

Inorganic Particle Analysis of Dental Impression Elastomers

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The aim of this study was to determine quantitatively and qualitatively the inorganic particle fraction of commercially available dental elastomers. The inorganic volumetric fraction of two addition silicones (Reprosil Putty/Fluid and Flexitime Easy Putty/Fluid), three condensation silicones (Clonage Putty/Fluid, Optosil Confort/Xantopren VL and Silon APS Putty/Fluid), one polyether (Impregum Soft Light Body) and one polysulfide (Permlastic Light Body) was accessed by weighing a previously determined mass of each material in water before and after burning samples at 600°C, during 3 h. Unsettled material samples were soaked in acetone and chloroform for removal of the organic portion. The remaining filler particles were sputter-coated with gold evaluation of their morphology and size, under scanning electron microscopy (SEM). Flexitime Easy Putty was the material with the highest results for volumetric particle fraction, while Impregum Soft had the lowest values. Silon 2 APS Fluid presented the lowest mean filler size values, while Clonage Putty had the highest values. SEM micrographs of the inorganic particles showed several morphologies - lathe-cut, spherical, spherical-like, sticks, and sticks mixed to lathe-cut powder. The results of this study revealed differences in particle characteristics among the elastomeric materials that could lead to different results when testing mechanical properties.

Key Words: Dental impression materials, scanning electron microscopy, filler.

INTRODUCTION

Making impressions to duplicate oral conditions and tooth morphology and constructing casts or models in gypsum are important steps in numerous prosthetic dentistry procedures (1,2). In order to produce an accurate impression, the materials should be fluid enough to seep around the oral tissues and viscous enough to remain contained in the tray. They should set into a rubbery solid within a reasonable amount of time and should not distort or tear when removed from the mouth (1).

There is a group of synthetic rubber impression

materials, known as elastomers or elastomeric impression materials, which were developed during World War II due to the difficulty to obtain natural rubber. These elastomers are materials formed of molecules (polymers) that are joined to each other by crosslinking in a process known as polymerization (1). According to the polymer composition, there are three kinds of dental elastomers: polysulfide, polyether and cured silicones (1-3).

Silicone materials are classified as condensation or addition silicones, depending on the reaction that produces polymerization (1,3,4). Condensation-cured materials are also known as polysiloxanes as they have alternating atoms of oxygen and silicone. They are

all two-component systems with a base and a catalyst paste. The base consists of siloxane and inorganic particles while the catalyst paste contains alkylsilicate and a tin-based activator. Setting occurs by crosslinking between the terminal hydroxyl groups and the alkyl, which produces alcohol as a byproduct. As alcohol is produced in the reaction, the set material distorts as it is released (1,3). Addition-cured silicones are also known as polyvinylsiloxanes (PVS) or vinyl polysiloxanes. They are also two-component materials and the setting occurs by crosslinking of vinyl groups in the base material with a hydride group in the catalyst paste via a platinum catalyst and there are no reaction byproducts (1,3). Inorganic particles are present in both pastes normally in the form of amorphous silica to add bulk and improve the properties of the paste (5).

The presence of particles is important on the strength of both materials and determines the viscosity (1) and accuracy (6,7). Craig and Sun (8) determined that there is a relationship between strain in compression and consistency as it is decreased from putty to light bodied consistency. Lu et al. (9) showed that there are differences in the mechanical properties of the impression materials correlated to their consistencies and Chen et al. (2) stated that higher filler component may increase the accuracy. The particle size is also important to be considered, as the smaller particles tend to aggregate among each other and the larger ones do not contribute to reinforcement (1).

To the best of our knowledge, no previous study has evaluated the inorganic fraction of dental elastomers, considering only the viscosities. Due to the importance of filler in the composition of impression materials, the aim of this study was to determine filler fraction and size of commercial dental elastomers. In addition, fillers were analyzed qualitatively by scanning electron microscopy (SEM) and their composition was determined by energy-dispersive x-ray spectroscopy (EDX).

