J Indian Prosthodont Soc (Oct-Dec 2013) 13(4):525–530
DOI 10.1007/s13191-012-0183-0

ORIGINAL ARTICLE

Comparative Evaluation of Dimensional Accuracy and Tensile Strength of a Type IV Gypsum Using Microwave and Air Drying Methods

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Received: 16 June 2012 / Accepted: 27 September 2012 / Published online: 4 October 2012

Abstract To evaluate dimensional accuracy and tensile strength of a type IV gypsum product, at different time intervals, dried in air or a microwave oven. Eighty specimens prepared from a cylindrical mould were used for measuring tensile strength (group A). Twenty specimens from a master die mould were used for determining dimensional accuracy (group B). In group A, 40 specimens were dried in open air at room temperature (A1). The other 40 were removed after 30 min and air dried for 20 min. These were subjected to microwave oven drying for 5 min (A2). Ten specimens each were tested under diametral compression at each of the following time periods: 1, 2, 4 and 24 h after drying. In group B, ten specimens were dried in open air at room temperature (B1). Ten specimens were removed from the mould after 30 min and air dried for 20 min. These were then dried in a microwave oven for 5 min (B2). The data was statistically analyzed using students unpaired "*t*" test. At all time intervals, diametral tensile strength (DTS) values for specimens dried in microwave oven were significantly higher than for those dried in open air. There were no significant differences between the dimensional accuracy of the two groups. In this study, microwave oven drying had a positive effect on the DTS of a type IV gypsum and the microwave oven dried specimens were as accurate as the air dried specimens over the same time period.

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Keywords Dimensional accuracy · Diametral tensile strength · Type IV gypsum · Microwave drying

Introduction

Type IV gypsum has been the most widely used material in dentistry for producing casts and dies.

Popularity of type IV gypsum is attributed to its ease of use, relatively quick setting, and reasonable accuracy. However, there are several disadvantages to its use in the fabrication of casts and dies, for example, poor abrasion resistance, potential variability in fine detail reproduction, inadequate tensile strength, and need for a waiting period prior to initiation of laboratory procedures [1].

Generally the strength of gypsum products is related to the water/powder ratio, mixing time, volume of mixture, chemical composition, and elapsed time after the cast is poured [2].

Although it is advised by manufacturers to wait for 24–48 h before manipulating gypsum casts/dies, dentists often find it necessary to work with casts/dies soon after they are poured. These "new" or "water soaked casts" have inadequate strength and surface hardness to be manipulated without damage [3]. In addition, eliminating excess water prior to manipulation is necessary as wet stone tends to clog up and reduce the cutting efficiency of instruments [1].

In dentistry, microwave radiation has been used for sterilization [4] and disinfection [5, 6], polymerization of acrylic resins [7, 8], processing for denture relines, repairs and rebases [9], and fabrication of provisional facial prostheses [10]. Drying type IV gypsum in a microwave can save considerable time. However, there is little research on the tensile strength and dimensional accuracy of type IV gypsum dried in this manner.

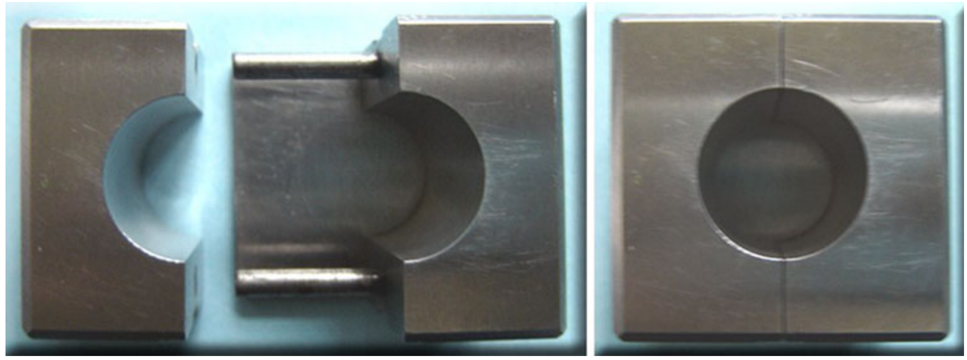

Fig. 1 Master metal mould for cylindrical specimens

The purpose of this study was to evaluate and compare the tensile strength and dimensional accuracy of a type IV gypsum product, at different time intervals, dried in either air or a microwave oven.

