UNIVERSIDADE ESTADUAL DE CAMPINAS - UNICAMP
FACULDADE DE ODONTOLOGIA DE PIRACICABA

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PROPRIEDADES FÍSICAS DE GESSOS ODONTOLÓGICOS TIPO IV DESIDRATADOS EM TEMPERATURA AMBIENTE E EM FORNO DE MICROONDAS

Dissertação de mestrado apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do título de mestre em Materiais Dentários.

Orientador: Prof. Dr. Rafael Leonardo Xediek Consani

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DEDICATÓRIA

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RESUMO

O objetivo deste estudo foi verificar a influência da secagem em temperatura de laboratório (25±4ºC) e em forno de microondas sobre propriedades físicas de gessos odontológicos tipo IV: Elite Rock, Shera Premium e Durone IV. Os gessos foram proporcionados e espatulados mecanicamente a vácuo seguindo as recomendações dos fabricantes e vazado no molde com auxílio de vibrador mecânico. No Capítulo 1 foi avaliada a alteração dimensional, resistência à compressão e reprodução de detalhes dos gessos desidratados em forno de microondas e em temperatura de laboratório. Dois protocolos de desidratação com diferentes períodos de avaliação foram utilizados para os ensaios de alteração dimensional, resistência à compressão e reprodução de detalhes dos gessos foram utilizados e divididos em 4 grupos com: G1- temperatura de laboratório (25 ± 4ºC) no período de 2 horas; G2- temperatura de laboratório após 24 horas; G3- temperatura de laboratório após 7 dias e G4- microondas com potência de 800 W por 5 minutos após 2 horas. No Capitulo 2 foi avaliada a influência de diferentes potências de radiação do forno de microondas durante a secagem dos gessos sobre a alteração dimensional e resistência à compressão. Foram utilizados seis diferentes métodos de secagem e divididos em seis grupos: G1-temperatura ambiente no período de 2 horas; G2- temperatura ambiente no período de 24 horas; G3- temperatura ambiente no período de 7 dias; G4- microondas com potência de 200 W por 5 minutos; G5- microondas com potência média 400 W por 5 minutos e G6- microondas com potência alta 800 W por 5 minutos. Nos capítulos 1 e 2 para o ensaio de alteração dimensional linear foram confeccionadas amostras utilizando matriz metálica com entalhes de diferentes profundidades e distância entre as linhas de 2,5 mm. Em seguida, a superfície foi escaneada com resolução de 1200 dpi e analisada com *software ImageTool* 3.0. Para o ensaio de resistência à compressão foram confeccionados moldes de silicone polimerizada por condensação, a partir de uma matriz metálica cilíndrica medindo 20 mm de comprimento por 10 mm de diâmetro. Para o ensaio de reprodução de detalhes

executada somente no capítulo 1, a análise foi feita na linha central da matriz reproduzida na superfície da amostra de gesso. Os dados foram submetidos à análise de variância (ANOVA) com dois fatores e as médias ao teste de Tukey com nível de 5% de significância. Capitulo 1: No G1, as três marcas comerciais apresentaram maiores níveis de expansão dimensional sendo diferentes estatisticamente entre os grupos 2, 3 e 4. Para o ensaio de resistência à compressão os gessos Elite Rock e Durone IV não apresentaram diferença estatística significante entre G2 e G4, exceto para Shera Premium não apresentando diferença entre G3 e G4. Os melhores índices de reprodução ocorreram para G3. Capítulo 2: Os valores de alteração dimensional para o gesso Elite Rock foram estatisticamente semelhantes entre G3 e G6 e entre G4,G5 e G2. O gesso Shera Premium apresentou maiores níveis de expansão para G1 em relação a G2, G3, G4, G5, G6. Para o gesso Durone IV o G5 apresentou os menos valores de expansão dimensional. Para as três marcas comerciais utilizadas, a desidratação em forno de microondas com potência de 200 W e em temperatura de laboratório após 7 dias promoveu resistência à compressão sem diferença estatística e significativamente maiores,com exceção do Shera Premium e Durone IV que produziram valores semelhantes com potência de 800 W e 400W respectivamente. Os gessos odontológicos tipo IV apresentaram melhora em suas propriedades com secagem em forno de microondas. A potência de 200 W a 800 W proporcionaram níveis de alteração dimensional similares aos desidratados em temperatura de laboratório após 24 horas e 7 dias. A potência de 200 W produz maiores valores de resistência à compressão para Elite Rock.

Palavras-chaves: Gesso odontológico, microondas, resistência à compressão, alteração dimensional linear, reprodução de detalhes.

ABSTRACT

The objective of this study was to assess the effect of drying laboratory temperature (25 ± 4 ° C) and in microwave oven on the physical properties of dental stone type IV: Rock Elite, Premium and Shera Durone IV. Plasters were proportionate and spatulate mechanically vacuum following the manufacturers recommendations and poured into the mold with the aid of mechanical vibrator. In Chapter 1 we evaluated the dimensional change, compressive strength and detail reproduction of plaster dried in a microwave oven and a temperature of the laboratory. Two different protocols of dehydration for tests of dimensional change, compressive strength and detail reproduction of plaster casts were used and divided into four groups: G1-temperature laboratory (25 ± 4 ° C) in 2 hours, G2-temperature laboratory after 24 hours, G3-temperature laboratory after 7 days and G4-microwave power of 800 W for 5 minutes after 2 hours. In Chapter 2 we evaluated the effects of different energy radiation from the microwave oven during the drying of plaster on the dimensional changes and compressive strength. We used six different methods of drying and divided into six groups: G1-room temperature within 2 hours; G2-room temperature within 24 hours; G3-temperature environment within 7 days: G4-microwave power of 200 W for 5 minutes and G5-average microwave power 400 W for 5 minutes, G6-high microwave power 800 W for 5 minutes. In chapters 1 and 2 for testing dimensional linear samples were prepared using metal matrix with notches of different depths and distance between lines of 2.5 mm. Then the surface was scanned at 1200 dpi and analyzed with software ImageTool 3.0. For testing the compressive strength silicone molds were prepared by condensation polymerized in cylindrical stainless steel mold measuring 20 mm by 10 mm in diameter. To test the reproduction of details performed only in chapter 1, the analysis was performed on the center line of the matrix reproduced on the sample surface of plaster. Data were subjected to analysis of variance (ANOVA) with two factors and the means with Tukey test at

5% level of significance. Chapter 1: In G1 of three commercial brands showed higher levels of dimensional expansion was statistically significant between groups 2,3 and 4. For testing the compressive strength gypsum Durone Elite Rock and IV showed no statistically significant difference between G2 and G4, except for Premium Shera no difference between G3 and G4. The highest rates of reproduction were to G3. Chapter 2: The values of dimensional change to the cast Elite Rock showed statistically similar values among and between G3 and G6, G4, G5 and G2. Gypsum Premium Shera had higher levels of expansion in G1 compared to G2, G3, G4, G5, G6. For plaster Durone IV G5 showed the least values of dimensional expansion. For the three trademarks used, drying in a microwave oven with a power of 200 W and temperature in the laboratory after 7 days promoted the compressive strength and not statistically significantly higher, except for the Premium and Shera Durone IV that produced similar values with power of 800 W and 400W respectively. Dental stone type IV showed improvement in their properties with drying in a microwave oven. The power of 200 W to 800 W gave similar levels of dimensional change in the dehydration temperature of the laboratory after 24 hours and 7 days. The power of 200 W produces higher values of compressive strength for Elite Rock.

