Comparative Study on the Characteristics of Silicone Elastomers used in Dental Impression Techniques

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Abstract. Even through nowadays technology and robotics seem to have taken a front seat on the medical field, there are still conventional methods being used in dental medicine, especially related to impression techniques. Elastomers such as silicone rubbers are widely used as impression materials for their suitable properties and behavior in dentistry. As the long-term success of the prosthetic therapy relies on the impression stage, it is important to know both the advantages and disadvantages of such materials. Nowadays in vitro and in vivo studies are being conducted to establish the precision and accuracy of these materials. In this study we report comparatively a number of characteristics determined by us under identical conditions for four such materials. Structural studies (through IR and XRF), morphology (through AFM and SEM) and surface (water contact angle) were performed. Based on the results obtained we can identify more or less significant differences between some characteristics, which could help to indicate the right material depending on the case requirements.

1. Introduction

Nowadays impression materials represent a useful tool in dental medical practice, being a method which does not require invasiveness for the patient and is easier and easier to use by dentists. These materials have been proved to be helpful and have, with the improvement of the obtaining technology in the last years, better qualities and characteristics which have been observed in both manipulation and results after usage. As the highest rate of usage is represented by elastic materials among impression materials, it is of high interest to know what requirements must these have and which are their eloquent traits for their medical application. Currently, in terms of classification of elastic materials or rubbery materials, two categories are underlined: synthetic elastomers represented by silicones, polyethers and polysulphides as well as elastic impression materials as reversible hydrocolloids (agars) and irreversible hydrocolloid (alginates). The silicone materials can also be differentiated by the method of polymerization on setting in condensation silicone impression materials and addition silicone impression materials [1-3]. A classification of above-mentioned materials is presented in Figure 1.
Impression is a mandatory step in the dental prosthesis algorithm. Synthetic elastomers are irreversible elastic materials, the most commonly used category for impression taking. They are indicated to record the impression of the prosthetic fields for the fixed prosthesis as well as for the functional impression of the partially/total edentulous prosthetic field.

When an impression material is used, dentists expect that material to pose a series of requirements such as accuracy, dimensional stability which is bound to accuracy, good manipulative and handling ease and good setting characteristics. To these factors, clinical considerations can also be factors involved in selecting and using an impression material.

By definition the role of impression materials is to record intraoral structures in detail (of both teeth and soft tissues) with good accuracy which are then used for the prostheses construction [1]. In medical practice, dentists choose materials like these based on a process of association between the chemical groups contained and the properties of materials that recommend them for the ultimate purpose. Even so, it is sometimes difficult to evaluate a product based on the data provided by the producers. In this study, we have evaluated in terms of structure, morphology and surface analysis four different commercially available impression materials used in medical practice, as clinical behavior depends on materials’ properties [4, 5]. For this, we selected and prepared as instructed by the producers’ indications four different materials represented by Coltene Speedex putty, Coltene Speedex light body, Lascod Silaxil light body and Zhermack Oranwash L light body. The light body and putty property expresses the materials viscosity [6-9].

As chemical composition, silicone condensation materials contain polydimethylsiloxane (PDMS) as the principal component and inert inorganic mass (usually pyrolytic silica and titanium dioxide) to increase the viscosity of the liquid precursor and improve the mechanical strength of the elastomer formed by crosslinking, for the main body paste. The accelerator paste can consist of tin octoat, tetraethyl orthosilicate and sometimes also of chromium oxide or palladium metal particles with the role of capturing hydrogen, which is not beneficial for the surface of the impression [10]. For the proposed studies, all the samples were pre-prepared in the same way as 10 mm thick films, cut at the same dimensions of 2 cm².

2. Structural study
2.1. FTIR-ATR analysis
Attenuated Total Reflectance Infrared (ATR-IR) spectra have been measured by using a Bruker Vertex 70 spectrometer (Bruker Optics, Ettlingen, Germany) equipped with a ZnSe crystal. The registrations were carried out in the 600-4000 cm⁻¹ spectral range, at room temperature with a resolution 4 cm⁻¹ and accumulation of 32 scans. All samples were cut at the same dimensions of 1 cm², with a thickness of 10 mm for this analysis.

