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In-Vitro Comparative Study Of Mechanical Properties Of Type V Die Stone And Epoxy Resins

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Abstract
Background: Since the advent of elastomeric impression materials, the use of indirect technique for fabrication of the prosthodontic restorations has become almost universal. Success of these techniques is dependent on the availability of a die material that meets certain mechanical criteria. Little published information is available comparing the properties of recently developed epoxy resin die materials and newly available type V die stone, which are claimed to be superior to conventional type IV gypsum, die materials.
Aims: This study compared the properties of two new die materials.
Methods and Material: The surface hardness, abrasion resistance, compressive and transverse strength of two recently introduced, type V die stone (Denflo HX) and new epoxy resin die material (Epoxy-Die), were studied.
Results: The epoxy resin exhibited much better abrasion resistance, compressive and transverse strength than the gypsum materials, which were similar in these properties. The type V gypsum exhibited the highest surface hardness, whereas the epoxy resin had the lowest value.
Conclusion: The resin products were significantly superior to the conventional type IV gypsum die materials. In general, the epoxy resin exhibited the best properties of the materials studied; however, its setting shrinkage may necessitate alterations in technique to achieve well-adapted castings

Key Words
Epoxy resin die materials, Type V die stone, Abrasion resistance, Compressive strength, Transverse strength

Introduction

The requirements of die material[1] include dimensional accuracy, acceptable detail reproduction, abrasion resistance, surface hardness, ease and efficiency of manipulation, compatibility with impression material and transverse strength. Dies are subjected to considerable flexural constraint when removed from impressions, duplicated to make refractory casts for dental ceramics or when dental restorations are seated. While no single die material possesses all the ideal properties for an indirect working model, gypsum products[2] have gained general acceptance because of their close approximation of critical properties of an ideal die material. The different die materials which are available today are type IV gypsum, Type V gypsum, epoxy resin, polyurethane resins, resin modified gypsum, electroformed dies etc. One of the most commonly used die material is Gypsum based, i.e. Type IV (high strength, low expansion) and Type V (high strength, high expansion) dental stones.

The purpose of this study is to evaluate and compare surface mechanical properties of commercially available improved gypsum product i.e. Type V die stone and a new epoxy die material. Each of these is claimed to have improved properties as compared with conventional Type IV gypsum die stone.

Methods And Materials

Materials used:

- Soft putty Poly vinyl siloxane impression material (AquasilTMsoft putty, Dentsply, Caulk)
- Light body polyvinyl siloxane impression material (AquasilTMLV, Dentsply, Caulk)
- Type V dental stone (Denflo HX)
- Epoxy resin (Diemet-e, Erkodent, Germany)
- Tray adhesive

Instruments and armamentarium:

- Stainless steel rectangular die (50 X 15 X 7mm)
- Stainless steel cylindrical die (10mm diameter and 30mm height)
- Instron testing machine
- Wear Testing Machine
- Microvicker Hardness Tester
- Precision Balance (ConTech)
- Vibrator

Vacuum mixer

This study was conducted to compare the surface mechanical properties (transverse strength, compressive strength, abrasion resistance, and surface hardness) of two die materials namely Epoxy resin and Type V die stone at the Department of Proshodontics, A.B. Shetty Memorial Institute of Dental Sciences, Mangalore(India) and at the Dept of Mechanical Engineering, NITK, Surathkal. Included in this study is a newly marked type V gypsum die stone, Denflo HX and a new epoxy resin die material, Diemet-e, Erkodent, Germany.
Specimen preparation and all tests are to be performed at room temperature. Forty specimens for each material are prepared and subjected to test for surface hardness, abrasion resistance, transverse and flexural strength. To conduct this study one master metal die and one cylindrical mould of a definite dimension were prepared. A custom impression tray was machined to fit over the master metal die. Individual impressions were made with polyvinyl siloxane impression material and the dies were prepared with: Type V die stone and Epoxy resin.