Key words: Dental stone, microwave energy, compressive strength, linear dimensional change, details reproduction.

SUMÁRIO

INTRODUÇÃO GERAL

A gipsita é um mineral encontrado na natureza e um dos produtos derivados desse mineral é o gesso odontológico ou sulfato de cálcio hemi-hidratado utilizado para confeccionar modelos a partir de moldes ou réplicas de tecidos bucais duros ou moles. Os modelos de gesso são materiais fundamentais na prática clínica e laboratorial em Odontologia, sendo necessário que reproduzam de maneira fiel às estruturas moldadas na cavidade bucal.

De acordo com a especificação n° 25 da Associação Dentária Americana (ADA), existem cinco classificações para os gessos odontológicos: gesso comum para moldagem (Tipo I), gesso comum para modelo (Tipo II), gesso pedra (Tipo III), gesso pedra de alta resistência (Tipo IV) e gesso pedra de alta resistência e alta expansão (Tipo V). Devido às características das partículas de pó de cada tipo de gesso variar em forma e densidade, os diferentes tipos de gesso necessitam de diferentes quantidades de água para estabelecer corretamente a relação pó-água e alcançarem as propriedades mecânicas consideradas ideais para a prática odontológica (Anusavice, 1996).

O critério para utilização de um determinado tipo de gesso estaria também baseado na dependência do uso clínico e, conseqüentemente, das propriedades mecânicas que esta indicação clínica em particular deveria exigir. Classificado como gesso tipo IV (alta resistência), o gesso especial apresenta grande resistência e dureza, além de reproduzir os detalhes com boa precisão e apresentar expansão de presa de 0,1% (Motta, 1991). Por essa razão, o gesso tipo IV é amplamente utilizado na confecção de troqueis e modelos de trabalho de prótese parcial fixa e prótese removível.

Para os troqueis, há necessidade de uma superfície resistente que permita o acabamento da cera de escultura com instrumento afiado, sem que ocorra desgaste e/ou ranhuras principalmente na margem cervical do troquel. Entretanto, uma das desvantagens do gesso tipo IV é justamente a possibilidade de ocorrer

abrasão do troquel quando da escultura do padrão de cera. Contudo, esse tipo de gesso tem sido comumente usado, pois tem custo relativamente baixo, permite fácil manipulação e apresenta compatibilidade com a maioria dos materiais para moldagem (Anusavice, 1996).

Os modelos de gesso devem apresentar resistências à compressão, abrasão e flexão e dureza de superfície para suportar os esforços ao qual serão submetidos durante o trabalho laboratorial, além de reproduzir com exatidão as estrutura do molde (Vanzillotta *et al*., 2002). O gesso deve aguardar de 24 a 48 horas antes de o modelo ser manipulado (Schwedhelm *et al*., 1997). No entanto, na rotina clínica, os cirurgiões-dentistas freqüentemente necessitam trabalhar os modelos logo depois de confeccionados. Nessas condições, esses modelos ainda apresentam resistência úmida, geralmente com resistência mecânica e dureza de superfície inadequadas ao uso protético (Luebke *et al*., 1985).

Entretanto, a remoção por meio da desidratação em temperatura ambiente (resistência seca) de todo excesso de água não relacionado à reação estequiométrica do gesso tem mostrado aumentar as propriedades mecânicas. No entanto, a gipsita é estável somente abaixo da temperatura de 40°C e a secagem em temperaturas mais altas deve ser evitada ou controlada com critério, porque podem causar contração volumétrica e redução da resistência mecânica (O'Brien, 1997).

Em estudo prévio, os modelos de gesso secos em forno de microondas como meio alternativo à secagem em meio ambiente mostraram resultados mecânicos promissores (Hersek *et al*., 2002). Além disso, a secagem do gesso em forno de microondas pode permitir um ganho de tempo considerável no trabalho diário.

O nível ideal de desidratação do gesso e a potência de radiação foram analisados por alguns estudos mostrando resultados conflitantes, quando se acredita que a alta potência de radiação por microondas seria a mais indicada (O'Brien, 1997); entretanto, outros autores relatam que a potência baixa seria a mais indicada para secagem dos modelos de gesso (Luebke *et al*., 1985).

Diante deste fato, o objetivo deste estudo foi avaliar as propriedades de resistência à compressão, estabilidade dimensional e reprodução de detalhes de gessos odontológicos tipo IV. Para isso, o estudo foi desenvolvido em dois capítulos. O Capítulo 1 avaliou a secagem em temperatura de laboratório e em forno de microondas e o Capítulo 2 qual potência de radiação das microondas seria mais indicada para secagem. A hipótese do trabalho seria que a secagem de modelos de gesso em forno de microondas não causaria influência deletéria nas propriedades mecânicas de gessos odontológicos.

Esta dissertação de mestrado foi apresentada no formato alternativo, de acordo com as normas estabelecidas pela Deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas.

CAPÍTULO 1

Linear dimensional change, compressive strength and detail reproduction in type IV dental stone dried at room temperature and in a microwave oven

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Objective: To compare the influence of drying techniques at laboratory temperature and using a microwave oven on the linear dimensional alteration, compressive strength and detail reproduction in type IV dental stones.

Materials and Method: Three type IV dental stone brands were selected: Elite Rock, Shera Premium and Durone IV. Two different drying protocols were used and 180 test samples were divided into 4 groups; G1- laboratory temperature (25 ± 4ºC) dried for 2 hours; G2- laboratory temperature dried for 24 hours; G3- laboratory temperature dried for 7 days and G4- microwave oven dried at 800 W for 5 minutes after 2 hours at laboratory temperature. After drying, samples were assayed for dimensional alteration, compressive strength and detail reproduction of the dental stone. The linear dimensional alteration and compressive strength data were submitted to analysis of variance (ANOVA, two-tailed) followed by the Tukey test with a 5% level of significance; reproduction of details were presented as percentages.

Results: For G1, all three commercial brands presented statistically greater levels of dimensional expansion than for groups 2,3 and 4. For the compressive strength test, Elite Rock and Durone IV had did not present any significant differences between G2 and G4, while Shera Premium did not present any difference between G3 and G4. The best reproduction levels were observed for G3.

Conclusion: Type IV dental stones presented improvements in their properties after microwave oven drying.

Keywords: dental stone, microwave, compressive strength, dimensional stability,

detail reproduction.

1. INTRODUCTION

Stone casts are key materials in dental laboratory and clinical practice and must reproduce, as faithfully as possible, structures obtained from molding. For this, precise techniques and suitable materials are required. The type IV dental stone is widely used for making dies and working patterns for fixed partial dentures and removable partial dentures, due to its superior mechanical properties, compared to the dental stone; such properties include increased strength, hardness and expansion[1].

