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CHARACTERIZATION OF SOME CONDENSATION SILICONE IMPRESSION MATERIALS

BY

OANA ŢĂNCULESCU[1*], DANIEL ŢÂMPU[2], MONICA ANDRONACHE[1], RALUCA MARIA MOCANU[1] and ADRIAN DOLOCA[1]

[1]"Gr.T.Popa" University of Medicine and Pharmacy of Iaşi,
Department of Fixed Prosthodontics
[2]"Petru Poni" Institute of Macromolecular Chemistry of Iaşi,
Polymer Physics and Structure Department

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Abstract. Dimensional stability, details reproduction and wettability are considered the main features of any impression material, and need to be taken into consideration to achieve a good restoration. In our study we focused on condensation silicones for social and economic reasons, these being the most frequently used materials in common practice. The used materials were: Stomaflex (Spofa – Czech Republic), Oranwash – Zetaplus (Zhermack- Italy), Optosil - Xantopren (Heraeus-Kulzer - Germany), Speedex (Coltène Whaledent- Switzerland).

For a quantitative dimensional stability assessment a new device that eliminates adhesion and friction was designed. Among the fluid impression materials, one can notice that the most stable from the dimensional point of view are those manufactured by Colthène-Whaledent - Speedex and the largest deformations have been recorded by Spofa - Stomaflex products.

In determining the degree of fidelity of the impression materials, grids of different sizes have been used as reference. It has been noticed that all the materials render well the details of the 2 grids (18 μm). The details of the third grid are rendered well only by Xantopren and Speedex, without voids or defects.

For the hydrophilic character of these materials, we measured the evolution of the contact angle between the artificial saliva and materials before setting and

*Corresponding author; *e-mail*: ot@umfiasi.ro

after setting. Data has been then recorded using DSA10 (Krϋss GmbH, Hamburg) droplet analysis. Speedex is the most hydrophilic material, followed by Xantopren, Oranwash VL, Oranwash L and Stomaflex.

Key words: condensation silicones, dimensional stability, details reproduction, hydrophilic character.

1. Introduction

Well-fitting indirect restorations can only be made if accurate models of the oral tissues obtained from high quality impressions are available. The accuracy of dental impressions depends on the composition and manipulation technique of the impression as well as on the die cast material itself (Chen *et al*., 2004; Shillingburg *et al*., 1997; Rosenstiel *et al*., 1998; Anusavice, 2008). Sometimes impression defects only become evident after the cast has been poured (Wassell *et al*., 2002). A number of studies have been carried out to evaluate the accuracy of impressions. In many, metal, ceramic or acrylic resin casts were used (http://www.coltene.com/).

Silicone impression materials are classified according to their method of polymerization on setting, in condensation curing (or Type I) silicones and addition curing (or Type II) silicones (Chen *et al*., 2004).

The condensation silicones are the most used impression materials in dental practice, hence the necessity of compiling a comparative study on these types of materials.

Dimensional stability, details reproduction and wettability are considered the main features of any impression material, and need to be taken into consideration to achieve a good restoration (Chen *et al*., 2004; Shillingburg *et al*., 1997). These physical properties differ consistently according to their chemical structure, making the clinical behaviour the most important criterion in choosing the impression material.

The above mentioned characteristics were studied for the materials presented in Table 1 (http://www.coltene.com/; http://www.en.zhermack.com/; http://www.spofadental.com/; http://www.calsun.com.my/).

Table 1
Studied Impression Materials

Impression material	Materials consistency	Manufacturer	Country of origin
Stomaflex	paste + solid	Spofa	Czech Republic
Oranwash - Zetaplus	paste + solid	Zhermack	Italy
	fluid + solid soft		
Optosil - Xantopren	paste + solid	Heraeus-Kulzer	Germany
Speedex	paste + solid	Coltène Whaledent	Switzerland

2. Dimensional Stability

Dimensional stability is a mandatory feature for exact reproduction of the dimensions and geometrical relations between the dental abutments.