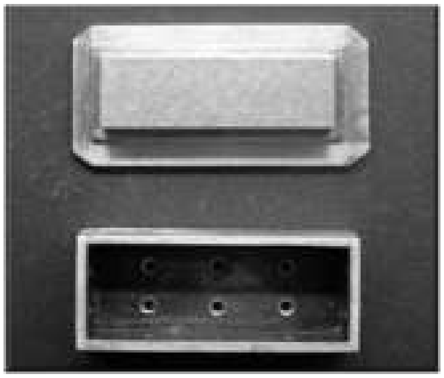


Figure-1: Rectangular Die

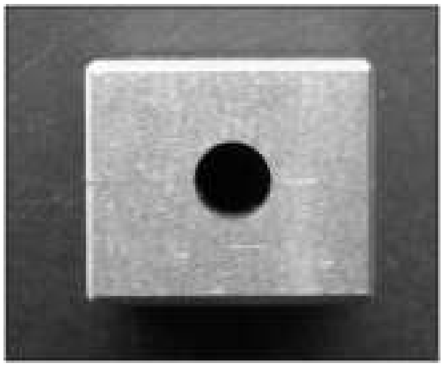


Figure-2: Top View Of Cylindrical Die

Figure-3: Die After Separation

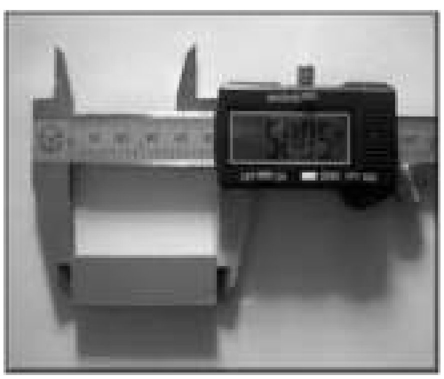


Figure-4: Measurement Oflength Of The Specimen

Figure-5: Measurement Ofwidth Of The Specimen

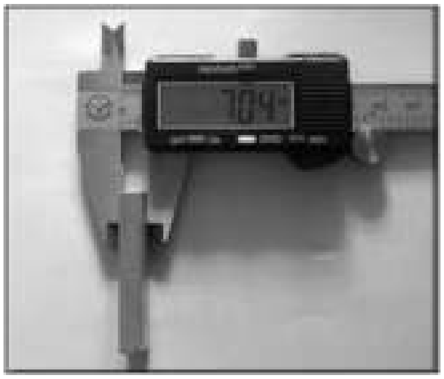


Figure-6: Measurement Ofthickness Of The Specimen

Fabrication of the Master die

Two master die were prepared by machining a medical grade 304, stainless steel. The prepared cylindrical master die **(Fig.-2 and 3)** had the following dimensions: height 20mm and diameter of 10 mm and used for evaluating compressive strength and abrasive resistance and another rectangular one 50 X 15 X 7 mm for evaluating surface hardness and flexural strength. **(Fig.-4, 5 and 6)**. The dies were finished and polished to provide a smooth, shiny polished and non corresponding surface to obviate any adhesion of the polyvinyl siloxane impression materials to the surface of the metallic die.

Fabrication of the Impression Tray

In order to make impressions of the master die, a custom impression tray was made from a 304 medical grade stainless steel. 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 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. The custom impression tray was designed to provide a uniform space of 3mm 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-1)**.

Fabrication of the Models

Eighty specimens, (ten for each mechanical properties) of two different materials namely Epoxy resin die material and Type V die stone, were made from individual impressions of the master die. The impressions of the die were made with soft putty polyvinyl siloxane impression material (AquasilTMsoft putty, Dentsply, Caulk) in the custom impression tray with light body polyvinyl siloxane impression material (AquasilTMLV, 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. The impressions were polymerized at 37°C and were stored at room temperature for about 60 minutes before pouring the die material.

Group I comprised of 40 Epoxy resin dies (Diemet-e, Erkodent, Germany) fabricated from individual impressions of the master metal die. Epoxy resin die material comprised of a resin, hardener and filler material. The resin and hardener were supplied in two dosing syringes by the manufacturer. The resin and hardener were dispensed into a measuring / mixing bowl until the graduation lines on the dosing syringes were reached. Two scoops of the filler material were then added to the mixture of resin and hardener as recommended by the manufacturer. The mix 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 a frequency of 50-60 Hz and was allowed to cure for 6-8 hours at ambient room temperature, after which the dies were recovered from the impressions.

Group II constituted forty Type V (Denflo HX, India) dies made from individual impressions of the machined master die using a standard water-powder ratio of 0.20. Type V die stone was initially hand mixed for 45 seconds to incorporate the powder and then a mechanical vacuum mixer (Multivaco4, Degussa) was used for 30 seconds to ensure a homogenous, bubble free mix. The resultant mixture was vibrated, 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.

Transverse Strength:

Transverse strength, flexural strength or modulus of rupture, as this property is variously called is essentially a strength test of abeam supported at each end, under a static load.

