Hugo Lemes Carlo

Influência do conteúdo volumétrico das partículas de carga nas propriedades mecânicas de diferentes materiais de moldagem

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Materiais Dentários.

Orientador: Prof. Dr. Mário Alexandre Coelho Sinhoreti

Co-orientador: Prof. Dr. Carlos José Soares

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PROF. DR. MARIO ALEXANDRE COELHO SINHORETI

PROF. DR. MARCELO COELHO GOIATO

PROF. DR. VANDERLEI LUIZ GOMES

PROF. DR. LOURENÇO CORRER SOBRINHO

Rafael L-X·Con
PROF. DR. RAFAEL LEONARDO XEDIEK CONSANI

Dedico este trabalho

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RESUMO

Baseado no entendimento incompleto de como o conteúdo de partículas de carga influencia nas propriedades mecânicas dos materiais de moldagem este estudo avaliou quantitativa e qualitativamente o conteúdo de partículas de carga inorgânicas presente em cinco marcas comerciais de alginatos (Jeltrate; Jeltrate Plus, Jeltrate Chromatic Ortho, Hydrogum e Ezact Krom) e nove marcas comerciais de siliconas de adição e/ou condensação nas consistências massa e/ou fluida (Clonage, Elite HD$^+$ Light Body, Express Light Body, Flexitime, Optosil P Confort/Xantopren VL Plus, Oranwash L, Reprosil A$^+$, Silon 2 APS e Virtual Extra Light Body). Foram realizados testes para determinar recuperação elástica e deformação sob compressão dos alginatos e dos elastômeros e estabelecer, dessa forma, uma correlação entre os resultados para partículas de carga e os testes mecânicos. O conteúdo volumétrico das partículas de carga foi determinado pesando-se as amostras submersas em água antes e após a queima das mesmas durante 3h a 450°C (alginatos) e a 600°C (siliconas). Quantidades determinadas de materiais não polimerizados foram lavadas em acetona e clorofórmio e recobertas com ouro para avaliação da morfologia e tamanho das partículas em M.E.V. A composição foi determinada por EDX. A recuperação elástica e a deformação sob compressão foram determinadas de acordo as especificações № 1563 (alginatos) e 4823 (elastômeros) da ISO. O alginatos Jeltrate e Jeltrate Plus apresentaram os maiores valores médios para quantidade volumétrica de partículas de carga (%) enquanto o material Hydrogum apresentou os menores valores. A silicone de adição Flexitime Easy Putty apresentou os maiores valores de quantidade volumétrica de partículas, enquanto que a silicone de condensação Xantopren VL Plus apresentou os menores. As partículas de carga dos alginatos apresentaram-se, de forma geral, como objetos esféricos e com perfurações. O material Hydrogum apresentou forma de bastões cilíndricos e perfurados. As siliconas apresentaram morfologias variadas – partículas trituradas, esféricas, esferóides, bastões cilíndricos perfurados e bastões misturados a partículas usinadas. O alginato Ezact Krom apresentou os maiores valores médios de tamanho de partícula, enquanto que o alginato Hydrogum as menores. A silicone de condensação Clonage massa apresentou os maiores valores médios de tamanho de partícula, enquanto a silicone de adição Elite HD os menores. A análise da composição das partículas apresentou o silício como o elemento em maior quantidade. Com relação aos resultados de recuperação elástica, o alginato Ezact Krom e as siliconas Reprosil A^+ massa e Flexitime fluida apresentaram os maiores valores de recuperação elástica, enquanto o alginato Jeltrate Plus e as siliconas Optosil P Confort e Clonage fluida apresentaram os menores. Os resultados de deformação sob compressão foram maiores para o alginato Jeltrate Plus e para as siliconas Silon 2 APS massa e fluida. Os menores resultados foram apresentados pelo alginato Ezact Krom e as siliconas Reprosil A^+ massa e Xantopren VL Plus. Todos os materiais estão em conformidade com a norma ISO №1563, mas nem todos estão em relação à norma №4823.

Palavras-chave: Alginatos, Moldagem dentária, Microscopia eletrônica de varredura, Raios X – difração, Elastômeros na odontologia

ABSTRACT

Based on the incomplete understanding on how filler features influence the properties of elastomeric impression materials, the purpose of this study was to determine the inorganic filler fraction and size of five commercially available alginates (Jeltrate; Jeltrate Plus, Jeltrate Chromatic Ortho, Hydrogum e Ezact Krom) and nine addition/condensation silicones using the putty/light consistence (Clonage, Elite HD$^+$ Light Body, Express Light Body, Flexitime, Optosil P Confort/Xantopren VL Plus, Oranwash L, Reprosil A$^+$, Silon 2 APS e Virtual Extra Light Body). A SEM/EDX analysis was done to qualitatively characterize the materials. Soon afterwards elastic recovery and strain in compression of the alginates and some the silicones was carried. The inorganic particles volumetric fractions were accessed by weighing a previously determined mass of each material in water before and after burning samples for 3 hours at 450ºC (alginates) and 600ºC (silicones). Unsettled materials were soaked in acetone and chloroform and sputter-coated with gold for SEM evaluation of fillers' morphology and size. The filler composition was determined by EDX. Elastic recovery and strain in compression tests were conducted according to ISO specification #1563 and 4823. Jeltrate and Jeltrate Plus presented the highest mean values of percentage content of inorganic particles in volume, while Jeltrate Chromatic Ortho presented the lowest values. Flexitime Easy Putty was the silicone with the highest mean value, while Xantopren VL Plus had the lowest value. The alginate fillers presented a circular appearance with helical form and various perforations. Hydrogum fillers looked like cylindrical, perforated sticks. SEM pictures of the silicone inorganic particles showed numerous morphologies – lathe-cut, spherical, spherical-like, sticks, and sticks mixed to lathe-cut powder. Ezact Krom was the alginate with the highest values for diameter size, while Hydrogum had the lowest. Clonage Putty showed the highest values, while Elite HD$^+$ Light Body presented the lowest values. The component in higher concentration in the materials is silicon. The alginate Ezact Krom, and the addition cure silicones Reprosil A$^+$ putty and Flexitime

Correct Flow had the highest mean values of elastic recovery, while the alginate Jeltrate Plus and the condensation cured silicones Optosil P Confort and Clonage Putty presented the lowest values. Strain in compression test showed the alginate Jeltrate Plus and the condensation cured silicones Silon 2APS Putty and Fluid as the materials with the highest values. The alginate Ezact Krom and the silicones Reprosil A^+ Putty and Xantopren VL Plus had lowest values. All materials are in conformity with the requirements of ISO specification #1563, but not all materials are in conformity with the requirements of ISO specification #4823.

Keywords: Alginates, Dental impression materials, Microscopy, electron, scanning, X ray – Difraction, Elastomers in dentistry.

SUMÁRIO

.

INTRODUÇÃO

Materiais de moldagem são utilizados em Odontologia para registro dos arcos dentários nas fases de pré e pós-tratamento ortodôntico, para tratamentos reabilitadores utilizando próteses fixa e removível e na confecção de modelos de estudo em diversas outras áreas (Anusavice, 2005; Doubleday, 1998; Giordano, 2000; Kim *et al.*, 2001). A construção de modelos e troqueis é uma etapa importante para diversos procedimentos e vários tipos podem ser confeccionados em gesso a partir de uma cópia negativa da cavidade bucal (molde) (Anusavice, 2005). Para que se obtenha um modelo preciso, os materiais de moldagem devem obedecer alguns requisitos: ser fluido o suficiente para adaptarem-se aos tecidos bucais; possuir viscosidade suficiente para ficarem contidos em uma moldeira, enquanto que na cavidade bucal devem transformar-se em um sólido borrachóide em curto espaço de tempo; após a reação de presa, não devem distorcer ou rasgar quando removidos da boca; moldes feitos destes materiais devem manter-se dimensionalmente estáveis até que o modelo seja vazado; o molde deve manter sua estabilidade dimensional após a remoção do modelo, para permitir que segundos modelos possam ser construídos a partir da mesma moldagem; devem ser biocompatíveis com os tecidos bucais (Anusavice, 2005).

Esses materiais podem ser classificados de acordo com suas características após a presa em anelásticos e elásticos. Os primeiros tornam-se tão rígidos após a presa que se forem removidos de áreas retentivas podem fraturar ou distorcer-se (Anusavice, 2005). A segunda classe de materiais possui deformação elástica, o que lhes permitem ser esticados ou comprimidos ligeiramente sem sofrer deformação permanente. Nessa classe de materiais estão incluídos os mais utilizados pelos profissionais, sendo eles, os hidrocolóides irreversíveis (alginatos) e os elastômeros não aquosos (poliéteres, polissulfetos, silicones de condensação e de adição) (Anusavice, 2005; Doubleday, 1998; Giordano, 2000).

Os alginatos foram desenvolvidos durante o início da década de 30 como substitutos ao ágar (hidrocolóide reversível) devido à escassez deste último causada pela I Guerra Mundial (Anusavice, 2005; Giordano, 2000). Normalmente estes materiais apresentam-se na forma de um sistema de dois componentes – pó e água – e a reação de presa tem início apenas após a mistura de ambos (Anusavice, 2005; Doubleday, 1998; Frey *et al.*, 2005; Giordano, 2000). A presa do material baseia-se numa reação do tipo sol-gel em que um alginato solúvel (de sódio, de potássio ou trietanolamina) reage com um sal parcialmente solúvel de cálcio (sulfato de cálcio) em presença de água dando origem a um gel de alginato insolúvel de cálcio (Anusavice, 2005; Doubleday, 1998; Giordano, 2000). Partículas de carga são adicionadas para aumentar a rigidez do gel, produzir uma superfície com textura lisa e assegurar que o gel tenha consistência firme e não pegajosa (Anusavice, 2005). Os fatores responsáveis pelo sucesso do material são a facilidade de manipulação e remoção da cavidade bucal, não exigir equipamentos sofisticados para sua manipulação, baixo custo, conforto para o paciente e hidrofilia (Anusavice, 2005; Giordano, 2000).

Após a II Guerra Mundial, avanços na tecnologia dos polímeros, devido à escassez de borracha natural, criaram materiais à base de borracha sintética, denominados elastômeros. Os materiais odontológicos baseados nessas borrachas sintéticas são chamados de materiais de moldagem elastoméricos e constituem-se em moléculas (polímeros) que se unem umas às outras durante a presa, formando longas cadeias com ligações cruzadas entre si, em um processo conhecido como polimerização (Anusavice, 2005). De acordo com a composição química do polímero base e com a reação de polimerização, esses materiais podem ser classificados em polissulfeto, silicone polimerizado por condensação, silicone polimerizada por adição e poliéter (Anusavice, 2005; Doubleday, 1998; Giordano, 2000; Mandikos, 1998).

Os materiais à base de silicone são classificados em condensação e adição de acordo com o tipo de reação química (Anusavice, 2005; Doubleday, 1998;

Giordano, 2000). Os materiais de presa por condensação, também conhecidos como poli-dimetil-siloxanos, possuem como componente básico uma molécula com um radical α-ω-hidroxipolidimetil siloxano. A polimerização ocorre pela formação de ligações cruzadas entre o grupamento terminal do polímero de silicone e silicatos de alquila tri e tetra-funcionais na presença de octoato de estanho. Como se trata de uma reação de polimerização por condensação há a formação de subproduto que neste caso específico trata-se de álcool etílico (Anusavice, 2005; Doubleday, 1998; Giordano, 2000). Os materiais são fornecidos pelos fabricantes em forma de pasta base e pasta catalisadora. Na pasta base, tem-se a presença do polímero base e partículas de carga, enquanto na pasta catalisadora apresenta-se o silicato de alquila e o ativador à base de estanho (Anusavice, 2005; Doubleday, 1998; Giordano, 2000). Os silicones com presa por adição, também conhecidas como poli-vinil-siloxanos ou vinil-polissiloxanos, têm sua reação de presa baseada na reação entre grupamentos terminais vinílicos com grupamentos hidretos via catalisador à base de sal de platina (Anusavice, 2005; Doubleday, 1998; Giordano, 2000). Não há a formação de subprodutos desde que sejam observadas as proporções corretas. Entretanto, uma reação secundária entre umidade e hidretos residuais do polímero base pode levar à liberação de gás hidrogênio da massa de material (Anusavice, 2005), o qual não promove distorção do material com sua liberação. Também possuem apresentação comercial na forma de duas pastas (pasta base e catalisadora) com diferentes viscosidades. A pasta base contém poli-metil-hidrogênio siloxano e outros pré-polímeros siloxanos. A pasta catalisadora contém di-vinil-polidimetil siloxano, outros pré-polímeros siloxanos e sal de platina como ativador. Partículas de carga estão presentes em ambas as pastas (Anusavice, 2005; Doubleday, 1998; Giordano, 2000).

