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Comparative Evaluation of Few Physical Properties of Epoxy Resin, Resin-Modified Gypsum and Conventional Type IV Gypsum Die Materials: An *in vitro* Study

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ABSTRACT

Aim: To compare and evaluate few physical properties of epoxy resin, resin-modified gypsum and conventional type-IV gypsum die material.

Materials and methods: In the present study, dimensional accuracy, surface detail reproduction and transverse strength of three die materials like epoxy resin (Diemet-E), resin-modified gypsum (Synarock) and conventional type-IV gypsum (Ultrarock) are analyzed. For dimensional accuracy, master die (Bailey's die) is used and calibrations were made with digital microscope. For surface detail reproduction and transverse strength, rectangular stainless steel master die (Duke's die) was used and calibrations were made with Toolmaker's microscope and Instron universal testing machine respectively. One-way analysis of variance (ANOVA) was performed on the means and standard deviation for groups of each test.

Results: The results of the study showed statistically significant difference among these materials in dimensional accuracy, surface detail reproduction and transverse strength.

Conclusion: Epoxy resin exhibited superiority in dimensional accuracy, surface detail reproduction and transverse strength and is nearest to the standards of accurate die material.

Keywords: Dimensional accuracy, Analysis of variance, Bailey's die, Duke's die.

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INTRODUCTION

In order to achieve a satisfactory restoration, the working cast or die must be dimensionally accurate and it should exhibit a certain degree of long-term accuracy. The prepared dies should resemble the prepared teeth as precisely as possible. This precision is affected by the quality of impression material and die material.

Conventional gypsum material exhibits continuous growth and progression of expansion.[1,2] This can profoundly influence the cast seating and fit. Minimal expansion may compensate for dimensional changes that are inherent in the fabrication of cast metal restoration.[3] Owing to several disadvantages of gypsum die material, such as low strength and poor abrasion resistance, other materials and techniques are widely devised and propagated.[4] Silver and copper electroplated die systems are accurate, abrasion resistant and nonabsorbent.[5,6] However, they have not been widely used because of their expense, technique sensitiveness, impression material incompatibility and toxicity.

Resinous die materials are more abrasion resistant and are stronger than gypsum material.[7,8] The high strength of resinous die material renders them extremely useful in the replication of long and thin preparations. The surface detail of epoxy resin is far superior to that of gypsum material. As is known, all resinous materials are subjected to polymerization shrinkage, whereas gypsum products undergo a setting expansion.[9] Owing to these adverse inherent properties, the superiority of one die material over another cannot be claimed unless they are compared in more than one essential physical property.

The purpose of study was to evaluate dimensional accuracy, surface detail reproduction and transverse strength of epoxy resin, resin-modified gypsum and conventional type IV gypsum die materials and to compare the broad cross-section of the properties tested.

MATERIALS AND METHODS

To conduct this study, two separate master model dies were prepared, i.e. Bailey's die and Duke's die with reference lines of definite dimensions scribed for measuring different properties (Figs 1 and 2). The reason why two different master dies were selected was due to their definiteness, standardarity and accuracy of the each master model for specific property and to coordinate these properties for overall superiority of the die materials compared.

A custom-made impression trays made of self-cure acrylic resin that fit over master metal dies are prepared with uniform space all over.[10] Individual impressions of metal dies are made with polyvinyl siloxane impression material and test die specimens are prepared.[11]

Bailey's Die

A master die was prepared by machining a 1/2 inch diameter brass rod with dimensions of cervico-occlusal length 12 mm, occlusal diameter of 9 mm, shoulder finish line 1 mm and 5º convergence angle.[12]

An axial vertical line that represents cervico-occlusal dimension is scribed on die. The axial vertical reference line referred to as dimension I. Two mutually perpendicular occlusal reference lines intersecting at the center of occlusal surface of the master die was scribed.[13] One of the occlusal reference lines was made to intersect at the axial vertical reference line and was referred to as dimension II. The other line drawn perpendicular to dimension II on the occlusal surface was referred to as dimension III. The die was finished and polished to provide a smooth, shiny polished and noncorresponding surface to obviate any adhesion of polyvinyl siloxane impression materials to surface of metallic die.