A stainless steel die of dimensions 50 X15X7 mm was used in this study. The custom impression tray was used, on which perforations were made at random and tray adhesive applied, for making impressions. The impression material was allowed to set and die was removed after the manufacturer recommended time period. Ten specimens were prepared for each die material and stored for 48 hours before testing. The finished width and thickness of each specimen were measured with a digital micrometer. The specimens were tested with a 3-point loading apparatus in a universal testing machine (Instron.) to failure at a crosshead speed of 1 mm/min. The ground side of each specimen was positioned so that it was in compression during the test. The span length used in the test was 40 mm. Load at fracture was used to compute transverse breaking strength in MPa. Means and standard deviations for the breaking strength were calculated from the 10 specimens in each group.

Compressive Strength

Cylindrical mould of 30mm height and 10mm internal diameter were used to fabricate the epoxy resin and type V die stone specimens. Specimens were removed after the materials were set and finished with by means of grinding in order to obtain flat parallel ends. These were then tested in universal testing machine (Instron) and subjected to compression loads at a crosshead speed of 1 mm per minute.

Surface Hardness

Vickers indentations were made on the lateral surfaces of the dies. Five indentations, 5 mm apart, were made on each die with a Microvickers Hardness Tester with a diamond indenter and a 300-gm load applied for 20 seconds. The average of the 5 readings for each specimen was used to calculate a group mean and standard deviation for 5 specimens of each die material.

Abrasion Resistance

Five specimens were poured with each die material and the dies were stored for 24 hours before testing. The abrasion apparatus is depicted in Figure 3The apparatus moves the abrader in a circular motion at 900RPM. Specimens are supported in a holder, so that a long line angle of the specimen is held in a vertical position for abrasion. Each specimen was run for 15sec on abrading wheels with a load of 100 Gm. Weight loss per unit area is reported for each cycle and averaged.

Results

1. The compressive strength of epoxy resin (59.4960MPa) was far better than that of type V die stone (17.7310MPa). **(Table I)** These mean difference were tested statistically by using student't' test. The difference between type V die stone and Epoxy Resin happened to be highly significant ($p < .0001$). Epoxy resin showed about 3 times increased compressive strength compared to the type V die stone.
2. The results also showed that the flexural strength of epoxy resin (56.2630MPa) was far better than that of type V die stone (13.9040MPa). **(Table II)** These mean difference were tested statistically by using student't' test. The difference between type V die stone and Epoxy Resin happened to be highly significant ($p < .0001$). Epoxy resin showed about 4 times increased transverse strength compared to the type V die stone.
3. The abrasion resistance in form of volume loss per unit area of epoxy resin (0.13280 mg/mm2) was less than that of type V die stone (0.24860mg/mm2).**(Table III)**. These mean difference were tested statistically by using student 't' test. The difference between type V die stone and Epoxy Resin happened to be highly significant ($p < .0001$). Epoxy resin showed about 2 times increased abrasion resistance when compared to the type V die stone.
4. It is also seen type V die stone had the highest value for the hardness (49 HV) than that of epoxy resin which had hardness value of 27 HV. The mean hardness value of the three groups **(Table IV)** that had been compared and was found highly statistically significant ($p <.0001$). Thus with regards to hardness, Type V die stone was superior to epoxy resin.
5. The abrasion resistance of Epoxy resin and the die stone has been

Table 1 : T-test (Group Statistics)

	Compressive Strength				Transverse Strength (Mpa)			
	N	Mean	Std. Deviation	Std. Error Mean	N	Mean	Std. Deviation	Std. Error Mean
Epoxy Resin	10	57.7860	5.7862	1.8298	10	54.9150	6.7440	2.1326
Die Stone	10	17.6310	2.0432	0.6461	10	13.9040	1.2622	0.3992

Table 2: Independent Samples Test (Compressive Strength, Transverse strength)

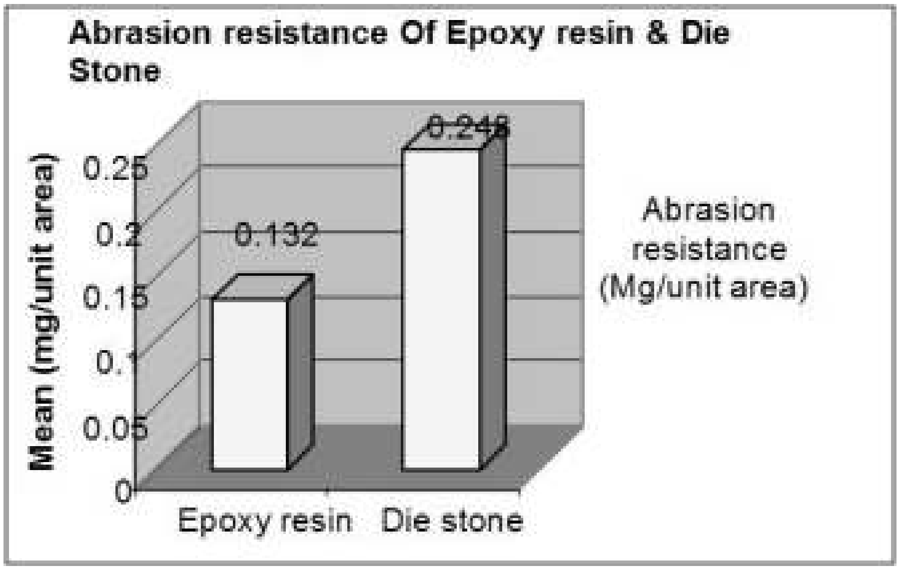
	t- test for Equality of Means						
	t	d.f	Sig. (2- tailed)	Mean Difference	Std. Error Difference	95% confidence interval of the difference	
						Lower	Upper
Compressive Strength	20.69	18	0.0001	40.1550	1.9405	36.078	44.232
Transverse Strength	18.90	18	0.0001	41.0110	2.1697	36.453	45.569