Objectives

This laboratory investigation was undertaken with the following objectives:

1 To evaluate the effect of microwave oven drying method on the tensile strength and dimensional accuracy of a type IV gypsum.
2 To evaluate and compare the effect of air drying and microwave oven drying method on the tensile strength of a type IV gypsum.
3 To determine the difference, if any, in the dimensional accuracy of dies dried in air or by microwave oven of a type IV gypsum.
4 To recommend a suitable method of drying type IV gypsum in order to save laboratory time.

Methodology

This study was conducted at the Department of Metallurgical and Material sciences, NITK, Surathkal. For measuring tensile strength, cylindrical specimens of 40 × 20 mm were obtained from a master mould of stainless steel. The master metal mould was prepared in two halves, wherein one of the halves could be slided manually on a 3 mm thickness base to approximate the other half to enclose a cylindrical cavity of 40 × 20 mm (Fig. 1).

For measuring dimensional accuracy, a master die was machined from stainless steel to a vertical dimension of 12 mm from the base of the master die to the occlusoaxial line. A 1.5 mm shoulder on the base was fabricated and a 5° convergence angle produced a 9 mm occlusal end diameter.

Two mutually perpendicular lines were drawn intersecting at the center of the occlusal surface. A circle (7 mm diameter) was scribed on the occlusal surface by using the intersection of the perpendicular lines as its center. The four points at which the circle intersected the perpendicular lines were used to record dimensions in the occlusal plane. One of these lines was referred to as dimension I. The other line drawn perpendicular to dimension I on the occlusal surface was referred to as dimension II.

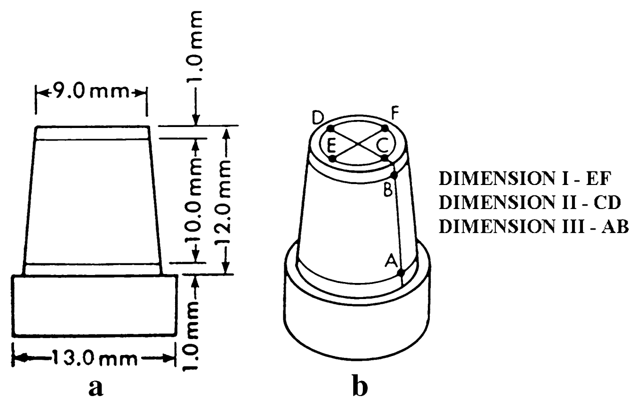

Fig. 2 Schematic representation of the master metal die

Two lines, each perpendicular to the long axis of the die, were scribed circumferentially on the surface of the axial wall of the die. One line was located 1 mm gingival from the occlusoaxial line angle and the other 1 mm occlusal from the axiogingival line angle.

An axial vertical reference line was scribed on the die joining the two circumferential reference lines. This axial vertical reference line between the circumferential reference lines was referred to as dimension III (Fig. 2a, b).

The die was finished and polished to provide a smooth and a shiny polished surface to obviate any adhesion of the polyvinyl siloxane impression material to the surface of the metallic die.

Fabrication of the Impression Tray

The master metal die was fixed on a base having elevated margins at its periphery, which acted as sleeves. These sleeves aided in guiding and stabilizing the custom

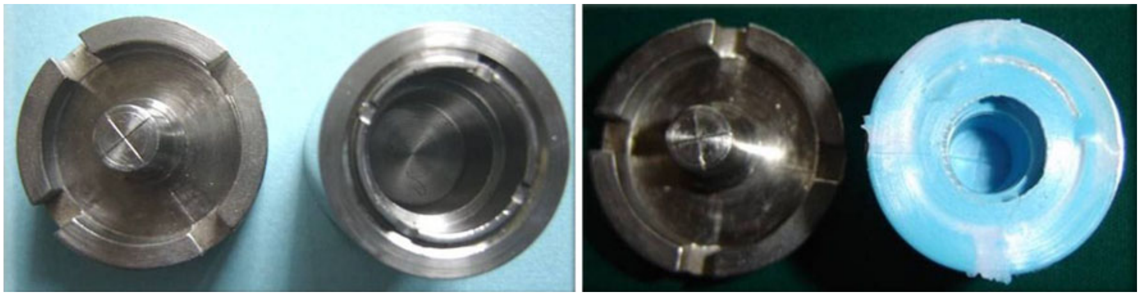

Fig. 3 Master metal die, impression tray and corresponding PVS impression

impression tray while making impressions of the die. Four escape ways were made on the sleeves of the base to facilitate the flow of excess impression material out, while making the impression.