MATERIAL AND METHODS

In the present study, we analyzed: 6 condensation silicones [Clonage Putty and Clonage Fluid (DFL, Rio de Janeiro, RJ, Brazil), Optosil P Confort and Xantopren VL Plus (Heraeus Kulzer, Hanau, Germany), Silon 2 APS Putty and Silon 2 APS Fluid (Dentsply Latin America, Petropolis, RJ, Brazil)]; 4 addition silicones [Flexitime Easy Putty and Flexitime Correct Flow (Heraeus Kulzer), Reprosil A⁺ Putty and Reprosil A⁺ Light (Dentsply Latin America)]; 1 polyether [Impregum

Soft Light (3M/ESPE, AG, Seefeld, Germany)]; and 1 polysulfide [Permlastic Light Body (Kerr Corporation, Orange, CA, USA)].

The volumetric fraction, morphology, size and composition of the filler particles of the commercial brands of dental elastomers were analyzed and are described in the following sections.

Volumetric Particle Fraction

The percentage of inorganic particles by volume was determined by calculating the difference between the mass of each material tested in air and in water (Archimedes' Principle) (10).

The materials were prepared according to the manufacturers' instructions and placed in an aluminum mold. Cylindrical specimens (12-mm diameter, 20-mm high) of each material were weighed in an analytical balance (JK 180; Chyo Balance Corp., Tokyo, Japan), accurate to 0.0001 g (n=5). The dry mass (Md) of the material after the setting time was determined in air. In order to determine the wet mass (Mi), a receptacle and a stainless steel mesh were placed onto the balance plate, filled with distilled water, and the specimen was immersed. The volume of the specimen after setting time was measured using the equation $V_s = M_d - M_i$.

The specimens were then burned in an oven (Bravac Ltda, São Paulo, SP, Brazil) to remove the organic phase, over 3 h gradually increasing the temperature from room temperature to 600°C (this temperature was previously determined in a pilot study). The resulting inorganic material was intact and pill-shaped. The mass in air (Mp) was then measured as described above. To determine the wet mass of the particles (Mpi), the specimens were triturated with a pestle and immersed in distilled water as described previously. The specimens were triturated to destroy air filled spaces in their interior. The volume of the inorganic particles was measured using the following equation: $V_p = M_p - M_{pi}$. The percentage of the inorganic phase by volume was calculated using the following equation: Inorganic particle percentage = $(V_p/V_s) \cdot 100$

Particle Morphology and Size

The morphology of the particles was determined by SEM micrographs. Unsettled amounts of each material (0.5 g) were submitted to the washing technique (11). The matrix was removed by dissolving each

material in 5 mL of acetone and centrifuging for 2 min at 1,000 rpm. This process was repeated 3 times. The remaining material mass was immersed 3 times in chloroform and centrifuged as described above for further washing and elimination of the matrix. The particles were then smeared in aluminum stubs (9), sputter coated with gold/palladium in high vacuum (SCD 050; Bal-tec AG, Liechtenstein), and examined with a scanning electron microscope (JSM-5600; JEOL Ltd. Tokyo, Japan) operating at 15 kv. Images were obtained at 1,200 \times magnification.

SEM micrographs were imported to the Image-Pro Plus 4.5 image analyzer software (Media Cybernetics Inc., Bethesda, MD, USA) and analyzed using the measurement tool. At least 20 particles of each material were analyzed during this procedure, determining the maximum, minimum and mean diameter size. Fillers' size was determined in micrometers (μm).

Particle Composition

Particle composition was determined using the powder obtained in the previous test by EDX. The particles were smeared in acrylic resin stubs, carbon coated (Denton Vacuum Desk II Sputtering; Denton Vacuum, Cherry Hill, NJ, USA), and then observed in a SEM/EDX integrated analysis system (SEM - JSM 5600; JEOL Ltd.; EDX: Vantage 1.4, Noran Instruments, Tokyo, Japan). Analyses were performed at a working distance (WD) of 20 mm, 10 kv, and variable spot size to obtain a deadtime of 20-25%. For each specimen three line scan analyses of 100 s were performed.

RESULTS

Volumetric Particle Fraction

The mean values of percentage content of inorganic particles in volume are listed in Table 1. Flexitime Easy Putty was the material with the highest mean value (73.68%), while Impregum Soft Light was the material with the lowest value (4.04%). It was typically observed that materials with high viscosity (putty consistency) presented greater amount of inorganic particle than the commercially corresponding materials with low viscosity (light bodied consistency).

