

Comparative evaluation of diametral tensile strength of phosphate-bonded investment (ringless) material by using air-drying method, conventional hot air oven, microwave oven, and combination of microwave and conventional oven: An *in vitro* study

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Abstract

Aim: This study evaluated and compared the diametral tensile strength (DTS) of phosphate-bonded investment (PBI) material (ringless) used in removable cast partial denture fabrication.

Settings and Design: Comparative - *In vitro* study.

Materials and Methods: The PBI material, Wirovest used in this study was subjected to four different drying methods. A total of 80 specimens were prepared in a cylindrical form; 20 specimens were air dried for 2 h, 20 specimens were dried in a conventional oven at 230°C for 1 h, 20 specimens were dried in microwave oven at 600 W for 10 min, and remaining 20 specimens were dried first in microwave oven and then in conventional oven. The dried specimens were tested at 2-h interval for diametral compression at a crosshead speed of 0.5 cm/min.

Statistical Analysis Used: Pairwise analysis.

Results: The microwave drying technique and drying by combination method resulted in greater DTS, respectively.

Conclusion: Within the limitation of this study, PBI specimens dried in microwave oven at 600W for 10 min increased the diametral strength and are also a time-saving procedure.

Keywords: Conventional oven drying, diametral tensile strength, microwave oven drying, phosphate-bonded investment material

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INTRODUCTION

Prosthodontics deals with the restoration and maintenance of oral function, comfort, appearance and health of the patient by the restoration of natural teeth and/or the

replacement of missing teeth and craniofacial tissues with artificial substitutes. Missing teeth can be replaced by a fixed or removable prosthesis. There are certain clinical

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situations, where implants or fixed partial dentures are contraindicated, due to cost, time, local, and systemic conditions; the role of cast partial dentures comes in play.

The designing and fabrication of a cast partial denture is a very technique sensitive and meticulous procedure in which the clinical, as well as laboratory procedures, hold equal importance. The success of laboratory procedures depends on the quality of investment materials which should withstand the loads during casting procedures without fracture to achieve accurately fitting of dental prosthesis. The rapid growth and increased use of higher melting alloys have resulted in increased use of phosphate-bonded investment (PBI) material.^[1]

PBI like other investments materials are brittle and may fracture at clamping points due to stress concentrations. Evaluating their performance test at working temperature provides the most accurate information about the strength of these materials. Diametral tensile strength (DTS) test method is recommended for brittle materials.

In dental laboratories, it is necessary to work on refractory casts soon after separating them from reversible hydrocolloid. Therefore to ensure it has adequate strength and sufficient surface hardness, it is necessary that it is moisture free. This also prevents distortion of the cast.^[2]

Drying the refractory cast at room temperature is one of the oldest methods and presently also many of the laboratories are working on the same methodology. As this method is time-consuming, so to overcome this conventional oven drying method was used but drying in conventional oven results in drying only of the external surface of the cast which does not result in complete drying of the cast, this leads to inadequate strength to work on it. The microwave oven technique suggested in recent years offers considerable time-saving advantages for drying of investment molds.^[2,3] Microwave oven results in removal of water from the innermost core to external surface. However, complete loss of water not only causes damage to the magnetron of the microwave but also cracking of refractory cast due to excessive desiccation. Hence, drying can be done by the combination of microwave oven and conventional oven. As combination method uses more time at controlled temperature, the purpose of this study was to evaluate and compare the DTS of PBI materials dried by using conventional oven, microwave oven, and combination of microwave and conventional oven.

Inclusion criteria

1. Investment material should follow all the specifications (samples dimension and properties of

investment material) provided by the American Dental Association (ADA) number 126

2. Investment material sachet should be used within 1 month from the manufacturing date for the study.

Exclusion criteria

1. Investment material more than 1 month old from manufacturing date should not be included for sample testing.

MATERIALS AND METHODS

The study was approved by the Institutional Ethics Committee V.S.P.M Dental College and Research Centre, Nagpur, Maharashtra, on December 20, 2012. The ethical number was VSPM/IEC/19/2012.

As it was *in vitro* study, informed consent was not required.

All the materials used in the current study are depicted in Table 1.