The main bands in the 4000-2500 cm⁻¹ spectral range (Figure 2(a)) are assigned to the O-H stretching vibrations from alcohol intermolecular bonded in the structure of the materials. The C-H
asymmetric and symmetric stretches in methyl and methylene groups of silicone components and alcohol can be observed at 2960-2920 cm\(^{-1}\) and 1860 cm\(^{-1}\), respectively. The 2000-1500 cm\(^{-1}\) spectral region is characteristic for O-H deformation vibrations (1650 cm\(^{-1}\)) and vinyl stretching vibrations at 1608 cm\(^{-1}\) (from catalyst paste and vinyl silicones). The bands at 1730, 1744 and 1780 cm\(^{-1}\) are characteristic for esters present in the structure of the activators, while the bands at 1950-1870 cm\(^{-1}\) are specific for H-bonding interactions between the OH groups appeared during the hydrolysis reaction of the alkyl silicate and carbonyl groups (C=O) from activator (stannous octoate) (Figure 2(b)). In the 1600-1300 cm\(^{-1}\) spectral region there are a number of absorption bands assigned to the C-O asymmetric stretches (1554 cm\(^{-1}\)) of stannous activator, C-H bending in methylene and methyl groups at 1460 cm\(^{-1}\) and strong S=O stretching vibrations in sulphate groups (gypsum filler) at 1410 cm\(^{-1}\) and 1378 cm\(^{-1}\) (Figure 2(c)). In the 1300-600 cm\(^{-1}\) spectral range the symmetric deformation vibrations of the Si-CH\(_3\) groups and the stretches of Si-O-Si and Si-C groups can be observed at 1260 cm\(^{-1}\), 1080-1010 cm\(^{-1}\) and 790 cm\(^{-1}\). The band at 868 cm\(^{-1}\) is assigned to the Si-O-C stretches in the silicate cross-linking agent, while the bands at 962, 934 and 902 cm\(^{-1}\) are due to the Si-O-C deformation vibrations (Figure 2(d)). The bands at 800-600 cm\(^{-1}\) are assigned to the C-H out-of-plane deformation vibrations and sulphate ions [11].

**Figure 2.** ATR-IR spectra of the four impression materials for Lascod Silaxil light body (B1), Zhermack Oranwash L light body (B2), Coltene Speedex light body (B3), Coltene Speedex putty (B5).
2.2. XRF analysis
Elemental composition of the samples was estimated by using X-Ray Fluorescence spectroscopy. XRF spectra were recorded on a X-Calibur Xenemetrix device equipped with a high-resolution detector and an X-ray tube, a chamber and a sample table that can accommodate various sample types and sizes and assisted with an Analytix software program. The tests were performed at ambient temperature of 32 °C and the results can be seen on Figure 3.

![XRF spectra](image)

**Figure 3.** XRF spectra for (a) Coltene Speedex putty, (b) Coltene Speedex light body, (c) Lascod Silaxil light body and (d) Zhermack Oranwash L light body.

The XRF spectra confirm, as well as the IR spectra, that the impression materials are in fact silicone materials, the presence of Si being shown on all the results, as an element as such or in a chemical group, respectively. The presence of Cu and Fe on the XRF spectra is attributed to any added dye and fillers and/or manipulation tools used for the preparation of the materials [12].

Ferric impurities are often present in the citrine, while trace amounts of iron, copper or zinc are present in rose quartz. Additionally, phosphate or aluminium can be found in the pink quartz.

| Material               | Atomic composition | Atomic ratio |
|-----------------------|--------------------|--------------|
|                       | Si     Ca     Cr    Zn   | Si/Ca   Si/Cr  Si/Zn |
| Coltene Speedex putty | 40120  17081  840  1323 | 3,35     88,7    70,4 |
| Coltene Speedex light body | 60438  4848 | 17,8         |
Beside quartz, calcium phosphate is usually added as filler in base paste (putty), while the stannous catalyst is present in catalyst paste, so that traces of these elements can been observed in the XRF spectra.

Atomic ratio calculated for the most representative elements present in the Coltene Speedex putty and light body materials is shown in Table 1.

3. Morphology study

3.1. AFM

As impression materials used in dental medicine have to be precise, accurate for the fidelity of the desired result in the prosthetic therapy, it is of high interest for them to be smooth and have as less discontinuities as possible with a low roughness to them as a main characteristic.

Figure 4. 2D and 3D topography of the four impression materials.
The less rough the surface of the impression material is, the more accurate will be the impression obtained with it, as even the smallest details are extremely significant [13-15].