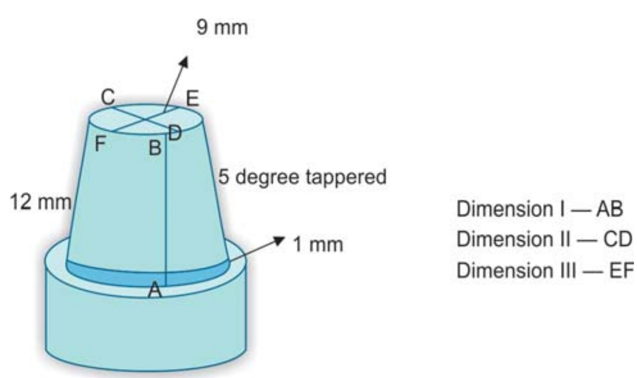


Fig. 1: Schematic representation of type-I master die (Bailey's die)

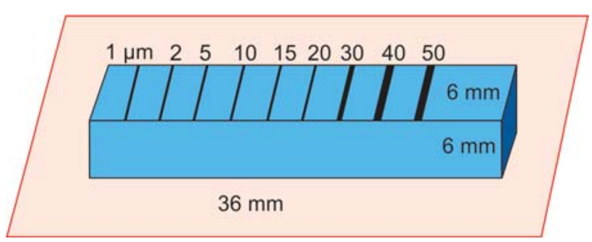


Fig. 2: Schematic representation of type II master die (Duke's die)

A total of 30 impressions of master die were taken using individual custom trays with polyvinyl siloxane impression material. Ten test specimens of each die material were prepared and evaluated for dimensional accuracy (Fig. 3).

Duke's Die

Other die is rectangular stainless steel master die of 6 × 6 × 36 mm with base extension over which nine engraved lines of varying width, i.e. 1, 2, 5, 10, 15, 20, 30, 40 and 50 µm are scribed on free surface of rectangle.[7] This was used for testing surface detail reproduction and transverse strength. For determining surface detail reproduction, 90% of each engraved line needed to be replicated to count it as present.

A total of 60 impressions of master die were taken by using individual custom trays with polyvinyl siloxane impression material. Twenty test specimens of each die material are prepared, out of which 10 specimens of each are used to test surface detail reproduction (Fig. 4) and other 10 specimens for transverse strength (Fig. 5).

FABRICATION OF SPECIMENS

Conventional type-IV [(Ultrarock) Kalabhai Karsan, India] dies made from individual impressions of machined master die using a standard water powder ratio of 0.023. Type IV die stone was initially hand mixed for 45 seconds to incorporate the powder and then a mechanical vacuum mixer (Multivac^R 4, Degussa) was used for 30 seconds to ensure a homogeneous, bubble-free mixture. The resultant mixture was vibrated on Unident vibrator, painted on the entire impression surface with a brush and then the remaining mixture was poured into the impression. The stone was allowed to set for 1 hour at ambient room temperature and 10 Bailey's test specimens were made for measuring dimensional accuracy and 20 Duke's test specimens were made to use 10 specimens each for surface detail reproduction and transverse strength.

Resin-modified gypsum (Synarock XR, DFS GmbH, Germany) dies made from individual impressions of machined master dies using a standard W-P ratio of 0.020. Resin-modified gypsum has 60 seconds total of mixing with 30 seconds of manual premix and 30 seconds mechanical vacuum mixing (Multivac^{R}4, Degussa). Manufacturer's instructions are followed in manipulation and pouring of dies with resin-modified gypsum. The resultant mixture

