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Evaluation of Elastomeric Impression Materials' Hydrophilicity: An *in vitro* Study

Procjena hidrofilnosti elastomernih otisnih materijala: istraživanje in vitro

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Abstract

Introduction: Hydrophilicity of dental impression materials is crucial for obtaining an accurate impression and necessary for the production of a well-fitting cast restoration. The most common technique for evaluation of hydrophilicity is a contact angle measurement. The aim of the present *in vitro* study was to compare the water contact angles of four groups of elastomeric impression materials, before and during setting. **Material and methods:** Flattened specimens (n=10) of tested impression materials were prepared by the use of a Teflon mold with specific dimensions. A 5μl droplet of deionized water fell on the specimen, and photos were taken using a Nikon D3200 DSLR camera and a 105 mm macro lens (Nikorr, Nikon) in specific time points. **Results:** The CAD/CAM material showed the highest contact angle measurements. The light body polyvinylsiloxane (PVS) material 1, polyether and vinylsiloxanether material showed comparable contact angle measurements especially at the initial time point. A statistically significant reduction of contact angles was reported during setup time for all PVS, PE and vinylsiloxanether materials, while the most expressed reduction of contact angle measurements, and thus the most significant increase of hydrophilicity were reported for light wash PVS material 2. **Conclusions:** The CAD/CAM impression material showed the most hydrophobic behavior. PVS materials showed excellent hydrophilicity. Polyether and polyvinyloxanether impression materials presented lower contact angle measurements, and thus superior hydrophilicity, compared with other tested materials initially and during setting. All tested impression materials presented a stepwise development of hydrophilicity during the setting stage.

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Introduction

Accuracy is the key word for an impression material to be considered clinically successful so that all the supragingival and subgingival prepared tooth details can be impressed and an accurate stone cast can be produced. Thus, accurate impression is necessary for the production of a well-fitting cast restoration (1, 2). Over the years, a variety of impression materials have been introduced in the field of prosthetic dentistry. Reversible hydrocolloids, alginate materials, polysulfides, condensation polysiloxanes, addition polyvinylsiloxanes (PVS) and polyethers (PE) are representative examples, each presenting advantages and drawbacks (3). Among the elastomeric impression materials, PVS and PE are the most commonly used materials in dental practice due to their favorable clinical properties and minimal dimensional change (4, 5).

Simplicity of use, high dimensional stability, and superior elastic recovery from undercuts, low viscoelastic properties,

Uvod

Preciznost je ključna riječ za otisne materijale koji se smatraju klinički uspješnima, a to znači da se mogu otisnuti svi detalji supragingivno i subgingivno od zuba i izraditi točan sadreni odljev. Zato je precizan otisak nužan za izradu dobro prilagođenoga nadomjestka (1, 2). Tijekom godina u području stomatološke protetike korišteni su različiti otisni materijali. Reverzibilni hidrokoloidi, alginati, polisulfidi, kondenzacijski polisiloksani, adicijski polivinilsiloksani (PVS) i polieteri (PE) reprezentativni su primjeri, no svaki ima prednosti i nedostatke (3). Među elastomernim otisnim materijalima u stomatološkoj praksi najčešće su korišteni PVS i PE zbog povoljnih kliničkih svojstava i minimalne promjene dimenzija (4, 5).

Jednostavnost uporabe, visoka dimenzijska stabilnost i elastični oporavak podminiranih mjesta, niska visokoelastična svojstva, karakteristike velikoga protoka i kratko stvrdnjahigh flow characteristics and short setting time are some of the advantages of the PVS impression materials (6, 7). These materials show hydrophobic behavior. As a result, moisture may negatively affect the accuracy of the definite impression (8, 9). Incorporation of several surfactants in the PVS matrix provides hydrophilic characteristics (7). PE impression materials containing copolymer tetrahydrofuran and ethylene oxide have a purely hydrophilic behavior, with the ability to wet the tooth and gingiva surface in presence of saliva (10).

A new impression material that combines the properties of PE and PVS, vinylsiloxanether or vinyl polyether siloxane has been introduced in the dental market since 2009 (Identium, Kettenbach Co, Eschenburg, Germany) (11). This material has been reported to combine easy removal of a PVS material with hydrophilic wetting properties of a polyether (12), which makes it a promising material for demanding prosthetic conditions in which both easy removal and moisture control are necessary, such as with narrow and deep gingival crevices (13).

The introduction of digital dentistry in recent years has led to the development of CAD/CAM systems that use an intraoral scanner for digital impression procedures and for patient comfort. This progress is followed by use of new impression materials, instead of conventional materials, which can be easily digitally scanned for impression making and a digital workflow (14).