This is shown by the results of Clonage Putty and Clonage Fluid (47.70% and 31.89%, respectively), Flexitime Easy Putty and Flexitime Correct Flow

(73.68% and 52.03%), Optosil P Confort and Xantopren VL Plus (24.74% and 23.45%), Reprosil A⁺ Putty and Reprosil A⁺ Light (32.66% and 29.04%), and Silon 2 APS Putty and Silon 2 APS Fluid (48.25% and 35.73%). Generally, it was observed that the addition cured silicones presented greater amount of fillers than condensation cured silicones when using the same classification of consistency (high viscosity or low viscosity), except for Reprosil silicone that presented filler fraction similar to condensation silicones. Polyether material showed the lowest amount of inorganic particle.

Particle Morphology and Size

The morphology of the particles is shown in the SEM micrographs presented as Figures 1 and 2. The materials particles had the appearance of lathe-cut powder, spherical objects, spherical-like objects, and sticks, which seemed to be a junction of various circular and perforated objects with a central hole and variable length and diameter. It was not possible to eliminate the polymeric matrix from Xantopren VL Plus even after dissolving each material over one week in acetone and chloroform. Also, it was still possible to see remaining portions of the polymeric matrix adjacent to the spherical fillers of Clonage Putty.

The maximum, minimum and mean length (for lathe-cut shape) or diameter (for spherical shape) values

Table 1. Mean values for volumetric particle fraction (%).

Material	Volumetric filler fraction
Clonage Putty	47.70
Clonage Fluid	31.89
Flexitime Easy Putty	73.68
Flexitime Correct Flow	52.03
Optosil P Confort	24.74
Xantopren VL Plus	23.45
Reprosil A ⁺ Putty	32.66
Reprosil A ⁺ Light	29.04
Silon 2 APS Putty	48.25
Silon 2 APS Fluid	35.73
Impregum Soft Light	4.04
Permlastic Light Body	39.35

of the inorganic particles are listed in Table 2. Clonage Putty showed the highest values (27.75 μm), while Silon 2APS presented the lowest values (4.60 μm). Because of the difference in the particle shape of Reprosil A⁺ Putty, Reprosil A⁺ Light and Impregum Soft Light,

which showed a considerable length to be measured, Table 2 presents their maximum, minimum and mean length values. It was not possible to measure the filler size of Xantopren VL Plus because of the difficulty to eliminate the polymeric matrix.