To make a die, a sturdy surface is required to allow the manufacture of the wax sculpture at the cervical margin, without abrasion of the cast. However, the disadvantage of type IV dental stone is precisely its ability to abrade the surface when it takes the form of the wax pattern [2]. Generally, the mechanical properties of gypsum products are related to the water/ powder ratio, mixing time, mixing volume, chemical composition, relative humidity, room temperature at which the material is stored, the remaining water and the time elapsed after the mold is cast[3]

Reports in the literature recommend waiting for 24 to 48 hours before manipulation of the stone model for the preparation of prosthetic pieces [4]. However, in clinical practice it is sometimes necessary to manipulate the models soon after their manufacture. These models generally have a resistant wet surface with low hardness, and are therefore considered unsuitable [5-6]. The removal of all the water in the models, not used by the stoichiometric reaction of calcium sulfate α- hemihydrate ($CaSO_4$ H_2O), by drying at room temperature is required to obtain adequate stone mechanical properties for the use of the model. Gypsum is stable only below 40 ° C and drying at higher temperatures should be managed with discretion since this may cause greater shrinkage, strength reduction and promote cracks in the model [7].

Microwave radiation has been widely accepted in dentistry [8-10] for the

disinfection of acrylic resins activation [11]. In a previous study, stone casts were dehydrated in a microwave oven as an alternative method of drying, this technique imparted increased mechanical properties to the models when dehydrated at room temperature [12]. As such, the aim of this study was to evaluate the compressive strength, dimensional stability and detail reproduction of three type IV dental stones following different drying techniques that employed laboratory temperature and microwave ovens. The working hypothesis is that these properties are not negatively influenced by the microwave dehydration of gypsum.

2. MATERIALS AND METHODS

Three type IV dental stones were selected; Durone IV, Elite Rock and Shera Premium, as listed in Table 1. All materials were mixed mechanically under vacuum, following the water/powder proportions recommended by the manufacturers. The stone cast was immediately poured into the mold with the aid of a mechanical vibrator.

Table 1- Product, manufacturer, water/powder ratio and batch number.

Product	Manufacter	Water/Powder	Batch No.
Durone IV	Dentsply, Petrópolis, Rio de Janeiro, Brazil	19 mL/100 g	635041
Elite Rock	Zhermack, Rovigo, Italy	20 mL/100 g	U110651/B
Shera Premium	Shera Werkstoff-Tecnologie, Lemforde, Germany	20 mL/100 g	92864

Two different protocols of drying for the tests of dimensional change, compressive strength and detail reproduction of stone casts were used and divided into four groups: G1- laboratory temperature (25 ± 4 ° C) drying for 2 hours, G2- laboratory temperature drying for 24 hours, G3- laboratory temperature drying for 7 days and G4-microwave oven power (800 W) for 5 minutes after 2 hours at laboratory temperature. A cup with 200 mL of distilled water was placed in the microwave to protect the magnetron [10].

Linear dimensional alteration:

For the analysis of dimensional alteration, forty samples were used for each stone brand, and divided into four groups (n = 10, each group). In the present study, a metallic matrix was used with two series of seven slots with depths ranging from 0.025 to 0.300 mm and 2.5 mm range in distance between the grooves, made in accordance with the rules established by specification n^{o} 25 of the ADA[13]. A PVC pipe of 30 mm in diameter by 15 mm high was placed on the metal matrix, so that the slot of 0.050 mm deep remained in the center of the ring diameter. The stone was weighed on an analytical balance (Sauter, model K1200, Switzerland) and mixed with deionized water and measured with a test tube, according to the proportions recommended by the manufacturers (Table 1) and manipulated mechanically by vacuum (Multivac 4, Degussa, Germany) with a speed of 450 rpm for 30 seconds. After mixing, the stone was poured inside the ring that was previously isolated with Vaseline under vibration. After 2 hours, samples were removed from the PVC pipe and the surface was subjected to the scanner (HP Scanjet 2410, São Paulo, Brazil) with a resolution of 1200 dpi and analyzed with software ImageTool 3.0. For image calibration the distance between lines 1 and 5 was used, comprising a 10-mm distance between them (Figure 1).

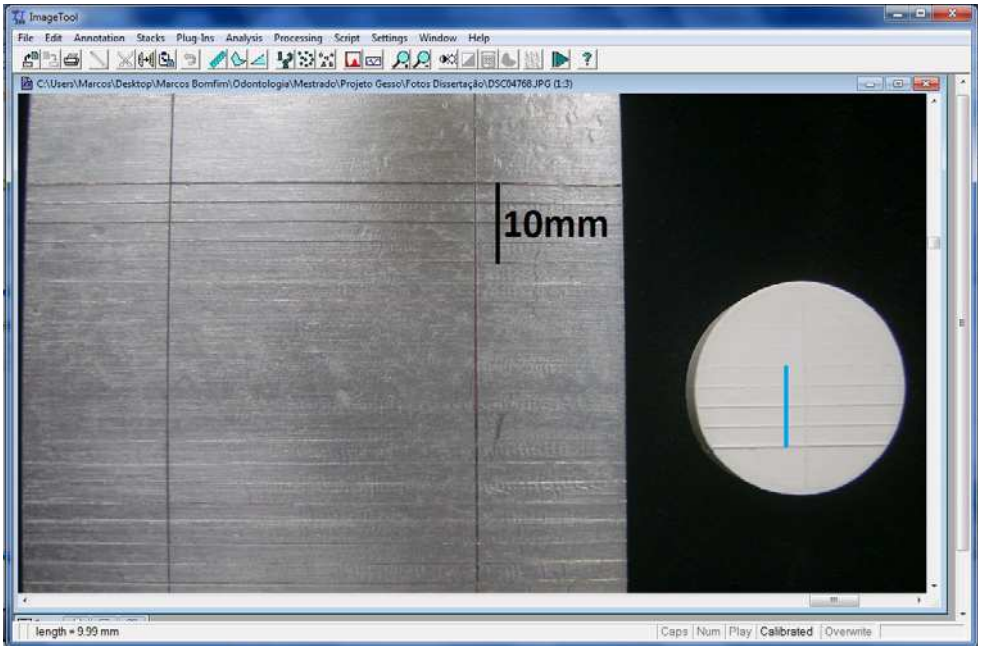

Figure 1 - Sample used for software analysis of dimensional alteration. Black line indicates calibrated scale blue line and the measurement of the sample.

Compressive strength:

To test compressive strength, forty samples were prepared and separated into 4 groups (n = 10). Specification N^{o} 25 of the American Dental Association (ADA) [13] provides for testing of compressive strength using samples of 40 mm in length and 20 mm in diameter. However, in the present study metal molds were used that were 20 mm in length and 10 mm in diameter (Figure 2), maintaining the length-diameter ratio recommended by ADA[13]. The metal molds were used to obtain the silicon die polymerized by condensation (Optosil Comfort, Heraus Kulzer, Hanau, Germany), in which the samples were made of stone. The tone was weighed on an analytical balance (Sauter, model K1200, Switzerland) and mixed with deionized water measured with a test tube, according to the proportions recommended by the manufacturers (Table 1) and manipulated

mechanically by vacuum (Multivac 4, Degussa, Germany) with a speed of 450 rpm for 30 seconds. The silicone molds were placed on a glass plate and filled with stone with the aid of a mechanical vibrator (Vibrator GC, MEG Chemical Co. Ltd.). After filling the mold, another glass plate was placed over the stone so that the ends of the sample remained flattened.