Since accuracy of dental impressions depends on flowing and wetting properties of the applied impression materials, hydrophilicity is regarded as a major influencing factor in the outcome of an impression (15). Several studies investigated wettability of the *already set* impression materials, showing no statistically significant differences between PVS and PE materials (16). However, wettability of an impression material *during its setting time* proved to be a field that needs further investigation (16, 17).

There are several methods for determining wettability of impression materials (15). Dynamic contact angle sessile drop goniometry and dynamic Wilhelmy tensiometry are commonly used (18, 19). Contact angle measurement was proved to be the most clinically relevant technique. Using this method, the investigator measures the contact angle of a distilled water droplet on a flat surface of a solid specimen of an impression material. The contact angle value may be affected by the drop volume that may be decreased due to evaporation (17). The lower the contact angle, the more increased is wettability and the greater is hydrophilicity (10).

The aim of the present *in vitro* study was to compare water contact angles of different impression materials, including one PE, two light wash PVS materials and a CAD/CAM scannable impression material, initially and during setting, in an effort to determine their surface wettability.

Methods and materials

Four groups of dental impression materials were used in laboratory conditions 230±10C, 50%±5% relative humidity) in this *in vitro* study. The groups were as follows:

1) Two light wash materials - PVS 1: polyvinyl siloxane

vanje neke su od prednosti PVS materijala za otiskivanje (6, 7). Ti su materijali hidrofobni. Zbog toga vlaga može negativno utjecati na preciznost konačnoga otiska (8, 9). Ugrađivanje surfaktanata u PVS matricu omogućuje hidrofilna svojstva (7). Materijali za otiske PE-a koji sadržavaju kopolimer-tetrahidrofuran i etilen-oksid potpuno su hidrofilni sa svojstvom ovlaživanja površine zuba i gingive u prisutnosti sline (10).

Novi otisni materijal koji kombinira svojstva PE-a i PVS-a, vinilsiloksanetera ili vinilpolietersiloksana predstavljen je na stomatološkom tržištu 2009. (Identium, Kettenbach Co, Eschenburg, Njemačka) (11). Istaknuto je da taj materijal kombinira jednostavno uklanjanje PVS-a sa svojstvima hidrofilnog vlaženja polietera (12), što ga čini obećavajućim materijalom za zahtjevne protetičke uvjete u kojima je potrebno jednostavno uklanjanje i kontrola vlage, kao što su uski i duboki sulkusi (13).

Primjena digitalne stomatologije posljednjih godina potaknula je razvoj CAD/CAM sustava koji ima intraoralni skener za digitalni otisak i povećava udobnost pacijenata. Taj napredak prati upotreba novih otisnih materijala umjesto onih konvencionalnih koji se mogu jednostavno digitalno skenirati radi uključivanja otisaka i imaju digitalni tijek rada (14).

Budući da točnost otiska ovisi o svojstvima tečenja i vlaženja otisnih materijala, hidrofilnost se smatra glavnim čimbenikom koji utječe na preciznost otiska (15). U nekoliko istraživanja autori su se bavili vlažljivošću već stvrdnutih otisnih materijala, ne pokazujući statistički značajne razlike između PVS-a i PE-a (16). No hodrofilnost otisnoga materijala tijekom stvrdnjavanja pokazala se kao područje koje treba dodatno istražiti (16, 17).

Nekoliko je metoda za određivanje hidrofilnosti otisnoga materijala (15). Uobičajeno se koriste goniometrija s dinamičkim kontaktnim kutom i dinamička Wilhelmyjeva tenziometrija (18, 19). Mjerenje kontaktnoga kuta pokazalo se klinički najrelevantnijom tehnikom. Tom metodom istraživač mjeri kontaktni kut kapljice destilirane vode na ravnoj površini čvrstog uzorka otisnoga materijala. Na vrijednost kontaktnog kuta može utjecati volumen kapljice koji se može smanjiti zbog isparavanja (17). Što je niži kontaktni kut, to je veće ovlaživanje i veća je hidrofilnost (10).

Cilj ovoga istraživanja *in vitro* bio je usporediti kontaktne kutove vode s različitim otisnim materijalima, uključujući jedan PE, dva rijetka PVS-a i CAD/CAM otiskivi otisni materijal na početku vezivanja i tijekom toga postupka, u nastojanju da se utvrdi njihova površinska vlažljivost.