For a qualitative visualization of the impression material contraction, the following approach has been used: a glass tub is filled with impression material so that the material is in full contact with the glass. It can be noticed that as the time passes the material breaks loose from the glass walls which demonstrates the contraction process that occurs in the silicon (Fig. 1).

For a quantitative dimensional stability assessment a new device was design (Fig. 2). The device was adapted to an IOR type optical research microscope (Fig. 3).

Description of the notation in Fig. 2: M − microscope for viewing the measuring divisions. For distance measurement: one division = 1, 3, 6, 10 μm according to objective/ocular settings, E − sample material placed in the standardized recipients each sample is marked (m) using a blade at a distance Lo = 70 mm, C − glass standardized recipients, F − fluor-polimer (Teflon) foil, thickness 20 μm, perfect optical transparency, low extensibility due to small thickness, S − optical glass, 1 x 30 x 90mm, P − glass textolite plate rigidly attached to S.

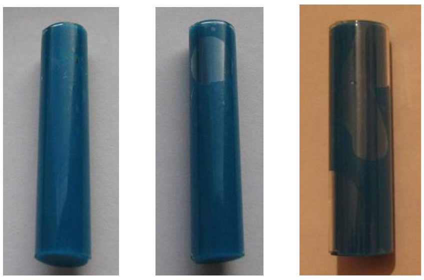


Fig. 1 − Qualitative experiment on condensation silicon contraction.

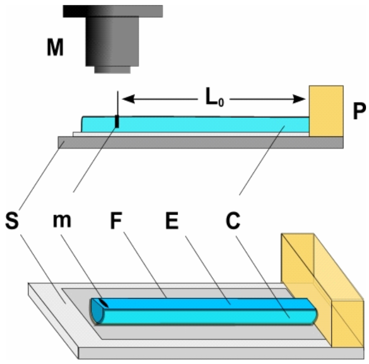


Fig. 2 − Silicon sample measuring device.

Fig. 3 − Adaptation of measuring device to microscope.

The silicon sample cannot extend to the left because of the plate P, it will only elongate to the right. The elongation will be measured with high precision by the optical system.

The silicon sample slides over multilayer material which eliminates friction and adhesion which otherwise could influence the outcome. The multilayer structure is as follows: vaseline/teflon foil/vaseline/standardized treated glass recipient.

The study was conducted by the “Petru Poni” Institute of Macromolecular Chemistry Polymer Physics and Structure Department in Iasi.

The impression material were prepared according to the indications of manufactures and set in a glass recipient. The recipient has a semicylindrical shape with the length of 70 mm and diameter of 8 mm. The glass of the recipient was first treated with H_2NO_3 to eliminate the OH- fragments from the surface which reduces adhesion. Additionally, the recipient is cover at the interior where the sample material is put with the Teflon foil 20 µm thick, optically transparent and a silicon Vaseline Moldosil L185 Li2 was applied on the both sides. This system eliminates adhesion and friction.

Using the microscope measurements have been performed at following time intervals: 1 min, 5 min, 15 min, 1 h, 2 h, 24 h, 72 h. For each material, 5 measurements have been made.

Relative elongation values versus time are listed in Table 2.

Table 2

Relative Deformations versus Time

Material	Material consistency	1 min	5 min	15 min	1 h	2 h	24 h	72 h
Stomaflex	solid (%)	−0.11	−0.13	−0.16	−0.18	−0.21	−0.22	−0.22
	paste (%)	−0.21	−0.38	−0.52	−0.66	−0.7	−0.95	−1.01
Oranwash L Zetaplus	solid (%)	−0,10	−0,12	−0,13	−0,15	−0,17	−0,18	−0,18
	paste (%)	−0,22	−0,39	−0,49	−0,55	−0,60	−0.65	−0,65
Oranwash VL Zetaplus Soft	solid (%)	−0.15	−0.18	−0.21	−0.24	−0.27	−0.29	−0.29
	fluid (%)	−0.26	−0.45	−0.59	−0.69	−0.77	−0.84	−0.84
Optosil Xantopren	solid (%)	−0.058	−0.062	−0.085	−0.092	−0.101	−0.109	−0.109
	paste (%)	−0.189	0.192	−0.196	−0.212	−0.215	−0.230	−0.230
Speedex	solid (%)	−0.002	−0.002	−0.003	−0.006	−0.008	−0.011	−0.011
	paste (%)	−0.016	−0.029	−0.038	−0.045	−0.052	−0.056	−0.056