According to the ADA specification number, 126 stainless steel cylindrical rods were machined to 20 mm in diameter and 40 mm in length to create space in rubber silicone mold to standardize the dimensions of the PBI material samples [Figures 1 and 2]. Silicone rubber was considered valid to facilitate the removal of the test samples without any damage.^[4]

All sachet of investment powder (one sachet of Wirovest - 400 g) were tumbled for 2–3 min in an 8 L plastic drum before use, in an attempt to overcome uneven particle size distribution. 200 g of powder was mixed in 30 ml of liquid for the fabrication of six samples. The wirovest (bego bremer, goldschlagerei wilh., herbst gmbh and co., germany) powder was weighed 0.1g on digital weighing machine, and begosol liquid (bego bremer, goldschlagerei wilh., herbst gmbh and co., germany) was measured to 0.1 ml accuracy for mixing to make study sample. The recommended powder was then added to the liquid in a clean rubber bowl and allowed to settle in water for 3 s. This technique minimized the amount of air incorporated

Table 1: Material used in the study

Sr no	Material	Manufacturer	Batch no
1.	Wirovest Phosphate bonded investment (ringless) material	Bego, Germany	02032350114
2.	Begosol	Bego, Germany	51090
3.	Doublident duplicating material	Willmann and Pein Gmbh, Hamburg	WP 5080
4.	Distilled Water	Glasmith Pharmaceutical, Nagpur	15639

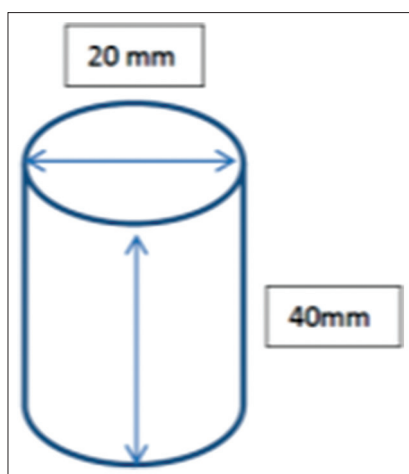


Figure 1: Sample dimensions

into the mix during the initial spatulation. It is then hand spatulated to get smooth consistency for 20 s followed by mechanical mixing in a vacuum mixer (SIRIO dental S.R.L., 47014 Meldola (FC) - Italy) to reduce the porosity at 1750 rpm under 12.7 kg for 20 s.^[5,6]

The mixture was then poured with gentle vibration (for 30 s) into the silicone mold and a glass slab was placed over the mold to ensure flat and parallel ends. After a setting period of 30 min, the cylindrical samples were carefully removed from the molds. All samples were coded and stored in the open air with a temperature range of $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 1 h. Any sample damaged on removal from the mold was discarded.^[6-8] The sampling method was same as done by Canay S *et al.* in their study, in this way, a total of 80 cylindrical samples were prepared from Wirovest investment materials.^[9]

Drying methods

Samples were subdivided into four groups: Group I – Control, Group II – Conventional hot air oven drying, Group III – Microwave oven drying, and Group IV – Combination of microwave and conventional hot air oven. Each containing 20 samples ($n = 80$).

- Group I: Twenty samples were dried in open air for 2 h. After that, they were tested under the universal testing machine
- Group II: Another 20 samples were dried in the conventional oven. Samples were kept in a digitally controlled hot air conventional oven at $230^{\circ}\text{C} (\pm 10)$ for 1 h. After 1 h, the samples were removed from the oven and left to cool at room temperature for 2 h, after that they were tested under universal testing machine
- Group III: Twenty samples were prepared for drying in microwave oven (Samsung model number: mc28h5135vk, Malaysia) had an output power of

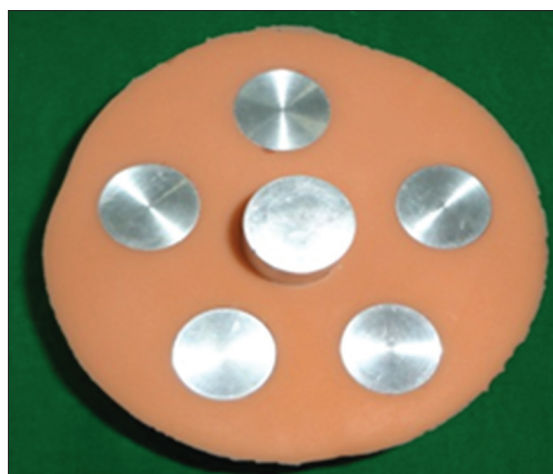


Figure 2: Silicone rubber mold for fabrication of study samples

600W and a frequency of 2450 MHz. Samples of Group III were kept for drying in microwave oven for 10 min with a rotating table. To ensure that the samples were adequately irradiated on all surfaces, they were first exposed for 5 min and subsequently turned upside down and irradiated again for the same amount of time.^[7] A microwave beaker containing 200 ml water, which acts as a heat sink, placed in the oven to protect the magnetron.^[9] After 10 min, 20 samples were stored at room temperature for 2 h. After that, these were tested under universal testing machine