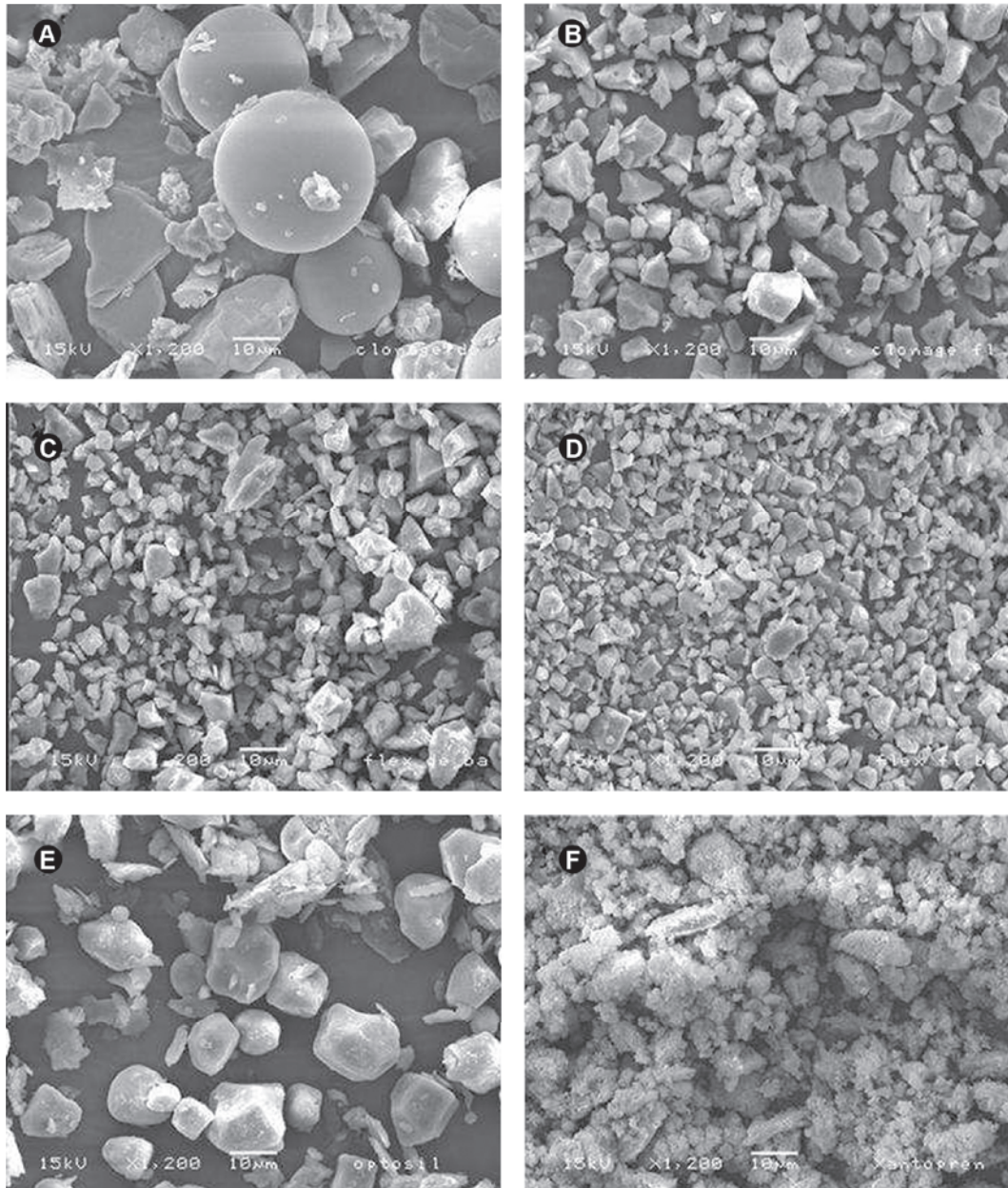


Figure 1. Panel of SEM micrographs of elastomeric materials (1,200 \times). A= Clonage Putty: large and spherical glass particles can be seen mixed with large lathe-cut particles; B= Clonage Fluid: lathe-cut particles can be seen, but smaller than in putty consistency; C= Flexitime Easy Putty: the material presents small lathe-cut particles mixed with larger particles of the same type; D= Flexitime Fluid: the fluid consistency of the Flexitime also presents lathe-cut particles, but only in smaller size; E= Optosil Confort: can be seen large vitreous particles with spherical-like form; F= Xantopren: it was not possible to completely eliminate the polymeric matrix to visualize the fillers, but the image suggests the presence of lathe-cut particles.

Particle Composition

Results for particle composition by EDX analysis are listed in Table 3. Silicon (Si) was the component

with highest concentration in the materials. In general the composition was the same for all materials with the presence of 100% Si composition. Clonage Putty presented Zinc (Zn), Si, Calcium (Ca), and Indium (I). Optosil P Confort presented Zn, Magnesium (Mg),