Relative deformations were determined using the formula: $\Delta L / L_0$

where: L_0 – initial sample length; ΔL – optically measured deformation.

In Fig. 4 the deformation variation for each paste and solid silicon material is presented. Thus the large difference between the paste silicon and solid silicon can be observed. The latter have a much greater dimensional stability which recommends them for utilization in combination for fluid silicones with the purpose of minimizing the contraction.

Because the material deformation is proportion to the quantity, one can stipulate the utilization of fluid silicones in small quantities so that the total deformation can be kept to a minimum and the cast will be more precise.

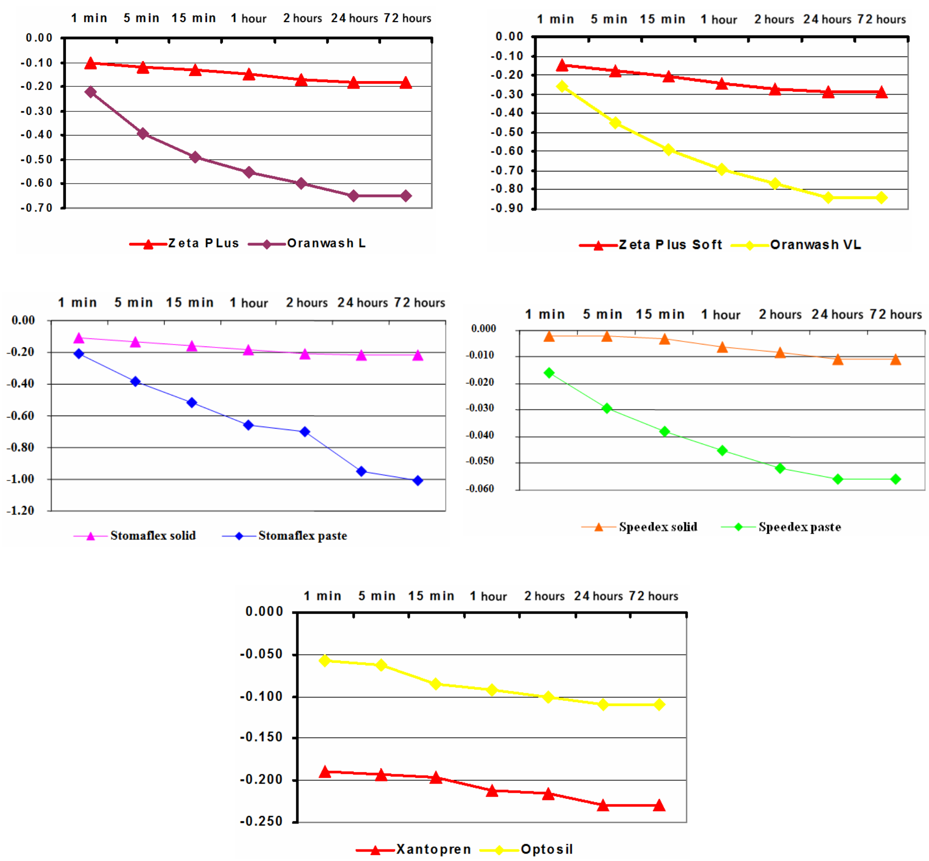


Fig. 4 – Evolution of deformation for each fluid silicone material and its solid counterpart.

