Evaluation of Elastomeric Impression Materials’ Hydrophilicity: An in vitro Study

Procjena hidrofilnosti elastomernih otisnih materijala: istraživanje in vitro

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, and the most expressed reduction of 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 material showed the most hydrophobic behavior. PVS materials showed excellent hydrophilicity. Polyether and polyvinylsiloxanether 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.

Abstract

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, alginates, 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, and hydrophilic nature make them the most commonly used materials in dental practice due to their favorable clinical properties and minimal dimensional change (4, 5).

Uvod

Preciznost je ključna riječ za otisne materijale koji se smatraju klinički uspješnima, a to znači da se mogu otsnuti svi detalji supragingivno i subgingivno od zuba i izraditi točan sadržani odljev. Zato je precizan otisak nužan za izradu dobro prilagođenog nadomjestka (1, 2). Tijekom godina u području stomatološke protetike koristi se različito otisni materijali. Reverzibilni hidrokolloidi, alginati, polisulfidi, kondenzacijski polisiloksani, adicijski polivinilsiloksani (PVS) i poliefteri (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 dimenzijalna stabilnost i elastični oporavak podminiranih mjesta, niska viskoelastična svojstva, karakteristike velikoga protoka i kratko stvrdnja-
high 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 gingival surface in presence of saliva (10).

A new impression material that combines the properties of PE and PVS, vinylsiloxanether or vinyl polymer siloxane has been introduced in the dental market since 2009 (Iden- tium, Kettenbach Co, Eschenburg, Germany) (11). This material has been reported to combine easy removal of a PVS material with hydrophilic wetting properties of a polymer (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 ± 10°C, 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 vлага može negativno utjecati na preciznost konačnoga otiska (8, 9). Ugrađivanje surfaktanata u PVS matricu omogućuje hidrofilnu svojstva (7). Materijali za otiske PE-a koji sadržavaju kopolimer-tetrahydrofuran i etilen-oksid potpuno su hidrofilni sa svojstvom ovlažavanja 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 stomatolokskom tržištu 2009. (Iden- tium, 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 koje se mogu jednostavno digitalno skenirati radi uključivanja otiska i imaju digitalni tijek rada (14).

Budući da točnost otiska ovisi o svojstvima tečenja i uvaženja otisnih materijala, hidrofilnost se smatra glavnim čimbenikom koji utječe na preciznost otiska (15). U nekoliko istraživanja autori su se bavili vlažlivošću već studiranih otisnih materijala, ne pokazujući statistički značajne razlike između PVS-a i PE-a (16). No hidrofilnost 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 (19). Mjerene kontaktnoga kuta pokazale se klinički najrelevantnijom tehnikom. Tom metodom istraživač mjeri kontaktni kut kapljice destilirane vode na ravnov 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 ovlažavanje i veća je hidrofilnost (10).

Cilj ovoga istraživanja in vitro bio je usporediti kontaktna kuta vode s različitim otisnim materijalima, uključujući jedan PE, dva riječka PVS-a i CAD/CAM otiskivski 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% relative vlažnosti zraka) u ovom istraživanju in vitro. Skupine su bile sljedeće:

1) dva riječka materijala – PVS 1: polivinilsiloksan (Varioti-
Elastomeric Impression Materials: Theodorou et al.

258

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 of 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 (Fig. 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).

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 poravnavali, 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 mjere. Parametri fotografije bili su sljedeći: f = 29, zatvarač od 1/125 s,
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 < 0.05). 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 kontaktne kutije 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.

| 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        | 56.5320                                | 50.9244                                |
| Std. Deviation • Std. devijacija       | 5.89244                                | 5.33345                                |
| ID Mean • Srednja vrijednost           | 102.5576                               | 99.8886                                |
| Std. Deviation • Std. devijacija       | 14.15034                               | 15.82237                               |

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

| Tested material • Testirani materijal | Statistical analysis • Statistička analiza | Significance level • Razina značajnosti (a<0.05) |
|----------------------------------------|-------------------------------------------|-----------------------------------------------|
| 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                                    |

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)
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° at initial time points tested (10, 15, 17). Light consistency of the tested PVS materials presented the values lower than 60°, while other studies reported contact angle values higher than 80° 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.

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° at initial time points tested (10, 15, 17). Light consistency of the tested PVS materials presented the values lower than 60°, while other studies reported contact angle values higher than 80° 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.

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° at initial time points tested (10, 15, 17). Light consistency of the tested PVS materials presented the values lower than 60°, while other studies reported contact angle values higher than 80° 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.

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° at initial time points tested (10, 15, 17). Light consistency of the tested PVS materials presented the values lower than 60°, while other studies reported contact angle values higher than 80° 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.
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 vinylsiloxane-another 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.

Superiority PE-a may be attributed to the superior 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 vinylsiloxane-another 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.