Figure 2 - Sample used for the testing of compressive strength

The samples were tested in a universal testing machine (Instron 4411, Corona, CA, USA) operating with a load cell of 5 kN with a displacement speed of 0.5 mm / min until failure.

Detail reproduction:

For the analysis of detail reproduction, forty samples were used for each brand of stone, which were divided into four groups (n = 10). The same metallic matrix was used for the preparation of samples for dimensional alteration testing. According to the American Dental Association (ADA), [13] a reproduction of a detail is considered satisfactory when a copy line of 0.050 mm in diameter is reproduced continuously across the stone model.

A PVC pipe was placed on the matrix (30 mm diameter, 15 mm high), so

that the notch on the metal matrix of 0.050 mm in depth was located in the center of the ring diameter. After mixing under vacuum, the stone was poured inside the ring that was previously isolated with vaseline under vibration. After two hours, the samples (Figure 3) were removed from the PVC pipe and surface conditions examined with a stereomicroscope (XLT30, Nova Optical Systems, Piracicaba - Sao Paulo - Brazil) at 25 times magnification.

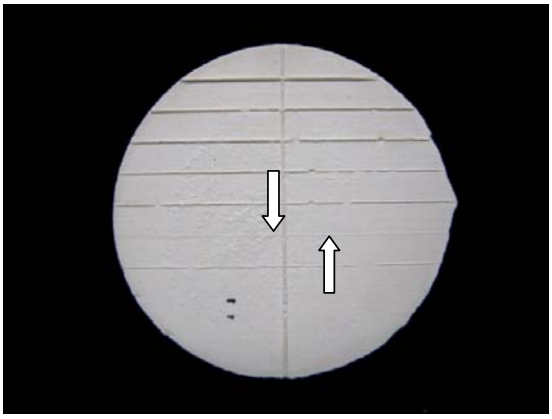

Figure 3 - Sample used in the reproduction of details (Arrows indicate the range of 0.050 mm).

Statistical analysis:

The results of the linear dimensional alteration and compressive strength were subjected to analysis of variance (ANOVA, two-tailed) followed by the Tukey test at a 5% level of significance. The values of the reproduction of details were given as a percentage.

3. RESULTS

Analysis of variance for testing linear dimensional alteration demonstrated the stone ($p = 0.00023$) and time ($p = 0.00001$) to be significant factors, together

with a stone-time interaction (p = 0.04924). The analysis of variance data are shown in Table 2.

Table 2 - Analysis of variance.

	Df	Sum of squares	Mean square	F value	P value
Stone	2	0.00163	0.00081	10.213	0.00023
Time	3	0.01048	0.00349	43.749	0.00001
Stone X Time	6	0.00104	0.00017	2.18	0.04924
Error	108	0.00862	0.00007		

Test results for the linear dimensional alteration of casts, depending on the drying method used, are illustrated in Table 3. For the Elite Rock, G1 showed the highest level of dimensional expansion, which was statistically higher than those of the groups 2,3 and 4. For G2, G3 and G4, Elite Rock did not demonstrate any statistically significant differences. The Shera Premium and Durone IV stones presented similar results to those of Elite Rock and the group with the highest expansion in the other groups. Comparing the results of the brands, G1 demonstrated no significant differences. For G2 and G3, Durone IV had the highest levels of expansion in relation to Shera Premium and Elite Rock. For G4, Shera Premium presented the lowest dimensional expansion.

Table 3 - Mean linear dimensional alteration (mm), standard deviation and percentage of dimensional alteration, as a function of casts and drying methods.

Dental Stone	G1	G2	G3	G4
Elite Rock	10.034±0.007 aA 0.34%	10.016±0.005 bB 0.16%	10.010±0.007 bB 0.10%	10.013±0.031 aB 0.13%
Shera Premium	10.032±0.007 aA 0.32%	10.012±0.006 bB 0.12%	10.010±0.004 bB 0.10%	10.005±0.010 bB 0.05%
Durone	10.032±0.009 aA 0.32%	10.023±0.004 aB 0.23%	10.016±0.010 aB 0.16%	10.015±0.007 aB 0.15%

Means followed by different small letters in each column and different capital letters in each row differ statistically by Tukey test (5%).

Analysis of variance for testing the compressive strength demonstrated that stone ($p = 0.00001$) and time ($p = 0.00001$) were significant factors and that there exists a stone-time interaction ($p = 0.00314$). The analysis of variance data are shown in Table 4.

Table 4 - Analysis of variance.

	Df	Sum of square	Mean square	F value	P value
Stone	2	3164.17	1582.08	55.10	0.00001
Time	3	12169.85	4056.61	141.30	0.00001
Stone x Time	6	617.07	102.84	3.58	0.00314
Error	108	3100.45	28.70		

Table 5 shows the values of compressive strength for the different drying methods. G1 made with Elite Rock stone presented the lowest values of compressive strength. G2 and G4 did not present statistically significant differences. Group 3 demonstrated significantly higher compressive strength values than G1, G2 and G4. Stone Shera Premium presented the highest resistance in groups 3 and 4, with no statistical difference between these groups. Lower resistance values are shown in G1, and intermediate values for G2. The Durone IV stone showed the highest strength values for G3 and the lowest values

for G1. No statistically significant differences were found between G4 and G2. When comparing the cast brands, Elite Rock presented the highest compressive strengths for G1, G2 and G3. For G4, the Durone IV stone had the lowest resistance values.

Table 5 - Mean values of compressive strength (MPa) and standard deviation, as a function of the interaction of the drying method and type of stone.

Dental Stones	G1	G2	G3	G4
Elite Rock	32.18±3.21aC	47.29±2.25aB	61.30±1.96aA	45.72±3.26aB
Shera Premium	21.65±4.29bC	37.70±6.32bB	43.03±7.19bA	43.18±5.76aA
Durone IV	18.87±2.89bC	38.16±6.30bB	45.48±6.12bA	34.23±4.04bB

Means followed by different small letters in each column and different capital letters in each row differ statistically by Tukey test (5%).

The test results for detail reproduction, as a function of drying method, are depicted in Figure 4 as percentages. Detail reproduction can be considered satisfactory when the cast maintains the continuous 0.050 mm line of the matrix. After reviewing the reproductions with a stereomicroscope, the G1 drying method presented the worst rates of reproduction for each cast. The highest rates of reproduction were found for G3.

