



**COMPARATIVE EVALUATION OF SURFACE HARDNESS OF TYPE IV GYPSUM  
PRODUCT USING THREE DIFFERENT DIE HARDENERS: AN IN – VITRO STUDY**

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**ABSTRACT**

**Statement of the problem:** A die, commonly made of Type IV gypsum, should be dimensionally stable, hard and resistant to inadvertent abrasion caused during its fabrication. To increase the surface hardness, coating of die hardeners has been recommended. But no conclusive data is available as some authors stated that the surface hardness increases while others were in view that it decreases or shows no effect on application die hardeners. Hence, this study was undertaken to evaluate and compare the surface hardness of Type IV gypsum products using different die hardeners. **Materials & Method:** A total of 60 samples, divided into four groups, were fabricated from the mould prepared by impression of the master die. Group I, II and III were treated with different types of die hardeners whereas, Group IV (control) was not treated with any type of die hardener. Micro Vickers hardness test was then conducted for the evaluation of surface hardness. **Results:** Statistically significant results ( $P < 0.05$ ) were found for surface hardness evaluation after the application of different die hardeners. Mean Surface hardness was highest with the control group followed by samples treated with mixture of vinyl and natural resins, Cyanoacrylate esters blended with acetone and least was seen with solution of phthalate resin. **Conclusion:** Die hardener coating applied on Type IV gypsum product decreases the surface hardness.

**KEYWORDS:** Die, Gypsum, Die hardener, Surface Hardness, Vickers hardness number.

**INTRODUCTION**

Individual dies used to fabricate fixed dental prosthesis should fulfil all mechanical properties like dimensional stability, hardness and resistant to inadvertent abrasion caused during fabrication of wax pattern. Laboratory technicians and dentists depends on these properties to predictably fabricate accurate and precise prostheses.<sup>[1]</sup> Different materials are used for the fabrication of dies like gypsum products, Electroformed dies (electroplated copper or silver), Epoxy resins and amalgam dies out of which Type IV gypsum product is the most commonly used material as fabrication of electroformed die and amalgam dies is more time consuming and special equipment are needed where chances of distortion are quite high.<sup>[2]</sup> The only disadvantage with gypsum material is its poor resistance to abrasion. Attempts have been made to overcome this defect with the use of so called gypsum hardener which improves the abrasion resistance but have relatively little effect on the hardness

of stone.<sup>[3]</sup> Different authors have different views regarding effect on surface hardness after applying die hardener. Various authors<sup>[4,5]</sup> were of the opinion that the hardness of die stone increases whereas, others<sup>[1,6]</sup> opined that there was decrease in surface hardness after application of die hardener. Some authors<sup>[7,8]</sup> were also of the view that applying die hardener on die stone caused no change in the hardness. There is no conclusive data available on the change in hardness on application of various die hardeners. This study was, therefore undertaken to evaluate and compare the surface hardness of Type IV gypsum products using different die hardeners.

**MATERIAL AND METHODS**

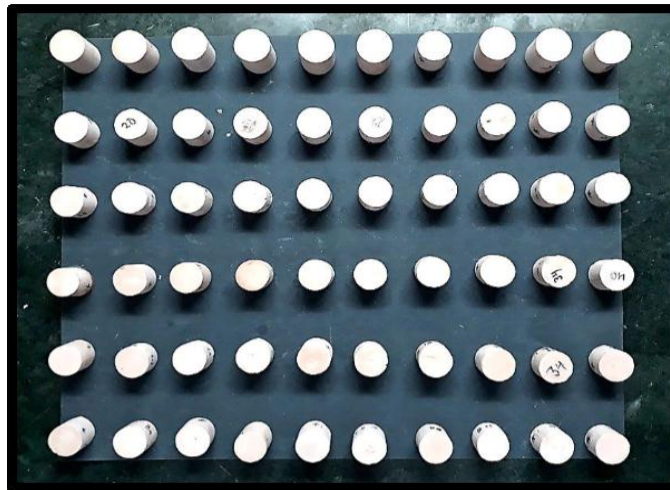
The test samples were fabricated from the mould prepared by impression of cylindrical rings of dimension 15 \* 15 mm, filled with mould plaster, with polyvinyl siloxane impression material (figure 1).