Superiority PE-a may be attributed to the superior 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 vinylsiloxane-another 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.
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 reduction; All impression materials presented statistically significant lower contact angles initially comparing to CAD; All impression materials developed a stepwise hydrophilicity.

Acknowledgment
The authors extend their gratitude to DETAX GmbH & Co. KG company for donating one of the tested materials (Detaseal® lite), and they are also grateful to Mrs. Lora Slini for performing statistical analysis of the results.

Conflict of interest
The authors declare that they have no conflict of interest.

Authors' contributions: K.T. and D.T. - conceived the experimental idea and planned the experimental protocol together and contributed to the writing as well, 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.

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 najznačajniji promjer smanjenje vrijednosti kontaktne kute. Svi otisni materijali imali su statistički značajno manje kontaktne kutove na početku u usporedbi s CAD-om. Svi otisni materijali postupno su postajali hidrofilni.

Zahvala
Autori zahvaljuju tvrtki DETAX GmbH & Co. KG na donaciji jednoga od ispitanih materijala (Detaseal® lite), a zahvaljuju i i Lori Slini na statističkoj analizi rezultata.

Sukob interesa
Autori nisu bili u sukobu interesa.

References
1. Takahashi H, Finger WJ. Dentin surface reproduction with hydrophilic and hydrophobic impression materials. Dent Mater. 1991 Jul;7(3):197-201.
2. Braden M, Inglis AT. Visco elastic properties of dental elastomeric impression materials. Biomaterials. 1986 Jan;7(1):45-8.
3. Hamalian AT, Nasr E, Chidiac JJ. Impression Materials in Fixed Prosthodontics: Influence of Choice on Clinical Procedure. J Prosthodont. 2011 Feb;20:153-60.
4. Rupp F, Aumann D, Jacobi A, Groten M, Geis-Gerstorfer J. Hydrophilicity of elastomeric non-aqueous impression materials during setting. Dent Mater. 2005 Feb;21(2):94-102.
5. Balkenhol M1, Wöstmann B, Kanehira M, Finger WJ. Shark fin test and impression quality: a correlation analysis. J Dent. 2007 May;35:409-15.
6. Tolidis K, Toporidis D, Gerasimou P, Theocharidou A, Boutsiouki C. Comparison of Elastomeric Impression Materials' Thixotropic Behavior. Eur J Prosthodont Restor Dent. 2013 Jun;21(2):75-8.
7. Nissan J, Lauper BZ, Brosh T, Assil D. Accuracy of three polyvinyl siloxane putty-wash impression techniques. J Prosthet Dent. 2000;83:161-5.
8. Johnson GH, Lepe X, Aw TC. The effect of surface moisture on the detail reproduction of elastomeric impressions. J Prosthet Dent. 2003;90:354-364.
9. Oh YI, Lee DY, Hwang SY, Kim KM. Effect of non-ionic surfactants on surface properties of hydrophilic polyvinyl siloxane impressions. Colloid Surface A. 2003;229:9-17.
10. Menees T, Radhakrishnan R, Ramp L, Burgess J, Lawson N. Contact angle of unset elastomeric impression materials. J Prosthet Dent. 2015 Oct;114(4):536-42.

Sažetak
Uvod: Hidrofilnost otisnih materijala ključna je za preciznost otiska i prijevođenje za izradu domjesta, te dobrijem dojmom. Najčešći komponenti za takav procjenu su mjerenje kontaktne kutove.

Materijali i metode: Spljošteni uzorci (n = 10) ispitanih otisnih materijala pripremljeni su u teflonskom kalupu specifičnog dimenzija. Kapljica deionizirane vode od 5 µl kapnuta je na uzorak, a fotografije su snimljene fotoparatom Nikon D3200 DSLR i 105 mm makroobjektivom (Nikkor, Nikon) u određenim vremenskim točkama. Rezultati: CAD/CAM materijal imao je najveći kontaktni kut. rijetki materijali poliviniloksin (PVS) 1, poliester i visniloksanatometeri imali su usporedive mjere kontaktne kute, posebno u početnoj vremenskoj točki. Statistički značajna smanjenja kontaktnih kutova zabilježeno je tijekom postavljanja za sve PVS-ove, PE-ove i visniloksanatom, a najznačajnije smanjenje, pa time i najznačajnije povećanje hidrofilnosti, zabilježeno je za rijeđak PVS 2. Zaključci: CAD/CAM otisni materijal imao je najhidrofobnija svojstva. PVS materijali imali su izvrsnu hidrofilnost. Poliesteri i poliviniloksanatomski otisni materijali imali su manji kontaktni kut, pa tako i veću hidrofilnost u usporedbi s drugim ispitanim materijalima na početku vezivanja i tijekom toga postupka. Svi ispitani otisni materijali postupno su razvijali hidrofilnost tijekom faze vezivanja.

MeSH pojmovi: stomatološki materijali za otiske; elastomeri; hidrofobno i hidrofilno međudjelovanje

Zaprimljen: 10. travnja 2021.  
Prihvaćen: 27. svibnja 2021.  