Independente da composição, certas propriedades físicas são necessárias para que os materiais desempenhem suas funções clínicas com sucesso. Algumas destas propriedades são: recuperação elástica, deformação sob compressão, resistência à compressão, tixotropia, compatibilidade com o gesso odontológico, rugosidade de superfície, resistência ao rasgamento, estabilidade dimensional e hidrofilia, entre outras (Donovan and Chee, 2004; Frey *et al.*, 2005; Murata *et al.*, 2004). Está normalmente convencionado que há uma correlação entre a quantidade de partículas de carga presente nesses materiais de moldagem e algumas propriedades mecânicas (Chen *et al.*, 2004; Corso *et al.*, 1998; Craig and Sun, 1994; Lu *et al.*, 2004; Mandikos, 1998). Segundo Anusavice (2005) e Doubleday (1998) a quantidade de partículas de carga determina a viscosidade. Craig e Sun (1994) demonstraram que há uma inter-relação entre deformação sob compressão e a consistência dos materiais e que esta é diminuída à medida que se diminui a viscosidade de massa para fluida. Lu *et al.* (2004) apresentaram resultados que mostram diferenças nos valores de propriedades mecânicas correlacionadas à viscosidade dos materiais. Chen *et al.* (2004) demonstraram que o aumento na quantidade de partículas de carga inorgânica de dois elastômeros experimentais melhorou a estabilidade dimensional dos materiais. Pôde-se observar, com base no levantamento bibliográfico, que não há estudos que avaliem as propriedades mecânicas dos materiais de moldagem com base nos valores de quantidade de partículas de carga, mas considerando apenas suas viscosidades.

Dessa forma, o objetivo deste trabalho foi avaliar a quantidade de partículas de carga de marcas comerciais de materiais de moldagem e determinar o seu efeito nas propriedades mecânicas dos mesmos. A presente tese foi dividida em quatro artigos que estão contemplados nos capítulos 1, 2, 3 e 4, cujos objetivos foram:

1) O primeiro estudo determinou a fração volumétrica e o tamanho médio das partículas de carga de silicones polimerizados por adição e/ou condensação. Além disso, essas partículas foram qualificadas através de análise em microscopia eletrônica de varredura (M.E.V.) e energia dispersiva de raios-X (E.D.X.).

2) No segundo estudo, foi determinada a fração volumétrica e o tamanho médio das partículas de carga de alginatos. Além disso, as partículas foram qualificadas através de análise em microscopia eletrônica de varredura (M.E.V.) e energia dispersiva de raios-X (E.D.X.).
3) O terceiro estudo avaliou os efeitos do conteúdo das partículas de carga sobre a recuperação elástica e deformação sob compressão de silicones polimerizados por adição e/ou condensação.
4) O quarto estudo avaliou os efeitos do conteúdo das partículas de carga sobre a recuperação elástica e deformação sob compressão de alginatos.

CAPÍTULO 1

Inorganic particle characterization of addition and condensation silicone impression materials.

Abstract

Based on the incomplete understanding on how inorganic particles features influence the properties of elastomeric impression materials, the purpose of this study was to determine the inorganic particle fraction and size of commercially available silicones. A SEM/EDX analysis was done to qualitatively characterize the materials. The inorganic particles volumetric fractions of five silicone rubbers were accessed by weighing a previously determined mass of each material in water before and after burning samples at 600ºC for 3 hours. Unsettled materials were soaked in acetone and chloroform and sputter-coated with gold for SEM evaluation of the morphology of the particles and size. The particle composition was determined by EDX. SEM pictures of the inorganic particles showed numerous morphologies – lathe-cut, spherical, spherical-like, sticks, and sticks mixed to lathe-cut powder. Flexitime Easy Putty is the material with the highest results for volumetric particle fraction, while Xantopren VL Plus had the lowest values. Silon 2 APS Fluid presented the lowest mean values of filler size, while Clonage Putty had the highest values. All materials presented high levels of silicon regardeless of the morphology.

Keywords: Elastomeric impression materials, Percentage of fillers, Filler morphology, SEM, EDX.

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). 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 in 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 that were developed during World War II due to the difficulty to obtain natural rubber. The elastomeric impression material consists of molecules (polymers) that are joined to each other by cross-linking in a process known as polymerization (1). According to the polymer composition, there are three kinds of dental elastomers: polysulfide, polyether and cured silicones (1-4).

Silicone materials are classified as condensation or addition depending upon the reaction producing the 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 cross-linking between the terminal hydroxyl groups and the alkyl which produces alcohol as a by-product. As alcohol is produced in the reaction, the set material distorts as it is released (1, 3, 4). Addition cured silicones are also known as polyvinylsiloxanes (PVS) or vinyl polysiloxanes. They are also two components and the setting occurs by cross-linking of vynil groups in the base material with a hydride group in the catalyst paste via a platinum catalyst and there are no reaction by-products (1, 3, 4). 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 particles presence is important on the strength of both materials and determines the viscosity (1, 3). Craig and Sun (6) 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.* (7) 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 important to be considered too, as the smaller particles tend to aggregate among each other and the larger ones do not contribute to reinforcement (1).

It was not found in literature, a study comparing silicone impression material's inorganic fraction and their mechanical and physical properties, just considering the viscosities. Considering the importance performed by the filler in the composition of the impression materials, the purpose of this study was to determine filler fraction and size of commercial condensation and addition silicones. Moreover, fillers were qualitatively analyzed by Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX)

Materials and Methods

The particles volumetric fraction, morphology, size and composition of five commercial brands (Table 1) of condensation and addition silicones were analyzed and described as follows.

Volumetric particle fraction

The percentage of inorganic particle by volume was determined by calculating the difference between the mass of each material tested in air and in water (Archimedes' Principle).(8)

Materials were manipulated according to the manufacturer's instructions and placed in an aluminum mold. Five cylindrical specimens (12-mm diameter, 20-mm high) of each material were weighed in an analytical balance (JK 180, Chyo Balance Corp., Tokyo, Japan), with an accuracy of 0.0001 g. The dried mass (Md) of the material after the setting time was determined in air. To determine the wet mass (Mi), a recipient and a stainless steel mesh were placed over the balance plate and filled with distilled water, and the specimen was immersed. The volume of the specimen after setting time was measured according to the following equation: Vs = Md – Mi. The specimens were then burned in an oven (Bravac Ltda, Sao Paulo, Brazil) to remove the organic phase, over 3 hours gradually increasing the temperature from room temperature to 600°C. 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 according to the following equation: Vp = Mp – Mpi. The percentage of the inorganic phase by volume was calculated using the following equation:

Inorganic particle percentage = (Vs/Vp)100.

Particle morphology and size

The morphology of the particles was determined by SEM images (JSM – 5600, JEOL Ltd. Tokyo, Japan). Unsettled amounts of each material (0.5 g) were submitted to the washing technique (9). This way, the polymeric matrix was removed by dissolving each material in 5 ml of acetone and centrifuging for 2 minutes at 1000 rpm. This process was repeated three times. The remaining material mass was next placed three times in chloroform and centrifuged as described above for a further washing and elimination of the matrix. The particles were then smeared in aluminum stubs, gold-sputter coated with gold/palladium in high vacuum (SCD 050, Bal-tec AG, Liechtenstein), and examined in a Scanning Electron Microscope operating at 15 Kv and magnificance of 1200x.

SEM pictures were imported to the Image-Pro Plus 4.5 image analyzer software (Media Cybernetics Inc., Bethesda, MD) where the images were analyzed by the measurement tool. The size of the particles was determined in micrometers (µm).

Particle composition

The particle composition was determined using the powder obtained in the previous test by Energy Dispersive X-ray analysis. The particles were smeared in acrylic resin stubs, carbon coated (Denton Vacuum Desk II Sputtering, Denton Vacuum, Cherry Hill, NJ), and then observed in a SEM/EDX (JSM – 5600, JEOL Ltd. Tokyo, Japan/ Vantage 1.4, Noran Instruments, Tokyo, Japan). Analyses were performed at a work 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 seconds were performed.

Results

Volumetric particle fraction

The mean values of percentage content of inorganic particles in volume are listed in Table 2. Flexitime Easy Putty is the material with the highest mean value (73.68%), while Xantopren VL Plus is the material with the lowest mean value (23.45%). 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 (medium or light bodied consistency). This is evidenced 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$^+$ Regular (32.66% and 29.04%), and Silon 2 APS Putty and Silon 2 APS Fluid (48.25% and 35.73%).

Particle morphology and size

The morphology of the particles is shown in the SEM images represented as Figures 1-10. The materials particles had the appearance of lathe-cut powder, spherical objects, spherical-like objects, and sticks that 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 a week in acetone and chloroform. Also, it is still possible to see the presence of remaining portions of the polymeric matrix adjacent to the spherical fillers of Clonage Putty.

The maximum, minimum and mean length (for lathe-cut particle shape) or diameter (for spherical shape) size values of the inorganic particles are listed in Table 3. Clonage Putty showed the highest mean value (27.75 µm), while Silon 2APS presented the lowest mean value (4.60 µm). Because of the difference of Reprosil A$^+$ Putty and Reprosil A$^+$ Regular particle shape that showed a considerable length to be measured, Table 3 presents their maximum, minimum and mean length values beyond the values for diameter. It was not possible to measure the filler size of Xantopren VL Plus because of the difficulty to eliminate the polymeric matrix.

Particle composition

Results for particle composition by EDX analysis are listed in Table 4. The component in higher concentration in the materials is silicon (Si). In general the composition was the same for all of the materials with the presence of Si in concentration of 100%. Clonage Putty presented Zinc (Zn), Si, Calcium (Ca), and

Indium (I). Optosil P Confort presented Zn, Magnesium (MG), Si, and Sodium (Na). Reprosil A^+ presented Zn, Germanium (Ge), Aluminum (Al), Si, Ca, and Antimony (Sb).

Discussion

Impression materials viscosity is determined by the amount of particles (2, 3, 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 similitude occurred with the materials 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 utilize plasticizers to control it. Another fact to be considered is that maybe the inorganic filler composition or polymeric matrix may influence on the flow of the material. Reprosil medium 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 material consistency difference (putty/fluid). It was not possible to determine if Optosil and Xantopren fillers differ between each other, but the impossibility to remove silicone rubber matrix from Xantopren may represent 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, 5-7, 10) as the consistency is decreased from putty to heavy, to medium or to light bodied materials (5, 6). Obviously, this is because less polymeric matrix is present in the composition of the materials (1). Chen *et al.* (2), Craig and Sun (6), Lu *et al.* (7), and Tjan and Li

(11), showed that material and consistency had significant influence on elastic recovery, permanent deformation, strain in compression, tear energy, tensile strength, thermal expansion, and dimensional stability. The light body consistency materials had lower elastic recovery than the heavy body materials (7). Higher strain in compression values indicate more flexibility; the putty or heavy body materials were stiffer than the light body materials tested (6, 7). Besides, heavy body impression materials had higher tear resistance than light body consistency (7). The tensile strength was higher for the heavy body consistency materials than for the light body (7). The higher the viscosity of the impression material, the lower the coefficient of thermal expansion (11). Large dimensional change was observed for light consistency silicones compared to putty consistency illustrating that the proportion of the particles modify the accuracy of the materials (2, 6). Unfortunately, it is not expected from Reprosil and Optosil/Xantopren to present the correlations mentioned, unless plasticizers, particles composition, linkage between particles and matrix or matrix composition has the capacity to modify, somehow , the properties of these materials.

SEM pictures of the inorganic particles showed numerous morphologies – lathe-cut, spherical, spherical-like, sticks, and sticks mixed to lathe-cut powder. Commonly, 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 process of silicon (Si). The stick-like particles are cell walls of algae from the division *Chrysophyta*, class *Bacillariophycea*. Termed diatomaceous earth, or diatomite, this material is mined and used for a variety of commercial purposes because of its worldwide range (12).