- Group IV: Samples of Group IV were kept for drying in microwave oven at 600 W for 5 min. Then, these were removed and kept in open air for 5 min to lower down the temperature. It was then placed in digitally programmed conventional hot air oven at 230°C for 30 min. These samples were then removed and kept for 2 h at room temperature followed by testing under universal testing machine.

Testing of samples

Twenty samples of each group were tested for DTS through a diametral compression test for maximum load at the point of sample fracture under INSTRON universal testing machine (Star Testing System, India) at a 5 mm/min crosshead speed using the following formula:

$$\text{DTS (MPa)} = \frac{2P}{\pi DT}$$

P = compressive load (Newton)

D = sample diameter (mm)

T = sample thickness (mm)

The results were recorded in MPa. The cylindrical samples were compressed between the table at the bottom and the

fracturing section until fractured [Figure 3]. The maximum load carried was recorded digitally and calculation of the DTS value was obtained from the maximum load at the point of sample fracture.

This is experimental study as the effect of each drying method on DTS of PBI material was checked. The pattern of fracture after load testing was also checked after drying for 2 h in each of the drying methods.

The study was done in 2013–2014 in which materials dried and tested, followed by statistical analysis.

RESULTS

Table 2 provides the descriptive statistics for the strength of Wirovest Bego material when exposed to different drying methods. The mean for Group I specimens was 1.00 (0.18) Mpa, whereas the mean for Group II specimens was 1.06 (0.09) Mpa, for Group III specimens was 3.05 (0.31) Mpa, and for Group IV specimens was 2.34 (0.3) Mpa. A bar chart representation of mean drying methods along with error bars for the material is shown in Figure 4.

The difference in the mean strengths due to different drying methods was evaluated for statistical significance through pair-wise analysis [Table 3]. It is evident that all the mean differences of paired combinations are significant except the difference between air-dried and conventional oven-dried methods.

DISCUSSION

When the investment was mixed with special liquid, the compressive strength of refractory casts significantly increases as stated by Luk and Darvell^[10] measured the effect of special liquid on strength under actual casting condition and found that the use of special liquid increases the strength of phosphate-bonded investments substantially. Li *et al.*^[11] also found the same results in their study. Therefore, in this study, investment material was mixed with special liquid of the same brand supplied by the manufacturers, i.e., Begosol, Bego.

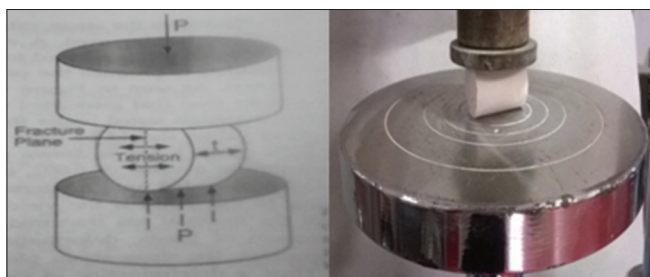


Figure 3: Testing of samples under universal testing machine

Luebke and Chan stated that microwave ovens cannot be used to dry extremely wet or water-soaked cast or dies, as the boiling free water may crack the casts or dies.^[4] Mahler also stated that 7% excess water remained in air-dried gypsum materials 1 h after mixing.^[12] In this study, samples after retrieval were kept for an hour at 20°C–22°C before any drying process, to allow for setting of the investment materials and loss of excess water.^[13]

In an air-dried method, the water is evaporated from the refractory samples by air pressure, heat and air movement which is present in the atmosphere. A previous study done by Luebke *et al.*^[3,4] and others, on air-drying methods suggested that refractory samples which were air-dried, gain sufficient strength after 24 h. The statistical analysis of the present study, Table 2 shows that the mean results obtained for Group I-A was 1.00 ± (0.18) Mpa, which showed that the DTS of Group I at 2 h interval was lower as compared to other groups. The cause of the lower strength after 2-h interval was the presence of free water in the investment material that weakens the structure. The result also agreed with Luebke and Schneider,^[3] Luebke and Chan,^[4] and Tuncer *et al.*^[14] In this study, the tested samples showed irregular pattern of fracture in small fragments.