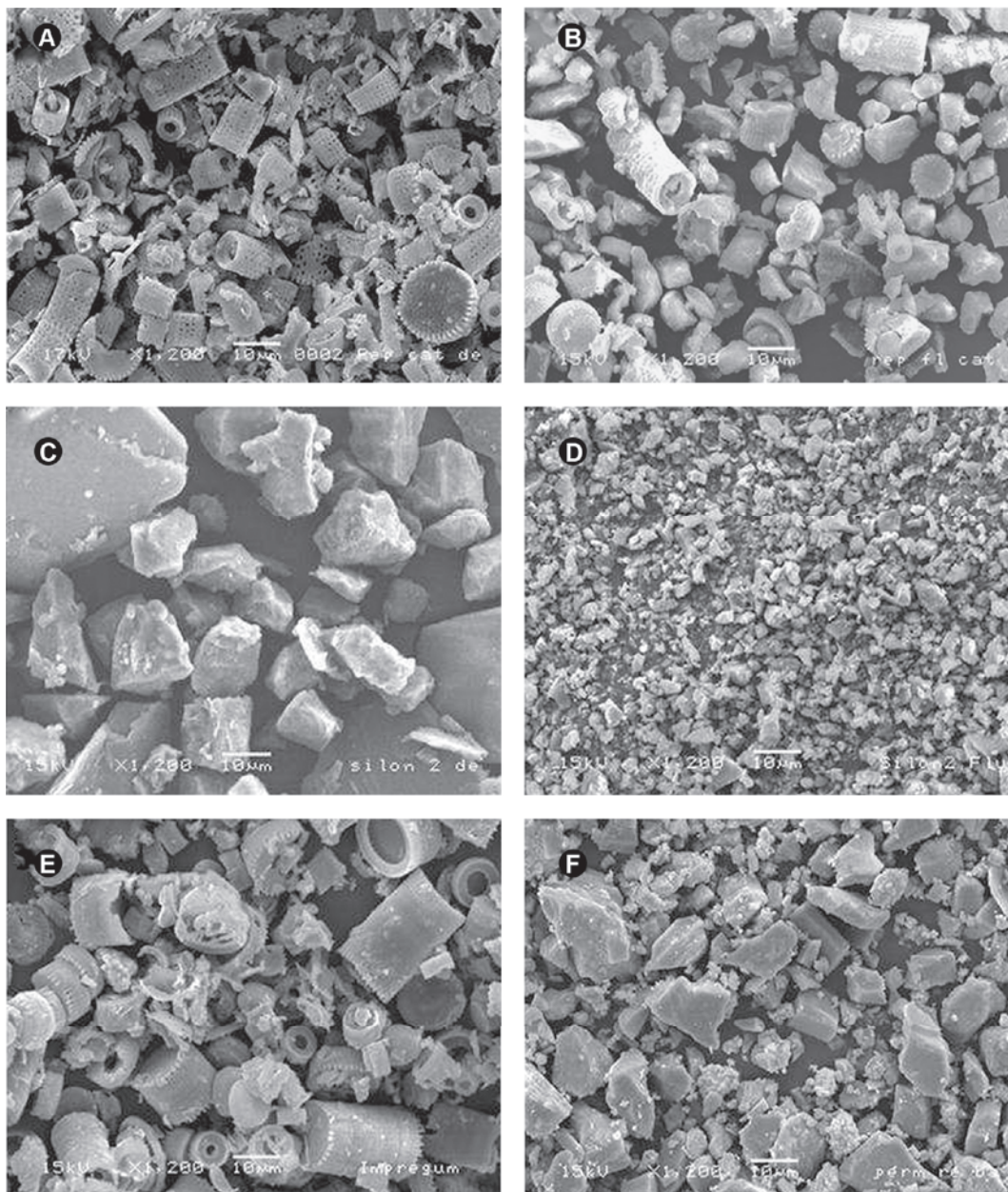


Figure 2. Panel of SEM micrographs of elastomeric materials (1,200×). A= Repsil A+ Putty: Diatomite particles can be seen with several shapes and sizes, predominating cylindrical and perforated sticks; B= Repsil A+ Regular: cylindrical diatomite particles can be seen mixed with big lathe-cut particles; C= Silon 2APS Putty: large particles with irregular form can be seen; D= Silon 2APS Fluid: the fluid consistency of Silon 2 APS presents small lathe-cut particles; and, E= Impregum Soft Light: Diatomite particles can be seen with several shapes and sizes, with a circular, cylindrical or helical structure presenting various perforations, and; F= Permlastic Light Body: large lathe-cut particles can see mixed with smaller particles. Residues of the polymeric matrix can also be seen.

Si, and Sodium (Na). Impregum Soft and Reprosil A⁺ presented Zn, Germanium (Ge), Aluminum (Al), Si, Ca, and Antimony (Sb).

DISCUSSION

The viscosity of impression materials is determined, in part, by the amount of particles (2,5) and the findings of this investigation showed that materials with high viscosity (putty consistency) presented greater amount of inorganic filler than the commercially corresponding materials with low viscosity (medium or light bodied consistency), as it was seen for Clonage (putty/fluid), Flexitime (putty/fluid), and Silon (putty/fluid). This also occurred with Reprosil (putty/fluid) and Optosil/Xantopren, although the percentage of fillers between high and low viscosity materials does not seem to determine effectively the consistency of the materials. It seems that the manufacturers of these products use plasticizers to control it. Another fact to be considered is that the inorganic filler composition or polymeric matrix may have some influence on the material flow. Reprosil light consistency differs from Reprosil putty due to the presence of stick fillers mixed to lathe-cut fillers. The different composition of the filler could be related to the difference in material consistency (putty/fluid). It was not possible to determine if Optosil and Xantopren fillers differ from each other, but the impossibility to remove

Table 2. Maximum, minimum and mean values for particle size (μm) of the evaluated materials.