The influence of the particle 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, therefore, 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% silicon composition and colloidal silica, which seems to be the main composition of silicone rubber materials (1). Materials presenting particles with other metals in composition – Zinc (Zn), Calcium (Ca), Indium (I), Magnesium (Mg), and Sodium (Na) – may be those which are originated from microsized metal oxides (1). Reprosil uses diatomite as filler particles. According to Bold and Wynne (12) the main composition of diatomite is silicon, 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 (13). The size and amount of particle may be related to silicone rubber accuracy. Xantopren VL Plus is the material with the lowest values for percentage content of inorganic particles in volume, therefore it is expected that it would be a material with nice results for detail reproduction although it is important to register its high hydrophobic matrix characteristic and because it was not possible to measure its volumetric filler fraction. Likewise, Silon 2APS Fluid could present good results for detail reproduction because of its lower mean values of filler size (4.60 µm). Flexitime Easy Putty and Clonage Putty are expected to present conflicting results because of their higher content of volumetric filler particle (73.68%) and mean values of filler size (27.75 µm) respectively.

Accurate casts and models are needful in a great number of dentistry procedures (1, 2), and the volumetric filler fraction is one of the factors affecting materials properties. This study determined the inorganic filler fraction and quality of some commercial brands of elastomeric rubber impression materials. It is now necessary to continue the research and 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

Conclusion

SEM pictures of the inorganic particles showed a wide range of morphologies. Flexitime Easy Putty is the material with the highest results for volumetric filler fraction, while Xantopren VL Plus had the lowest values. Silon 2APS Fluid presented the lowest mean values of filler size while Clonage Putty had the highest values. All materials presented high levels of silicon regardless of the morphology.

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Tables

Table 1 – Materials tested, manufacturer and batch number.

Material	Type	Manufacturer	Batch number
Clonage Putty	condensation silicone	DFL, Rio de Janeiro, Brazil	5111362
Clonage Fluid	Condensation silicone	DFL, Rio de Janeiro, Brazil	5111363
Flexitime Easy Putty	addition silicone	Heraeus Kulzer, Hanau, Germany	220402
Flexitime Correct Flow	addition silicone	Heraeus Kulzer, Hanau, Germany	220388
Optosil P Confort	Condensation silicone	Heraeus Kulzer, Hanau, Germany	230363
Xantopren VL Plus	Condensation silicone	Heraeus Kulzer, Hanau, Germany	210743
Reprosil A^+ Putty	addition silicone	Dentsply Latin America, Petrópolis, Brazil	377613
Reprosil A^+ Regular	addition silicone	Dentsply Latin America, Petrópolis, Brazil	378204
Silon 2 APS Putty	condensation silicone	Dentsply Latin America, Petrópolis, Brazil	1743-4
Silon 2 APS Fluid	condensation silicone	Dentsply Latin America, Petrópolis, Brazil	349629

Table 2 –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^+ Regular	29.04
Silon 2 APS Putty	48.25
Silon 2 APS Fluid	35.73

Table 3 –Maximum, minimum and mean values for materials particle size (μm)

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 – length	24.04	5.47	10.43
Reprosil A^+ Putty – diameter	16.78	4.94	8.16
Reprosil A^+ Regular – length	18.50	3.29	9.69
Reprosil A^+ Regular – diameter	12.73	5.59	8.42
Silon 2 APS Putty	22.24	9.64	15.13
Silon 2 APS Fluid	7.56	2.73	4.60

Table 4 – 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^+ Regular (diatomite)	Zn (2.34), Ge (1.2), Al (1.29), Si (88.5), Ca (1.58), Sb (5.21)
Reprosil A^+ Regular (lathe-cut filler)	Si (100)
Silon 2 APS Putty	Si (100)
Silon 2 APS Fluid	Si (100)

Figures

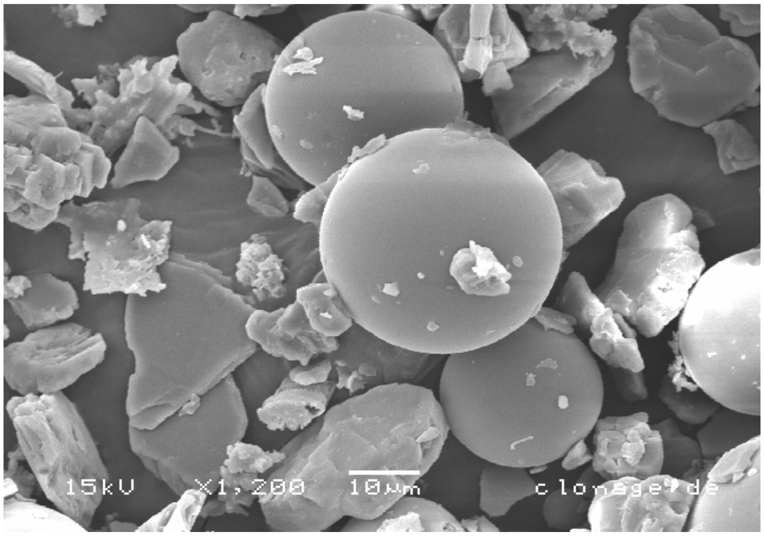

Figure 1 – SEM image of Clonage Putty presenting spherical particles (1200x).

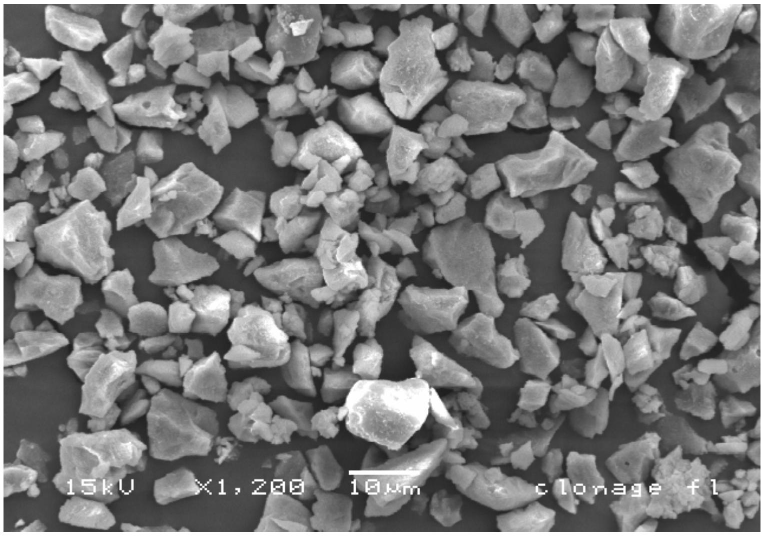

Figure 2 – SEM image of Clonage Fluid presenting lathe-cut particles (1200x).

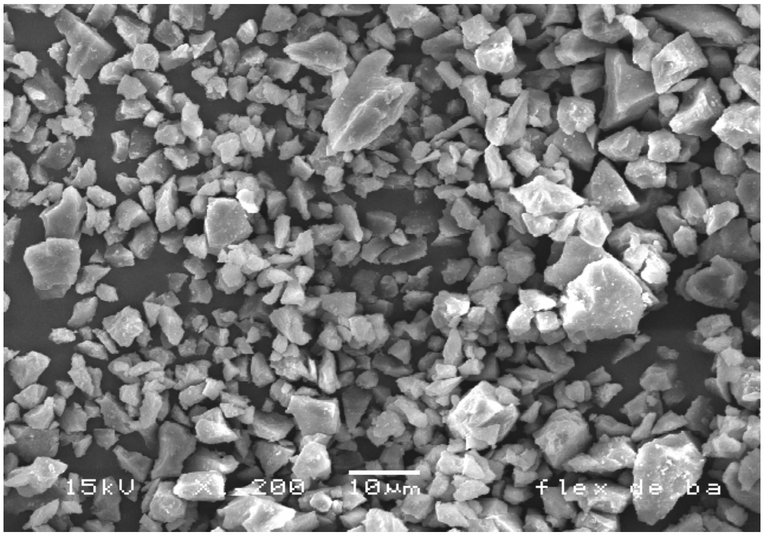

Figure 3 – SEM image of Flexitime Easy Putty presenting lathe-cut particles (1200x).

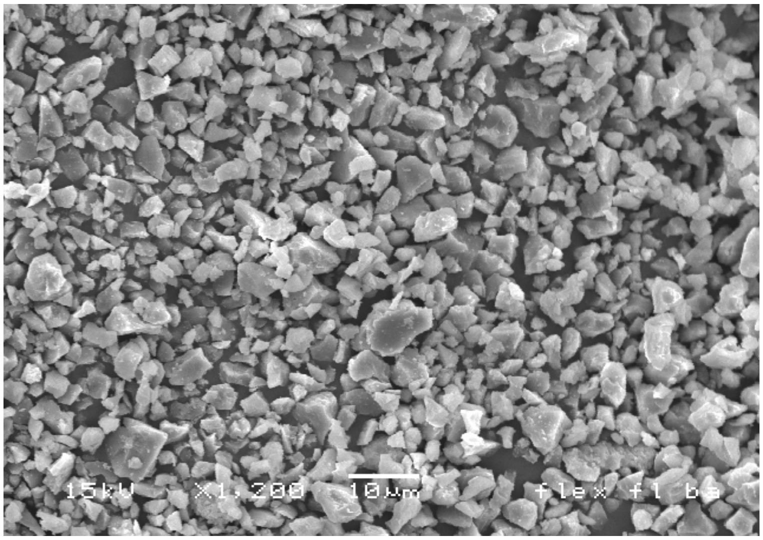

Figure 4 – SEM image of Flexitime Fluid presenting lathe-cut particles (1200x).

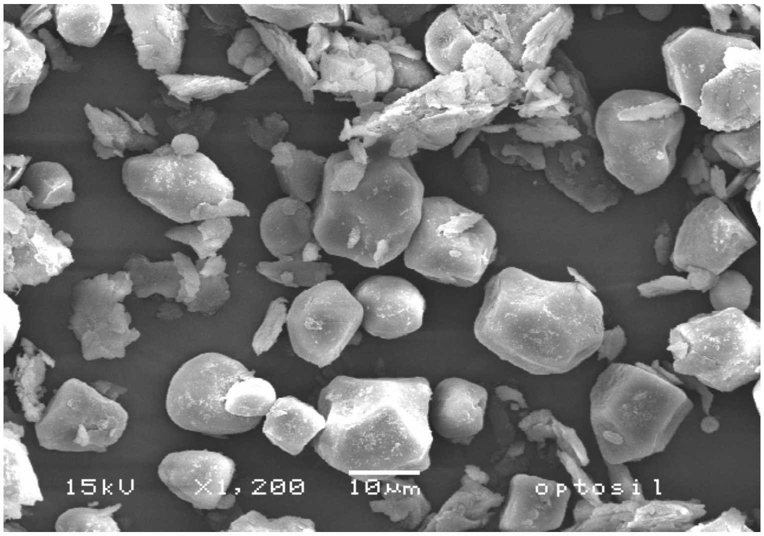

Figure 5 – SEM image of Optosil P Confort presenting spherical-like particles (1200x).

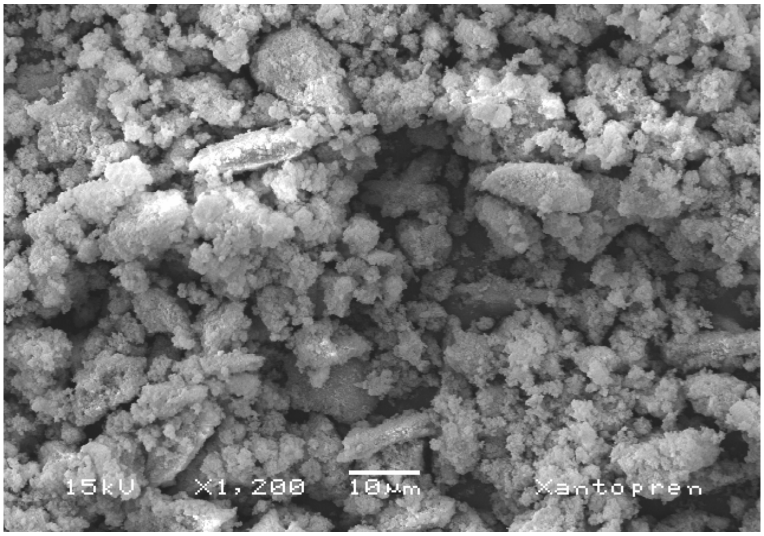

Figure 6 – SEM image of Xantopren. It was not possible to eliminate the polymeric matrix to visualize the fillers. (1200x).