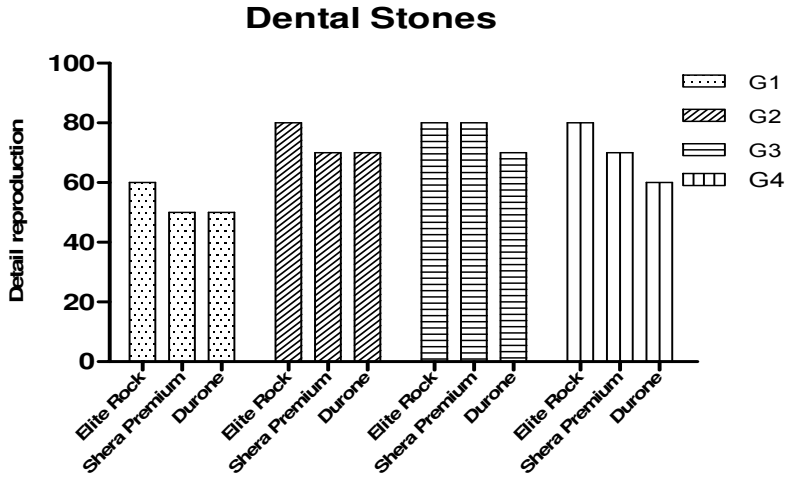

Figure 4 – Graphic of the results of the detail reproduction of casts (%).

4. DISCUSSION

This study was conducted to verify that the drying of dental stone type IV in a microwave oven, to gain time, would not have a negative influence on the linear dimensional alteration, compressive strength and detail reproduction of commercial dental stone. Overall, the results showed that the three marks of gypsum type IV were affected differently by the drying methods. The working hypothesis that these properties would not be negatively influenced by the dehydration of gypsum was confirmed.

Linear dimensional alteration: Capacity-setting expansion of stones is an important factor for many dental applications. The models must have slightly larger dimensions than the shaped structures to ensure that the dental pieces are adequately for prosthetic manufacturing, offsetting the contraction of the material for molding. The expansion of the mass of stone, resulting from the hydration of calcium sulfate during mixing, promotes the reaction of crystallization where crystal growth occurs from the crystallization nuclei, generating volumetric expansion that can range from 0.06 to 0.5% [2].

Specification n^{o} 25 ADA[13] recommends a type IV dental stone setting expansion of up to 0.1% at 2 hours after mixing. Another important fact to consider is that the setting expansion is inherent in the crystallization of the stone[14] and occurs according to the process of calcination of dental stone used to obtain the hemihydrate [6].

The setting expansions obtained in this study for samples subjected to drying at laboratory temperature and in a microwave oven for three different brands of gypsum (Table 2) were higher than those advertised on the product labels. The three brands showed no statistically significant difference between expansion levels for G2, G3 and G4 and lower levels of expansion for G1 for the three brands tested. The difference between the expansion mentioned by the manufacturer and

those obtained in this study could be due to the environmental conditions of the laboratory, where the samples were prepared in a room without temperature and humidity control, with the intention of establishing environmental similarity to the clinical or prosthetic laboratory conditions.

Compressive strength: The strength of gypsum-based products is usually expressed in terms of compressive strength, which is directly related to the material's ability to resist fracture when subjected to compressive strength. This mechanical property is linked to other important properties of the cast, especially to hardness and wears resistance. Thus, the importance of the compressive strength of gypsum is important for the manufacture of rehabilitation work in dentistry.

The compressive strength values found for the different drying techniques (Table 4) indicate that the models dehydrated using the microwave oven at 2 hours (G4) after manipulation showed average values of compressive strength with no statistical difference when compared with those dried at laboratory temperature for 7 days (G3, Shera Premium stone) and with no statistical difference from samples dried at laboratory temperature for 24 hours (G2) to, Elite Rock and Durone IV). Similar results were found in the study by Tuncer et al. (1993) [15], who evaluated drying techniques that employed a microwave oven and conventional oven during periods of 2, 4, 24 and 48 hours. However, data were different from those of Leubke & Schneider (1985) [16], who evaluated eight brands of type IV dental stone dried in a microwave oven with 1450 W for 5 minutes. These authors found no statistically significant differences between the values of compressive strength for the samples dried in a microwave oven and at room temperature, considering that this result may have been influenced by the use of high-power microwaves. The samples dried in a microwave oven and at laboratory temperature for 24 hours and 7 days demonstrated statistically similar values of compressive strength, suggesting that the casts could be used to manufacture prosthetic pieces at two hours after mold filling.

Hersek et al. (2002) [7] analyzed the diametral tensile strength of casts that were dried in a microwave oven at 10 minutes after the initial setting and

concluded that drying increased the resistance of the gypsum due to the rapid evaporation of the water remaining in the mass, due to the heat produced by the microwave radiation. Periods of between 10 and 60 minutes before drying in the microwave oven were also tested in this study to observe the dehydration of gypsum. For these times, models dried in a microwave oven presented little resistance to compression, fracturing easily with very low charge, justifying the two-hour period chosen before microwave drying samples for the testing of compressive strength.

Detail reproduction: The quality of detail reproduction of the metal matrix may have been influenced by the smaller size and regularity of the particles of calcium sulfate α- hemihydrate between casts. The Elite Rock and Shera Premium brands proved consistently less viscous during tapping, compared with Durone IV, which could have influenced the flow of material on the metal matrix and allow easier penetration of the gypsum and consequently better reproduction of the 0.050 mm line.

Due to the lack of further research in the literature about the reproduction of details in casts that are microwave oven dried, it is not possible to establish parameters for comparison with the results of this study. However, the results of dimensional alteration and compressive strength were consistent with the few studies reported in the literature [7,15,16], also demonstrating the influence of microwave oven drying on the dehydration of gypsum type IV. The comparison of the results of dimensional alteration and compressive strength of the marks cannot be discussed given that the manufacturers did not provide the complete chemical composition of their products.

However, the results of this study support the view that microwave drying method can be used without causing deleterious effects on the linear dimensional alteration, resistance to compression and reproduction of detail in models of type IV gypsum, when compared to drying of the material at room temperature.

5. CONCLUSION

Within the limitations of the study it was concluded that:

• The microwave oven dried dental stone showed linear dimensional alteration that was similar to that observed after laboratory temperature drying for 24 hours and 7 days.

• When analyzing the stone-drying method interaction, the compressive strength of models dried in a microwave oven was similar to those of samples dried at laboratory temperature for 24 hours, with the exception of Shera Premium gypsum, which demonstrated similar results following microwave and laboratory temperature drying for 7 days

• The microwave drying method reproduced similar levels of detail to those observed for samples dried at laboratory temperature for 24 hours and 7 days with the exception of Durone IV.

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CAPÍTULO 2

Influence of microwave oven power level on the linear dimensional change and compressive strength of dental gypsum type IV

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Objective: To investigate the influence of the power level of a microwave oven on the linear dimensional change and compressive strength of dental gypsum type IV.
Materials and Methods: Three brands of gypsum type IV were selected: Elite Rock, Shera Premium and Durone IV. Six different drying methods were used: G1-laboratory temperature (25 ± 4 °C) after 2 hours, G2-temperature laboratory for 24 hours, G3-temperature laboratory for 7 days; G4-low microwave power (200 W) for 5 minutes; G5-average microwave power (400 W) for 5 minutes, G6-high microwave power (800 W) for 5 minutes. Data were subjected to ANOVA-two-factors and Tukey test at a 5% level of significance.
Results: The values of dimensional change for the Elite Rock cast were statistically similar for the G3 and G6, G4, G5 and G2 groups. Gypsum Shera Premium demonstrated higher levels of expansion in G1 compared to G2, G3, G4, G5 and G6. For Durone IV, G5 presented the lowest values of dimensional expansion. For the three brands used, the techniques of drying in a microwave oven with a power of 200 W and drying at laboratory temperature for 7 days presented the highest compressive strength, although not statistically significantly higher than that of the other techniques. The exceptions were for Shera Premium and Durone IV, which produced similar values at microwave powers of 800 W and 400W, respectively.
Conclusion: The microwave power levels of 200 W to 800 W produced similar levels of dimensional change in the dehydration temperature of the laboratory after 24 hours and 7 days.