Fig. 3: Dies of different materials for dimensional accuracy

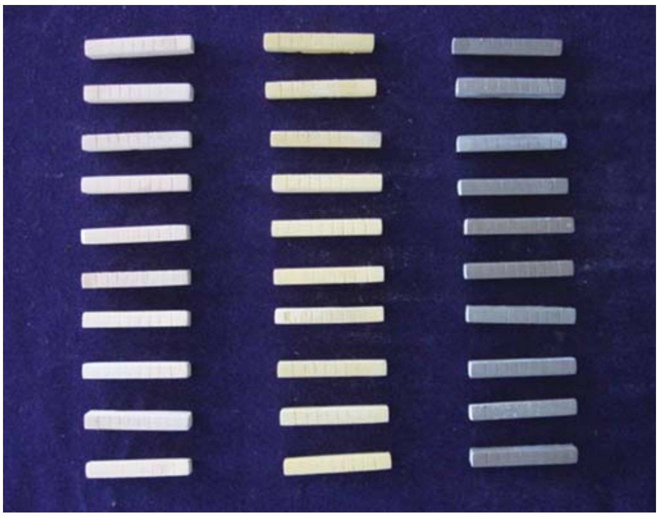


Fig. 4: Dies of different materials for surface detail reproduction

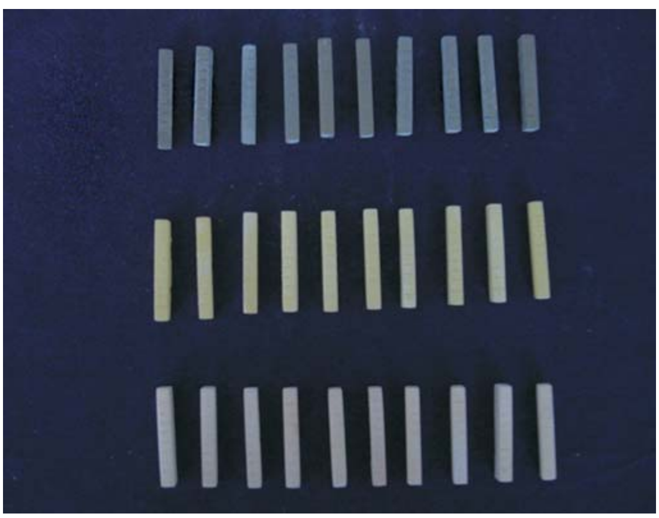


Fig. 5: Dies of different materials for transverse strength

vibrated on Unident vibrator, painted on the entire impression surface with a brush and then the mixture poured into the impression. Resin-modified gypsum was allowed to set for 1 hour at ambient room temperature and 10 Bailey's test specimens were made for measuring dimensional accuracy and 20 Duke's test specimens were made to use 10 specimens each for surface detail reproduction and transverse strength.

Epoxy resin dies (Diemet-E, Erkodent, Germany) fabricated from individual impressions of master metal die. Epoxy resin die material comprised of a resin, hardener and filler material. The resin and hardener were dispensed into a measuring/mixing bowl until the graduation lines on the dosing syringes were reached. Two scoops of filler material were then added to the mixture of resin and hardener as recommended by the manufacturer. The mixture was then spatulated for 30 seconds in the measuring/mixing bowl supplied by the manufacturer. The epoxy resin die material was vibrated into the impression using a vibrator at frequency of 50 to 60 Hz and was allowed to cure for 6 to 8 hours at ambient room temperature, after which the dies were recovered from impressions. A total 10 Bailey's test specimens were made for measuring dimensional accuracy and 20 Duke's test specimens were made to use 10 specimens each for surface detail reproduction and transverse strength.

To evaluate and compare dimensional accuracy of conventional type IV gypsum, resin-modified gypsum, epoxy resin of 10 Bailey's test specimens with assigned nomenclature are measured in a sequence to evaluate the mean for each property. All the measurements were recorded by one investigator and with the models at an ambient room temperature and humidity ($22.1^{o} \pm 0.2^{o}$C and 60% ± 10%). A digital micrometer (Mitutoyo, Japan) was used to measure the dimensions of each model, upto an accuracy of 0.01 mm (Fig. 6). The mean of each dimension, measured three times on the master die, was used as basis for determining the percent relative changes in that dimension of each sample.