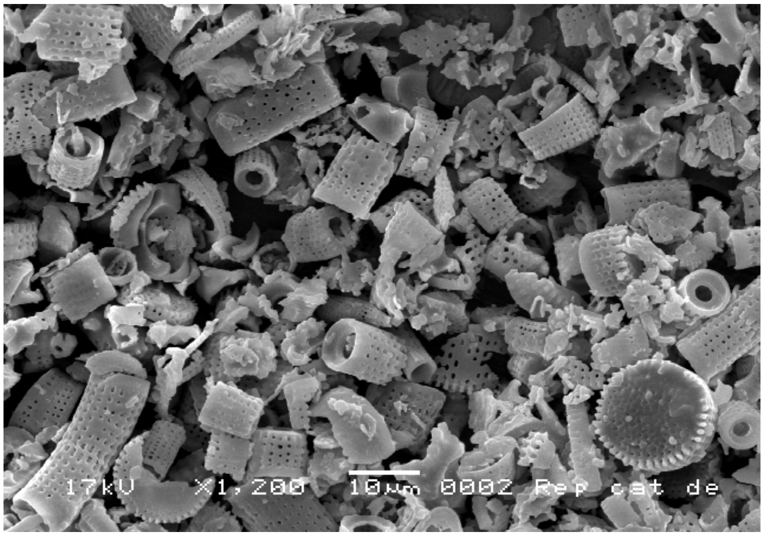

Figure 7 – SEM image of Reprosil A^+ Putty presenting stick particles (1200x).

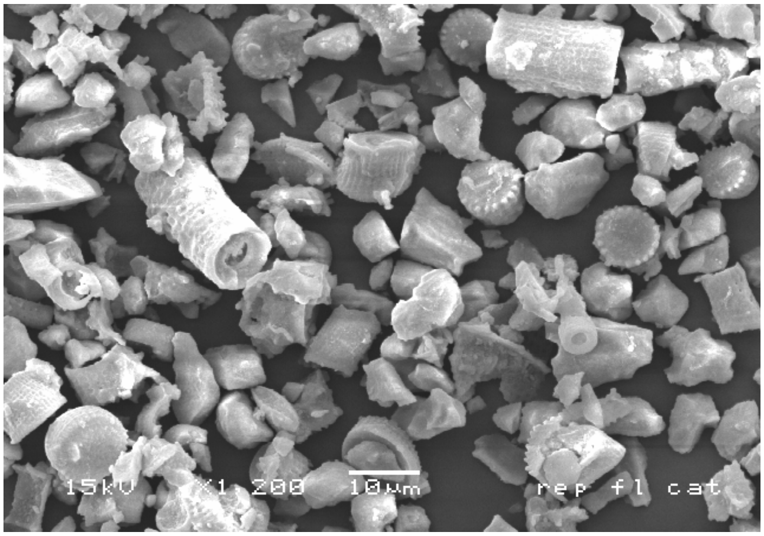

Figure 8 – SEM image of Reprosil A^+ Regular presenting stick particles mixed to lathe-cut (1200x).

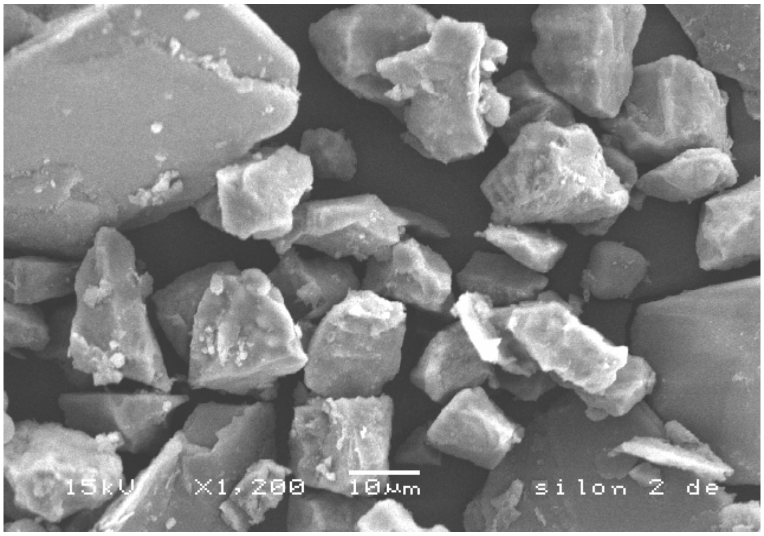

Figure 9 – SEM image of Silon 2APS Putty presenting lathe-cut particles (1200x).

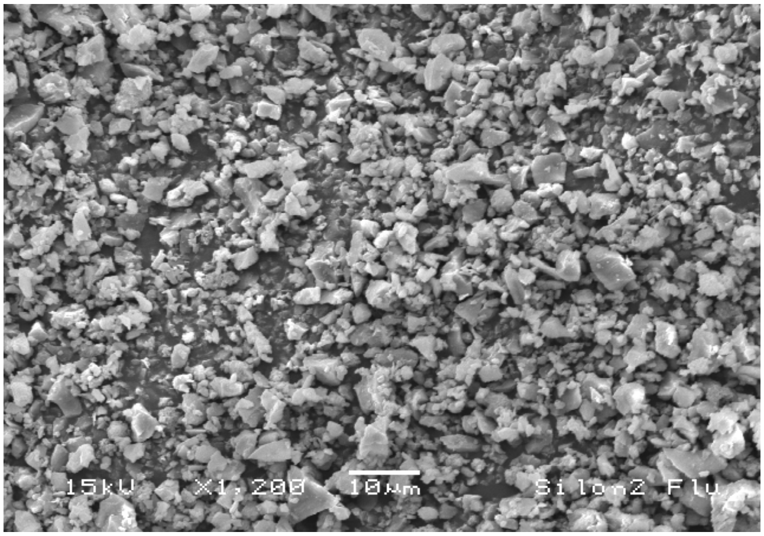

Figure 10 – SEM image of Silon 2APS Fluid presenting lathe-cut particles (1200x).

CAPÍTULO 2

Qualitative and quantitative analysis of dental alginates' inorganic fraction

(Trabalho submetido ao Journal of Prosthodontics)

Abstract

Purpose: The aim of this study was to determine the inorganic filler fraction and size of commercial available alginates. A SEM/EDX analysis was done to qualitatively characterize the materials.

Materials and Methods: The inorganic particles volumetric fractions of five alginates – Jeltrate (J), Jeltrate Plus (JP), Jeltrate Chromatic Ortho (JC), Hydrogum (H) and Ezact Krom (E) was accessed by weighing a previous determined mass of each material in water before and after burning samples at 450ºC, during three hours. Unsettled materials were soaked in acetone and chloroform and sputter-coated with gold for SEM evaluation of fillers' morphology and size. The filler composition was determined by (EDX).

Results: The results for the volumetric inorganic particle content were (%): J – 48.33, JP – 48.33, JC – 33.79, H – 37.55 and E – 40.55. The fillers presented a circular appearance with helical form and various perforations. Hydrogum fillers looked like cylindrical perforated sticks. The mean values for the alginates filler size were (µm): J – 12.91, JP – 13.67, JC – 13.44, E – 14.59 and H – 9 (diameter), 8.81 (length). Composition was similar for all materials with the presence of Zn, Ge, Br, Ca and Sb. Hydrogum was the unique material to present Al in the composition.

Conclusions: Jeltrate Chromatic Ortho is the material with the lowest results for volumetric filler fraction while Jeltrate and Jeltrate Plus had the highest values. All materials presented high levels of silicon on the diatomite filler composition. Hydrogum had the lowest mean particle diameter and Ezact Krom the highest.

INDEX WORDS: Alginate, Percentage of fillers, Filler morphology, SEM, EDX.

Introduction

Impression materials are used in dentistry for pre-treatment planning, fabrication of fixed or removable prostheses and pos-treatment records.[1, 2] These materials can be allocated in two families: elastic and inelastic ones. Elastic impression materials returns to their original configuration after being removed from undercuts in the mouth due to elastic recovery.[2, 3] Originally developed in the 1930s, alginate (irreversible hydrocolloid) is classified as an irreversible hydrocolloid material, having a widely use in dentistry due to easy manipulation, fairly comfortable to the patient, inexpensive, and hydrophilic (contact angle 37º).[1, 3-5]

Alginates are commonly used as a two-component system – powder and water. The reaction does not start until the dry powder is mixed with water.[1, 3] It is suggested to tumble the alginate powder in a closed container before use to establish uniform distribution of the material's ingredients. During this process an aerosol is formed by airborne particles that could be hazardous to dental professionals causing pleural and peritoneal malignances.[6, 7]

Irrespective of composition, impression materials physical properties are required to warrant clinical success. Elastic recovery, strain in compression, compressive strength, thixotrophy, compatibility with dental stones, surface roughness, tear energy, dimensional stability and hydrophilicity are some of these important properties.[5, 8, 9] Nonetheless, alginate has some disadvantages as it poor reproduces surface details, it is not dimensionally stable on storage and it is usually best poured immediately.[1-3]

Lim *et al.*[10] and Beaty *et al.*[11] showed that filler fraction affects composite resins wear, uniaxial tensile strength, Young's modulus in slow compression, Knoop hardness, water sorption, and toothbrush abrasion resistance. Similar to composite resins the filler fraction may also have an effect on alginate properties. Although Giordano,[2] Anusavice[3] and Murata *et al*[9]. did not make a correlation between inorganic particles and alginate properties, the authors state that fillers size and content are closely related to accuracy and compressive strength results.

Therefore, the aim of this study was to investigate the filler fraction and size of commercial alginates. Moreover, fillers were qualitative analyzed by Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX).

Materials and Methods

The filler volumetric fraction, morphology, size and composition of five commercial brands of alginate were analyzed (Table 1) as described below.

Volumetric filler fraction

The percentage of inorganic particle by volume was determined calculating the difference between the mass of each material tested in air and in water (Archimedes' Principle).[12]

Materials were manipulated according to the manufacturer's instructions and placed in an aluminum matrix. Five cylindrical specimens of each material with 12mm in diameter and 20mm in height were weighed in an analytical balance (JK 180, Chyo Balance Corp., Tokyo, Japan), with an accuracy of 0.0001g. The dried mass of the material after the setting time was determined (Md) in air. In order to determine the wet mass a recipient and a stainless steel mesh were placed over the balance plate and filled with distilled water were the specimen was immersed (Mi). The volume of the specimen after setting time was measured according to the following equation: Vs = Md – Mi. The specimens were then burned to remove the organic phase in an oven (Bravac Ltda, Sao Paulo, Brazil), gradually increasing the temperature from room temperature to 450°C, during of three hours. The resulting inorganic material was intact and pill shaped. The mass in air was then measured as described above (Mp). To determine the wet mass of the particles (Mpi) the specimens were immersed in distilled water as described above and at this time the pill shape was disarranged because of the contact with water. The volume of the inorganic particles was measured according to the following equation: Vp = Mp – Mpi. The percentage of the inorganic phase by volume was calculated using the following equation: inorganic particle percentage = (Vs/Vp)100.

Filler morphology and size

The morphology of the fillers was determined by SEM images (JSM – 5600, JEOL Ltd. Tokyo, Japan). Unsettled amounts of each material (0.5g) was submitted to the washing technique.[13] The matrix was removed by dissolving each material in 5ml of acetone and centrifuging for 2 min at 1000rpm. This process was repeated three times. The remaining material mass was next placed three times in chloroform and centrifuged as described above for a further washing and elimination of the matrix. The fillers were then smeared in aluminum stubs, gold-sputter coated with gold/palladium in high vacuum (SCD 050, Bal-tec AG, Liechtenstein) and examined in a Scanning Electron Microscope operating at 15Kv.