In order to make impressions of the master die, a custom impression tray was machined from a stainless steel rod. The custom impression tray was designed to provide a uniform space of 3 mm around the master die. The open end of the custom impression tray had four grooves which were to be aligned along the escape ways of the base, in order to facilitate easy flow of excess impression material out and to establish a metal to metal contact (Fig 3).

Methodology

A total of 100 specimens were prepared from a type IV gypsum, namely Kalrock (Kalabhai Karsan, India).

Eighty cylindrical specimens ($n = 80$) were used for measuring the tensile strength (group A). Twenty die specimens were used for determining the dimensional accuracy (group B).

In group A, half of the specimens ($n = 40$) were dried in open air at room temperature (control group) (A1).

The other half ($n = 40$) were removed from the mould after 30 min and were later left for another 20 min in open air. These specimens were then coded. These were then subjected to microwave oven drying for 5 min (test group) (A2).

Within these groups (A1) and (A2), ten specimens each were tested under diametral compression at each of the following time periods: 1, 2, 4 and 24 h after drying.

In group B, half of the specimens ($n = 10$) were dried in open air at room temperature (control group) (B1).

The other half ($n = 10$) were removed from the mould after 30 min and left for 20 min for further drying in open air. These specimens were then coded. These were then dried in a microwave oven for 5 min (test group) (B2).

These groups (B1) and (B2) were used to determine dimensional accuracy.

The microwave used in this study, (BPL SANYO-BMO 700 T) had an output power of 1,200 W and a frequency of 2,450 MHz. The samples were dried at 50 % of the output power (600 W) for 5 min. A container with 500 ml of water was placed in the microwave oven as a heat sink, to protect the magnetron from excess energy.

Fabrication of Specimens

The powder was weighed to 0.001 mg using an electronic balance (Contech) and mixed with distilled water dispensed by a measuring cylinder using a standard water powder ratio of 0.23 as specified by the manufacturer. Type IV gypsum was initially hand mixed using a rubber bowl and spatula for 45 s and then a mechanical vacuum mixer (Multivac® 4, Degussa) was used for 30 s to ensure a homogenous, bubble free mix. For group A cylindrical specimens, the mixture was vibrated into the mould, and a glass slab was placed over the mould to ensure flat and parallel ends.

For group B die specimens, impressions of the master die were made with soft putty polyvinyl siloxane impression material (Aquasil™ soft putty, Dentsply, DeTrey) in the custom impression tray with light body polyvinyl siloxane impression material (Aquasil™ UltraLV, Dentsply, caulk) syringed onto the master die. Tray adhesive (caulk tray adhesive, Dentsply) was used to retain the impression material on the custom impression tray provided by the manufacturer.

Type IV gypsum mixture was vibrated, painted on the entire impression surface with a brush and then the remaining mixture was poured into the impression.

Checking for Tensile Strength

In this study, the diametral tensile strength (DTS) of the specimens was investigated through a diametral compression test or an indirect tensile test. The DTS tests were made on Instron machine (model 4206) at a crosshead speed of 0.1 cm/min.

A compressive load was applied on the curved surface of the cylindrical specimen placed between two flat and parallel metallic jigs. The fracture of the specimen i.e. ideal fragmentation into two segments (Fig. 4) which is

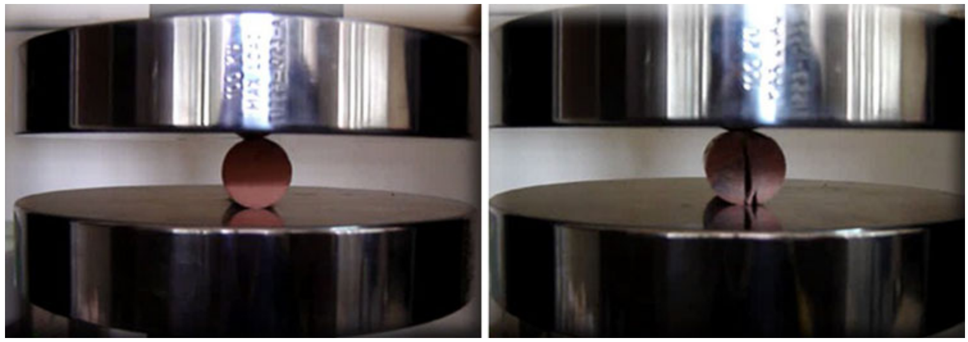

Fig. 4 Positioning and fracture of specimens in the Instron machine

characteristic of diametral compression tensile test was noted and the load values were recorded in kilo-Newton. DTS was recorded in MPa and computed according to the formula:

DTS $= 2P/\pi$dt ($P =$ load, $D =$ diameter, and $T =$ thickness).