In Fig. 5 a comparison between fluid silicon is shown; the same for solid silicones. Among the fluid impression materials, one can notice that the most stable from the dimensional point of view are those manufactured by Colthène-Whaledent - Speedex and the largest deformations have been recorded by Spofa - Stomaflex products. The differences are quite big having an impact in the clinical practice. The second place for the dimensional stability is taken by Xantopren which had good performances, followed by Oranwash. Although the difference between the 2 products of Zermack, it can be noticed that low viscosity material (Oranwash VL) experienced larger deformations.

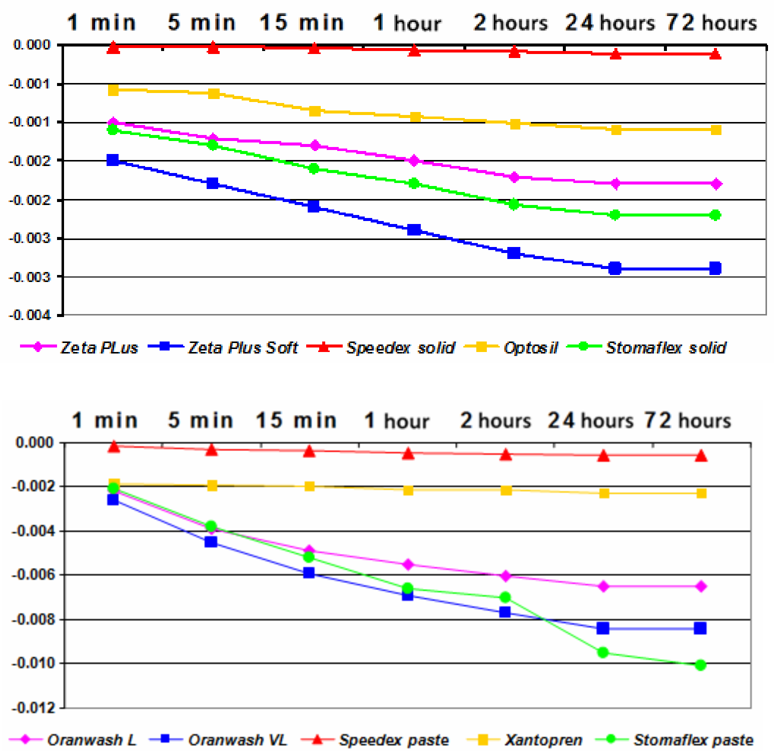


Fig. 5 – Comparison between solid and fluid impression materials.

The same hierarchy is determined also for the solid silicones, where Speedex is the most dimensional stable material and is followed by Optosil, Zetaplus, Zetaplus Soft, Stomaflex. In this respect a very large difference has been observed between Speedex and Stomaflex, in both categories.

3. Material Fidelity

In determining the degree of fidelity of the impression materials, grids of different sizes have been used as reference. These grids are used in chemical industry. The grids have been laid down at the base of metallic box recipients (Fig. 6). A very fine oil has been first applied to the grid. Between the bottom of the recipient and the grid, a thin Teflon foil has been placed. This was also immerses in Moldosil silicon vaseline, and placed on a glass plate which was treated for removing the OH- fragments on the surface. These measurements were taken so that the impression material could not adhere to the grid or tot the bottom of recipient and to be easily removed after the material set. This way the precision of the detail reproduction in the impression material can be better observed on the microscope.

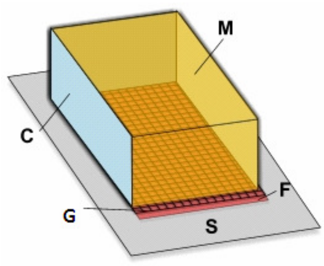


Fig. 6 − Design of the recipient used for detail rendering precision.

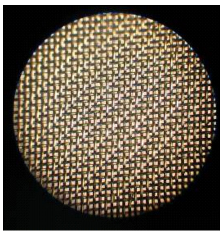
The notations in the Fig. 6 are: M − impression material, C − metallic box recipient, G − grid impression references, F − isolating foil, S − treated glass.

Readings have been made using the optical microscope using 3 types of oculars according to necessities and using a scale with divisions (Table 3).