SEM pictures were imported to the Image-Pro Plus 4.5 image analyzer software (Media Cybernetics Inc., Bethesda, USA) were the images were analyzed by the measurement tool. Fillers' size was determined in micrometers (µm).

Filler composition

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

Results

Volumetric filler fraction

The mean values of percentage content of inorganic particles in volume are listed in table 2. Jeltrate and Jeltrate Plus presented the highest mean values (48.33%), while Jeltrate Chromatic Ortho presented the lowest ones (33.79%). Ezact Krom and Hydrogum showed 40.55% and 37.55% respectively.

Filler morphology and size

The morphology of the fillers is showed by the SEM images in Figures 1-5. The inorganic particles of the materials presented several shapes and sizes. In general, materials' particles had the same appearance, with a circular structure and a helical form with various perforations. The unique exception was Hidrogum that looked like cylindrical perforated sticks.

The maximum, minimum and mean diameter size values of the inorganic particles are listed in Table 3. Ezact Krom showed the highest values for diameter size. Because of the difference of Hydrogum particle shape that had a considerable length to be measured, Table 3 presents its maximum, minimum and mean length values beyond the values for diameter.

Filler composition

Results for filler composition by EDX analysis are listed in Table 4. The component in higher concentration in the materials is silicon (Si). In general the composition was the same for all materials with the presence of Zinc (Zn), Germanium (Ge), Bromine (Br), Calcium (Ca) and Antimony (Sb). Hydrogum was the unique material to present Aluminum (Al).

Discussion

The objective of the present study was to analyze qualitatively and quantitavely the inorganic particle content of some commercial brands of alginates. The findings of this investigation showed Jeltrate Chromatic Ortho as the material with the lowest results for volumetric filler fraction (33.79%) while Jeltrate and Jeltrate Plus had the highest values (48.33%). Thus, it is expected that the increase of alginate on Jeltrate Chromatic Ortho to cause an alteration on stability due to the fact that gels are invariably subject to changes in dimension by syneresis, evaporation and imbibition of water.[3]

It was not found in literature, a study comparing dental alginates' inorganic fraction and their mechanical and physical properties. Differences among materials are not directly related to fillers content [7, 14] but it seems to be very important to be considered.[2, 3, 9]

The inorganic particles observed by SEM pictures are cell walls of algae from the division Chrysophyta, class Bacillariophycea. The members of this class, referred to as diatoms, are essentially unicellular, although chains of cells and colonial aggregations may occur.[15] There are records of these algae dating from the Cretaceous. The classification of the diatoms is almost entirely based on the structure and ornamentation of the cell wall, which is termed the frustules.[15] Diatom frustules are resistant to natural degradation, and their accumulation over geologic periods has resulted in significant deposits. Termed diatomaceous earth, or diatomite, this material is mined and used for a variety of commercial purposes because of its worldwide range.[15]

There are no special interaction known between fillers and gel fibrils demanding the use of diatomite as alginates inorganic fractions and it was quite easy to separate them by the washing technique.[13] Possibly diatomite was chosen as inorganic fraction because of its low cost.[15] When added in proper amounts, it can optimize the strength and stiffness of the alginate gel, produce a smooth texture and ensure a firm gel surface that is not tacky. It also aids in forming the sol dispersing the alginate powder particles in water. Without the filler, the gel formed lacks firmness and exhibit a sticky surface covered with an exudate produced by synerisis.[3]

All materials presented high levels of silicon on their composition, being the predominant component. As described by Bold and Wyne,[15] the main composition of the diatomite is silicon and it has been shown to be an absolute requirement for mitosis and frustule formation. The composition of the materials was similar varying the percentage among the components with the exception of Hydrogum that presented an extra component - aluminum. The presence of aluminum just in Hydrogum and the small differences presented by the materials in the percentage content of each component may be related to the different origin of the diatomite.

The size and amount of filler and the gel fibrils are related to the alginate accuracy.[2, 3] This way, it is expected from Hydrogum to be the material with the best results for detail reproduction because of its lowest mean diameter (9μm) and low results for volumetric filler fraction, while Jeltrate and Jeltrate Plus are expected to present conflicting results. There is difference in the morphology of Hydrogum and the other materials. It seems that the diatomite in its composition is a colonial aggregation belonging to a different order, suborder or genera from the other materials.

Inhalation of aerosols arising from alginates is potential hazardous to dentists and their assistants during a long period.[6] The degree of malignance is related to the size of the fibers rather to their composition.[16] Particles with less than

3µm in diameter and more than 20µm in length present the greatest hazard.[17] Woody et al.[6] monitored and characterized aerosols particles from two alginates and showed that 10% to 15% percent of the particles had dimensions less than 3µm in diameter and more than 20µm in length. The fillers observed in the present work vary from 34.92µm to 13.26µm in diameter and from 15.25µm to 5.42µm in length. Apparently, these particles would not be an etiologic factor in fibroses of the lungs. Additionally, the manufactures attempt to produce “dust-free” alginates by incorporating additives (glycols) and suppressing the aerosol formation.[7]

The findings of this investigation lead to questions about results on the inorganic filler fraction of dental alginates correlation to their mechanical properties. It is now necessary to continue the research, answer the assumed questions and make a suitable correlation between fillers and results for elastic recovery, strain in compression, dimensional stability, radiodensity, detail reproduction, and others.

Conclusion

Jeltrate Chromatic Ortho is the material with the lowest results for volumetric filler fraction while Jeltrate and Jeltrate Plus had the highest values. All materials presented high levels of silicon on the diatomite filler composition. Hydrogum had the lowest mean particle diameter and Ezact Krom the highest.

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Tables

Table 1 – Materials tested, manufacturer and batch number

Material	Manufacturer	Batch number
Jeltrate	Dentsply Latin America, Petropolis, Brazil	156999
Jeltrate Plus	Dentsply Latin America, Petropolis, Brazil	288721
Jeltrate Chromatic Ortho	Dentsply Latin America, Petropolis, Brazil	142603
Hydrogum	Zhermack, Rovigo, Italy	21834
Ezact Krom	Vigodent, Rio de Janeiro, Brazil	078/05

Table 2 –Mean values for alginates volumetric filler fraction (%)

Material	**Volumetric filler fraction**
Jeltrate	48.33
Jeltrate Plus	48.33
Jeltrate Chromatic Ortho	33.79
Hydrogum	37.55
Ezact Krom	40.55

Table 3 –Maximum, minimum and mean values for alginates filler size (µm)

Material	Maximum	Minimum	Mean
Jeltrate	20.93	7.86	12.91
Jeltrate Plus	17.25	10.73	13.67
Jeltrate Chromatic Ortho	21.60	7.28	13.44
Hydrogum (diameter)	13.26	5.19	9.00
Hydrogum (length)	15.25	5.42	8.81
Ezact Krom	34.92	8.05	14.59

Table 4 – Composition of the materials by EDX analysis

Material	**Composition (%)**
Jeltrate	Zn (1.94), Ge (1.89), Br (1.19), Si (91.21), Ca (1.55), Sb (2.22)
Jeltrate Plus	Zn (1.65), Ge (3.16), Br (1.63), Si (85.53), Ca (3.84), Sb (4.26)
Jeltrate Chromatic Ortho	Zn (3.19), Ge (3.16), Br (1.63), Si (86.55), Ca (3.95), Sb (3.15)
Hydrogum	Zn (2.33), Ge (0.98), Al (1.15), Si (89.9), Ca (1.69), Sb (4.76)
Ezact Krom	Zn (4.75), Br (2.2), Si (92.09), Ca (0.76), Sb (0.2)

Figures

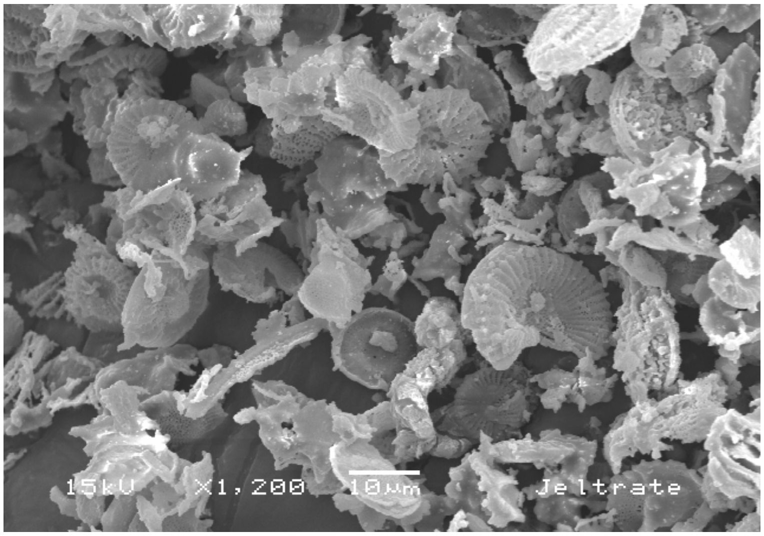

Figure 1 – Typical scanning electron photomicrograph of Jeltrate inorganic fraction (x1200).

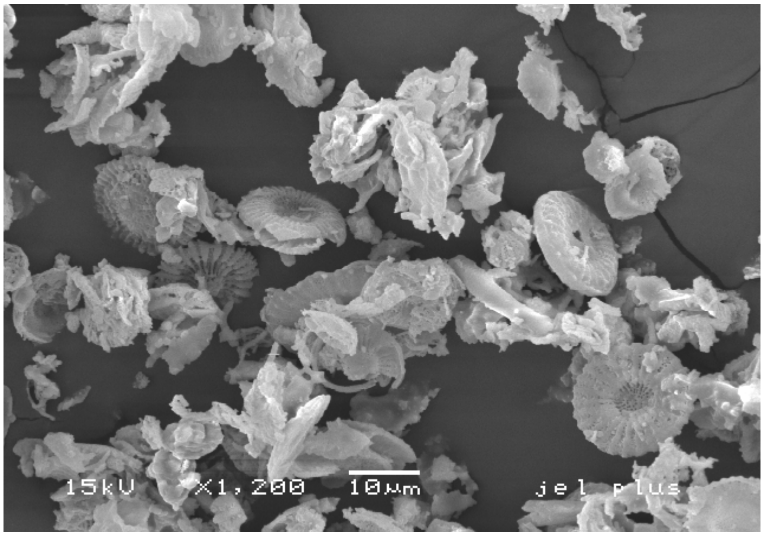

Figure 2 – Typical scanning electron photomicrograph of Jeltrate Plus inorganic fraction (x1200).

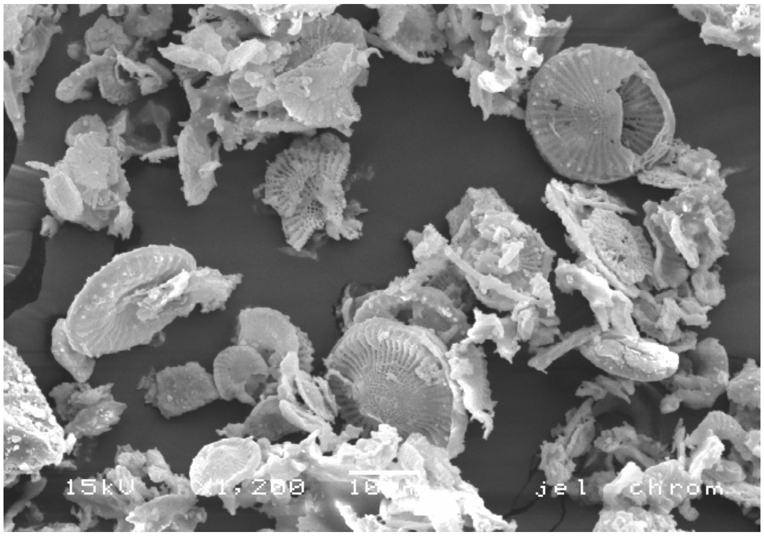

Figure 3 – Typical scanning electron photomicrograph of Jeltrate Chromatic Ortho inorganic fraction (x1200).