To evaluate and compare surface detail reproduction of conventional type IV gypsum, resin-modified gypsum, epoxy resin of 10 Duke's test specimens of each material with assigned nomenclature are measured under Toolmaker's microscope (Metzer Opto Electronical Instruments Pvt Ltd, Bombay) with low angle lighting to determine the narrowest line seen on each die specimen (Fig. 7). At least 90% of the line needed to be replicated to count as present. The narrowest line seen on all specimens of each die material needed to be recorded if there is any variation.[7]

To evaluate and comparative transverse strength of conventional type IV gypsum, resin-modified gypsum, epoxy resin of 10 Duke's test specimens of each material with assigned nomenclature tested with Instron universal

Fig. 6: Digital micrometer (Mitutoyo, Japan)

Fig. 7: Toolmaker's microscope (Metzer Opto Electronical Instruments Pvt Ltd, Bombay)

testing machine. Epoxy resin needs longer setting time to attain strength when compared to the type IV gypsum and resin-modified gypsum. So, uniform time of storage of 48 hours before testing was maintained for all specimens of three different materials. The irregular free surface of each specimen was ground flat and parallel to opposite surface and finished with 600 grit SiC paper.[13] The specimens were tested with 3 point loading apparatus in universal testing machine (Sintech 1123, Renew, Minneapolis, Minn) (Fig. 8). The ground side of each specimen was positioned so that it was in compression during the test. Load at fracture was used to compute transverse breaking strength of the test specimens of three die materials.

The measurements of all test specimens of three die materials were evaluated by tabulation and statistical analysis is done to draw results for inference with broad comparison.

DISCUSSION

In order to achieve a satisfactory restoration, the working cast or die must be dimensionally accurate and it should exhibit a certain degree of long-term accuracy.[9] The prepared dies should resemble the prepared teeth as precisely as possible. This precision is affected by the quality of impression material and die material.[11]

The ideal requirements of die material are dimensional accuracy, acceptable detail reproduction, abrasive resistance, surface hardness, ease and efficiency of manipulation, compatibility with impression material, lack of toxicity and transverse strength.[14,15] In the present *in vitro* study, dimensional accuracy, surface detail reproduction and transverse strength of conventional type IV, resin-modified gypsum and epoxy resin die materials are evaluated.

Various methods have been reported in the literature to measure the dimensional accuracy of stone dies. In this study, a digital micrometer (Mitutoyo, Japan) with an accuracy of 0.01 mm was utilized.

For surface detail reproduction, Toolmaker's microscope (Metzer Opto Electronical Instruments Pvt Ltd, Bombay) was used to visualize the narrowest line recorded (90% of line has to be read in order to count it is replicated.[7] According to ADA specification number 25, for dental gypsum products minimum width line to be replicated to be used as die material is 50 micrometer.[1]

For transverse strength, test specimens are subjected to three point loading on Instron Universal testing machine (Sintech 1123, Renew, Minneapolis, Minn).

Fig. 8: Instron or Universal testing machine (Sintech 1123, Renew, Minneapolis, Minn)

Dimensional Accuracy

NR Chaffee et al[16] (1997) in their study of comparison of dimensional accuracy of improved dental stone and epoxy resin die material demonstrated epoxy die system provided a greater degree of dimensional accuracy comparable to gypsum when used with addition silicon impression material.

In the present study for dimensional accuracy, three dimensions I, II and III of master die were measured thrice and mean is calculated. The mean of dimension I is 11.2033, mean of dimension II is 9.9133 and mean of dimension III is 9.9067 mm. This mean of dimensions is compared with test specimen measurements of each material. From these measurements it is inferred that type IV gypsum die stone and resin-modified gypsum showed an increase in dimensions due to expansion. This increase is more in type IV gypsum compared to resin-modified gypsum. Epoxy resin on other hand showed shrinkage. Owing to this variation, the die materials were compared with master die. All these die materials showed statistically significant differences circumferentially and cervico-occlusally. Epoxy resin was found to be most dimensionally accurate die material followed by resin-modified gypsum and conventional type IV gypsum (Graphs 1 to 3).

