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Comparison of the Effect of Addition of Cyanoacrylate, Epoxy Resin, and Gum Arabic on Surface Hardness of Die Stone

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Abstract

Purpose: To observe the effects of incorporating cyanoacrylate, epoxy resins, and gum arabic on the abrasion resistance of type IV gypsum die materials.
Materials and Methods: Forty specimens were prepared and divided into four groups (10 specimens in each group), namely group A (control), group B (die stone mixed with cyanoacrylate), group C (die stone mixed with epoxy resin), group D (die stone mixed with gum arabic). All the specimens were subjected to abrasion testing, wear volume analysis, Fourier transform infrared spectroscopy (FT-IR), and scanning electron microscope (SEM) analysis.
Results: Abrasion testing showed maximum wear in the control group and minimum wear in the gum arabic group. Intergroup differences were statistically significant ($p < 0.001$). The largest mean difference was between control and gum arabic. The lowest was between cyanoacrylate and the control group. The mean wear volume was lowest in the gum arabic group (4.23 mm^3) and highest in the control group (6.78 mm^3). The FT-IR graphs of the gum arabic models showed the presence of CH_2, which is responsible for its binding activity. SEM revealed that the irregular particles of gum arabic display an interlocking arrangement. This jigsaw puzzle pattern results in stronger physical bond formation.
Conclusion: Observations from this study showed that the addition of gum arabic increases resistance to abrasion in type IV gypsum. Cyanoacrylates are good adhesives as well, but a major drawback is that they have very low resistance to chemical action with water and physical actions such as sunlight. Epoxy resins are powerful adhesives, but they attain their full efficiency when cured with heat. Cyanoacrylate and epoxy resin displayed poor physical bonding, primarily because of inhomogeneity.

Gypsum is a mineral composed of calcium sulfate dihydrate, with the chemical formula $CaSO_4.2H_2O$. Gypsum is found in nature as flattened, often twined crystals and transparent cleavable masses known as selenite. It is also available in compact and granular forms.[1]

Gypsum is widely used for preparation of dental casts for records and laboratory procedures.[4,7] The basic process of manufacturing all types of gypsum products consists of dehydration (calcining) by heating to remove the water constituent. The gypsum is heated in an autoclave under steam pressure, or it can be dehydrated in boiling 30% calcium chloride solution. The powder obtained by this procedure is the densest of all types of dental stone, and is described as a high strength dental stone called Densite, or type IV die stone.

Cast hardness and abrasion resistance are of critical value for working casts and dies that undergo the rigors of waxup, fit check of castings, and their final finishing. To increase surface hardness of dental stone, surface coatings or various treatments have been recommended.[2] Materials such as cyanoacrylate, die sealants, and resins have been found to increase surface hardness and reduce surface fracture at critical marginal areas of dies.[6]

The purpose of this study was to observe the effects of incorporating cyanoacrylate, epoxy resins, and gum arabic in type IV gypsum die materials with respect to abrasion resistance.[3]

Materials and methods

The study was performed at the Department of Prosthodontics, Saraswati Dental College Lucknow, India; Department of Materials Science Engineering, Indian Institute of Technology, Kanpur, India; and Department of Chemistry, Banaras Hindu

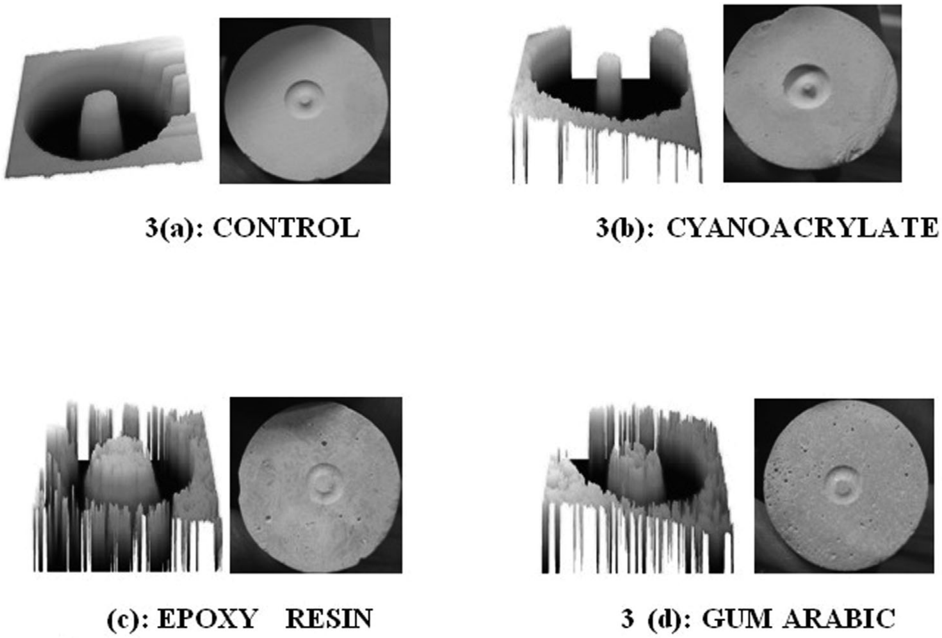

Figure 1 Laser profilometry.

University (BHU), Varanasi, India. The study consisted of four groups: A (control) and B, C, D (experimental). Each group had 10 specimens. Thus, in all, 40 specimens were tested.

Group A (control): 10 specimens were prepared as per the manufacturer's instructions with no modification (i.e., 23 mL of water with 100 g of Type IV gypsum).

Group B (die stone mixed with cyanoacrylate): 10 specimens were prepared by incorporating 0.2 mL of cyanoacrylate (Pidilite, Mumbai India) with 100 g of Type IV gypsum in 23 mL of water.