The results were subjected to statistical analysis using students unpaired "t" test.

Measurements for Dimensional Accuracy

To compare the dimensional accuracy of type IV gypsum, dimensions I, II and III on each model were measured in an ordered sequence to evaluate the mean. All the measurements were recorded by one investigator. A digital micrometer (Mitutoyo, Japan) was used to measure the dimensions on each model, upto an accuracy of 0.001 mm, aided by the two ends of a vernier caliper which were used to orient to the points to be measured.

The mean of each dimension, measured three times on the master die, was used as the basis for determining the percent relative changes in that dimension of each sample according to the formula:

$$\text{Percent relative change}_t = \frac{\text{Dimension } i \text{ sample} - \text{Dimension } i \text{ masterdie} \times 100}{\text{Dimension } i \text{ master die}}$$

where i refers to the dimension number.

The measurements were then statically analyzed using students unpaired "t" test to compare the percentage relative change from the master die between air and microwave dried specimens at different time intervals.

Results

The comparison of DTS values between air dried and microwave oven dried specimens at 1, 2, 4 and 24 h are presented in Table 1. The values indicate a statistically significant increased DTS for microwave oven dried specimens at all time intervals as compared to their air dried counterpart specimens ($p < 0.001$ vhs). The DTS values show that microwave oven dried specimens at 4 h are as strong as air dried samples at 24 h ($p = 0.961$).

Table 1 Comparison of the mean DTS values (MPa) of air and microwave dried specimens

Group	Time (h)	N	Mean	SD	t
Air	1	10	2.46250	0.133193	8.340
MW		10	2.98670	0.147518	$p < 0.001$ vhs
Air	2	10	2.65710	0.137041	15.282
MW		10	3.42480	0.080357	$p < 0.001$ vhs
Air	4	10	3.41260	0.052855	41.710
MW		10	5.04460	0.111875	$p < 0.001$ vhs
Air	24	10	5.04220	0.105860	15.014
MW		10	6.48440	0.284714	$p < 0.001$ vhs

Table 2 shows students unpaired "t" test for comparing percentage relative change values from the master die between air and microwave oven dried samples at the same time intervals of 1, 2, 4 and 24 h. All p values were statistically not significant.

Discussion

Cast and die materials must reproduce an impression accurately and remain dimensionally stable under normal conditions of use and storage. Not only should the cast be accurate, but it should also satisfactorily reproduce fine detail and have a smooth, hard surface. Such an accurate cast or die must also be strong and durable and withstand the subsequent manipulative procedures without fracture [11].

Though type IV gypsum products continue to be the most popular die materials, important properties of tensile strength and dimensional accuracy need to be evaluated.

The strength of gypsum products is generally expressed in terms of compressive strength, although tensile strength should also be considered in order to secure a satisfactory guide to the total strength characteristics [12]. Thus tensile strength is a property of significance.

Table 2 Comparison of the mean percentage relative change from the master die of air dried and microwave oven dried specimens at different time intervals

Group	Time (h)	Dimension	N	Mean	SD	t
Air	1	I	10	0.096207	0.0368607	0.157000
MW			10	0.098996	0.0423171	$p = 0.877$ ns
Air	2		10	0.100390	0.0346558	0.112000
MW			10	0.101785	0.0186488	$p = 0.912$ ns
Air	4		10	0.101785	0.0218492	0.126000
MW			10	0.103179	0.0272595	$p = 0.901$ ns
Air	24		10	0.103179	0.0209920	0.152000
MW			10	0.104573	0.0199905	$p = 0.881$ ns
Air	1	II	10	0.090618	0.0188764	0.152000
MW			10	0.092012	0.0219939	$p = 0.881$ ns
Air	2		10	0.090618	0.0188764	0.152000
MW			10	0.092012	0.0219939	$p = 0.881$ ns
Air	4		10	0.092012	0.0209891	0.000000
MW			10	0.092012	0.0209891	$p = 1$ ns
Air	24		10	0.094800	0.0261229	0.000000
MW			10	0.094800	0.0183544	$p = 1$ ns
Air	1	III	10	0.059088	0.0393103	0.053000
MW			10	0.060026	0.0400858	$p = 0.958$ ns
Air	2		10	0.060026	0.0065579	1.555000
MW			10	0.064716	0.0069205	$p = 0.137$ ns
Air	4		10	0.072219	0.0117395	0.661000
MW			10	0.075971	0.0135916	$p = 0.517$ ns
Air	24		10	0.090039	0.0166609	0.493000
MW			10	0.092853	0.0069205	$p = 0.628$ ns