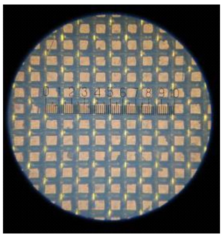
Table 3
Dimensional Correspondence

Ocular	Division size
x6	1 div. = 10 μm
x10	1 div. = 6 μm
x20	1 div. = 3 μm

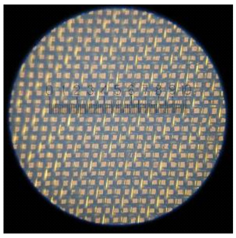
The grids were in three sizes rare, medium and fine (Fig. 7). The latter is water tight.



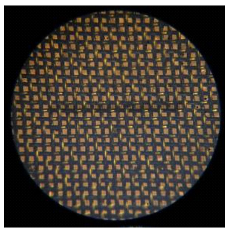
Rare grid - microscopic imagine (objective - x6)



Rare grid - microscopic imagine (objective - x10)



Medium grid - microscopic imagine (objective - x10)



Fine grid - microscopic imagine (objective - x20)

Fig. 7 – Types of grids.

Preliminary grid measurements are presented in Table 4. Fluid impression materials must reproduce details of approximately 20 μm in size. The used grids have details of 6 to 42 μm, being efficient in evaluating impression materials.

Table 4
Grid Dimension - Reference

Grid type	Wire		Gap	
	no. of divisions	dimension [μm]	no. of divisions	dimension [μm]
Fine grid (x20)	2	6	3	10
Medium grid (x10)	3	18	4	24
Rare grid (x10)	5	30	7	42

Fluid impression materials must reproduce details of approximately 20 μm in size. The used grids have details of 6 to 42 μm, being efficient in evaluating impression materials. The samples have been analyzed under microscope checking how the grid details have been rendered. The wires of the grids produce a negative impression while the gaps produced a positive impression. Fig. 8 shows different images obtained with the optical microscope.

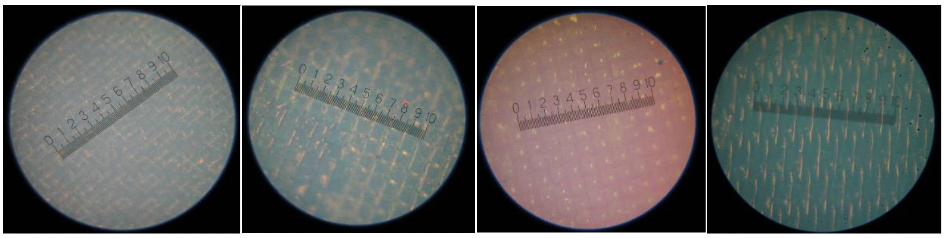


Fig. 8 − Images acquired with the optical microscope.

It has been noticed that all the materials render well the details of the 2 grids. The details of the third grid are rendered well only by *Xantopren* and *Speedex*, without voids or defects. A good fidelity has been shown by *Oranwash* VL. The lowest detail rendering capacity has been shown by *Stomaflex* which did not consistently reproduce the grid gaps. This was because the material was not able to penetrate into these gaps. The evaluation of the detail rendering fidelity has been qualitative. An exact hierarchy of the fluid condensation silicones was difficult to establish but it was shown that the purpose these materials to render details of 20 μm in size is fulfilled.

The solid silicones managed to reproduce the rare grid, and *Speedex*, *Optosil* and *Zetaplus Soft* were capable of reproducing also the medium grid.

4. Wettability

The hydrophile characteristic of the impression materials represents one of the most important properties for the clinical success. In the literature little information about the impressions materials before setting exists. On the other hand the hydrophile aspect is clinical relevant during the working time. In that period of handling, the impression material, by injecting or loading the material in the impression trey, the surface layer of the impression material comes in the contact with the moisture. In order to obtain a high quality impression, the material has to be hydrophile.

The aim of this study was to investigate the hydrophilic character of the fluid impression material and to find a determination method of this property before the setting has occurred.