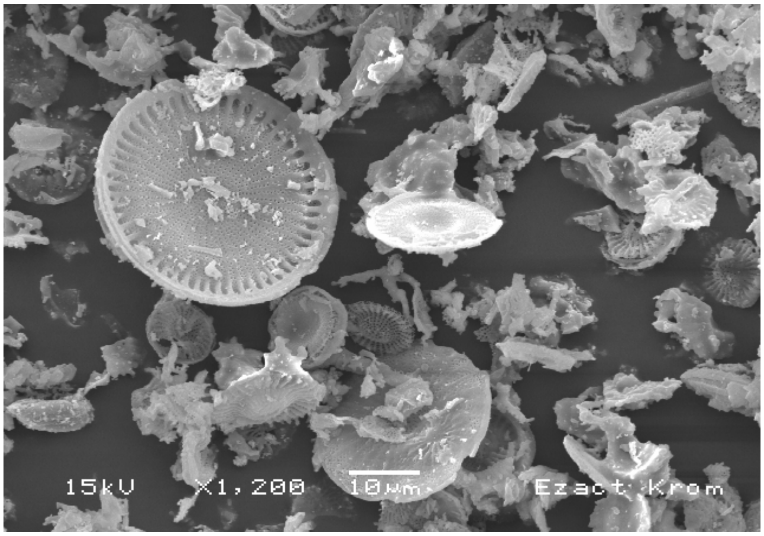

Figure 4 – Typical scanning electron photomicrograph of Ezact Krom inorganic fraction (x1200).

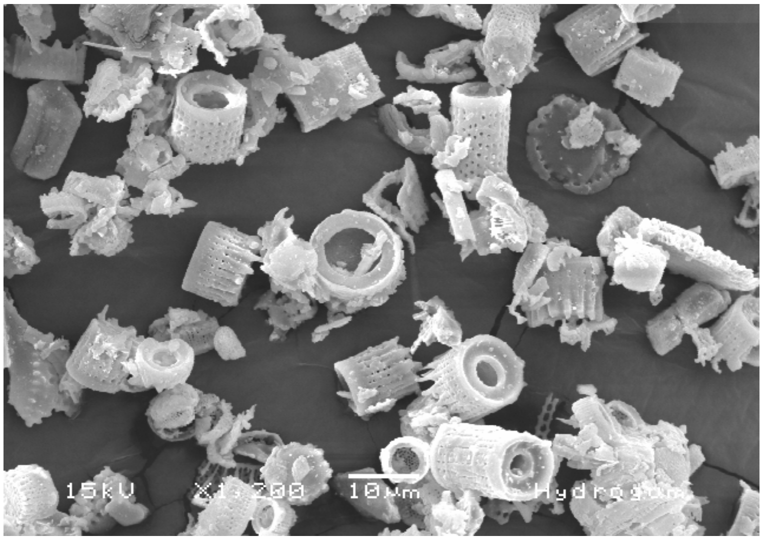

Figure 5 – Typical scanning electron photomicrograph of Hydrogum inorganic fraction (x1200).

CAPÍTULO 3

Mechanical properties of silicone impression materials by volumetric filler content

Abstract

The objective of this study was to evaluate the effects of silicone impression materials filler fraction on the properties of elastic recovery and strain in compression. Five silicones – Reprosil A^{+} (R), Flexitime (F), Clonage (C), Silon 2 APS (S), and Optosil/Xantopren (O/X) with their putty (P)/light (L) consistency were used to measure elastic recovery (K) and strain in compression (E). The tests were conducted according to ISO specification #4823. The specimens (n=5) were prepared by mixing the materials according to the manufacturer's instructions. Values for filler fraction were used from a previous study. Data was submitted to 2-way ANOVA and Tukey's test. Results of elastic recovery (%) were: R – 99.78^{A} (P), F – 99.52^{AB} (P), C – 99.31^{B} (P), S – 97.89^{C} (P), and O – 97.81^{C} (P); R – 99.75^{A} (L), F – 99.8^{A} (L), X – 99.68^{A} (L), S – 99.64^{A} (L), and C – 99.50^{A} (L). The results of strain in compression test (%) were: S – 2.14^{A} (P), F – 2.08^{A} (P), O – 1.73^{A} (P), C – 1.59^{A} (P), and R – 1.19^{A} (P); S – 21.12^{A} (L), C – 10.93^{B} (L), R – 8.34^{C} (L), X – 6.00^{D} (L), and F – 5.98^{D} (L). Not all materials are in conformity with the requirements of ISO specification #4823. The light body consistency materials had higher elastic recovery and strain in compression than the heavy body materials. The addition cured silicones showed the highest values of elastic recovery.

KEYWORDS: Elastomeric impression materials, Percentage of fillers, Elastic Recovery, Strain in compression.

Introduction

Making impressions to register or duplicate the form and relations of the tooth and the surrounding oral tissues is an integral part of fixed prosthodontic, operative dentistry, removable prosthodontic, and implant dentistry treatment [1-5]. Important requirements of impression materials are ease of manipulation, compatibility of cast materials, and precise replication of oral tissue due to dimensional stability, hydrophilicity, high tear strength, low setting shrinkage, and good elastic recovery, [1, 2, 6].

Today there are many kinds of dental impression materials and they are classified into elastic and inelastic, and aqueous and nonaqueous [1, 2]. The rubberlike impression materials, also known as elastomers, are nonaqueous elastic impression materials and according to the polymer composition may be classified as condensation silicone, addition silicone, polysulfide and polyether [1-3, 6, 7].

Polydimethyl siloxane (condensation silicone) and polyvinylsiloxane (addition silicone) are two component systems (base and catalyst paste) based on the siloxane polymer, however the presence of differing terminal groups accounts for their different classification based on the reaction producing the polymerization [2, 4, 6, 7].

It is usually accepted that there is a correlation between some properties of the elastomeric impression materials and filler fraction [3, 4, 8-10] but, Carlo *et al.* [11] analyzing the volumetric filler fraction of various commercially available elastomers verified that this correlation may not always be maintained. The authors [11] supposed that not only the percentage of fillers between high and low viscosity materials seem to determine, effectively, the properties of the materials.

The objective of this study was to evaluate the effects of silicone impression materials filler fraction on the properties of elastic recovery and strain in compression. The hypothesis tested was that there were differences in properties among the materials not only related to volumetric filler content.

Materials and Methods

The silicone impression materials tested in this study are listed in Table 1. The materials were prepared by hand-mixing using the ratio and time specified in the manufacturer's instructions.

Elastic recovery (K) and strain in compression (E) were tested according to ISO #4823 [12]. For measurement of recovery from deformation (K), the specimens (12.5 mm diameter x 20 mm high) (n=5) were deformed by 30% in length within 1 s and maintained this deformation for 5 s in a universal testing machine (Instron 4411, Instron Inc. Canton, MA. USA). Two minutes after releasing the load the change in length (ΔL) was measured. K (%) was calculated as 100 x (L – ΔL)/L, while L is the original length (20 mm).

The strain in compression test (E) was carried out on a compression apparatus described In ISO international standard [12]. The specimens (n=5) were prepared with the same specifications of the previous test and subjected to a load of 1.22 N for 30 s and the change in length (h_1) was measured. The load was removed and the specimen subjected to a load of 12.25 N gradually, over a period of 10 s. The load was maintained during 30 s and the change in length (h_2) measured. E (%) was calculated as 100 x ($h_1 – h_2$)/h_0, where h_0 is the original length (20 mm).

Data of recovery from deformation and strain in compression presented a normal and homogeneous distribution and two-way ANOVA following Tukey's test ($\alpha = 0.05$) were employed on the statistical analysis.

Results

The 2-way ANOVA test (Table 2) showed that there were significant differences among materials, viscosity, and the interaction material/viscosity with respect to recovery from deformation and strain in compression.

Table 3 show means, statistical categories, and standard deviations defined by Tukey's test for elastic recovery of the materials. For all putty materials tested, K was greater than 97.81%. Optosil presented the lowest data (97.81%), being statistically the same as Silon (97.89%). Reprosil presented the highest values of K (99.78%), being statistically the same as Flexitime (99.52%). Clonage presented intermediate value (99.31%). For the fluid materials Flexitime presented the highest values o K (99.78%), but there is no statistically significant difference from the other materials.

Data from strain in compression of the materials tested are showed in Table 4. The results of the putty materials presented Silon with the highest values of E (2.14%), but there is no statistically significant difference from the other materials. For fluid materials Silon showed the highest data of E (21.12%), being statistically different from other materials. Flexitime and Xantopren presented the lowest values of E (5.98% and 6.0%, respectively). Clonage (10.93%) and Reprosil (8.34) presented intermediate values.

Tables 3 and 4 show statistical analysis of elastic recovery and strain in compression of the materials considering the relation between putty and light consistency. Strain in compression were statistically different between putty and fluid consistency for all materials. Elastic recovery were statistically different between putty and fluid consistency of Silon 2 APS and Optosil/Xantopren.

Discussion

The ideal impression material should exhibit maximum energy absorption with minimal distortion to maintain the integrity of the impression and to warrant clinical success [2, 4, 10]. Elasticity is inherent to all of the elastomeric impression materials as they are polymers with highly flexible kinked segments that allow freedom of movement. Under a load, the flexible kinked segment of these polymers will uncoil allowing movement. The degree to which this occurs is a measure of the material elastic recovery [4, 13]. Strain in compression test is a method of measuring the flexibility/stiffness property ranges of materials so as to determine whether the set materials, when formed as impressions, can be removed from the mouth without injury to impressed oral tissues, and will have adequate stiffness, in the more flexible portions of impressions, to resist deformation when model-forming products are poured against them [12]. The degree of cross-linking of the polymer strands, temperature, and the rate of applied stress may be related to these mechanical properties [13].

The elastic recovery of the silicone impression materials tested met the requirement of ISO 4823, which establishes a recovery greater than 96.5 % [12]. The results of the putty materials presented Reprosil with the highest values (99.78%), and did not differ to Flexitime (99.52%). Optosil presented the lowest results (97.81%), being statistically the same as Silon (97.89%). It was expected that the flexible kinked segment of the polymers would allow polymeric matrix movement and, the greater the volume of the polymeric matrix, the greater the elastic recovery values would be. Actually, this was observed at Reprosil (32.66% - filler content [11]) but not to Optosil (24.74% - filler content [11]). The low values of Optosil elastic recovery may be explained by a low degree of polymer cross-linking or a worse interaction (linkage) between filler and polymer [2]. Flexitime showed higher values of inorganic particles content (73.68% [11]) than Reprosil did, but their results of K are statistically the same. It seems that the filler quality, the low degree of cross-linking or the linkage between filler and polymer may be affecting the results.

In the same way Flexitime and Clonage, and Silon and Optosil posses different volumetric filler content [11] but similar statistical results of K. Again the polymer quality, the low degree of cross-linking or the linkage between filler and polymer may be affecting the results. The analysis of the fluid materials showed Flexitime with the highest mean values (99.80%), but there is no statistically significant difference from the other materials. Not only has the volumetric filler content seemed to be the factor affecting impressions elastic recovery. The degree of cross-linking of the polymers, the fillers size, morphology, and composition may also have contributed to these results.

ISO international standard [12] requires a minimum of 0.8% and a maximum of 20% for putty elastomeric impression materials strain in compression and a minimum of 2.0% and 20% maximum for fluid materials. Of the materials tested just Silon fluid did not meet ISO requirement (21.12%). Silon putty presented the highest values of E (2.14%), but there is no statistically significant difference from the other materials. Silon fluid showed the highest data of E, being statistically different from Clonage (10.93%). Flexitime and Xantopren presented the lowest values of E (5.98% and 6.0%, respectively). As it happened for elastic recovery test not only the filler content seems to be affecting strain in compression. Polymer composition, degree of cross-linking, filler size, morphology, and composition may also be affecting strain in compression.

The light body consistency materials had higher elastic recovery and strain in compression than the heavy body materials. These results agree with the results of Craig and Sun [9] and in part with those of Lu *et al.* [10]. The authors established that the light body consistency materials tested had lower elastic recovery than the putty materials [10] and higher strain in compression than the putty materials [9, 10]. It seems to be obvious that the most correct would be flexible materials to show higher values of elastic recovery. In general, K and E results were statistically different among materials with the exception of Reprosil, Clonage, and Flexitime.

The same elastic behavior presented by the high and low consistency material may warrant better clinical success.