Surface Detail Reproduction

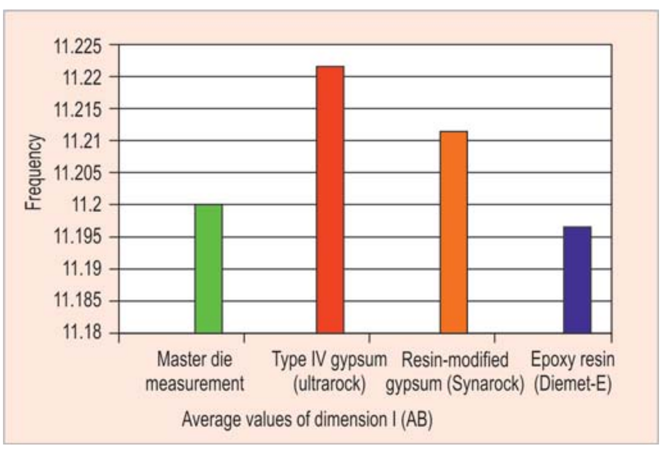
Jacinthe M Paquette,[3] Daniel Aiach,[17] NR Chaffee,[16] etc. have reported superior abrasion resistance and excellent replication of surface detail by epoxy resin die material and showed much difference when compared with gypsum die material. In the present study for suface detail repoduction, the narrowest line read on each test specimens of each die material are visualized using Toolmaker's microscope. Compatibility between impression material and die material is critical for evaluation of surface details and was taken care. At least 90% of line is needed to be replicated to count as present.[7] The results of present study has shown surface details of epoxy resin is superior which can reproduce 1 μm narrow line, next best being resin-modified gypsum (15 μm) and finally is conventional type IV gypsum material (20 μm) (Graph 4).

Transverse Strength

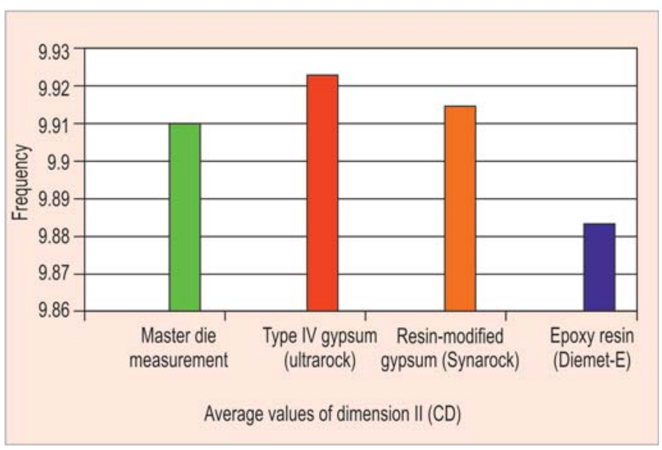
Phillip Duke et al[7] (2000) reported higher values of transverse strength ranging from 82.8 to 91.8 MPa for epoxy die material. Gypsum-based die materials tested were brittle and exhibited very little deformation before fracture.

In the present study, 10 specimens prepared for each die material are stored for 48 hours before testing.[7] The specimens were tested with 3 point loading apparatus in Universal testing machine (Sintech 1123, Renew, Minneapolis, Minn) at crosshead speed of 0.1 mm/min. Load at fracture was used to compute transverse breaking strength in MPa. The results of this study had shown significant difference in transverse strength of the die materials studied. Epoxy resin demonstrated highest transverse strength or transverse breaking strength with an average of 87.69 MPa for all the test specimens. For resin-modified gypsum it is 27.75 MPa and least for type IV gypsum 24.24 MPa (Graph 5).

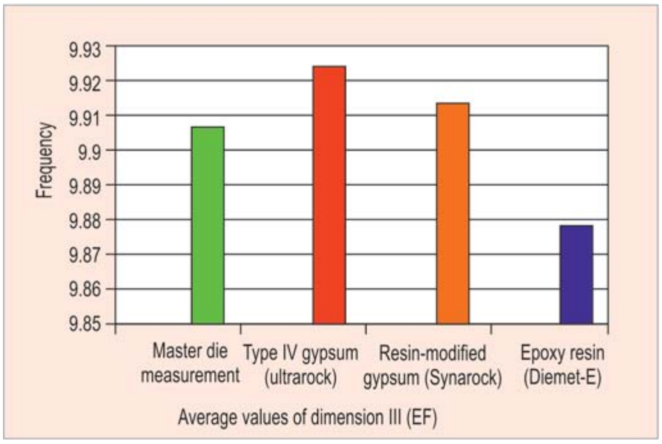
One-way analysis of variance (ANOVA) is performed by tabulating the statistical results. The measurements after statistical analysis reflected significant difference in dimensional accuracy, surface detail reproduction and transverse strength. Individual die material influenced all the selected properties. Within the limitations of this study, epoxy resin was found to be the most dimensionally accurate with finest surface detail reproduction and highest transverse strength among all the three die materials studied.[18,19]