Material	Maximum	Minimum	Mean
Clonage Putty	36.81	17.69	27.75
Clonage Fluid	15.68	3.62	8.91
Flexitime Easy Putty	15.48	4.29	8.44
Flexitime Correct Flow	10.55	3.29	5.53
Optosil P Confort	18.46	5.63	11.66
Xantopren VL Plus	---	---	---
Reprosil A ⁺ Putty	24.04	5.47	10.43
Reprosil A ⁺ Light	18.50	3.29	9.69
Silon 2 APS Putty	22.24	9.64	15.13
Silon 2 APS Fluid	7.56	2.73	4.60
Impregum Soft Light	22.60	4.50	12.76
Permlastic Light Body	17.76	2.25	10.91

silicone rubber matrix from Xantopren may indicate that there are differences on the linkage between fillers and matrix or matrix composition influencing on viscosity.

There is a correlation between some properties of the elastomeric impression materials and particle fraction (2,8,9,12) as the consistency decreases from putty to heavy, medium or light bodied materials (5, 8). It is obviously because less amount of polymeric matrix is present in the composition of the materials (1). Several authors (2,8,9,13) have shown that material and consistency had a significant influence on elastic recovery, permanent deformation, strain in compression, tear energy, tensile strength, thermal expansion, and dimensional stability. The light bodied materials had lower elastic recovery than the heavy bodies materials (9). Higher strain in compression values indicate more flexibility. In the present study, the putty or heavy bodied materials were stiffer than the light bodied materials (8,9,14). Furthermore, heavy bodied impression materials have been shown to have higher tear resistance than those with light body consistency (9). The tensile

Table 3. Composition of the materials particles by EDX analysis.

Material	Composition (%)
Clonage Putty	Zn (11.92), Si (68.3), Ca (18.33), I (1,45)
Clonage Fluid	Si (100)
Flexitime Easy Putty	Si (100)
Flexitime Correct Flow	Si (100)
Optosil P Confort	Zn (6.39), Mg (15.30), Si (72.89), Na (5.42)
Xantopren VL Plus	---
Reprosil A ⁺ Putty	Zn (2.14), Ge (0.86), Al (1.23), Si (89.4), Ca (1.83), Sb (4.54)
Reprosil A ⁺ Light (diatomite)	Zn (2.34), Ge (1.2), Al (1.29), Si (88.5), Ca (1.58), Sb (5.21)
Reprosil A ⁺ Light (lathe-cut filler)	Si (100)
Silon 2 APS Putty	Si (100)
Silon 2 APS Fluid	Si (100)
Impregum Soft Light	Zn (4.23), Ge (0.96), Al (2.55), Si (82.2), Ca (2.21), Sb (7.85)
Permlastic Light Body	Si (100)

strength has been found to be higher for the heavy bodied materials than for the light bodied ones (9). The higher the viscosity of the impression material, the lower the thermal expansion coefficient (13). Large dimensional changes were observed for light consistency silicones compared to those with putty consistency, illustrating that the proportion of particles does modify the accuracy of the materials (2,8). Based on results of this study, it is not expected from Reprosil and Optosil/Xantopren to present the correlations mentioned above because it seems that the plasticizers, particle composition, linkage between particles and matrix, and matrix composition can somehow modify the properties of these materials.

It was also observed that the addition-cured silicones and polysulfide presented larger amount of particles than the condensation-cured silicones and polyether considering the same consistency (high viscosity or low viscosity). It seems that condensation-cured silicones do not allow incorporating a maximum amount of particles in the polymeric matrix as it is possible to do with the addition-cured silicones or they are supposed to display acceptable properties with less particles.

The panels of SEM micrographs of the inorganic particles (Figs. 1 and 2) showed numerous morphologies - lathe-cut, spherical, spherical-like, sticks, and sticks mixed to lathe-cut powder. Usually, colloidal silica or microsized metal oxide is added as particles (1). Particles with the lathe-cut pattern are commonly produced by grinding or milling glasses (1). Spherical particles are obtained by pyrolytic or precipitation of Si. The stick-like particles are cell walls of algae from the division *Chrysophyta*, class *Bacillariophyceae*. Termed diatomaceous earth or diatomite, this material is mined and used for a variety of commercial purposes because of its worldwide range (15).