Keywords: dental stone, microwave, dimensional change, compressive strength.

1. INTRODUCTION

Dental stone is widely used in Dentistry and Prosthodontics and the success of indirect restorations is related not only to the procedures of molding, but also to the development of models with the highest fidelity to the molded structures. Due to superior mechanical properties compared to ordinary gypsum, gypsum type IV (high-strength dental stone) is the most suitable for the manufacture of dies and work patterns due to its high strength, hardness and expansion[1].

The mechanical properties of gypsum products are related to the water/powder ratio, mixing time, mixing volume, chemical composition, relative humidity, and room temperature in which the material is stored and the time elapsed after the mold is cast [2]. For adequate use of stone models, it is necessary to wait 24 to 48 hours for the remaining water contained in the mass[3] to be eliminated to promote resistance and sufficient surface hardness[4].

Microwave radiation is a widely-accepted technique in dentistry for disinfection [5, 6] polymerization of acrylic resins and wax removal from molds [7]. In previous studies, researchers have related the drying of stone models in conventional ovens and microwave ovens as an alternative means to reduce the waiting time before use [4.8]. However, gypsum is stable only below 40 °C and drying at higher temperatures should be handled with discretion. At high temperatures the evaporation of water in the remaining mixture can cause shrinkage, reduction of mass resistance[9] and cracking of the models, reducing their mechanical properties [10].

The optimal level of drying and radiation power has been analyzed in some studies that present conflicting results. Thus, some authors[11] believe that high power microwave radiation is most recommended; however, another study reports that low power may be more suitable for drying plaster models [12].

Given these considerations, this study aimed to investigate the influence of different power levels of microwave radiation on the linear dimensional change and

compressive strength of dental stone type IV. The study hypothesis is that different powers of microwave radiation used for drying promote different effects on the dimensional changes and compressive strength of gypsum type IV.

2. MATERIALS AND METHODS

Three type IV dental stone brands were selected; trademarks Durone IV, Elite Rock and Shera Premium, as listed in Table 1.

Table 1- Product, manufacturer, water/powder ratio and batch number for dental stones employed in the study.

Product	Manufacturer	Water/Powder	Batch No.
Durone IV	Dentsply, Petrópolis, Rio de Janeiro, Brazil	19 mL/100 g	635041
Elite Rock	Zhermack, Rovigo, Italy	20 mL/100 g	U110651/B
Shera Premium	Shera Werkstoff-Tecnologie, Lemforde, Germany	20 mL/100 g	92864

Six different drying methods were used: G1- laboratory temperature (25 ± 4°C) for 2 hours, G2- laboratory temperature for 24 hours, G3- laboratory temperature for 7 days; G4-low microwave power (200 W) for 5 minutes; G5-average microwave power (400 W) for 5 minutes, G6-high microwave power (800 W) for 5 minutes. The microwave oven used a programmed maximum power of 800 W and

frequency of 2450 MHz (Panasonic NN7809 B, Manaus, Amazonas, Brazil). The samples dried at laboratory temperature (25 ± 4°C) were used as controls. A cup with 200 mL of distilled water was placed in the microwave to protect the magnetron[13].

Linear dimensional change:

For the analysis of dimensional alteration, sixty samples were used (n = 10) for each stone brand, which was divided into four groups. In the present study a metallic matrix with two series of seven slots were used with depths ranging from 0.025 to 0.300 mm and a 2.5 mm range of distance between the grooves, made in accordance with rules established by specification no. 25 of the ADA[13]. A PVC pipe was placed (30 mm diameter and 15 mm height) on the metal matrix, so that the slot of 0.050 mm depth remained in the center of the ring diameter. The stone was weighed on an analytical balance (Sauter, model K1200, Switzerland) and mixed with deionized water and measured with a test tube, according to the proportions recommended by the manufacturers (Table 1) and manipulated mechanically by a vacuum (Multivac 4, Degussa, Germany) with a speed of 450 rpm for 30 seconds. After mixing, the stone was poured inside the ring, previously isolated with Vaseline under vibration. After 2 hours, samples were removed from the PVC pipe and the surface recorded using a scanner (HP Scanjet 2410, São Paulo, Brazil) with a resolution of 1200 dpi and analyzed by ImageTool 3.0 software. For image calibration the distance between lines 1 and 2 was used, comprising the range of 2.5 mm distance between them (Figure 1).

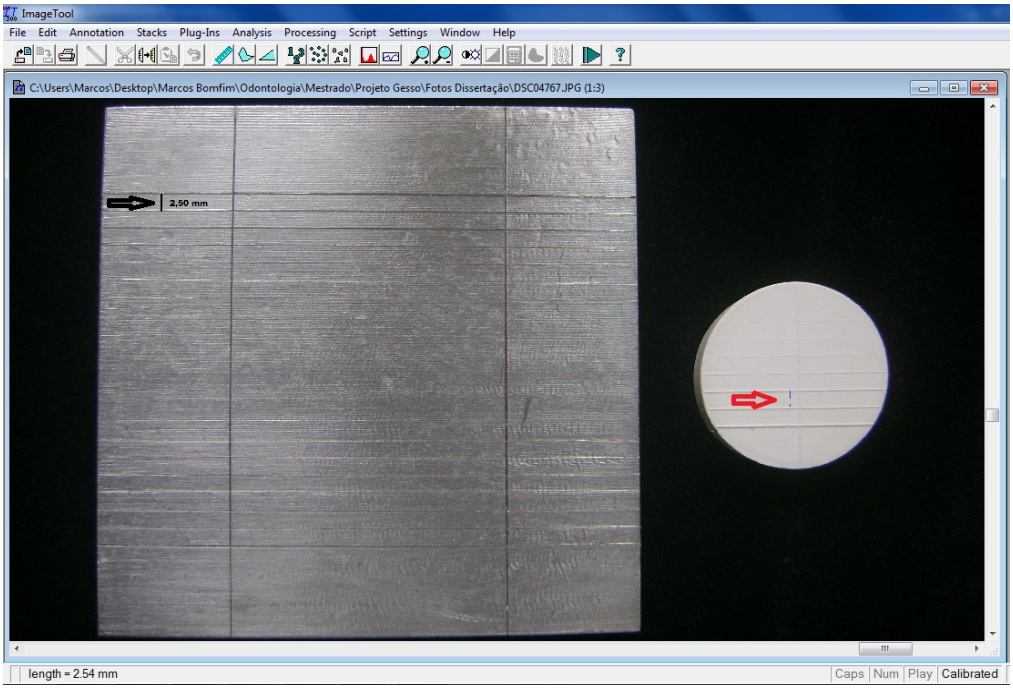

Figure 1 - Sample for analysis of dimensional alteration in software. Black line indicates calibrated scale red line and the measurement of the sample.