The degree of wettability in impression materials has a major importance by the impact that it has into two fundamental phases of the clinical-technological process: impression and model casting.

In the first phase this property has an impact on the quality of the impression in a wet environment like gingival sulcus; in the second phase it has an impact on the details rendered by the casted mold and on the facilitating the casting process.

The hydrophilic or hydrophobic character of an impression material is given by the surface tension developed in the material. This can be evaluated by the contact angle between a water droplet and the respective surface (Fig. 9).

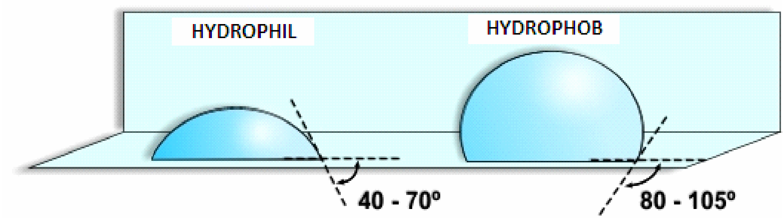


Fig. 9 − Contact angle measurement.

The surface tensions results from the relation between cohesion forces and adhesion forces. If the material is hydrophilic it will develop adhesion forces to the water molecules with which it will comes in contact. This will deform the water droplets which become flat. If the material is hydrophobic, this phenomenon does not occur.

Because measuring with high precision surface tension and contact angles implies standardized protocol, and usage of special equipment and calibration, this study employs a qualitative assessment method. This method compares the contact angles between investigated impression materials and an artificial saliva droplet (*Afnor* - S90-701).

For determining the hydrophilic characteristic the contact angles have been measured at room temperature. The working time recommended by manufacturer has been used. The contact angles have been compared between the 2 phases: before the setting (impression phase) and after setting (model cast phase) for each material.

Determined quantities of impression materials have been placed down on glass plates obtaining a thin layer of material in order to prevent the saliva droplet to become immersed in the material mass, thus altering the outcome. A digital camera has been placed on the same level as the glass plate for recording the process.

After 45 sec from the beginning of the mixing material, one droplet (5 μm) has been placed on the surface of the material. Data has been then recorded using DSA10 (Krüss GmbH, Hamburg) droplet analysis, that implies also a video analyses. The software allows the recording of 25 angles/sec during a 20 sec per interval. The high resolution measurement allows the determination of initial contact angle when the water droplet comes in contact with the material.

The obtained movies are in .avi format and static images have been acquired from it at some time intervals. The images obtained from different materials at the same moments in time have been compared. The results have been statistically analyzed using ANOVA ONE–WAY and Turkey ($p<0.05$).

The sequences in determining the contact angle are:

1. Placement of the water droplet;
2. Initial contact angle (advanced);
3. The water droplet after 2, 4, 6, 10, 15, 20 sec.

First of all, the wettability of the impression materials during the setting is much lower than after the setting. This has a direct impact during the impression phase when some materials cannot penetrate the gingival sulcus which is a wet environment.

Most condensation silicones are hydrophobic, which indicates a perfect drying of the sensitive areas.

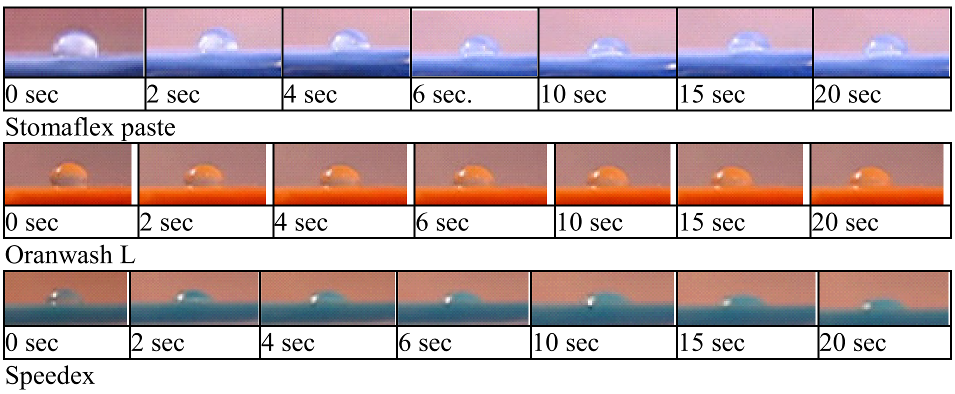


Fig. 10 – Acquired images.