The addition cured silicones showed the highest values of elastic recovery as they are frequently reported to be the most ideally elastic material because of the polymer composition [2, 4, 10]. Besides, Optosil/Xantopren presented the lowest values of strain in compression. The inorganic particles are routinely surface treated to provide better adhesion to the polymeric matrix [2]. Carlo et al. [11] showed that there is such tight linkage between filler and polymer of Xantopren that this may be the reason for E results.

Several authors showed that there is a correlation between elastomeric materials viscosity and their physical properties [3, 4, 8-10] but, the volumetric filler content seems not to be the only factor affecting materials elastic recovery and strain in compression. The degree of cross-linking of the polymers, the polymer composition, the filler size, morphology, and composition may affect these results too. New researches are necessary to understand how each material element influences on physical properties to achieve decisive characteristics for clinical success.

Conclusion

One of the materials tested is not in conformity with the requirements of ISO specification #4823. The light body consistency materials had higher elastic recovery and strain in compression than the heavy body materials. The addition cured silicones showed the highest values of elastic recovery.

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Tables

Table 1 – Materials tested, manufacturer, and batch number

Material	Type	Volumetric Filler Content (%)*	Manufacturer	Batch number
Clonage Putty	CS	47.70	DFL, Rio de Janeiro, Brazil	5111362
Clonage Fluid	CS	31.89	DFL, Rio de Janeiro, Brazil	5111363
Flexitime Easy Putty	AS	73.68	Heraeus Kulzer, Hanau, Germany	220402
Flexitime Correct Flow	AS	52.03	Heraeus Kulzer, Hanau, Germany	220388
Optosil P Confort	CS	24.74	Heraeus Kulzer, Hanau, Germany	230363
Xantopren VL Plus	CS	23.45	Heraeus Kulzer, Hanau, Germany	210743
Reprosil A^+ Putty	AS	32.66	Dentsply Latin America, Petrópolis, Brazil	377613
Reprosil A^+ Regular	AS	29.04	Dentsply Latin America, Petrópolis, Brazil	378204
Silon 2 APS Putty	CS	48.25	Dentsply Latin America, Petrópolis, Brazil	1743-4
Silon 2 APS Fluid	CS	35.73	Dentsply Latin America, Petrópolis, Brazil	349629

* According to Carlo *et al*[11]. CS – Condensation silicone. AS – Addition silicone

Table 2 – Summary of 2-way ANOVA.

	Elastic Recovery		**Strain in compression**	
	F value	***p* value**	**F value**	***p* value**
Material	23.51	< 0.00001	63.60	< 0.0011
Viscosity	122.03	< 0.00001	1512.18	< 0.00001
Mat * Visc	20.14	< 0.00001	58.34	< 0.00001

Table 3 – Mean values, standard deviations and statistical categories defined by Tukey's test for elastic recovery (K) of the materials.

Material	**Elastic Recovery (%)**	
	Putty	**Fluid**
Reprosil A^{+}	99.78 (0.20)^{A,a}	99.75 (0.04)^{A,a}
Flexitime	99.52 (0.36)^{AB,a}	99.80 (0.07)^{A,a}
Clonage	99.31(0.04)^{B,a}	99.50 (0.12)^{A,a}
Silon 2 APS	97.89 (0.57)^{C,b}	99.64 (0.19)^{A,a}
Optosil/Xantopren	97.81 (0.52)^{C,b}	99.68 (0.21)^{A,a}

Groups identified by different letters are significantly different ($p < 0.05$). Capital letters were used to compare groups in the vertical lines and lower case letters were used to compare groups in the horizontal lines.

Table 4 – Mean values, standard deviations and statistical categories defined by Tukey test for strain in compression (E) of the materials.

Material	Strain in compression (%)	
	Putty	Fluid
Reprosil A^+ Putty	1.19 (0.36)^{A,b}	8.34 (0.15)^{C,a}
Flexitime Easy Putty	2.08 (0.22)^{A,b}	5.98 (0.38)^{D,a}
Clonage Putty	1.59 (0.18)^{A,b}	10.93 (0.78)^{B,a}
Silon 2 APS Putty	2.14 (0.42)^{A,b}	21.12 (2.23)^{A,a}
Optosil/Xantopren	1.73 (0.23)^{A,b}	6.00 (0.44)^{D,a}

Groups identified by different letters are significantly different ($p < 0.05$). Capital letters were used to compare groups in the vertical lines and lower case letters were used to compare groups in the horizontal lines.

CAPÍTULO 4

Effect of volumetric filler content on mechanical properties of alginate impression materials

Abstract

This study examined the correlation between volumetric filler fraction and mechanical properties of commercially available alginates. Five alginates – Jeltrate (J), Jeltrate Plus (JP), Jeltrate Chromatic Ortho (JC), Hydrogum (H), and Ezact Krom (E) were used to measure elastic recovery (K) and strain in compression (E). The tests were conducted according to ISO specification #1563. The specimens (n=5) were prepared by hand-mixing the alginate powder with distilled water using the ratio and time specified in the manufacturer's instructions. Values for filler fraction were used from a previous study. Data was submitted to 1-way ANOVA and Tukey's test. Results for elastic recovery (%) were: E – 97.25^{A}, J – 96.84^{A}, JC – 97.23^{A}, H – 96.96^{A}, and JP – 95.83^{B}. The means for strain in compression test (%) were: E – 10.96^{B}, J – 12.05^{B}, JC – 12.01^{B}, H – 11.55^{B}, and JP – 18.65^{A}. All materials are in conformity with the requirements of ISO specification n.1563

INDEX WORDS: Alginate, Percentage of fillers, Recovery from deformation, Strain in compression.

Introduction

Alginate impression materials, also known as irreversible hydrocolloids, are a two-component system – powder and water – having a wide use in dentistry [1-4]. The sol-gel reaction creates a hydrophilic elastic gel formed by a reaction of a dry powder of a soluble salt of alginic acid and a slowly soluble calcium salt (calcium sulfate) mixed with water [1, 2]. Alginates are relatively inexpensive, easy to manipulate and to remove from the mouth, comfortable for the patient, easy to flow in wet or bloody conditions, and the stone is capable to readily wet their surface [1, 3].

These qualities alone are not the main reasons for alginate to be the most commonly used material in the world [4]. Adequate mechanical properties ensure alginates to make and pour accurate impressions by supporting stresses during the removal from the mouth until the elastic strain. ISO 1563 [5] specifies the requirements for properties of dental alginates as compatibility with gypsum, reproduction of detail, elastic recovery, strain in compression, and compressive strength.

The inorganic filler fraction of various commercially available alginates was analyzed by Carlo *et al.* [6]. At that moment the authors determined that Jeltrate and Jeltrate Plus presented a filler content of 48.33%, Jeltrate Chromatic Ortho presented 33.79%, Hydrogum 37.55% and Ezact Krom 40.55%. The authors mentioned that during a database search it was not found a study comparing the inorganic fraction of materials and their mechanical and physical properties. The authors also showed that differences among materials may not be directly related to filler content [7, 8] but, it seems to be meaningful to be considered [1, 3, 9].

Then, the objective of this study was to evaluate the effects of filler fraction of dental alginate impression materials on the elastic recovery and strain in compression. The hypothesis tested was that there were differences in properties among the materials related to volumetric filler content.

Materials and Methods

The impression materials used in this study are listed in Table 1. The materials were prepared by hand-mixing the alginate powder with distilled water using the ratio and time specified in the manufacturer's instructions.

Elastic recovery (K) and strain in compression (E) were tested according to ISO 1563 [5]. For measurement of elastic recovery (K), the specimens (12.5 mm diameter x 20 mm high) (n=5) were deformed to a height of 16 mm (20 %) within 1 s and maintained in this deformation for 5 s in a universal testing machine (Instron 4411, Instron Inc. Canton, MA. USA). Forty seconds after releasing the load the change in length (ΔL) was measured. K (%) was calculated as 100 x (L – ΔL)/L while L is the original length (20 mm).

The strain in compression test (E) was carried out on a compression apparatus described in ISO international standard [5]. The specimens (n=5) were prepared with the same dimension of the previous test and subjected to a load of 125 g, thereby producing a stress of approximately 0.01 N/mm^2, for 30 s and the change in length (h_1) was measured. The load was removed and after 30 s the specimen subjected to a load of 1250 g, producing a stress of approximately 0.1 N/mm^2, for 30 s and the change in length (h_2) measured. E (%) was calculated as $100 \times (h_1 - h_2)/h_0$ where h_0 is the original length (20 mm).

Data for elastic recovery and strain in compression presented a normal and homogeneous distribution and one-way ANOVA following Tukey's test ($\alpha = 0.05$) were employed on the statistical analysis.

Results

The 1-way ANOVA test (Table 2) showed that there were significant differences (*p* = .00005 and *p* = .00011) among materials with respect to elastic recovery and strain in compression. Table 3 lists means, statistical categories defined by Tukey's test and standard deviations (SD) of the tested properties. For all materials tested, the elastic recovery was greater than 95%; Jeltrate Plus presented the lowest value (95.83%), being statistically different from the other materials. For strain in compression, all the materials showed values below 20%; Jeltrate Plus had significantly higher values (18.65%) than the other materials tested.

Discussion

Adequate mechanical properties assure the impression materials to sustain stresses during impression technique, maintaining dimensional stability and integrity, and warranting clinical success [10]. Elastic recovery test is a method of determining whether the materials possess the elastic properties required to recover adequately after deformation occurring when the materials are removed from the mouth [4, 10, 11], and the greater the elastic recovery, the more accurate the impression shall be [4, 12]. Strain in compression measures the flexibility/stiffness of impression materials and indicates whether (1) the polymerized impression can be removed from the mouth without injury to impressed oral tissues, (2) the polymerized impressions will have adequate stiffness in the more flexible portions of impressions to resist deformation when gypsum products are poured against them, and (3) the poured gypsum cast can be removed from the impression without fracture [4, 10, 11, 13].

The elastic recovery of the five alginates met the requirement of ISO 1563, which requires a recovery greater than 95% [5]. Ezact Krom showed the highest value (97.25%) and was statistically equivalent to Jeltrate (96.84%), Jeltrate Chromatic Ortho (97.23%), and Hydrogum (96.96%). Jeltrate Plus presented the lowest value (95.83%), being statistically different from the other materials. During the material sol-gel reaction adjacent molecules of alginate become cross-linked forming a molecular complex (polymer network) constituting the gel structure [1]. This reaction may be classified as a form of polymerization because cross-linking occurs guaranteeing elasticity characteristic to the material [1]. In addition, Carlo *et al.* [6] showed that there are differences in alginates filler fraction that would affect materials mechanical properties. Thus, It was expected that alginates with low filler content would present higher values of elastic recovery. In fact, this was observed at Ezact Krom, Jeltrate Chromatic Ortho, and Hydrogum. There must be other powder components affecting Jeltrate results. Frey *et al.* [4] measured elastic recovery of 96.36% for Jeltrate, which seems to be similar to values observed in this study. On the other hand, Murata *et al.* [9] measured recovery of 97.1% for Jeltrate Plus, which does not seem to be similar to the values in this study.

For strain in compression test, all the materials showed values within the limits of the ISO specification #1563 [5] (not less than 5% or more than 20%). Ezact Krom showed the lowest value (10.96%) and was statistically equivalent to Jeltrate (12.05%), Jeltrate Chromatic Ortho (12.01%), and Hydrogum (11.55%). Jeltrate Plus had significantly higher values (18.65%) than the other materials tested, being statistically different from the other materials. Frey et al. [4] and Murata et al. [9] also measured strain in compression for Jeltrate (14.1%) and Jeltrate Plus (approximately 16.4%); their results seem to follow up the results of this study (deformation is higher for Jeltrate Plus than for Jeltrate) but their data do not seem to be similar to the values in this study. It is supposed that Jeltrate/Jeltrate Plus powder components alter somehow the gel structure development, which becomes more flexible /stiff affecting the results of recovery from deformation and strain in compression.