Compressive strength:

Sixty specimens were prepared for each brand and divided into six groups (n = 10). Specification No. 25 of the American Dental Association (ADA) [14] recommends testing of compressive strength using samples 40 mm in length and 20 mm in diameter. However, in the present study, we used metal molds of 20 mm in length and 10 mm in diameter (Figure 2), keeping the length-diameter ratio recommended by ADA[14]. The metal molds were used to obtain the silicon die polymerized by condensation (Optosil Comfort, Heraus Kulzer, Hanau, Germany), in which the samples were made of stone.

The stone was weighed on an analytical balance (Sauter, model K1200,

Switzerland) and mixed with deionized water measured with a test tube, according to the proportions recommended by the manufacturers (Table 1) and manipulated mechanically by vacuum (Multivac 4, Degussa, Germany) with a speed of 450 rpm for 30 seconds. The silicone molds were placed on a glass plate and filled with stone with the aid of a mechanical vibrator (Vibrator GC, MEG Chemical Co. Ltd.). After filling the mold, another glass plate was placed over the stone so that the ends of the samples remained smooth and flat.

Figure 2 - Sample used in the testing of compressive strength

The samples were tested in a universal testing machine (Instron 4411, Corona, CA, USA) operating with a cell load of 5 kN with a displacement speed of 0.5 mm / min until failure.

Statistical analysis:

The results for the linear dimensional alteration and compressive strength were subjected to analysis of variance (ANOVA, two-factors) and the means with Tukey test at a 5% level of significance.

3. RESULTS

Analysis of variance for testing linear dimensional changes showed that the stone ($p = 0.00006$) and time ($p = 0.00001$) were significant factors in these parameters and showed a stone/time interaction ($p = 0.07401$). The data of the analysis of variance are shown in Table 2.

Table 2 - Analysis of variance.

	Df	Sum of square	Mean square	F value	P value
Stone	2	0.00189	0.00094	12.49	0.00006
Time	5	0.01189	0.00237	31.31	0.00001
Stone x Time	10	0.00132	0.00013	1.74	0.07401
Error	162	0.01230	0.00007		

The mean results of linear dimensional changes of dental stone are listed in Table 3. Gypsum Elite showed no statistically significant differences between G2, G4 and G5. No significant difference was observed between G6 and G3. Group 1 presented a higher dimensional expansion. Gypsum Shera Premium demonstrated significant statistical difference between G1 and G2 to G6. For Durone IV, no statistically significant differences were found in the dimensional change between G3, G4 and G6. No significant difference was observed between G1 and G2 with G5 (which demonstrated lower value). Comparing the results among the trademarks tested, G1 did not demonstrate any significant difference. For G2, Durone IV demonstrated higher levels of expansion in relation to the dimensional change of Shera Premium and Elite Rock. For G4 and G5 IV, no statistically significant difference in the values of dimensional change was seen.

Table 3 - Mean linear dimensional alteration (mm), standard deviation and percentage of dimensional alteration due to the different drying methods.

Excluído: ¶

Stone	G1	G2	G3	G4	G5	G6
Elite Rock	2.535±0.010aA 1.4%	2.520±0.006aB 0.8%	2.504±0.006bC 0.16%	2.509±0.005aC 0.36%	2.513±0.009aB 0.52%	2.506±0.009bC 0.24%
Shera Premium	2.531±0.007aA 1.24%	2.514±0.006bB 0.56%	2.510±0.004aB 0.40%	2.509±0.007aB 0.36%	2.513±0.006aB 0.52%	2.505±0.010bB 0.20%
Durone IV	2.532±0.009aA 1.28%	2.526±0.010aA 1.04%	2.518±0.012aB 0.72%	2.518±0.012aB 0.72%	2.512±0.006aC 0.48%	2.517±0.008aB 0.68%

Means followed by small letters in each column differ and means followed by capital letters in a line differ in each row between them, Tukey test (5%).

Analysis of variance for testing the compressive strength showed that the stone (p = 0.00001) and time (p = 0.00001) were significant factors and that there was a stone-time interaction (p = 0.00314). The data for the analysis of variance are shown in Table 4.

Table 4 - Analysis of variance.

	Df	Sum of squares	Mean square	F value	P value
Stone	2	4536.59	2268.29	75.64	0.00001
Time	5	14176.97	2835.39	94.55	0.00001
Stone x Time	10	1285.08	128.50	4.28	0.00314
Error	162	4857.85	29.98		

The mean compressive strength (MPa) and standard deviation as a function of the interaction and stone drying method are listed in Table 5. For the Elite Rock stone, G3 and G4 promoted slightly higher resistance values that were not statistically significantly higher than for the other methods, whereas G1

demonstrated the lowest strength. G2 and G6 did not demonstrate any statistically significant difference in resistance values. G5 showed intermediate resistance values, relative to G4 and G6. For Shera Premium, higher statistical similarity was promoted by the G3, G4 and G6. G5 presented values similar to those of G2. For Durone IV, no statistical difference was observed between G3, G4 and G5. G6 had lower strength values for G4 and G5. When comparing the brands tested, Elite Rock gypsum had the highest resistance to compression, with the exception of G6, producing a similar result for Shera Premium.

Table 5 - Mean values of compressive strength (MPa) and standard deviation as a function of the interaction of drying method and type of stone.

Drying Method	Dental stone		
	Elite Rock	Shera Premium	Durone IV
G1	31.87±3.99 dA	20.65±4.09 cB	16.87±2.43 dB
G2	48.24±2.86 cA	37.78±7.32 bB	40.16±7.18 bB
G3	60.25±1.96 aA	45.03±8.19 aB	47.38±6.95 aB
G4	55.58±4.98 aA	47.58±5.76 aB	48.07±6.07 aB
G5	50.66±5.37 bA	37.68±7.31 bB	42.80±4.12 aB
G6	45.30±4.00 cA	42.58±5.83 aA	34.60±4.77 cB

Means followed by different small letters in each column and different capital letters in each row differ statistically by Tukey test (5%).

4. DISCUSSION

This study aimed to analyze which strength of microwave radiation is most suitable for the dehydration of dental stone type IV and also assess the influence of power on the linear dimensional change and compressive strength of dental stone. The study hypothesis that different powers of microwave radiation used for drying would promote different effects on the dimensional changes and compressive strength of dental stone type IV was partially accepted.

Dimensional change: The dimensional changes of dental plaster can significantly affect the laboratory applications of gypsum in dentistry. The specification nº 25 ADA [14] allows for a type IV plaster setting expansion of up to 0.1% in 2 hours. In general, the expansions of samples obtained in this study for specimens submitted to drying methods in a microwave oven and at laboratory temperature for three different brands of stone were higher than those advertised on the label of the material by manufacturers. The difference in expansion observed and that advertised on the label of the product by the manufacturer may be explained by environmental conditions in the laboratory sample preparation, i.e. samples were prepared in a room without any control for temperature and humidity, with the intention of establishing similarity to the conditions employed by private clinics or prosthetic laboratories.