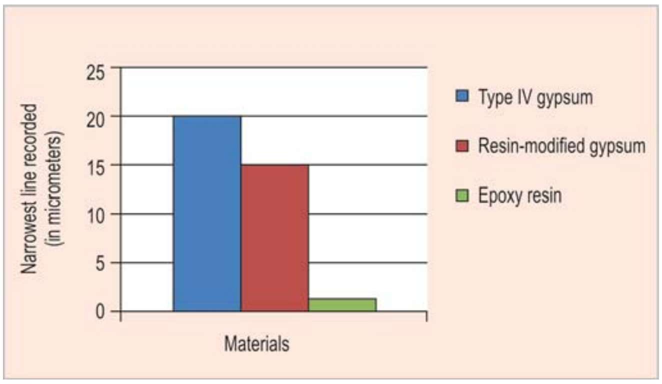
Graph 1: Comparison of mean values of dimension I (AB) of master die measurement with type IV gypsum (Ultrarock), resin-modified gypsum (Synarock) and epoxy resin (Diemet-E)



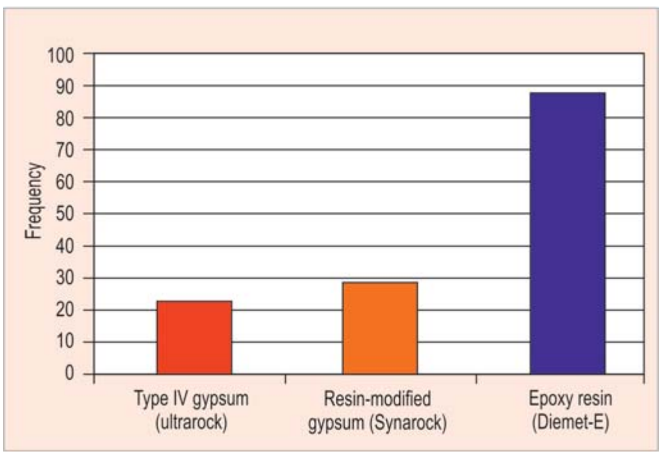
Graph 2: Comparison of mean values of dimension II (CD) of master die measurement with type IV gypsum (Ultrarock), resin-modified gypsum (Synarock) and epoxy resin (Diemet-E)



Graph 3: Comparisons of mean values of dimension III (EF) of master die measurement with type IV gypsum (Ultrarock), resin modified gypsum (Synarock) and epoxy resin (Diemet-E)



Graph 4: Comparison of surface detail reproduction of type IV gypsum (Ultrarock), resin-modified gypsum (Synarock) and epoxy resin (Diemet-E) die materials with the narrowest line recorded



Graph 5: Comparison of mean values of transverse breaking strength of type IV gypsum (Ultrarock), resin-modified gypsum (Synarock) and epoxy resin (Diemet-E) transverse breaking strength

CONCLUSION

Within the limitations of this study, the following conclusions were drawn.

Epoxy resin die material had net shrinkage in contrast to the gypsum-based material which had net expansion. Overall percentage of contraction exhibited by epoxy resin is less than percentage of expansion exhibited by gypsum-based die materials. Hence, epoxy is the most dimensionally accurate die material followed by resin-modified gypsum. Surface detail reproduction of epoxy resin is finest which can read even 1 μm narrow line, next best being resin-modified gypsum 15 μm and finally is conventional type IV gypsum material 20 μm. Transverse strength of epoxy resin is far superior to type IV gypsum and resin-modified gypsum. Resin admix gives additional strength to gypsum-based die materials.

The individual variabilities in setting expansion and shrinkage of die materials evaluated shown epoxy resin to be the dimensionally accurate with finest surface detail reproduction and highest transverse strength of the three die materials studied. However, there is scope for further studies in this subject.

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