**Figure 1: Steel pipe filled with plaster on glass slab for master die fabrication and mould made of polyvinyl siloxane material.**

A total of 60 test samples were made and divided into four main groups of 15 samples each based on type of die hardeners used (figure 2). Group 1- Samples treated with mixture of vinyl and natural resins Die Hard (MDM

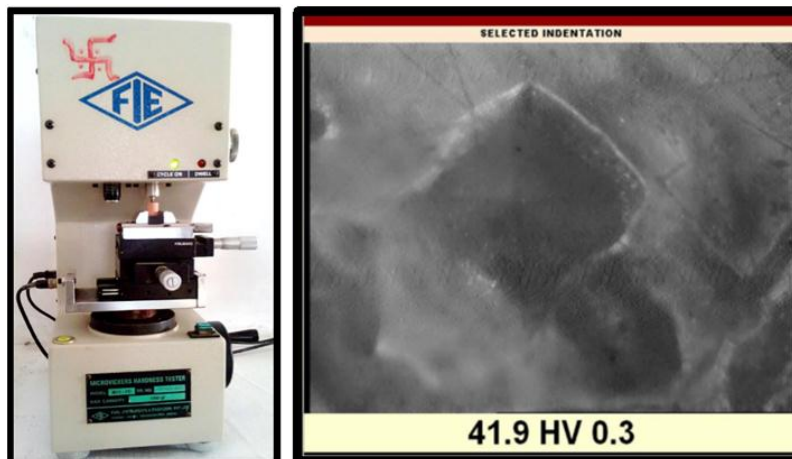
Co.), Group 2- Samples treated with solution of phthalate resin Durol E (Bego), Group 3- Samples treated with Cyanoacrylate esters blended with acetone and Group 4- Control group (No die hardener used).



**Figure 2: Test Samples for evaluation of surface hardness (60).**

One face of each specimen was applied with a single coat of a die hardener which was air thinned and allowed to dry. The hardness of samples under various groups were then tested using Micro Vickers hardness tester

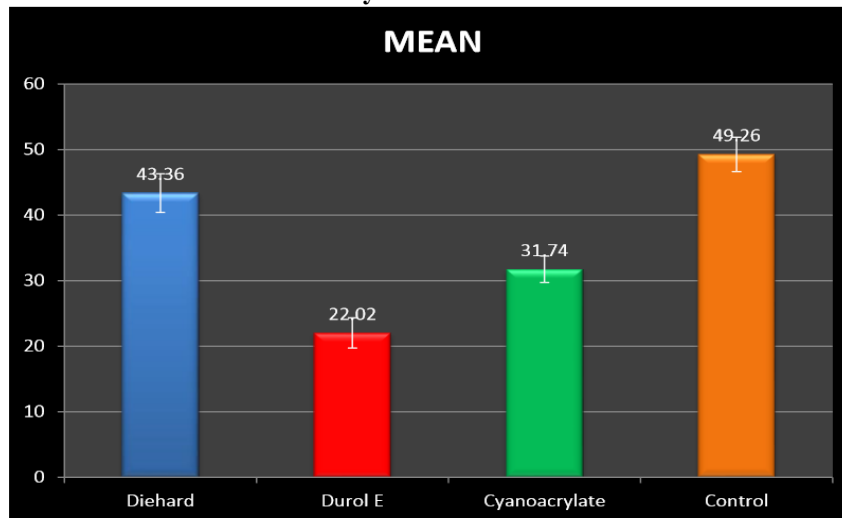
(model MV-1 PC) (figure 3). Each sample was tested 3 times at 3 different points using 300 gm of load for 20 sec. The mean of all three readings was calculated and taken up for statistical analysis.