Materijali i metode

Četiri skupine otisnih materijala korištene su u laboratorijskim uvjetima (230 ± 10 C, 50 % ± 5 % relativne vlažnosti zraka) u ovom istraživanju *in vitro*. Skupine su bile sljedeće:

1) dva rijetka materijala – PVS 1: polivinilsiloksan (Varioti-

(Variotime, Heraus Kulzer Michui Chemicals) and PVS 2: polyvinyl siloxane (Detaseal lite, hydroflow impression silicone, Detax)

2) A soft polyether impression material PE (Impregum, 3M ESPE)
3) A CAD/CAM scannable polyvinylsiloxane CAD
4) Hybrid vinylsiloxanether impression material ID (Identium, Kettenbach Co, Eschenburg, Germany)

All materials except PE were provided in cartridges together with their mixing tips. The soft base and catalyst were set in the Pentamix TM 3 Automatic Mixing Unit and specific mixing tips were used (20).

Hydrophilicity was evaluated by water contact angle measurement for each material before and during setting. Flattened specimens were prepared by the use of a Teflon mold with specific dimensions. A mixing tip was always embedded in the mold in order to avoid air entrapment and subsequent bubble formation. The Teflon molds were overfilled and a flattened surface of each impression material was obtained by means a glass slab that slid over the impression material after its initial infusion in the mold (Fig. 1). Ten (10) specimens for each impression material were created, which made 50 specimens in total.

A 5μl droplet of deionized water was collected in a calibrated micropipette and positioned above the flattened specimen surface (Figure 2) (8, 15). The droplet fell on the specimen and photos in specific time intervals were taken. (Figure 3)

The imaging of the droplet was standardized for all impression materials. Photos were taken using a Nikon D3200 DSLR camera and a 105 mm macro lens (Nikorr, Nikon). In order for the camera and the specimen to be aligned, the DSLR camera was set on a 3 mm height basis and the specimen on a 6 mm height basis. The distance between the edge of the lens and the edge of the specimen was set at 12.5 mm

me, Heraus Kulzer Michui Chemicals) i PVS 2: polivinilsiloksan (Detaseal lite, Detax)

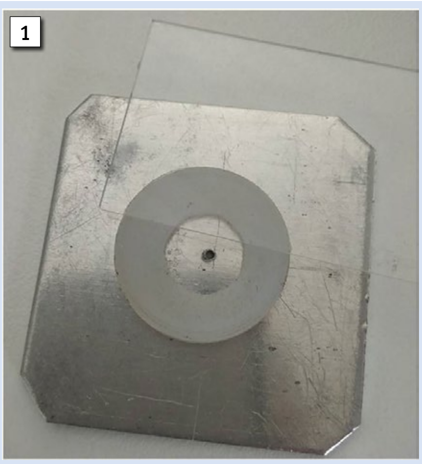
2) mekani polieterski otisni materijal PE (Impregum, 3M ESPE)
3) CAD/CAM polivinilsiloksan koji se može skenirati CAD
4) hibridni vinilsiloksaneter ili drugi otisni materijal ID (Identium, Kettenbach Co, Eschenburg, Njemačka)

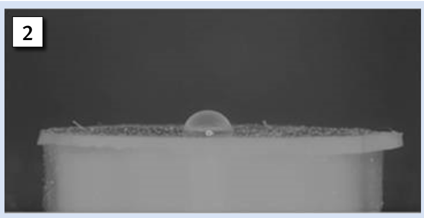
Svi materijali, osim PE-a, isporučuju se u patronama zajedno s nastavcima za miješanje. Mekana baza i katalizator postavljeni su u Pentamix TM 3 jedinicu za automatsko miješanje i korišteni su posebni nastavci za miješanje (20).

Hidrofilnost je procijenjena mjerenjem kontaktnoga kuta s vodom za svaki materijal prije vezivanja i tijekom toga postupka. Spljošteni uzorci pripremljeni su u teflonskom kalupu specifičnih dimenzija. U kalup je uvijek bio umetnut nastavak za miješanje da bi se izbjeglo uključivanje zraka i poslije stvaranje mjehurića. Teflonski kalupi su prepunjeni, a spljoštena površina uzorka svakoga otisnoga materijala postignuta je s pomoću staklene ploče koja je kliznula po otisnome materijalu nakon početnoga ulijevanja u kalup (slika 1.). Za svaki otisni materijal napravljeno je deset (10) uzoraka, što je ukupno činilo 50 uzoraka.

