	3
Meliodent (x)		109,2	110,3	148,75
Ivocap (x)		130,1	150,65	156,25
Vrijednosti t-testa		2,80*	3,51*	1,25

Rasprava

Testovi čvrstoće na savijanje smatraju se prikladnima za polimerne dentalne materijale (23-26). U usporedbi s tim testovima izravna mjerenja vlačne čvrstoće tehnički se teže obavljaju, ne omogućuju testiranje samo izabrane površine uzorka na vlak i ne odražavaju deformaciju pri savijanju koja se događa pri žvačnom opterećenju. Kompresivna čvrstoća je samo indirektno, i to vrlo kompleksno, povezana s određenim vrstama loma kod vlačnih i smičnih opterećenja. Mjerenje dijametralne vlačne čvrstoće zahtijeva, pak, od materijala da ne pokazuje sposobnost plastičnog tečenja, što nije svojstveno većini dentalnih polimernih materijala.

Molekule vode prodiru u prostore između polimernih lanaca, zadržavaju se u njima i razmiču polimerne lance. Tako mogu smekšati proteznu bazu, jer apsorbirana voda djeluje kao plastifikator PMMA, što proizlazi iz interakcije s polimernom struk-

Discussion

Flexural strength tests are considered to be appropriate for polymer dental materials (23-26). In comparison to these tests, direct measurements of tensile strength are technically more demanding, do not enable testing of the selected surface of the sample and do not resemble the flexural deformation that occurs during masticatory load. Compressive strength is only indirectly, and in a very complex way, correlated to specific fractures that occur during flexural and tensile load. Measurement of diametric tensile strength requires that a material does not represent the characteristic of plastic flow, which is not inherent to most of the dental polymer materials.

Molecules of water that penetrate into the areas between polymer chains remain there and separate these chains. In that way they may soften the denture base, since absorbed water can act as PMMA turom te smanjuje mehanička svojstva gradivnoga materijala, a posljedica je smanjenje čvrstoće na savijanje i modusa elasticiteta (27).

Ulazak vode primarno je uzrokovan difuzijom, a dio apsorpcije polarnošću polimernih lanaca koja se javlja zbog nezasićenih veza unutar samih molekula i neuravnoteženih međumolekularnih sila (27).

Zato su za istraživanja u području dentalnih materijala nužni postupci umjetnog ostarivanja kao što su skladištenje pod vodom i cikličke promjene temperature, kako bi se odredila trajnost mehaničkih svojstava materijala u zahtjevnom mediju usne šupljine (28).

Različiti autori koriste se različitom duljinom skladištenja u vodi, pa i različitim temperaturama - sobnom ili 37° C. No, važno je istaknuti da se bitno smanjenje vrijednosti čvrstoće na savijanje događa tijekom prva četiri tjedna imerzije u vodi, dok u sljedećem razdoblju više nema statistički znatnog smanjenja čvrstoće na savijanje (29). Zato je i u ovom istraživanju uporabljeno skladištenje uzoraka u vodi na temperaturi od 37° C i to 4 tjedna.

No, u ovom istraživanju nisu se smanjile vrijednosti čvrstoće na savijanje nakon četverotjednog pohranjivanja uzoraka u vodi temperature 37° C, nego je ona čak i porasla za 15% kod uzoraka izrađenih postupkom Ivocap. Nameće se zaključak da je pohranjivanje uzoraka u vodi prouzročilo relaksaciju stresa u samom materijalu nastalog polimerizacijskom kontrakcijom (30, 31), što se pokazalo kao mogući uzrok za povišenje vrijednosti čvrstoće na savijanje ispitivanih polimernih materijala.

Toplinski učinci termocikliranja kojima se oponaša unos tople i hladne hrane i/ili pića, mogu imati znatan utjecaj na mehanička svojstva polimernih materijala (32, 33) te na boju, sjaj, površinsku hrapavost i abrazijsku otpornost (33).

U ovom radu postupak termocikliranja nije uzrokovao pad vrijednosti čvrstoće na savijanje nego - dapače - porast, a osobito u skupini tlačno-toplinski polimeriziranih uzoraka (za 35%). Čini se da je u ovoj podskupini uzoraka porast temperature kod termocikliranja potaknuo efekt produžene polimerizacije, a posljedica je smanjenje rezidualnoga monomera, što je pak rezultiralo poboljšanjem mehaničkih svojstava polimernoga materijala, pa tako i povišenjem vrijednosti čvrstoće na savijanje. Velike standardne devijacije, uobičajene za takva istraživanja (28), uzrok su što nije i statistički potvrđeno znatno povišenje vrijednosti čvrstoće na savijanje nakon termocikliranja tlačno-toplinski polimeriziranih uzoraka, iako su te vrijednosti bile za 35% ve-

plastificator, a fact that emanates from the interaction with the polymer structure. It diminishes the mechanical properties of the material, resulting in lower flexural strength and lower modulus of elasticity (27).