With the purpose of obtaining fidelity characteristics, an Atomic Force Microscope Park SYSTEMS XE-100 with liquid cell and thermal control (ILX Lightwave LDT-5948 Precision Temperature Controller) was used for the morphology study of the impression materials selected and both 2D and 3D topography images can be observed in Figure 4. The estimated roughness values based on these images are between 124 nm for the Lascol Silaxil sample and 510 nm for the Coltene Speedex light body sample, the values for the other samples being in the first part of this field.

3.2. SEM analyses

As impression materials based on crosslinked silicone are solid inorganic materials, Scanning Electron Microscopy - SEM, was also conducted on film surface using an ESEM Quanta 200 Scanning Electron Microscope (Netherlands) operating at 20 kV. In dental practice, impressions taken from patients often have to maintain their shape for longer periods of time [16, 17]. As such, morphology characteristics were obtained for the materials tested after 14 days after their preparation. SEM images captured for our samples are presented in Figure 5.

![Figure 5. SEM images of impression materials (a) Coltene Speedex putty, (b) Coltene Speedex light body, (c) Lascol Silaxil light body, (d) Zhermack Oranwash L light body.](image)

Based on the surface images obtained through SEM analysis the three samples of light body impression materials Coltene’s Speedex, Lascol Silaxil and Zhermack Oranwash L seem to have close morphologies. However, unlike AFM images indicating roughness on small areas (40x40 µm), SEM images taken on larger areas indicate surface heterogeneities of materials created by the presence of additions. According to them, contrary to the AFM results, the Coltene Speedex light body sample
appears to have the most uniform surface, followed by Lascod Silaxil light body Zhermack Oranwash L light body and Coltene Speedex putty.

4. Surface study
The hydrophilic surface with a smaller static contact angle and larger work of adhesion values are those characteristics sought for the impression materials [2].

For the hydrophilic or hydrophobic characterization of the materials measurements of contact angle and work of adhesion were conducted using a CAM 101 (KSV Instruments, Helsinki, Finland) system, equipped with a liquid dispenser, video camera and drop shape analysis software. In this regard, experimental liquids were prepared and used (distilled water and a 6.8 pH buffer solution for the physiological simulation of saliva), while their surface tension was determined by using a Sigma 700 Force Tensiometer. The obtained results 72.8 N/m for distilled water and 59.9 N/m for the simulated saliva, were used in the calculations for the adhesion work $1+\cos \theta \times 72.8$ for water and $1+\cos \theta \times 59.9$ for simulated saliva. All measurements were made at room temperature.

Taking under consideration the nature of the materials used – biomaterials – one might expect that impression materials would have to be hydrophobic in order to achieve the best results when used. In this case the adhesion and cohesion forces which occur in surface tension when the materials come in contact with water would not make the material adhere to the water, which is the expected outcome [18-20].

In Figure 6 and Figure 7 are represented the static contact angle and the work of adhesion.

As expected, the Coltene Speedex light body sample with the highest roughness has the smallest contact angle and therefore the largest adhesion work.

Correlating the contact angle values with AFM and SEM data, it could be said that the AFM provides more relevant data, as measurement technique. The hydrophilic surface and a small contact angle represent those characteristics sought for the impression materials.

5. Conclusion and perspectives
Silicone elastomer impression are among the most used impression materials in dental medicine. Four impression materials, commercially available, in light body form and putty have been tested in terms of structure, morphology and surface.

The structural study using Fourier Transform Infrared in Attenuated Total Reflected mode and X-Ray Florescence proves that these are in fact silicone-based materials, Si being the main element present on all spectra obtained, in different atomic ratios to other elements in all four materials.

The morphological studies were conducted by AFM and SEM. The AFM results that proved to be the most relevant from this perspective, indicate the Lascod Silaxil material as having the least
roughness and as a result potential for rendering the details with the highest accuracy. However, roughness influences wettability, meaning that higher roughness will decrease the contact angle, as found in the Coltene Speedex light body sample, which is desirable for impression materials. Therefore, it can be concluded that none of these materials is ideal, each having its performance and limitations, which can be preferred depending on the situation.

For a better understanding of the structural part we intend to also study the materials by EDAX microanalysis (Energy Dispersive X-ray Analysis).

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