Kapljica deionizirane vode od 5 μL navučena je u kalibriranu mikropipetu i postavljena iznad površine uzorka (slika 2.) (8, 15). Kapljica je pala na uzorak i snimljene su fotografije u određenim intervalima (slika 3.).

Snimanje kapljice standardizirano je za sve otisne materijale. Fotografije su snimljene fotoaparatom Nikon D3200 DSLR i makroobjektivom od 105 mm (Nikorr, Nikon). Kako bi se fotoaparat i uzorak poravnali, DSLR kamera postavljena je na visinu od 3 mm, a uzorak na visinu od 6 mm. Razmak između ruba leće i ruba uzorka postavljen je na 12,5 mm s pomoću digitalne pomične mjerke. Parametri fotografije bili su sljedeći: f = 29, zatvarač od 1/125 s,





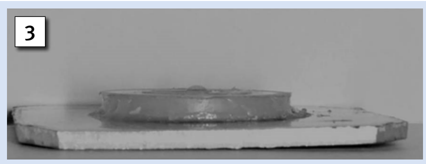


Figure 1 The Teflon mold used in this experiment is shown. It is stabilized on a metal base, and a glass slab is used to achieve flattened surface. The hole at the center denoted with an arrow was designed as an escape route for excess impression material to avoid air entrapment and subsequent bubble formation.

Slika 1. Teflonski kalup upotrijebljen u ovom pokusu – stabiliziran je na metalnoj podlozi, a za postizanje spljoštene površine korištena je staklena ploča; rupa u sredini označena strelicom dizajnirana je kao izlaz za višak otisnoga materijala da bi se izbjeglo uključivanje zraka i poslije stvaranje mjehurića

Figure 2. Flattened impression materials specimens were prepared and a specific value drop fell on the surface.

Slika 2. Uzorci spljoštenoga otisnoga materijala pripremljeni su i kapljica specifične vrijednosti pala je na površinu

Figure 3 Photos were taken after the drop felt on the specimen’s surface.

Slika 3. Fotografije su snimljene nakon što je kap pala na površinu uzorka by the use of a digital caliper. The photography parameters were the following: f=29, shutter of 1/125 sec, ISO 100 film, TTL ring flash with a shutter of ¼ sec.

Digital pictures were taken at two time points for all specimens; immediately after mixing of each impression material (t1) and at 50% of the suggested working time according to the manufacturer's instructions for each impression material (t2).

For contact angle calculation, drop analysis program plugged in Image j software was used (21, 22).

The Wilcoxon matched-pair test was applied in order to explore the relation between two different time periods of the same material. The distribution of the materials in the same time period was compared and assessed using non-parametric tests, that is the Mann-Whitney and Kruskal-Wallis tests, in order to detect differences in distribution of sample populations. The significance level was set at 0.05 throughout the analysis. Statistical analysis was performed by using IBM SPSS 25.

Results

Contact angle values are presented in Table 1. A comparison of the contact angle measurements of the impression materials initially, after mixing, revealed statistically significant differences (p<.05). All groups presented statistically significant lower contact angles initially comparing to the CAD material. Although the PE material showed superior behavior concerning hydrophilicity among all the tested groups, a comparison of contact angles t1 between the PE and PVS1 material did not reveal any significant differences (Table 2).

The contact angles measured during setting were significantly lower compared with those measured at initial time

ISO 100 film, TTL prstenasta bljeskalica sa zatvaračem od ¼ sek.

Digitalne slike snimljene su u dvije vremenske točke za sve uzorke; odmah nakon miješanja svakoga otisnoga materijala (t1) i pri 50 % predloženog radnog vremena prema uputama proizvođača za svaki otisni materijal (t2).

Za izračun kontaktnoga kuta korišten je program za analizu kapljica koji je priključen na softver Image j (21, 22).

Wilcoxonov test uparenih uzoraka primijenjen je da bi se istražila veza između dvaju različitih vremenskih razdoblja istog materijala. Raspodjela materijala u istom razdoblju uspoređena je i ocijenjena neparametrijskim testovima, odnosno Mann-Whitneyjevim i Kruskal-Wallisovim testom da bi se otkrile razlike u raspodjeli populacije uzoraka. Razina značajnosti tijekom analize postavljena je na 0,05. Statistička analiza obavljena je u softveru IBM SPSS 25.

Rezultati

Vrijednosti kontaktnoga kuta prikazane su u tablici 1. Usporedbom mjerenja kontaktnoga kuta otisnih materijala na početku miješanja i poslije toga postupka, otkrivene su statistički značajne razlike (p < 05). Sve su skupine pokazale statistički značajno manje kontaktne kutove na početku u usporedbi s CAD materijalom. Iako je PE materijal pokazao bolje ponašanje kad je riječ o hidrofilnosti među svim ispitivanim skupinama, usporedba kontaktnih kutova t1 između PE i PVS1 materijala nije otkrila značajne razlike (tablica 2.).