**Figure 3: Evaluation of Surface Hardness (Microvickers) and Pyramid shaped indentation of Vickers indenter on computer screen.**

**RESULTS**

The following results were obtained from this study



**Figure 4: Mean surface hardness of type IV gypsum product after application of different die hardeners.**

Figure 4 shows Vickers hardness value (VHN) for each group after applying different die hardeners. Mean surface hardness of Group IV (control) was highest 49.26(±2.61) among all the groups followed by Group I 43.36(±2.96) and Group III 31.74(±2.01). Group II showed the least mean surface hardness 22.0(±2.31).

**Table 1: Comparison of surface hardness of type IV gypsum product using different die hardeners after applying One Way ANOVA (Analysis of Variance).**

	Sum of Squares	Df	Mean Square	F	Sig
Between Groups	6651.576	3	2217.192	353.865	.000
Within Groups	350.876	56	6.266		
Total	7002.452	59			

Table 1 shows one way AVOVA test for the analysis of significance in comparing surface hardness of type IV gypsum product using different die hardeners. Results showed statistically significant difference between the surface hardness.

**Table 2: Post- hoc analysis.**

p value	Group I	Group II	Group III	Group IV
Group I	-	0.00*	0.00*	0.00*
Group II	-	-	0.00*	0.00*
Group III	-	-	-	0.00*
Group IV	-	-	-	-

\* Significance of relationship at p < 0.05

**Table 2.1.**

		Mean Difference (I-J)	Std. Error	Sig.
GP1	GP2	21.43333*	.91401	.000
	GP3	11.71333*	.91401	.000
	GP 4	-5.80667*	.91401	.000
GP2	GP 3	-9.72000*	.91401	.000
	GP 4	-27.24000*	.91401	.000
	GP 1	-11.71333*	.91401	.000
GP3	GP 2	9.72000*	.91401	.000
	GP 4	-17.52000*	.91401	.000
	GP 1	5.80667*	.91401	.000
GP4	GP 2	27.24000*	.91401	.000
	GP 3	17.52000*	.91401	.000

Table 2 and 2.1 shows on applying Tukey post hoc test for intergroup comparison the mean surface values for groups were significantly different while comparing Group 1 with other groups. Similarly group 2, group 3

and group 4 with the other group also showed significant difference (p=0.000).

## DISCUSSION

In the present study a single coat of die hardener (mixture of resin and ketone)<sup>[7]</sup> is applied which is similar to the study done by Sudhakar et al and Harris et al. Single resinous coating<sup>[5]</sup> applied with a film thickness of 2µm are meant to reduce the surface abrasion and surface fracture especially at critical margin areas<sup>1</sup> without altering any dimensions of the finished die appreciably. 18-20 Increase in number of coating applied will result in dimensional changes (8 µm) and further decrease in mechanical properties like surface hardness and abrasion resistance.<sup>[4,5]</sup>

Results of the present study showed that Mean surface hardness (VHN) of control group (no die hardener) was highest 49.26 (±2.61) followed by vinyl and natural resins (Die Hard, MDM Co.) 43.36 (±2.96), Cyanoacrylate esters 31.74 (±2.01) and least was seen with phthalate resin (Durol E, Bego) 22.0 (±2.31). This difference was in accordance with the studies conducted by other authors<sup>[1-10]</sup> where in control group with application of no die hardener produced significantly harder surfaces (KHN) as compare to those applied with die hardener. Sanad et al in his study used model sealants and acrylic resin for increasing the surface hardness and found similar results with hardness of control group (RHN) 45.5 (±3.0) and resin treated group of 44.8 (±2.4). They stated that on evaporation of the solvent, acrylic resin remained as a rigid material which surrounded the gypsum crystals imparting strength and abrasion to resistance, whereas surface hardness of dry stone was not improved as acrylic resin is not a very hard material. However when compared to other studies<sup>[4,11]</sup> highest value of surface hardness was found to be with the application of die hardener specimen as compared to control group. The authors were of the view that after the application of single coat of die hardener solution penetrates to the greatest depth and resulted in the formation of protective layer that may hold the surface particles together which increases the surface hardness whereas application of 3-8 layers of die hardener may affect the surface topography and decreases the surface hardness. While other authors<sup>[7,12]</sup> opined that after the application of die hardener no significant effect of increase or decrease in surface hardness of stone was seen as results shown by HE et al in Nano indentation test with the control group 32.36 (±6.22) and applying die hardener 30.79 (±5.13). SEM (scanning electron microscope) explains the effect of die hardener application on stone surface which penetrated into surface specimen to depth of 3-5 µm, filling subsurface voids and sealing the gypsum by capillary action reduced surface abrasion and surface water absorption of die material. Similarly Khan et al and Schneider et al found an increase in surface hardness after application of die hardener as measurement was done by abrasion and scratch test.<sup>[13]</sup> Toreskog et al mentioned that higher hardness number for die stone does not give an indication that resistance to abrasion is also greater as hardness is only one of the many factors that can affect