The results obtained from the image capture (Fig. 10) are presented in Fig. 11. It can be observed the *Speedex* has the biggest degree of tolerance to water (hydrophilia) which confirms the superiority of this material. *Stomaflex* is the most hydrophobic, characteristic which is also shown by *Oranwash L*. *Oranwash VL* shows a hydrophilic character being placed immediately under the 70° limit for the contact angle. *Xantopren* has a high degree of hydrophilia which recommends it in clinical situations where a dry environment cannot be obtained.

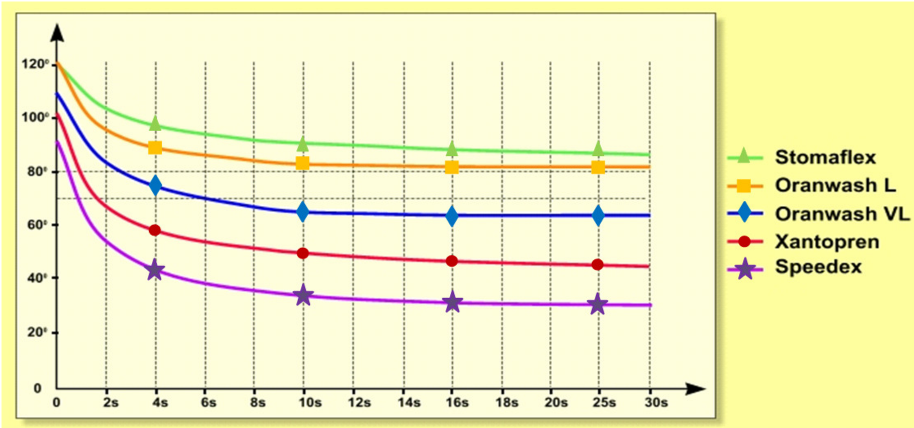
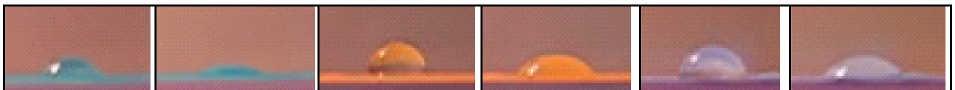


Fig. 11 − Contact angle evolution immediately after contact.

Images with the best (*Speedex*) and the worst (*Stomaflex*) materials are shown and also with *Oranwash L* which is the most frequently used in dental clinics because of the acceptable price/quality ratio (Fig. 12).



initial	after 20 sec	Initial	after 25 sec	initial	after 30 sec
Speedex		Oranwash L		Stomaflex	

Fig. 12 − Wettability of some of the materials after setting.

After the setting the wettability increases, being favorable to model casting. Again *Speedex* shows to be the most hydrophilic, the contact angle being under 10° which is achieved in fewer than 20 sec. As the images show all the materials have a better wettability after the setting, but the time interval for reaching the stability varies from one material to another.

The longest material for reaching stability is found in *Stomaflex* (30 sec) and the final contact angle is between 30 - 40°. *Xantopren* has a very good properties being similar to *Speedex*. *Oranwash VL* is superior to *Oranwash L*, the differences being significant.

5. Conclusions

1. The study presented in this paper has shown considerable differences between the studied materials, especially between *Speedex-Coltène*, the most stable and *Stomaflex - Sphofa*, the least stable. *Oranwash L* is more dimensional stable then *Oranwash VL* which can be explained by different viscosity between the 2 materials.