Kontaktni kutovi izmjereni tijekom vezivanja bili su značajno niži u usporedbi s onima izmjerenima u početnim vre-

Table 1 Mean values and standard deviation of contact angles measurements for all tested materials
Tablica 1. Srednje vrijednosti i standardne devijacije mjerenja kontaktnih kutova za sve ispitivane materijale

Case summaries • Sažetak			
Tested material • Testirani materijal		Time point_t1 • Vremenska točka_t1	Time point _t2 • Vremenska točka _t2
PVS 1	Mean • Srednja vrijednost	56.0158	49.0618
	Std. Deviation • Std. devijacija	1.88897	1.57554
PE	Mean • Srednja vrijednost	59.5220	41.3034
	Std. Deviation • Std. devijacija	9.12284	8.60896
PVS 2	Mean • Srednja vrijednost	70.8020	45.8863
	Std. Deviation • Std. devijacija	5.30755	4.82622
ID	Mean • Srednja vrijednost	56.5320	50.9244
	Std. Deviation • Std. devijacija	5.89244	5.33345
CAD	Mean • Srednja vrijednost	102.5576	99.8886
	Std. Deviation • Std. devijacija	14.15034	15.82237

Table 2 Statistically significant differences between contact angle measurements (significance level is (a<0.05)
Tablica 2. Statistički značajne razlike između mjerenja kontaktnoga kuta (razina značajnosti je a < 0,05)

Tested material • Testirani materijal	Statistical analysis • Statistička analiza	Significance level • Razina značajnosti (a<0.05)	
		t1	t2
PE- PVS1	Independent-sample Mann-Whitney U test • Nezavisni uzorak Mann-Whitney U test	0.721	**0.038**
PE- PVS2	Independent-sample Mann-Whitney U test • Nezavisni uzorak Mann-Whitney U test	**0.004**	0.568
PE-ID	Independent-sample Mann-Whitney U test • Nezavisni uzorak Mann-Whitney U test	0.622	0.065

points for all the tested groups. Moreover, all tested impression materials presented a stepwise development of hydrophilicity in the setting stage, which was not observed at the initial time point t1. The PE presented lower measured contact angle values both at t1 and t2 examined time points. At t2, the PVS 2 showed the most pronounced reduction in contact angle measurements, and thus the most significant increase of hydrophilicity. As a result, no statistically significant difference was reported between the contact angle values of PVS2 and PE at t2 (during setting) (Table 2).

Discussion

It is known that water interaction with elastomeric dental impression materials may compromise the quality and accuracy of impressions (14). Contact angle measurements have been suggested for hydrophilicity evaluation of elastomeric impression materials. Drop analysis used in this *in vitro* experiment has been reported as an alternative method for contact angles measurements and analysis of hydrophilic properties of unset PE, ID, CAD and PVS1 and PVS 2 impression materials (15). Impression material samples of 2mm thickness were chosen because it was recommended by most manufacturers (20). It has been speculated that not only lower but also higher thickness of impression material could compromise the final result as well (23, 24).

Contact angles values measured in this experimental study were lower compared with those reported in other *in vitro* experiments (10, 15). More specifically, the tested PVS materials presented contact angle values higher than 70^0 at initial time points tested (10, 15, 17). Light consistency of the tested PVS materials presented the values lower than 60^0, while other studies reported contact angle values higher than 80^0 for similar materials (10, 15). These differences could be attributed to the consistency of the tested PVS impression materials and to differences in the experimental protocols (25).

The theory argues that surfactants in PVS materials reduce contact angles by either migrating to the PVS surface and increasing its wettability (23), or releasing from the PVS surface and reducing the surface tension of the wetting liquid (13). An experiment by Balkenhol et al. (18) discovered that PVS surfactants were present within the wetting liquids exposed to set PVS materials. In the current study, surfactants of PVS materials were released from impression materials surface interacting with the dropped water droplet causing a downregulation at contact angle values, and consequently increasing wettability and hydrophilicity of impression material. Unlike water, saliva contains dissolved proteins and salts. Saliva also contains mucins, which cause strong adhesiveness and increase its viscosity. If surfactants could dissolve in saliva, they might not be able to overcome the surface tension produced by adhesive forces of mucins (26). Protocols using drops of saliva may report higher contact angle values, as PVS materials with surfactants may not achieve low contact angles when they come in contact with saliva. Low contact angles measured in this *in vitro* study – using water for hydrophilicity measurements - could not be directly correlated with the clinical condition.