the abrasion resistance which is a three body wear mechanism as former is mainly two body. The increased hardness with no die hardener application in the present study could be due to differences in measurement technique since hardness is an operationally defined physical property of materials. The surface hardness measured by conventional microhardness (Vickers measurements) in the present study was found to be lower due to the nature of the surface hardness measurement of the coating film itself, and not the film/gypsum matrix.

## Limitations

The present study had certain limitations. Die hardener was manually applied and thickness may vary according to consistency of hardener used. Only single coat of die hardener was evaluated. Surface hardness was measured in the present study whereas abrasion resistance is also equally important for the fabrication of accurate die. So, furthermore studies are suggested to evaluate the abrasion resistance and application of multiple coatings of different die hardeners applied with standardisation of thickness on type IV gypsum product.

## CONCLUSION

**Within the limitations of this study the following conclusions can be drawn**

1. Die hardener coating applied in this study decreased the surface hardness of gypsum product.
2. Among the different die hardeners evaluated, mixture of vinyl and natural resins (Die hard) showed highest value for the surface hardness followed by Cyanoacrylate esters and phthalate resin (Durol E, Bego) respectively.

## REFERENCES

1. Harris PE, Scott H, Lindquist TJ, Stanford CM. Alteration of surface hardness with gypsum die hardeners. *J Prosthet Dent*, 2004; 92(1): 35-38.
2. Rosenstiel SF, Land MF, Fujimoto J. *Contemporary Fixed Prosthodontics*. Mosby Elsevier India, 2007; 3: 529-530.
3. Craig RG. *Restorative dental materials* Mosby: St. Louis, 2006; 12: 391-416.
4. Fukui H, Lacy AM, Jendresen MD. Effectiveness of hardening films on die stone. *J Prosthet Dent*, 1980; 44(1): 57-63.
5. Ghahremannezhad HH, Mohamed SE, Stewart PG, Weinberg R. Effects of cyanoacrylates on die stone. *J Prosthet Dent*, 1983; 49(5): 639-46.
6. Fan P L, Power J M, Reid B C. surface mechanical properties of stone, resin and metal dies. *JADA*, 1981; 103: 408-11.
7. He LH, Vuuren LJV, Plantiz N, Swain MV. A micro mechanical evaluation of the effects of die hardener on die stone. *Dental materials journal*, 2010; 29(4): 433-437.
8. Lee WS, Kim JH, Kim TS, Chin HY. A comparison of surface hardness and microstructural characteristics between a type IV stone with and

- without die hardening treatment and a polyurethane resin die material. Korean journal of dental technology, 2012; 34(3): 227-235.
9. Bajada S B, Makinson O F. the effect of some surface treatments to dental modelling stones. Australian dental journal, 1974; 118-21.
  10. Kim YR, Park JM, Song KY. The influence of the die hardener on gypsum die. Korean journal of dental prosthodontics, 2007; 45(4): 546-553.
  11. Campangi W V. Measurement of coating agents used for surface protection of stone dies. J Prosthet Dent, 1986; 55(4): 470-74.
  12. Dulaimi SF. Evaluation of the effect of microwave irradiation and die hardener application on surface hardness of type IV dental stone. Tikrit journal of dental sciences, 2016; 4: 129-135.
  13. Khan FM, Dall Q A, Sajid K. Effect of cyanoacrylate as surface hardener on gypsum die material. JLUMHS, 2012; 11(3): 185-189.