Tensile strength is generally determined by a uniaxial tension test. Since such a test is difficult to perform for brittle materials, another test called as the diametral compression test has become popular. In this method a compressive load is applied by a flat plate against the side of a cylindrical specimen. The vertical compressive force along the side of the specimen produces a tensile stress that is perpendicular to the vertical plane that passes through the center of the specimen. Fracture occurs along this vertical plane. The ideal fracture of specimens into two fragments suggests the reliability of the test. In such a situation, the tensile stress is directly proportional to the compressive load applied. It is computed by the formula: tensile strength $= 2P/\pi$dt. (P = load, d = diameter and t is the thickness) [13]. Gypsum being a brittle material [14], this test was chosen to determine the tensile strength.

Gypsum die materials have been reported to exhibit a setting expansion of 0.01–0.1 %. This minimal expansion has been said to compensate for the dimensional changes inherent in the fabrication process of an indirect restoration [15].

The dental stones are compatible with all impression materials [16]. In this study, PVS impression material was used to procure moulds, obtained after making an impression of a master metal die, for fabrication of dies made of the type IV gypsum under investigation for determining dimensional accuracy.

When time is a serious consideration, it is desirable to work with gypsum casts soon after separation from the impressions. Since wet casts usually have inadequate strength and surface hardness, dentists normally must wait for 24–48 h before manipulating gypsum casts [3]. To save time, drying by microwave oven has been suggested [16].

Microwaves are electromagnetic waves produced by a generator called a magnetron. Domestic microwave ovens use a frequency of 2,450 MHz which gives a wavelength of about 12 cm [7].

A study compared the DTS of microwave and oven dried investment materials. The microwave used in the study had an output power of 600 W. It was concluded that microwave drying of type III investment materials at 600 W for 10 min was, apart from strengthening the material, time saving for the dental laboratory [15].

In another study, which evaluated the DTS of five type IV gypsum at different time intervals using microwave and air drying methods, it was found that at all time intervals, the DTS values for microwave dried specimens were significantly higher than air dried specimens [3].

In accordance with the aforementioned studies, in the present in vitro study, drying type IV gypsum in a microwave oven had a positive effect on the DTS of Kalrock. Tensile strength of microwave dried specimens was more than air dried specimen at all time intervals (Table 1). This may be due to the loss of excess water by microwave drying. A good explanation to this effect may be that as the last traces of water leave, fine crystals of gypsum precipitate. These anchor the larger crystals which causes an increase in strength [12].

The setting reaction of gypsum causes a decrease in the volume of the reactants early in the setting process when the mix is fluid. However, once the mix becomes rigid, an expansion is observed that results from growth pressure of the gypsum crystals that form [1].

Type IV gypsum show an expansion of 0.01–0.1 %. Typically over 75 % of the expansion observed at 24 h occurs during the first h of setting [15]. Both air and microwave dried specimens in this study showed a mean percentage relative change from the master die. (0.1 %-maximum).

The dimensional changes of microwave dried specimens were not significant when compared to the air dried specimens at all time intervals.

Conclusion

This study supports the use of microwave oven for rapid drying of type IV gypsum. This method not only saves time, but also makes it possible to obtain a stronger specimen that is more resistant to damage during normal laboratory handling.

This study has supplied some preliminary information about the use of microwave oven in drying a type IV gypsum. However, follow up testing must be performed on: the optimum length of microwave drying, different microwave programmes for drying, effect of microwave oven drying on other properties, ideal time of microwave oven drying after mixing dental stone, and the effect on various other gypsum products.

Within the limitations of this in vitro study, the following conclusions can be drawn:

1 Drying type IV gypsum, namely Kalrock, for 5 min in a microwave oven at 600 W produced specimens that demonstrated stronger DTS values than air dried specimens over the same time period at all time intervals
2 Differences in the dimensional changes for microwave oven dried die specimens and air dried specimens were not statistically significant over the same time period
3 Microwave dried specimens at 4 h were as strong as 24 h air dried specimens
4 There was no statistical difference in the percentage relative change between the microwave oven dried specimens at 4 h and the 24 h air dried specimens
5 However, 24 h air dried samples were stronger than microwave oven dried specimens at 2 h and there was a statistically significant difference in percentage relative change in dimension III between the dies dried at these time intervals
6 Microwave radiation at 600 W for 5 min is acceptable for drying type IV gypsum used in the study

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