menskim točkama za sve ispitane skupine. Štoviše, svi ispitani otisni materijali postupno su razvijali hidrofilnost u fazi vezivanja, što nije učeno u početnoj vremenskoj točki t1. PE je imao niže izmjerene vrijednosti kontaktnoga kuta u ispitivanim vremenskim točkama t1 i t2. Pri t2, PVS 2 je pokazao najizraženije smanjenje kontaktnoga kuta, pa tako i najznačajnije povećanje hidrofilnosti. Kao rezultat toga nije zabilježena statistički značajna razlika između vrijednosti kontaktnoga kuta PVS2 i PE pri t2 (tijekom vezivanja) (tablica 2.).

Rasprava

Poznato je da interakcija vode s elastomernim otisnim materijalima može ugroziti kvalitetu i preciznost otiska (14). Mjerenje kontaktnoga kuta predloženo je za procjenu hidrofilnosti elastomernih otisnih materijala. Analiza kapljice korištena u ovom eksperimentu *in vitro* ponuđena je kao alternativna metoda za mjerenje kontaktnih kutova i analizu hidrofilnih svojstava nevezanih otisnih materijala PE, ID, CAD i PVS1 i P VS 2 (15). Uzorci otisnoga materijala debljine 2 mm odabrani su jer ih je preporučila većina proizvođača (20). Pretpostavljalo se da ne bi samo manja, nego i veća debljina otisnoga materijala mogla ugroziti konačni rezultat (23, 24).

Vrijednosti kontaktnih kutova izmjerene u ovom eksperimentalnom istraživanju bile su niže u usporedbi s onima zabilježenima u drugim pokusima *in vitro* (10, 15). Točnije, ispitivani PVS materijali imali su vrijednosti kontaktnih kutova veće od 700 u početnim ispitivanim vremenskim točkama (10, 15, 17). Rijetka konzistencija ispitivanih PVS materijala imala je vrijednosti niže od 600, a u drugim su istraživanjima autori izvijestili o vrijednostima kontaktnih kutova većima od 800 za slične materijale (10, 15). Te se razlike mogu pripisati konzistenciji ispitivanih PVS materijala i razlikama u eksperimentalnim protokolima (25).

Teorija tvrdi da površinski aktivne tvari u PVS materijalima smanjuju kontaktne kutove migriranjem na površinu i povećanjem vlažnosti (23), ili otpuštanjem s površine i smanjenjem površinske napetosti tekućine za ovlaživanje (13). Eksperiment Balkenhola i suradnika (18) otkrio je da su PVS surfaktanti u tekućinama za vlaženje izloženi stvrdnutim PVS materijalima. U ovom istraživanju su površinski aktivne tvari PVS materijala oslobođene s površine otisnoga materijala u interakciji s ispuštenom kapljicom vode, uzrokujući smanjenje vrijednosti kontaktnoga kuta i posljedično povećavajući vlažnost i hidrofilnost otisnoga materijala. Za razliku od vode, slina sadržava otopljene bjelančevine i soli. U njoj su također mucini koji izazivaju snažnu ljepljivost i povećavaju njezinu viskoznost. Ako bi se tenzidi mogli otopiti u slini, možda ne bi mogli prevladati površinsku napetost koju stvaraju adhezivne sile mucina (26). Protokoli koji upotrebljavaju kapljice sline mogu rezultirati većim vrijednostima kontaktnoga kuta jer PVS materijali sa surfaktantima možda neće postići male kontaktne kutove kada dođu u doticaj sa slinom. Mali kontaktni kutovi izmjereni u ovom istraživanju *in vitro* – s pomoću vode za mjerenje hidrofilnosti – nisu se mogli izravno povezati s kliničkim stanjem.

The superiority of PE could be attributed to the intrinsic hydrophilicity of PE impressions. Other studies (19) showed that PE favored moist surfaces producing precise reproductions despite the presence of moisture. Also, Shah et al. (27) concluded that PE has a significantly better accuracy than polyvinyl siloxane. A 3D laser scanner was used to measure plaster models obtained with a double-phase 1-step impression in the absence of moisture. In contrast, in the present *in vitro* study, the main goal was to evaluate hydrophilicity and therefore impression accuracy in the presence of moisture. This difference could provide explanation for the absence of statistically significant differences among PE, ID and PVS 2 materials in this study.