Since many years, the conventional oven has been used in dentistry for different purpose.^[15] One of the functions of conventional oven is to dry refractory cast as recommended by Marxkors.^[16] Conventional oven dries the refractory cast by surrounding it with hot air. It saves time as compared to the air-drying method. However, after drying in conventional oven for 60 min, still there are some remnants of water present. The mean DTS for Group II was 1.06 ± (0.09) Mpa. The mean DTS was lower than Group III and Group IV. This is because of the composition of the phosphate-bonded investment material. This agrees with Luk and Darvell^[17]

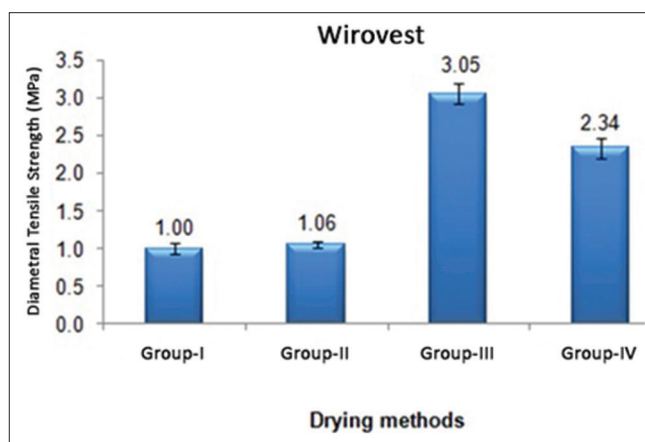


Figure 4: A bar chart representation of mean of all drying methods

Table 2: Descriptive statistics for strength of Wirovest material exposed to different drying methods

Method comparison	Absolute mean difference	P
Air Dried vs. Oven Dried	0.093	0.282
Air Dried vs. Microwave	2.033	< 0.001
Air Dried vs. Microwave and convention	1.467	< 0.001
Oven Dried vs. Microwave	1.94	< 0.001
Oven Dried vs. Microwave and convention	1.374	< 0.001
Microwave vs. Microwave and convention	0.566	< 0.001

Table 3: Pair wise mean difference of strength between different drying method

Parameter	Drying methods			
	Air Dried	Oven Dried	Microwave	Microwave and convention
N	20	20	20	20
Mean (Mpa)	1.00	1.06	3.05	2.34
SD (Mpa)	0.18	0.09	0.31	0.30
Range (Mpa)	0.69 - 1.30	0.92 - 1.23	2.24 - 3.47	1.29 - 2.77
Median (Mpa)	1	1.06	3.16	2.37

who suggested that the high-temperature compressive strength was greatly decreased by thermal transformation and thermal decomposition. They further stated that quartz and cristobalite are the two allotropic forms of silica that are commonly employed for the formation of dental investments. There is an α - to β -phase transformation for cristobalite and quartz from 220°C to 270°C and at 573°C, respectively, which lead to volumetric expansion on heating. Such expansion has been utilized to compensate for much of the alloy thermal contraction, but it must lead to a slightly less dense mass which may, therefore, weaken it. In the case of cristobalite, the expansion is isotropic and the change is rapid, this would create a differential shearing movement between the refractory material and the binder, creating stress and therefore potentially crack nucleation sites. In the case of quartz, the expansion is anisotropic and may exaggerate the differential shear and thus the localized cracking and disruption of contact points within the mass. Moore and Watts^[18] stated that calcium and sodium are present in the phosphate-bonded investment material. Both calcium and sodium can form metal ammonium phosphates, similar to that of magnesium, which decompose and form pyrophosphates when heated and decreases in strength. According to Luk and Darwell,^[19] variation in strength with temperature and between brands of investment material indicates that strength depends very much on composition and the amount of heat supplied. There was very little damage to the edges of the fractured samples observed in the present study. Hence to save time

and to increase strength and surface hardness, microwave oven drying method has been introduced.^[3,4]