2. The least capable in rendering details are *Oranwash L* and *Stomaflex*. However, because of the liquid catalyzer for *Stomaflex*, a higher fluidity of the material can be obtained, and therefore a higher degree of detail rendering. Risks linked to catalyzer overdosing must also take into account.

3. *Speedex* and *Xantopren* have the highest hydrophilic degree due to lower surface tensions. *Oranwash L* and *Stomflex* have a high hydrophobic degree which is characteristic for traditional condensation silicones.

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EVALUAREA PROPRIETĂŢILOR MATERIALELOR DE AMPRENTĂ DE TIP SILICONIC

(Rezumat)

În practica curentă, ca material de amprentă, sunt folosiţi cu precădere siliconii de condensare de unde şi necesitatea realizării unui studiu comparativ pe acest tip de material.

Materialele luate în studiu sunt cele mai des utilizate dintre siliconii de condensare şi au caracteristici destul de diferite, susţinute atât prin compoziţia chimică, cât şi prin proprietăţile fizice pe care le au, reflectate în comportamentul clinic al acestor materiale. Materialele provin de la patru producători şi au fost alese câte două consistenţe - cremă şi solid.

Proprietăţile asupra cărora ne-am concentrat în cercetare sunt: stabilitatea dimensională, gradul de fidelitate al materialului şi umectabilitatea.

Pentru verificarea stabilităţii dimensionale sub aspect cantitativ, s-a realizat un dispozitiv adaptat la un microscop optic de cercetare tip IOR. În cazul materialelor fluide, se constată că cele mai stabile dimensional sunt cele ale firmei Colthène-Whaledent - Speedex, iar cele mai mari deformări le-au înregistrat produsele firmei Spofa - Stomaflex. Următoarele materiale ca stabilitate dimensională sunt Xantopren, cu performanţe destul de bune, urmate de Oranwash. Aceeaşi ierarhizare se menţine şi în cazul siliconilor de consistenţă solidă, unde Speedex este cel mai stabil dimensional, urmat de Optosil, Zetaplus, Zetaplus Soft, Stomaflex. Trebuie remarcată diferenţa foarte mare care există între Speedex şi Stomaflex, la ambele categorii de materiale.

Pentru determinarea gradului de fidelitate al materialelor de amprentă s-au folosit ca referinţe site din plasă de diferite mărimi, utilizate în industria chimică. Sitele folosite au dimensiuni cuprinse între 6 şi 42 microni, fiind eficiente în evaluarea materialelor de amprentă. S-a observat că toate materialele redau profilul primelor două reţele, iar a celei de-a treia site doar Xantoprenul şi Speedex redau fidel, fără goluri sau retezarea reliefului pozitiv. O fidelitate bună a dovedit şi Oranwash VL. Cel mai slab s-a dovedit a fi Stomaflexul, care nu s-a dovedit constant în reproducerea ochiurilor reţelei.

Hidrofilitatea materialelor de amprentă reprezintă una dintre cele mai importante proprietăţi pentru obţinerea succesului clinic. Pentru determinarea hidrofilităţii s-a ales măsurarea unghiului de contact. S-a verificat dacă unghiurile de tangenţă sunt diferite în cele două faze ale materialului de amprentă. S-au înregistrat apoi datele folosind Sistemul de analiză a formei picăturilor DSA10 (Krüss GmbH, Hamburg) ce implică şi o analiză video. Se poate observa că materialul de amprentă Speedex prezintă cea mai mare toleranţă pentru apă (hidrofilie), ceea ce dovedeşte încă o dată superioritatea acestui material. Stomaflexul, o dată în plus, se dovedeşte a fi cel mai hidrofug, dar nici Oranwash-ul L nu este cu mult mai bun. Oranwash VL se dovedeşte a fi hidrofil, plasându-se imediat sub limita de 70°, unghi final de contact. Xantoprenul are un grad crescut de hidrofilie, ceea ce îl recomandă deplin în situaţiile clinice în care uscarea nu poate fi controlată.