In this study, the level of linear dimensional changes in samples submitted to the powers of 200 W to 800 W presented positive results in relation to dimensional changes following laboratory temperature drying for 24 hours (G2) and 7 days (G3). Samples that were dried in the microwave, independently of the radiation power used showed a lower level of dimensional expansion, in comparison with specimens that were dried at laboratory temperature for 2 hours.

The values of compressive strength of the samples that were dried in a microwave oven with a power of 200 W (G4) showed values similar to those dried at laboratory room temperature for 7 days (G3), with the exceptions of the Shera Premium and Durone IV brands, which also demonstrated similar values for 800 and 400 W respectively. These data suggest that the low radiation power output allows slower water dispersion in the dough, without affecting the physical properties of the stone. Tuncer et. Al [10] reported that microwave ovens should not be used to dehydrate models that are extremely damp or wet, due to the rapid boiling of free water and the formation of cracks in the stone. In the same study, the authors concluded that the use of low power improves the resistance to compression found at high power, corroborating our findings.

In a similar study, Luebke and Schneider [15] drew the same conclusion, in

a study that employed eight different brands of dental stone that were dried in a microwave oven for 5 minutes with a maximum power output of 1450 W. For G6, Durone IV presented compressive strength values that were statistically lower than those of Elite Rock and Shera Premium. Some cracks or fissures were easily visible on the surface of the Durone IV specimen, probably causing the reduction in strength compared to the other brands. When comparing the results for compressive strength, a comparison of the components of the products is desirable; however manufacturers did not provide the complete chemical composition of their products.

Despite the change in the size of the specimens, as determined by the ADA[14], the size ratio for the compressive strength test results was maintained positively compared to samples that were conventionally dried at room temperature. Results were considered positive, demonstrating that there is no need to wait for 24 and 48 hours for plaster models to dry, rather a dehydration in a microwave oven may be used to save time.

5. CONCLUSION

Microwave radiation at powers of 200 W to 800 W provided similar levels of dimensional change to that observed following dehydration at laboratory temperature for 24 hours and 7 days. The power of 200 W produces higher values of compressive strength for the Elite Rock, in contrast to Durone IV and Shera Premium , which produce similar results using 200 W to those found for 400 W and 800 W, respectively.

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CONSIDERAÇÕES GERAIS

A especificação n° 25 da *Associação Dentária Americana* (ADA), estabelece cinco classificações para os gessos odontológicos, sendo: gesso comum para moldagem (Tipo I), gesso comum para modelo (Tipo II), gesso pedra (Tipo III), gesso pedra de alta resistência (Tipo IV) e gesso pedra de alta resistência e alta expansão (Tipo V). Em nosso estudo foi utilizado o gesso Tipo IV haja vista que é o material utilizado para vazamento de modelos que necessitam de alta precisão para execução de procedimentos odontológicos indiretos.

Neste trabalho, o objetivo do Capítulo 1 foi avaliar resistência à compressão, estabilidade dimensional e reprodução de detalhes de gessos odontológicos tipo IV desidratados em temperatura de laboratório e em forno de microondas e para o Capítulo 2 avaliar qual nível de potência de radiação seria o mais indicado para desidratação de modelos de gesso. A hipótese do trabalho que a desidratação de modelos de gesso em forno de microondas não causaria influência negativa nas propriedades físicas dos gessos odontológicos foi parcialmente aceita.

No Capítulo 1 os resultados mostraram que a secagem com microondas dos gessos depois de 2 horas de manipulados proporcionou maiores valores de resistência à compressão em relação aos gessos com secagem em temperatura de laboratório por 2 horas. A estabilidade dimensional e a reprodução de detalhes das amostras secas com microondas foram similares às amostras secas em temperatura de laboratório após 24 horas e 7 dias. No capitulo 1, foram discutidos os resultados, sendo sugerido que os modelos secos em forno de microonda apresentam melhorias nas propriedades analisadas neste estudo. Os resultados descritos na (Tabela 2 e 4) sugerem os modelos de gesso poderiam ser utilizados para confecção de trabalhos protéticos após 2 horas do preenchimento do molde.

Resultados similares ao do capitulo 1 foram encontrados no estudo de (Tuncer *et al*. 1993) o qual avaliaram o uso de potência de radiação alta e baixa em períodos de 2, 4, 24 e 48 horas.

O objetivo do Capítulo 2 deste estudo foi avaliar a influência de diferentes níveis de potência de radiação de microondas sobre as propriedades físicas de gessos odontológicos tipo IV. Os valores de resistência à compressão para os gessos estudados secos em forno de microondas com potência de 200 a 800 W (G4 e G6) foram significativamente maiores que o grupo seco em temperatura ambiente por 2 horas, no entanto espécimes secos após 2 horas com potência de 200 W apresentam valores semelhantes aos secos em temperatura ambiente no período de 7 dias. O uso de baixa potência de radiação permite uma saída mais lenta da água contida na massa sem afetar as propriedades físicas do gesso sugerindo ser a mais indicada para desidratação de espécimes de gesso. Resultados esses confirmado com estudo de (Luebke e Schneider, 1985).

Em relação à análise alteração dimensional linear dos capítulos 1 e 2 os resultados mostraram que o uso de forno de microondas influenciou de forma positiva comparado aos espécimes desisdratados em temperatura de laboratório no período de 24 horas e 7 dias. Nos dois estudos os valores de alteração dimensional foram maiores que o anunciado nos rótulos dos produtos, fato este justificado devido às condições ambientais do laboratório quando do preparo das amostras em sala sem temperatura e umidade controladas, com a intenção de estabelecer similaridade com as condições clínica ou do laboratório protético.

Quanto a análise descritiva de reprodução de detalhes descritas no capitulo 1 (Gráfico 1) apenas em forma de percentagem mostrou valores semelhantes para os gessos secos em forno de microondas e para os espécimes desidratados em temperatura de laboratório no período de 24 horas e 7 dias

CONCLUSÃO GERAL

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

1. Os gessos desidratados em forno de microondas mostraram alteração dimensional linear similar aos desidratados em temperatura de laboratório após 24 horas e 7 dias.
2. As potências de 200 W a 800W influenciaram de forma positiva a alteração dimensional, produzindo valores similares aos secos em temperatura de laboratório após 24 horas e 7 dias
3. Na interação gesso-método de secagem, para o Capítulo 1, a resistência à compressão para os modelos desidratados em microondas foi semelhante aos desidratados em temperatura de laboratório após 24 hosras, exceto para o gesso Shera Premium que mostrou valores semelhantes aos secos em temperatura de laboratório após 7 dias.
4. A radiação por microondas com potência de 200 W proporcionou maior resistência à compressão aos gessos odontológicos tipo IV,exceto para Shera Premium e Durone IV que mostraram aumento da resistência com as potências de 800 e 400 W respectivamente.
5. O método de secagem em microondas reproduziu níveis de detalhes similares aos da secagem em temperatura ambiente em 24 horas e 7 dias, exceto, para o Durone IV.

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ANEXOS

Capítulo 1

Capítulo 2

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