In microwave oven drying method, the heat is produced directly inside the sample; it takes much less time than the conventional hot air oven. For testing the investment materials that exhibit limited plastic deformation, the DTS has been used successfully.^[20,21] The mean for Group III was $3.05 \pm (0.31)$ Mpa. Study of Luebke and Chan^[4] suggested that drying in microwave oven at high power (1485 W) decreases the strength of investment material during the first 2 h with fewer cracks and holes in the materials. This might be obtained by microwave drying (1485 W) at 2 h interval after mixing because less excess water would remain in the material. Tuncer *et al.*^[14] found many holes and cracks that were easily seen on the outer surface of the specimens, which were easily broken by handling at the high-power level (1485 W) was used with 5 min and 15 min exposure time. Sharma *et al.* found that microwave radiation at 600 W for 5 min is acceptable for drying type IV gypsum at different time interval.^[22] While on the contrary, a study done by Canay S *et al.*^[9] found that microwave oven drying for 10 min at 600 W significantly increased the DTS of investment at 2 h interval.^[23] This variation in the result of all the studies could have been because of different power levels. In the present study, the same power level and time were used for drying the phosphate-bonded investment materials, i.e., 600 W and 10 min at 2 h interval. Testing the specimens at 2 h interval for Group III investment material had a greater DTS as compared to other methods because microwave acts directly on water molecule present in refractory samples, due to which it results in drying the sample rapidly.

To ensure complete drying of the refractory cast from the core to the external surface, and to increase strength as well, drying was done using combination of microwave oven (5 min at 600 W) followed by the conventional oven (30 min at 230°C) by applying more controlled heat. As this is a combination method, consideration was given to the time as well as increased strength of the refractory cast. The mean DTS for Group IV was $2.34 \pm (0.3)$ Mpa, which was lower than the Group III but significantly higher than the Group I and Group II. The increase strength was because of microwave oven drying for 5 min and there was no additional benefit of drying in conventional oven. It is assumed that at 230°C in conventional oven, there was shearing movement between the refractory material and the binder, which was creating stress. This alteration in composition of investment material affects the strength negatively. The effect of the α - to the β -phase transformation of silica on the compressive

strength of investment had been studied by Ohta *et al.*^[24] They reported a large decrease in compressive strength for phosphate-bonded investment material. While Ohno *et al.*^[25] claimed that the temperature of the displacing transformation of quartz and cristobalite seems to be associated with a decrease in strength to a greater extent, Hence the refractory cast drying with microwave drying and combination method is recommended over conventional oven and air drying because it gives more strength and surface hardness and also takes less time when compared with other drying methods.

Microwave oven method results in the removal of water from the innermost core to the external surface. However, complete loss of water not only causes damage to the magnetron of the microwave but also cracking of refractory cast due to excessive desiccation which results in failure of the prosthesis. Drying can also be done efficiently by a new combination of the microwave oven and conventional oven method. This ensures not only complete drying of the refractory cast from core to the external surface but also increases the strength by applying more controlled heat.

Laboratory procedures are of utmost importance same as that of clinical procedures, hence drying of the refractory cast is also one of the important procedures because it increases the surface hardness and DTS of the refractory cast in a relatively short period, thus maintaining the accuracy of the prosthesis.

Limitation of the study

The curvature of the refractory cast follows the contours of the anatomic tissues. The cylindrical sample were used for the test, the same curvature could not be simulated in this study.

Scope for further studies

The favorable results of this study encourage the scope for future studies. The effect of microwave drying on other physical and mechanical properties of PBI material like dimensional accuracy and surface hardness can be tested.

CONCLUSION

Within the limitation of the study, the following outcomes were drawn.

When the entire spectrum of this study analyzed among all four drying methods, the primary outcome of this study was that microwave oven drying method and combination method showed statistically significant results. Both the methods increase the DTS when compared

with conventional oven and air-drying method. As time and strength is more important factors in the study because in dental laboratories, it is necessary to work on refractory casts soon after separating them from reversible hydrocolloid. Therefore to ensure it has adequate strength and sufficient surface hardness within short spans of time, it is obligatory that it is moisture free so that further casting work can be carried out without damaging the refractory cast. The secondary outcome of this study was that when time is considered as an important factor, both the methods showed effective drying in short period, but in between microwave oven-drying and combination method, microwave drying method takes less time than combination drying method. Hence, it can be used in day-to-day laboratory procedures for the fabrication of the refractory cast for cast partial denture.

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Conflicts of interest

There are no conflicts of interest.

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