Water entry is primarily caused by diffusion, and partly by the polarity of polymer chains that is caused by unsaturated molecules and unbalanced intermolecular forces (27).

Therefore, the investigation of dental materials requires procedures of artificial ageing, such as underwater storage and cyclic changes of temperature, in order to determine the longevity of mechanical properties of materials in the demanding environment such as the oral cavity (28).

Different authors use different periods of underwater storage, as well as different temperatures – room temperature or 37°C. It is important to stress that the important decrease of flexural strength value occurs during first four weeks of immersion, while the next period does not present statistically significant decrease (29). It is the reason why we used immersion in water at 37°C, during four weeks.

But, we did not measure the decrease of flexural strength values after four weeks immersion in water at 37°C. On the contrary, the valued increased, even 15% for samples produced by Ivocap procedure. One can consider that the immersion in water caused relaxation of the stress in the material that occurred during polymerization shrinkage (30, 31), which has been proven to be the possible cause for the increase of the flexural strength values for the tested polymer materials.

Effects of thermocycling that imitates cold and hot food/beverages can have a significant impact on mechanical properties of polymer materials (32, 33), as well as on the color, surface smoothness and resistance to abrasion (33).

Thermocycling procedure in this experiment did not exhibit a decrease of flexural strength values of samples, but – on the contrary – it resulted in an increase of values, especially in pressure-heat polymerization samples (35%). It seems that the increase of temperature in this sample subgroups resulted in the effect of prolonged polymerization, which can, in turn, result in the decrease of the residual monomer volume, enhancing mechanical properties of the material and increasing the flexural strength values. Great values of standard deviations, usual for this type of experiment (28), have caused the lack of statistical confirmation of significant increase of flexural strength values afće od onih izmjerenih odmah nakon polimerizacije i pohranjivanja u vodi. Nakon termocikliranja, u podskupini uzoraka izrađenih injekcijskim postupkom Ivocap, samo se neznatno povećala čvrstoća na savijanje (4%) u odnosu prema skupini uzoraka pohranjenih u vodi, te se može zaključiti da su vrijednosti čvrstoće na savijanje bile postojane bez obzira na provedene postupke umjetnog ostarivanja. Postojane vrijednosti čvrstoće na savijanje zapažene su i u istraživanju Archadiana i suradnika (34), iako su bile nižih vrijednosti (oko 100 MPa) u odnosu prema dobivenima u ovom istraživanju (130-156 MPa). Rezultati u ovom istraživanju također su viših vrijednosti i od onih Karacaera i suradnika (35) dobivenih injekcijskom metodom Palajet.

Rezultati t-testa pokazuju da su uzorci izrađeni injekcijskim postupkom Ivocap imali statistički znatno više vrijednosti čvrstoće na savijenje od uzoraka izrađenih klasičnim postupkom tlačno-toplinske polimerizacije, i to u skupinama ispitanima odmah nakon polimerizacije i pohranjivanja u vodi. To valja pripisati maloj poroznosti toga materijala s direktnim utjecajem na mehanička svojstva, na što su upozorili i drugi autori. Osim toga valja istaknuti da od dviju uobičajenih tehnika izradbe pomičnih proteza - ubrizgavajuća (injekcijska) i kompresijska tehnika (tlačno-toplinska) (36, 37) - ubrizgavajuća tehnika rezultira manjom polimerizacijskom kontrakcijom (36) i zato omogućuje veću točnost izradbe proteze u odnosu prema kompresijskoj tehnici (37). Manja polimerizacijska kontrakcija znači i manje šupljina u polimeru, pa je zato takav polimer otporniji na apsorpciju vode sa svim njezinim već opisanim posljedicama, a osobito ako su potaknute cikličkim promjenama temperature.

Zaključci

Na temelju rezultata dobivenih u istraživanju može se zaključiti da injekcijski Ivocap Plus High Impact materijal ima konstantno visoke vrijednosti savijne čvrstoće bez obzira na postupke umjetnoga ostarivanja, pa se svakako može preporučiti u kliničkoj praksi. No, uobičajeni tlačno-toplinsko polimerizirajući materijal Meliodent Heat Cure, iako nižih vrijednosti čvrstoće na savijanje, pokazao je porast vrijednosti nakon postupka termocikliranja. Zato valja preporučiti produženu polimerizaciju materijala iz te skupine, bez obzira na naputke proizvođača o trajanju (kratkoći) polimerizacije, kako bi se postigla bolja mehanička svojstva, a time i klinički dugotrajniji protetski rad.

ter thermocycling of pressure-heat polymerization samples, although the values were increased for 35% when compared to the values measured in samples immediately after polymerization and immersion in water. After thermocycling, the flexural strength value in the sample subgroup that was produced by Ivocap injectional procedure only slightly increased (4%), when they were compared to the samples immersed in water. It can be concluded that flexural strength values remained stable during artificial ageing procedures. Archadian et al. observed stable values (34), although they were lower (around 100 MPa) than in our study (130-156 MPa). The results of our study were higher than in the study of Karacaer et al. (35) that used Palacet injectional method.