The influence of particles on the strength of a silicone elastomer is critical (1). The selection and pretreatment of the particle are of extreme importance because silicones have a low cohesive energy level and, thus, a weaker intermolecular interaction. The particles are routinely surface treated to provide better adhesion to the polymeric matrix (1). This is the reason why it was impossible to remove the polymeric matrix from Xantopren VL Plus. The linkage between particles and polymer was so tight that even after dissolving Xantopren and over a week in acetone and chloroform it was not possible to remove the silicone rubber.

The EDX analysis showed that the majority of

the materials presented 100% Si composition, which seems to be the main composition of silicone rubber materials (1). Materials presenting particles with other metals in their composition - Zn, Ca, I, Mg and Na - may be those that are originated from microsized metal oxides (1). Impregum Soft and Reprosil use diatomite as filler particles. According to Bold and Wynne (15), the main element in the composition of diatomite is Si, which has been shown to be an absolute requirement for perfect cell functioning.

Silicone impression materials are considered to be the best in reproducing surface details. The International Organization for Standardization (ISO) states that the elastomeric impression materials must reproduce lines of 75, 50 and 20 μm - width according to the classification determined by the international standard (16). The size and amount of particles could be related to silicone rubber accuracy. Impregum Soft Light has diatomite as filler, which is relatively larger than the fillers of the others materials. However, as it has the lowest values of inorganic particles in volume, and thus it is expected that it would be a material with nice results for detail reproduction. Likewise, Xantopren VL Plus is the condensation silicone with the lowest values for percentage content of inorganic particles in volume. In this case, it is also expected that it would be a material with good results for reproduction of details although it is important to mention its high hydrophobic matrix and the fact that it was not possible to measure its volumetric filler fraction. Likewise, Silon 2 APS Fluid could present good results for detail reproduction because of its lower mean filler size values (4.60 μm), in spite of presenting a high hydrophobic matrix as well. Permlastic Light Body also has hydrophobic matrix and filler particles of intermediate size and amount compared to other materials.

Accurate casts and models are needful in a large number of dental procedures (1,2,17,18), and the volumetric filler fraction is one of the factors affecting materials properties (19). This study determined the inorganic filler fraction and quality of some commercial brands of elastomeric rubber impression materials. Further research is necessary to make a suitable correlation between particles, polymer matrix, and results for elastic recovery, strain in compression, dimensional stability, radiodensity, and detail reproduction, among other properties.

Within the limitations of the proposed analyses, the SEM images of the inorganic particles showed a

wide range of morphologies. Flexitime Easy Putty was the material with the highest results for volumetric filler fraction, while Impregum Soft Light had the lowest values. Silon 2 APS Fluid presented the lowest mean values of filler size while Clonage Putty had the highest values. The results of this study revealed differences in particle characteristics among the tested materials that could lead to different results when testing mechanical properties.

RESUMO

O objetivo desse estudo foi determinar quantitativamente e qualitativamente a fração de partículas inorgânicas de elastômeros dentais disponíveis comercialmente. A fração volumétrica de dois silicões por adição (Reprosil Denso/Fluido e Flexitime Denso/Fluido), três silicões por condensação (Clonage Denso/Fluido, Optosil/Xantopren, e Silon 2 APS Denso/Fluido), um poliéter (Impregum Soft Light) e um polissulfeto (Permlastic Light Body) foi determinada pela pesagem prévia de uma determinada massa de cada material em água antes e após a queima das amostras a 600°C, por 3 h. Amostras de material não polimerizado foram imersas em acetona e clorofórmio para a remoção da parte orgânica. As partículas de carga remanescentes foram cobertas com uma camada de ouro para avaliação da sua morfologia e tamanho, em microscopia eletrônica de varredura. O material Flexitime Denso foi o material com maior fração volumétrica de partículas de carga, enquanto que o Impregum teve menor fração volumétrica. Silon 2 APS Fluido apresentou partículas de carga de menor tamanho, enquanto que o Clonage Denso apresentou as maiores partículas. A observação em MEV, mostrou partículas de carga com vários tipos de morfologia (esféricas, irregulares, semi-esféricas, retangulares e mistura de retangulares/irregulares). Os resultados desse estudo mostraram diferenças nas partículas de carga dos materiais, as quais podem levar a diferentes resultados em suas propriedades mecânicas.

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