There is a wide range of scientific papers concerning contact angle values that are reported in the literature (10,12,26). Different experimental protocols do not allow the comparison between contact angle values. The volume of droplet, the choice of saliva or distilled water and the time points at which photo images are taken affects the contact angles values. Although they differ when experimental design differs, it is supported that ID and PE usually presented lower contact angle values compared to other impression materials. This comparable and superior hydrophilicity was established in many studies and is more pronounced in case of using saliva instead of water droplets (10, 11, 26). More specifically, the hybrid impression material which has been introduced in dental market as Identium, and characterized as vinylsiloxanether material, combines the ease of removal of PVS with the hydrophilicity wetting properties of polyether (12).

In line with the findings of Menees et al. (10), PE showed the smallest deviations after setting, according to contact angle measurements and thus the best hydrophilic behavior. PVS 2 indicated statistically significant differences of contact angle measurements between the two examined time points, presenting consequently a statistically significant improvement of hydrophilicity, which is important for the accuracy of impression when moisture is present. This result could be attributed to the composition of this elastomeric impression material (PVS 2 Detaseal), containing polydimethylsiloxane with functional groups and fillers and pigments additionally, while the catalyst additionally contains platinum complex compound.

Several factors, including the hydrophilicity measured with contact angle values, material thickness and the impression materials types are of great importance and are related with accuracy of impressions either at every day clinical practice (fixed or removable prosthodontics) (28) or at innovative protocols in maxillofacial prosthetics (29). The limitation of this study was that specimens of impression materials were flat. Furthermore, deionized water was used instead of saliva, which is in agreement with other similar studies (6, 10). Artificial saliva does not represent clinical conditions, as saliva viscosity and composition varies among different persons (30, 31). Attachment of saliva drops to the tip of a calibrated pipette could affect contact angle measurements negatively. Higher variability and contact angle values were recorded in studies using saliva.

Superiornost PE-a mogla bi se pripisati unutarnjoj hidrofilnosti otisaka od toga materijala. U drugim istraživanjima (19) autori su pokazali su da PE favorizira vlažne površine na kojima daje preciznu reprodukciju unatoč prisutnosti vlage. Shah sa suradnicima (27) također je zaključio da PE ima znatno veću preciznost od polivinilsiloksana. 3D laserski skener korišten je za mjerenje sadrenih modela dobivenih dvofaznim otiskom u jednom koraku u odsutnosti vlage. Suprotno tomu, u ovom istraživanju *in vitro* glavni je cilj bio procijeniti hidrofilnost, a time i preciznost otiska u prisutnosti vlage. Ta razlika mogla bi objasniti to što u ovom istraživanju ne postoje statistički značajne razlike između PE, ID i PVS 2 materijala.

Mnogobrojni su znanstveni radovi o vrijednostima kontaktnoga kuta koji su objavljeni u literaturi (10, 12, 26). No različiti eksperimentalni protokoli ne dopuštaju usporedbu njihove vrijednosti. Volumen kapljice, izbor sline ili destilirane vode i vrijeme u kojemu se snimaju fotografije utječu na vrijednosti kontaktnoga kuta. Iako se razlikuju kada se razlikuje eksperimentalni dizajn, ID i PE obično su imali manje vrijednosti kontaktnih kutova u usporedbi s drugim otisnim materijalima. Ta usporediva i superiorna hidrofilnost ustanovljena je u mnogim istraživanjima i izraženija je u slučaju korištenja sline umjesto kapljica vode (10, 11, 26). Točnije, hibridni otisni materijal koji se na stomatološkom tržištu može nabaviti pod nazivom Identium, a okarakteriziran kao vinilsiloksaneter, kombinira jednostavnost uklanjanja PVS-a sa svojstvima vlaženja polietera hidrofilnosti (12).

U skladu s nalazima Meneesa i suradnika (10), PE je pokazao najmanja odstupanja nakon vezivanja prema mjerenjima kontaktnoga kuta, pa tako i najhidrofilnije svojstvo. PVS 2 upozorio je na statistički značajne razlike mjerenja kontaktnoga kuta između dviju ispitivanih vremenskih točaka, što je posljedično statistički značajno poboljšanje hidrofilnosti, a to je važno za preciznost otiska kada je prisutna vlaga. Taj rezultat mogao bi se pripisati sastavu toga elastomernoga otisnoga materijala (PVS 2 Detaseal) koji sadržava polidimetilsiloksan s funkcionalnim skupinama te punila i pigmente, a u katalizatoru je dodatno kompleksni spoj platine.