T-test results show that the samples produced by the Ivocap injectional procedure had statistically higher values of flexural strength when compared to the classical pressure-heat polymerization technique and water storage. It should be attributed to the low porosity of the material that directly influences the mechanical properties. Other authors addressed this as well. It must be singled out that out of the two usual techniques of removable denture production – injectional and compression technique (pressure-heat) (36, 37) – the injectional technique results in lower polymerization shrinkage (36), therefore enabling greater precision in the production when compared to the pressure-heat polymerization procedure (37). Lower polymerization shrinkage means less porosity; such polymer is more resistant to water absorption and all of its consequences, especially if they are triggered by temperature changes.

Conclusions

Based on the results of this study, we can conclude that Ivocap “Plus” High Impact injectional material has constantly high values of flexural strength, with no regards to artificial ageing procedures. It can be safely recommended for clinical work. The pressure-heat polymerization material Meliodent Heat Cure, although showing lower flexure strength values, gave higher values after thermocycling. Therefore, one should propose a prolonged polymerization of material in this group, not regarding the manufacturer’s instructions on the (short) duration of the polymerization, in order to achieve better mechanical propertied of the material, and with it longer clinical duration of the product.

Abstract
Fractures of polymer material are one of the most frequent reasons for repair of removable dentures. Therefore, there is a continuous strive to strengthen them, and polymer materials of high resistance to fracture are being developed. The aim of this study was to determine the flexural strength of Ivocap "Plus" High Impact (Ivoclar Vivadent, Schaan, Liechtenstein) material using the method of "short bar" together with the injectional method of polymerization, as well as comparison of flexural strength of Meliodent Heat Cure material with the use of classic pressure-heat polymerization, immediately after polymerization and after artificial ageing by storage at 37°C during 28 days and thermocycling. The study showed significantly higher values of flexural strength (130.1-150.65 MPa) of Ivocap "Plus" compared to Meliodent (109.2-110.3 MPa) in groups of samples tested after polymerization and storage in water. In the group of samples tested after thermocycling there was no significant difference (148.75 compared to 156.25 MPa). Furthermore, there was no significant difference between the samples of same material although the procedure of artificial ageing was performed.

Received: December 1, 2005
Accepted: May 11, 2006

Address for correspondence
Ante Lončar D.D.S., Ph.D.
DZ "Zapad"
Prilaz baruna Filipovića 11
HR-10000 Zagreb
Croatia
anteloncar@zg.t-com.hr