Table 3: Group Test (Abrasion Resistance, Surface Hardness HV Vickers Hardness Number)

	Abrasion Resistance (mg/unit area)				Surface Hardness HV Vickers Hardness Number			
	N	Mean	Std. Deviation	Std. Error Mean	N	Mean	Std. Deviation	Std. Error Mean
Epoxy Resin	5	0.13280	0.0164	0.0073	5	27.00	3.39	1.52
Die Stone	5	.24860	0.267	0.0119	5	49.00	3.39	1.52

Table 4: Independent Samples Test (Abrasion Resistance, Surface Hardness HV Vickers Hardness Number)

		t- test for Equality of Means						
		t	d.f	Sig. (2- tailed)	Mean Difference	Std. Error Difference	95% confidence interval of the difference	
							Lower	Upper
Epoxy Resin	Materials	-8.27	8	0.0001	-0.11580	0.0140	-0.1481	-0.0835
Die Stone	Equal variance assumed	-10.26	8	0.0001	-22.00	2.14	-26.95	-17.05



Bar Chart -1: The Abrasion Resistance Of Epoxy Resin And The Die Stone

Bar Chart -2: The Compressive Strength, Flexure Strength And Surface Hardness Of Epoxy Resin And Die Stone

depicted **(Bar Chart 1)**

6. The compressive strength, Flexure strength and Surface hardness of Epoxy Resin and Die Stone have also been depicted **(Bar Chart 2)**

Discussion

The ultimate goal of a Prosthodontist is to fabricate a successful restoration. The contributory factors for this success include a perfect diagnosis, treatment planning and proper execution of the clinical and laboratory steps along with patient education and motivation followed by a thorough follow-up and maintenance. In order to achieve a satisfactory restoration, the working cast or die must be dimensionally accurate[10], able to reproduce fine detail, resistant to abrasion, hard and of enough strength, as casts are subjected to considerable flexural constraints when removed from impressions, if duplicated to make refractory casts for dental ceramics, or when dental restorations are seated. When we describe the strength of an object or a material we are most often referring to the maximum stress that is required to cause fracture or a specified amount of plastic deformation. In the present study two aspects of strength i.e. the flexural and compressive strengths, were assessed in order to secure a satisfactory guide to the total strength characteristics.

Flexural Strength

Type V die stone has limited flexural strength, and this may predispose working casts to fracture when they are removed from impressions. By making casts harder, manufacturers have encouraged brittleness[4]. This fragility is particularly obvious with long and narrow tooth preparations. Epoxy resins, on the other hand, have traditionally exhibited superior mechanical qualities and acceptable dimensional stability[10] and the degree varied with the type of impression materials and epoxy resin used[3]. In the present study, the mean flexural strength exhibited by the Type V die stone specimens (13.904 MPa) was significantly less than that of the epoxy die material specimens (56.263 MPa). This ratio difference (1:4) is in concurrence with two studies conducted where they concluded that epoxy resin had four times the flexural strength than that of dental stone.

All the gypsum-based die materials tested were brittle and exhibited very limited deformation before fracture[11]. The epoxy die material, however, was much less stiff than the gypsum products and displayed significant elastic deformation before failure. Clearly, the epoxy resin will absorb much more energy before fracturing and should be less susceptible to breakage if dropped or handled roughly.

Compressive Strength

The principal requisites of a die are strength, hardness, abrasion resistance and minimum setting expansion.