It was expected that the content of inorganic particle, although not the remarkable characteristic to be considered [7, 8], would display an important role when appraising mechanical properties of the dental alginates [1, 3, 9]. The hypothesis tested that there would be differences in the properties of the material related to filler content was accepted in part, since materials were not significantly affected only by volumetric filler content. The flexible materials would be expected to have less cross-linking, or less fillers, so they would be expected to be weaker than the stiffer materials and more easily torn[10]. Jeltrate and Jeltrate Plus presented[6] the highest volumetric filler content (48.33%) among materials but showed statistically different behaviors for elastic recovery and strain in compression probably due to powder components affecting gel structure. Jeltrate Chromatic Ortho was the material with the lowest results for volumetric filler fraction (33.79%) and presented results statistically equivalent to Jeltrate. Elastic recovery and strain in compression were inversely correlated as it was described by Lu *et al.*[10]. Higher elastic recovery was related to lower strain in compression (stiffer material).

These differences would be attributed to the following: (1) Trisodium phosphate concentration. Trisodium phosphate is a retarder used in alginates composition to control gelation process[1]. Maybe the presence of higher concentrations of retarder could potentially influence the properties of the material; (2) Dust suppression-additives concentration. Veres et al.[8] showed that the properties of stress, strain, and deformation were significantly changed by higher concentrations of dust suppression-additives; (3) Fluoride concentration. Lee et al.[7] showed that the presence of NaF and SnF_2 alters the mechanical properties of alginate due to the strong activity of fluoride ions and their affinity to Ca ions that may have some destructive effect on alginate impression material; and (4) Cross-linking. Differences in cross-linking among the molecules of the alginic acid salt during the gelation process could lead to materials with worst properties.

It was remarkably observed in this study that the volumetric filler content seems not to be the only factor affecting materials elastic recovery and strain in compression. The powder components and the degree of cross-linking between alginate molecules alter the matrix formed after the gelation process affecting these results. New researches are necessary to understand how each component influences on physical properties and thus creating a material with components in equilibrium and with decisive characteristics for their clinical success.

Conclusion

All materials are in conformity with the requirements of ISO specification #1563. Volumetric filler content affected, in part, the mechanical properties of the tested materials. Ezact Krom, Jeltrate, Jeltrate Chromatic Ortho, and Hydrogum showed the highest values for recovery from deformation and the lowest for strain in compression.

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Tables

Table 1 – Materials tested, manufacturer, and batch number

Material	**Volumetric filler content (%)***	**Manufacturer**	**Batch number**
Jeltrate	48.33	Dentsply Latin America, Petropolis, Brazil	156999
Jeltrate Plus	48.33	Dentsply Latin America, Petropolis, Brazil	288721
Jeltrate Chromatic Ortho	33.79	Dentsply Latin America, Petropolis, Brazil	142603
Hydrogum	37.55	Zhermack, Rovigo, Italy	21834
Ezact Krom	40.55	Vigodent, Rio de Janeiro, Brazil	078/05

* According to Carlo *et al*.[6]

Table 2 – Summary of 1-way ANOVA.

	Elastic recovery		**Strain in compression**	
	F value	***p* value**	**F value**	***p* value**
Material	14.46	< 0.00005	12.13	< 0.0011

Table 3 – Mean values (%) and statistical categories defined by Tukey's test (SD) for elastic recovery (K) and strain in compression (E) of the tested materials.

Material	K (%)	E (%)
Jeltrate	96.84^{A} (0.71)	12.05^{B} (4.3)
Jeltrate Plus	95.83^{B} (0.8)	18.65^{A} (1.02)
Jeltrate Chromatic Ortho	97.23^{A} (0.75)	12.01^{B} (0.67)
Hydrogum	96.96^{A} (0.78)	11.55^{B} (0.74)
Ezact Krom	97.25^{A} (0.76)	10.96^{B} (0.39)

Groups identified by different letters are significantly different ($p < 0.05$). Capital letters were used to compare groups in the vertical lines.

CONSIDERAÇÕES GERAIS

Propriedades mecânicas consideradas como ideais, garantem ao material de moldagem suportar tensões durante o processo de moldagem. Isso confere estabilidade de cópia e integridade ao material e, desta forma, determinam seu sucesso clínico (Lu *et al.*, 2004). O material de moldagem ideal é aquele que consegue absorver a maior quantidade de energia que lhe cause deformação, conseguindo manter a sua integridade (Anusavice, 2005; Lu *et al.*, 2004; Mandikos, 1998). O objetivo do presente estudo foi o de determinar quantitativamente e qualitativamente as partículas de carga inorgânicas incorporadas a marcas comerciais de materiais de moldagem e determinar a influência das mesmas em algumas propriedades mecânicas, além de tentar estabelecer uma correlação entre ambas.

O Capítulo 1 do presente estudo demonstrou que há uma grande variedade de formas, tamanhos e composição das partículas de carga presentes nas siliconas odontológicas. Os materiais de alta viscosidade (tipo “massa”) apresentaram maior quantidade de partículas de carga em sua composição do que os materiais de média e baixa consistência embora, para algumas marcas comerciais (Reprosil e Optosil/Xantopren), esse não pareça ser o motivo específico que determine a diferença entre as viscosidades apresentadas pelos mesmos. Supõe-se que os fabricantes conseguem controlar a viscosidade de seus materiais trabalhando com plastificantes (espessantes) de consistência ou, que o tipo de partícula de carga (tamanho e composição) e/ou qualidade da matriz polimérica possa influenciar nestas características. Além disso, talvez as ligações existentes entre a partícula e a matriz, quando extremamente estáveis e duradouras, possam também influenciar neste aspecto. Ainda com relação à quantidade de partículas de carga apresentadas pelos materiais, foi observado que as siliconas por adição possuem maior quantidade de cargas inorgânicas do que as siliconas por condensação. Talvez os materiais de presa por condensação não permitam a incorporação de carga como o fazem os materiais de presa por adição ou, simplesmente, as primeiras não necessitem da mesma quantidade de carga para apresentarem propriedades satisfatórias. Todos os materiais apresentaram altas concentrações de silício na sua composição.

O conteúdo de partículas inorgânicas dos alginatos foi analisado no Capítulo 2. As partículas apresentaram-se como paredes celulares externas de algas depositadas o longo de gerações geológicas e, de maneira geral, não parece haver diferença significativa entre marcas comerciais, com exceção do material Hydrogum, que apresentou diferença na forma e presença de um elemento na composição de sua partícula que leva a supor tratar-se de algas de uma ordem, sub-ordem ou gênero diferente dos demais. Todos os materiais apresentaram altas concentrações de silício na sua composição. Não foi encontrada uma interação específica entre as fibrilas do gel de alginato e as partículas de diatomácea que justifique o seu uso específico como carga inorgânica dos alginatos. Possivelmente, a escolha se baseie no baixo custo que a diatomácea incorpora aos custos do processo industrial (Bold and Wynne, 1985).

As propriedades de recuperação elástica e deformação sob compressão das siliconas foram analisadas no Capítulo 3 e, além disso, tentou-se estabelecer uma correlação entre o estudo das partículas de carga do Capítulo 1 e os resultados dos testes das propriedades dos materiais. Todos apresentaram valores de recuperação elástica que atendem ao mínimo (96,5%) exigido pela especificação №4823 da Associação internacional para Padronização (ISO 4823:2000). Esperava-se que os resultados obtidos para recuperação elástica dos materiais fossem determinados pela quantidade de matriz polimérica de cada marca comercial e viscosidades estudadas onde, quanto maior a quantidade de matriz polimérica e quanto mais fluida a consistência maiores seriam os resultados da propriedade em estudo. Nem todos os materiais apresentaram essa correlação. A composição da matriz polimérica de cada marca comercial, o grau de ligação cruzada entre os polímeros, o tamanho, morfologia e composição das partículas de carga parecem influenciar conjuntamente essa propriedade. De maneira geral foi observado que os materiais de viscosidade fluida (regular e leve) apresentam-se com maiores valores de recuperação elástica do que os materiais de alta viscosidade (massa). A norma №4823 da ISO (ISO 4823:2000) determina o mínimo de 0,8% e o máximo de 20% como resultado para deformação sob compressão de materiais elastoméricos na consistência de massa (“putty”) e mínimo de 2,0% e máximo de 20% para os materiais de consistência regular e leve. Todos os materiais atendem ao valor mínimo e máximo exigidos pela norma, com exceção da silicone de presa por condensação Silon 2 APS fluido (21,12%). Assim, como o foi verificado para o teste de recuperação elástica parece que os resultados do teste de deformação sob compressão não são determinados exclusivamente pela quantidade de partículas de carga presentes na composição do material. A composição da matriz polimérica de cada marca comercial, o grau de ligação cruzada, o tamanho, morfologia e composição das partículas de carga também parecem influenciar significativamente essa propriedade. Os resultados para recuperação elástica e deformação sob compressão apresentaram-se em quase toda a sua totalidade inversamente proporcionais como descrito por Lu *et al.* (2004).

O Capítulo 4 trata da análise da recuperação elástica e deformação sob compressão dos alginatos estudados estabelecendo-se, também, uma correlação entre as partículas de carga dos materiais (avaliadas no Capítulo 2) e os resultados das propriedades em estudo. A especificação №1563 da ISO (ISO1563:1990) determina que os valores de recuperação elástica para alginatos deve ser superior a 95% e todos os materiais em estudo estão em conformidade com a norma. Para o teste de deformação sob compressão, todos os materiais apresentaram resultados dentro dos limites impostos pela norma ISO (ISO1563:1990). Durante a reação de presa dos alginatos uma molécula de alginato solúvel liga-se a outra formando um complexo molecular. Essa estrutura do gel pode ser considerada como uma cadeia polimérica e é ela quem garante elasticidade à massa de material geleificado. Da mesma forma que para os elastômeros, esperava-se que materiais possuindo menor quantidade de partículas de carga apresentassem maiores valores de recuperação elástica e deformação sob compressão. Todos os materiais testados apresentaram essa correlação com exceção do Jeltrate Plus. É provável que haja na composição do pó do material Jeltrate Plus algo que altere suas propriedades determinando esse comportamento. Essas diferenças talvez possam ser atribuídas à presença de fosfato de sódio (retardador) em alta concentração influenciando na qualidade da matriz geleificada. Além disso, a presença de aglomeradores de partícula (supressores de formação de poeira) ou altas concentrações de fluoretos podem estar alterando as propriedades do material. Os resultados de recuperação elástica e deformação sob compressão apresentaram-se inversamente relacionados aos materiais estudados.

Modelos confeccionados com alta precisão são necessários em um grande número de procedimentos odontológicos. Alguns estudos demonstraram haver uma correlação entre a quantidade de partículas de carga e propriedades mecânicas de elastômeros (Chen *et al.*, 2004; Corso *et al.*, 1998; Craig and Sun, 1994; Lu *et al.*, 2004; Mandikos, 1998). Infelizmente, não foi possível determinar a mesma correlação para os alginatos com base em pesquisa na literatura. Porém, os resultados do presente estudo demonstraram que o conteúdo de partículas de carga não parece ser o único fator a influenciar na consistência, recuperação elástica e deformação sob compressão dos materiais de moldagem. Aditivos misturados ao pó dos alginatos, o grau de ligações cruzadas da cadeia polimerizada, a composição da matriz polimérica das siliconas antes da reação de polimerização, o tamanho, morfologia e composição da partícula de carga também exercem papel significativo na determinação das propriedades dos materiais.

CONCLUSÃO

Dentro das limitações do presente estudo as seguintes conclusões podem ser definidas:

1. Os materiais para moldagem à base de alginato e silicone testados apresentaram ampla distribuição de morfologia, tamanho e quantidade de partículas de carga;

2. Todos os materiais apresentaram altas concentrações de silício na sua composição;

3. Todos os alginatos apresentam resultados de recuperação elástica e deformação sob compressão em conformidade com o determinado pela norma ISO №1563;

4. Nem todas as siliconas apresentam resultados de recuperação elástica e deformação sob compressão em conformidade com o determinado pela norma ISO №4823

5. O conteúdo de partículas inorgânicas não parece ser o único fator a influenciar na consistência, recuperação elástica e deformação sob compressão dos materiais de moldagem estudados.

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* De acordo com a norma da UNICAMP/FOP, baseada na norma do International Committee of Medical Journal Editors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

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ANEXO

1. Comprovante de envio de trabalho – Artigo do Capítulo 2.

De: onbehalfof@scholarone.com em nome de alethea_gerding@dentistry.unc.edu
Enviado em: sexta-feira, 25 de abril de 2008 09:58
Para: sinhoret@fop.unicamp.br
Assunto: Journal of Prosthodontics Submission

25-Apr-2008

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