Key words
polymers; dental materials; dentures

References

1. Vallittu PK, Lassila VP, Lappalainen R. Evaluation of damage to removable dentures in two cities in Finland. Acta Odontol Scand. 1993;51(6):363-9.
2. Hargreaves AS. The prevalence of fractured dentures. A survey. Br Dent J. 1969;126(10):451-5.
3. Darbar UR, Huggett R, Harrison A. Denture fracture - a survey. Br Dent J. 1994;176(9):342-5.
4. Zissis AJ, Polyzois GL, Yannikakis SA. Repairs in complete dentures: Results of a survey. Quintessence Dent Technol. 1997;23:149-55.
5. Schneider RL. Diagnosing functional complete denture fractures. J Prosthet Dent. 1985;54(6):809-14.
6. Lassila V, Holmlund I, Koivumaa KK. Bite force and its correlations in different denture types. Acta Odontol Scand. 1985;43(3):127-32.
7. Rangert BR, Sullivan RM, Jemt TM. Load factor control for implants in the posterior partially edentulous segment. Int J Oral Maxillofac Implants. 1997;12(3):360-70.
8. Gunne J, Rangert B, Glantz PO, Svensson A. Functional loads on freestanding and connected implants in three-unit mandibular prostheses opposing complete dentures: an in vivo study. Int J Oral Maxillofac Implants. 1997;12(3):335-41.
9. Smith DC. The acrylic denture. Mechanical evaluation, mid-line fracture. Br Dent J. 1961;110:257-67.
10. Vallittu PK. Fracture surface characteristics of damaged acrylic-resin-based dentures as analysed by SEM-replica technique. J Oral Rehabil. 1996;23(8):524-9.
11. Beyli MS, von Fraunhofer JA. An analysis of causes of fracture of acrylic resin dentures. J Prosthet Dent. 1981;46(3):238-41.
12. Matthews E, Wain EF. Stresses in denture bases. Br Dent J. 1956:100;167-71.
13. Uzun G, Hersek N, Tincer T. Effect of five woven fiber reinforcements on the impact and transverse strength of a denture base resin. J Prosthet Dent. 1999;81(5):616-20.
14. Neihart TR, Li SH, Flinton RJ. Measuring fracture toughness of high-impact poly(methyl methacrylate) with the short rod method. J Prosthet Dent. 1988;60(2):249-53.
15. Rodford RA. Further development and evaluation of high impact strength denture base materials. J Dent. 1990;18(3):151-7.
16. Strohaver RA. Comparison of changes in vertical dimension between compression and injection molded complete dentures. J Prosthet Dent. 1989;62(6):716-8.
17. Anderson GC, Schulte JK, Arnold TG. Dimensional stability of injection and conventional processing of denture base acrylic resin. J Prosthet Dent. 1988;60(3):394-8.
18. Salim S, Sadamori S, Hamada T. The dimensional accuracy of rectangular acrylic resin specimens cured by three denture base processing methods. J Prosthet Dent. 1992;67(6):879-81.
19. Ivoclar. SR Ivocap system: Instructions for use. Schaan: Ivoclar AG; 1995.
20. Lončar A, Vojvodić D, Komar D. Fiber-Reinforced Polymers - Part I: Basic and Construction Problems. Acta Stomatol Croat. 2006;40(1):72-82
21. Hansson O. Strength of bond with Comspan Opaque to three silicoated alloys and titanium. Scand J Dent Res. 1990;98(3):248-56.
22. Berg CA, Tirosh J, Israeli M. Analysis of Short Beam Bending of Fiber Reinforced Composites - Testing and Design. Philadelphia: American Society for Testing and Materials; 1970.
23. Ladizesky NH, Cheng YY, Chow TW, Ward IM. Acrylic resin reinforced with chopped high performance polyethylene fiber--properties and denture construction. Dent Mater. 1993;9(2):128-35.
24. Vallittu PK, Lassila VP, Lappalainen R. Transverse strength and fatigue of denture acrylic-glass fiber composite. Dent Mater. 1994;10(2):116-21.
25. Vallittu PK. Flexural properties of acrylic resin polymers reinforced with unidirectional and woven glass fibers. J Prosthet Dent. 1999;81(3):318-26.
26. Kanie T, Fujii K, Arikawa H, Inoue K. Flexural properties and impact strength of denture base polymer reinforced with woven glass fibers. Dent Mater. 2000;16(2):150-8.
27. Anusavice KJ. Phillips' Science of dental materials. 10th ed. Philadelphia: W. B. Saunders Co; 1996.
28. Vojvodić D. Ispitivanje različitih sustava veznih posrednika u fiksnoj protetici [dissertation]. Zagreb: Stomatološki fakultet; 1996.
29. Vallittu PK. Effect of 180-week water storage on the flexural properties of E-glass and silica fiber acrylic resin composite. Int J Prosthodont. 2000;13(4):334-9.
30. Schneider W, Powers JM, Pierpont HP. Bond strength of composites to etched and silica-coated porcelain fusing alloys. Dent Mater. 1992;8(3):211-5.
31. Saygili G, Sahmali SM, Demirel F. The effect of placement of glass fibers and aramid fibers on the fracture resistance of provisional restorative materials. Oper Dent. 2003;28(1):80-5.
32. Oshida Y, Hashem A, elSalawy R. Some mechanistic observation on water-deteriorated dental composite resins. Biomed Mater Eng. 1995;5(2):93-115.
33. Drummond JL, Bapna MS. Static and cyclic loading of fiber-reinforced dental resin. Dent Mater. 2003;19(3):226-31.

34. Archadian N, Kawano F, Ohguri T, Ichikawa T, Matsumoto N. Flexural strength of rebased denture polymers. J Oral Rehabil. 2000;27(8):690-6.
35. Karacaer O, Polat TN, Tezvergil A, Lassila LV, Vallittu PK. The effect of length and concentration of glass fibers on the mechanical properties of an injection- and a compression-molded denture base polymer. J Prosthet Dent. 2003;90(4):385-93.
36. Nogueira SS, Ogle RE, Davis EL. Comparison of accuracy between compression- and injection-molded complete dentures. J Prosthet Dent. 1999;82(3):291-300.
37. Huggett R, Zissis A, Harrison A, Dennis A. Dimensional accuracy and stability of acrylic resin denture bases. J Prosthet Dent. 1992;68(4):634-40.