Hardness

The Vickers micro hardness test was chosen for this study, because one of the materials tested was an epoxy resin. Epoxy resins are known to exhibit elastic recovery after indentation. Because this elastic recovery occurs mainly along the shorter diagonal of the vickers indentations, a more accurate measure of the hardness is obtained from the length of the long diagonal that exhibits very little elastic recovery. This study reported **(Table 3)** Vickers hardness values ranging from 45 to 51 for the type V die stone and 29 to 39 for epoxy resin die material. These values are in relative agreement to those in another study[5] investigating type IV that found Knoop values of 21.6 for Vel-Mix and Silky-Rock. The resin-containing gypsum die materials, epoxy resin, were not more resistant to indentation than type V die-stone. The results of this study also are in agreement with other studies.[6],[7] which concluded that epoxy resin die materials are less resistant to indentation than type V gypsum-based die materials.

Abrasion Resistance

The results of this study agreed with other studies.[8] which concluded that the abrasion resistance of epoxy resin die materials was higher than that of gypsum-based die materials.

As confirmed by other studies.[8],[9] no correlation between hardness and abrasion resistance was encountered in our study. The die material that was the least resistant to indentation, Epoxy-Die was the most resistant to abrasion. Conversely Die-Stone, which was the most resistant to indentation, was the least resistant to abrasion. Hardness may be of limited value for comparing die materials for clinical use. Among the die materials tested, epoxy resin was the most resistance to wear test done i.e. most abrasive resistant whereas Denflo HX was almost half the resistant to abrasion than epoxy resin. To obtain these properties α-hemihydrate of the Densite type is used. The cuboidal shaped particles and the reduced surface area produce such properties without undue thickening of the mix. Type V die stone meets most of these requirements. The chief disadvantage of Type V die stone is its susceptibility to abrasion during carving of the wax pattern. Inspite of this disadvantage, it is the most widely used die material. Types V die stones exhibit a higher compressive strength as well as a higher setting expansion than does Type IV die stone. The rationale for this increase in setting expansion is that certain newer alloys, such as base metal, have a greater casting shrinkage than do the traditional noble metal alloys. Thus, higher expansion is required in the stone for the die to aid in compensating for the alloy solidification shrinkage. The use of a Type V die stone may also be indicated when inadequate expansion may have been achieved during the fabrication of cast crowns. The use of Type V die stones should be avoided in the production of dies for inlays since the higher expansion may lead to unacceptably tight fits [3].

Conclusions

Within the limits of this study, the following conclusions were drawn:

- The properties of the resin-modified gypsum die materials were significantly different than those of type V die stone.
- The epoxy resin die material was markedly superior in abrasion resistance, compressive and transverse strength to type V die stone studied.
- The epoxy resin die material has a lower surface hardness than that of type V die stone.
- Surface hardness does not correlate with the other properties tested and may not be a good measure of performance for these materials.

References

1. Stephen F. Rosenstiel. Contemporary fixed prosthodontics. 3rd ed. Mosby Inc. 2001.
2. Anusavice K.J. Phillip's science of dental materials. 11th ed. Philadelphia; WB Saunders: 2004.
3. Reza H. Heshmati, William W.Nagy, Carl G.Wirth,Virendra B. Dhuru. Delayed linear expansion of improved dental stone. J Prosthet Dent 2002; 88:26-31.
4. Aiach D, Malone WF, Sandrik J. Dimensional accuracy of epoxy resins and their compatibility with impression materials. J Prosthet Dent 1984; 52: 500 – 4.
5. Chaffee NR, Bailey JH, SherrardDJ. Dimensional accuracy of improved dental stone and epoxy resin die materials. Part I: Single die. J Prosthet Dent 1997; 77:131- 35.
6. Philip Duke, B. Keith Moore, Steven P. Haug, Carl J. Andres. Study of the physical properties of type IV gypsum, resin containing and epoxy die materials. J Prosthet Dent 2000; 83:466-73.
7. Moser JB, Stone DG, Willoughby GM. Properties and characteristics of a resin die material. J Prosthet Dent 1975; 34: 297–304.
8. Gujjarlapudi MC, Reddy SV, Madineni PK, Ealla KK Comparative evaluation of few physical properties of epoxy resin, resin-modified gypsum and conventional type IV gypsum die materials: An in vitro study.JContemp Dent Pract. 2012 Jan 1;13(1):48-54.
9. Gerald T. Nomura, Morris H. Reisbick, Jack D. Preston. An investigation of epoxy resin dies. J Prosthet Dent 1980; 44:45-50
10. Gerard Derrien, George Sturtz. Comparing of transverse strength and dimensional variations between die stone, die epoxy resin, and die polyurethane resin. J Prosthet Dent 1995; 74:569-74.
11. Schwedhelm ER, Xavier Lepe. Fracture strength of Type IV and V die stone as a function of time. J Prosthet Dent 1997; 78:554-9

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