Adresa za dopisivanje
Dimitrios Toporidis, PhD  
Aristotle University of Thessaloniki  
School of Dentistry  
Department of Prosthodontics, University Campus, Dentistry Building,  
GR 54124, Thessaloniki, Greece  
tel/faks. +30 2310995947  
dtortop@dent.auth.gr
11. Enkling N, Bayer S, Jöhrn P, Mericske-Stern R. Vinysiloxanether: a new impression material. Clinical study of implant impressions with vinysiloxanether versus polyether materials. Clin Implant Dent Relat Res. 2012 Mar;14(1):144-51.
12. Walker MP, Alderman N, Petrie CS, Melander J, McGuire J. Correlation of impression removal force with elastomeric impression material rigidity and hardness. J Prosthodont. 2013 Jul;22(5):362-6.
13. Schulein TM. Significant events in the history of operative dentistry. J Hist Dent. 2005 Jul;53(2):63-72.
14. Ahlholm P, Sipila K, Vallittu P, Jakonen M, Kotrantza U. Digital versus Conventional Impressions in Fixed Prosthodontics: A Review. J Prosthodont. 2018 Jan;27:1:35-41.
15. Kugel G, Klettke T, Goldberg JA, Benchimol J, Perry RD, Sharma S. Investigation of a New Approach to Measuring Contact Angles for Hydrophilic Impression Materials. J Prosthodont. Mar-Apr 2007;16(2):84-92.
16. Michalakis KK, Bakopoulou A, Hirayama H, Gareffis DP, Gareffis PD. Pre- and Post- set Hydrophilicity of Elastomeric Impression Materials. J Prosthodont. Jul-Aug 2007;16(2):238-48.
17. Hosseinpour D, Berg JC. The Dynamic Interaction of Water with Four Dental Impression Materials during Cure. J Prosthodont. 2009 Jun;18(4):292-300.
18. Balkenhol M, Eichhorn M, Wostmann B. Contact angles of contemporary type 3 impression materials. Int J Prosthodont. Jul-Aug 2009;22(4):396-8.
19. Walker MP, Petrie CS, Haj-Ali R, et al: Moisture effect on polyether and polyvinylsiloxane accuracy and detail reproduction. J Prosthodont. 2005;14:158-163.
20. Punj A, Bompolaki D, Garacaoa J. Dental Impression Materials and Techniques. Dent Clin North Am. 2017 Oct;61(4):779-796.
21. Stalder AF, Melchiort, MüllerM, SageD, BluT, UnserM. Low-Bond Axisymmetric Drop Shape Analysis for Surface Tension and Contact Angle Measurements of Sessile Drops. Colloids and Surfaces A: Physicochemical and Engineering Aspects. 2010;364:72-81.
22. Stalder AF, Kulig G, Sage D, BarbieriL, Hoffmann P. A Snake-Based Approach to Accurate Determination of Both Contact Points and Contact Angles. Colloids and Surfaces A: Physicochemical and Engineering Aspects. 2006;286:92-103.
23. Nissan J, Gross M, Shifman A, Assif D. Effect of wash bulk on the accuracy of polyvinylsiloxane putty-wash impressions. J Oral Rehabil. 2002 Apr;29(4):357-61.
24. Satheshe B, Haralur, MajedSaadToman, Abdullah Ali Al-Shahrami, Abdullah Ali Al-Qarni. Accuracy of Multiple Pour Cast from Various Elastomer Impression Methods. Int J Dent. 2016;2016:7414737.
25. Basapogu S, Pilla A, Pathipaka S. Dimensional Accuracy of Hydrophilic and Hydrophobic VPS Impression Materials Using Different Impression Techniques - An InvitroStudy. J Clin Diagn Res. 2016 Feb;10(2):ZC56-9.
26. Stober T, Johnson GnL, Schmitter M. Accuracy of the newly formulated vinyl siloxanether elastomeric impression material. J Prostheth Dent. 2010 Apr;103(4):228-39.
27. Shah S, Sundaram G, Bartlett D, Sherriff M. The use of a 3D laser scanner using super imposition software to assess the accuracy of impression techniques. J Dent. 2004 Nov;32(8):653-8.
28. Kršek H, Dulčić N. Functional Impressions in Complete Denture and Overdenture Treatment. Acta Stomatol Croat. 2015 Mar;49(1):45-53.
29. Tasopoulos T, Kouveliotis G, Polyzois G, Karathanassi V. Fabrication of a 3D Printing Definitive Obturator Prosthesis: a Clinical Report. Acta Stomatol Croat. 2017 Mar;51(1):53-58.
30. Marcinkowska-Gapińska A, Linkowska-Świdzińska K, Świdziński T, SurdackaA. Rheological parameters of saliva in comparison with taste examination. Biorheology. 2018;55(1):51-60.
31. Schwarz WH. The rheology of saliva. J Dent Res. 1987;66:660-6.