Nekoliko čimbenika, uključujući hidrofilnost izmjerenu vrijednostima kontaktnoga kuta, debljinu materijala i vrste otisnih materijala, iznimno su važni i povezani su s preciznošću otisaka u svakodnevnoj kliničkoj praksi (fiksna ili mobilna protetika) (28) ili prema inovativnim protokolima u maksilofacijalnoj protetici (29). Ograničenje ovog istraživanja bilo je to što su uzorci otisnoga materijala bili ravni. Nadalje, umjesto sline korištena je deionizirana voda, što je u skladu s drugim sličnim istraživanjima (6, 10). Umjetna slina ne pokazuje kliničko stanje jer viskoznost i sastav sline variraju od čovjeka do čovjeka (30, 31). Pričvršćivanje kapi sline na vrh kalibrirane pipete moglo bi negativno utjecati na mjerenje dodirnoga kuta. Veća varijabilnost i vrijednosti kontaktnoga kuta zabilježene su u istraživanjima sa slinom.

Conclusion

Within the limitations of this *in vitro* study, the following conclusions can be made: PE and ID showed superior behavior concerning hydrophilicity both initially and during setting; The PVS materials showed excellent hydrophilicity. PVS 1 presented comparable hydrophilicity with PE and ID initially, while PVS 2 showed the most pronounced contact angle value reduction; All impression materials presented statistically significant lower contact angles initially comparing to CAD; All impression materials developed a stepwise hydrophilicity.

Zaključak

Unatoč ograničenjima u ovom istraživanju *in vitro* mogu se donijeti sljedeći zaključci: PE i ID pokazali su superiorno svojstvo kad je riječ o hidrofilnosti te na početku vezivanja i tijekom toga postupka; PVS materijali pokazali su izvrsnu hidrofilnost. PVS 1 je na početku bio, kad je riječ o hidrofilnosti, usporediv s PE-om i ID-om, a PVS 2 je pokazao najizraženije smanjenje vrijednosti kontaktnoga kuta. Svi otisni materijali imali su statistički značajno manje kontaktne kutove na početku u usporedbi s CAD-om. Svi otisni materijali postupno su postajali hidrofilni.

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Conflict of interest

The authors declare that they have no conflict of interest.

Sukob interesa

Autori nisu bili u sukobu interesa.

Authors' contributions: **K.T.** and **D.T.** - conceived the experimental idea and planned the experimental protocol together and contributed to the writing as well, but mainly to the review of the reports and revisions; **A.T., K.T.** - performed the experiments in coordination. They contributed to this manuscript both by writing reports and by completing the final version of this manuscript.

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Sažetak

Uvod: Hidrofilnost otisnih materijala ključna je za preciznost otiska i prijeko potrebna za izradu nadomjestka s dobrim dosjedom. Najčešća tehnika za njezinu procjenu jest mjerenje kontaktnog kuta. Cilj ovoga istraživanja *in vitro* bio je usporediti kontaktne kutove vode u četirima skupinama elastomernih otisnih materijala, prije vezivanja i tijekom vezivanja. **Materijali i metode:** Spljošteni uzorci (n = 10) ispitanih otisnih materijala pripremljeni su u teflonskom kalupu specifičnih dimenzija. Kapljica deionizirane vode od 5 μL kapnuta je na uzorak, a fotografije su snimljene fotoaparatom Nikon D3200 DSLR i 105 mm makroobjektivom (Nikorr, Nikon) u određenim vremenskim točkama. **Rezultati:** CAD/CAM materijal imao je najveći kontaktni kut. Rijetki materijali polivinilsiloksan (PVS) 1, polieter i vinilsiloksaneter imali su usporedive mjere kontaktnoga kuta, posebno u početnoj vremenskoj točki. Statistički značajno smanjenje kontaktnih kutova zabilježeno je tijekom postavljanja za sve PVS-ove, PE-ove i vinilsiloksan, a najizraženije smanjenje, pa time i najznačajnije povećanje hidrofilnosti, zabilježeno za rijedak PVS 2. **Zaključci:** CAD/CAM otisni materijal imao je najhidrofobnija svojstva. PVS materijali imali su izvrsnu hidrofilnost. Polieterski i poliviniloksaneterski otisni materijali imali su manji kontaktni kut, pa tako i veću hidrofilnost u usporedbi s drugim ispitivanim materijalima na početku vezivanja i tijekom toga postupka. Svi ispitani otisni materijali postupno su razvijali hidrofilnost tijekom faze vezivanja.

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