Group C (die stone mixed with epoxy resin): 10 specimens were fabricated by incorporating 0.2 mL of epoxy resin (araldite) with 100 g of Type 1V gypsum in 23 mL of water.

Group D (die stone mixed with gum arabic): 10 specimens were fabricated by incorporating 2.5 g of gum arabic (Fisher Scientific, Mumbai, India) with 100 g of Type IV gypsum in 23 mL of water.

Abrasion testing

The equipment used was a pin-on disc Tribometer (T3 400; Nanovea, Irvine, CA). The tribology parameters used for the experiment are as follows. The stainless steel abrader was spherical with a 3 mm diameter. The load applied and maintained throughout was 5 N, and the speed was kept constant at 150 rpm. The duration for which each specimen was subjected to experimentation was 30 minutes, wherein a total distance of 42.39 m was covered.

The weight of the test specimens prior to testing was recorded using a digital weighing machine (Mettler AE 100; Mettler Toledo, Leichester, UK). The specimen was then locked into position on the abrasion testing machine and subjected to sliding wear for the specified duration. Once the cycle was completed, the specimen was unloaded from the device and weighed again. The difference between the initial and final weight provided the numerical value of the mass loss owing to wear. This procedure was repeated for each specimen, and all observations were recorded. Specimens were subjected to wear volume analysis using a laser profilometer (PGK 120; Mahr, Göttingen Germany).

Wear volume analysis

Each specimen was subjected to an infrared laser beam of 780 nm wavelength. Using the wear volume obtained from the software, the wear rate was calculated. This was followed by hardness and modulus calculation using the CSM Micro-Hardness Tester (also known as the Vickers' Hardness Test). Each specimen was indented only once (Fig 1). The objective was to derive information about specific properties, including hardness, elastic modulus, fracture toughness, and formation and to achieve a clearer understanding of the substrate/additive interactions.

Fourier transform-infrared spectroscopy (FT-IR)

The specimens were crushed to powdered form and mixed homogenously with potassium bromide. Small pellets of uniform size were prepared from this mixture. These pellets were kept for observation in the Varian 3100 FT-IR spectroscope, and graphs were obtained. By performing this test, the quality or consistency of the specimen as well as the presence of various compounds in it was determined. This was to verify that the additive had spread uniformly throughout the die stone mix.

Table 1 Abrasion resistance test with difference in weight loss

Group	Number of specimens	Mean weight loss (g)	Std. deviation	Minimum weight loss (g)	Maximum weight loss (g)
Control	10	0.021190	0.0070549	0.0080	0.0287
Cyanoacrylate	10	0.018340	0.0053932	0.0099	0.0256
Epoxy resin	10	0.014210	0.0079264	0.0011	0.0241
Gum arabic	10	0.006380	0.0024073	0.0029	0.0102
Total	40	0.015030	0.0081198	0.0011	0.0287

$F = 11.246$; $p < 0.001$.

Table 2 Wear volume analysis

Specimens	Mean wear volume (mm^3)	Mean wear rate (mm^3/Nm)
Control	6.78	4.91×10^{-2}
Cyanoacrylate	5.93	4.30×10^{-2}
Epoxy	5.36	3.89×10^{-2}
Gum arabic	4.23	3.07×10^{-2}

Table 3 Intergroup comparison of abrasion resistance

Comparison	Mean diff. (g)	SE	*p*
Control vs cyanoacrylate	0.0029	0.0027	0.722
Control vs epoxy resin	0.0070	0.0027	0.066
Control vs gum arabic	0.0148	0.0027	<0.001
Cyanoacrylate vs epoxy resin	0.0041	0.0027	0.436
Cyanoacrylate vs gum arabic	0.0120	0.0027	0.001
Epoxy resin vs gum arabic	0.0078	0.0027	0.032

Scanning electron microscopy

The specimens were thoroughly crushed and dried completely. All specimens were tested under a scanning electron microscope (SEM) at a magnification of 500×. Data were analyzed using SPSS v15.0 (SPSS Inc., Chicago, IL). Distributions were checked for normality using Kolmogorov Smirnov test. As all four distributions were found to be normal, a parametric evaluation plan was adopted. Intergroup comparisons were done using ANOVA. Between-group differences were analyzed using the Tukey HSD test. The confidence level of the study was kept at 95%; hence, $p < 0.05$ indicated a statistically significant difference.

Results

Table 1 shows the results of the abrasion resistance test with difference in weight loss. Table 2 shows the wear volume analysis of the four groups.

$$\text{Wear rate} = \frac{\text{wear volume(mm}^3)}{\text{Load} \times \text{no.of cycles} \times \text{stroke length}}.$$

Laser profilometry of the wear-tested specimens was done to visualize the surface topography. Table 3 shows intergroup comparison of wear resistance.

The largest difference was observed in the control group and the smallest in the gum arabic group. Intergroup differences were statistically significant ($p < 0.001$). The mean difference was largest between the control and gum arabic, and the smallest was between the cyanoacrylate and control groups.

Statistically, no significance was observed between control, cyanoacrylate, and epoxy resin groups ($p > 0.001$). Gum arabic had a significantly lower mean value as compared to the other groups.

FT-IR spectroscopy

On performing the tests, four graphs were obtained for four specimens (Figs 2 to 5). These graphs depict different peaks, which denote the presence of different chemical groups. The first two consecutive peaks, around 3400 to 3600 cm^{-1}, denoted a hydroxyl group due to the presence of water. Water was used as a vehicle. Peaks around levels 1600 cm^{-1} signified the presence of a sulfate group, which was present in gypsum. The peak around 2924 cm^{-1} depicted the presence of a vinyl group that imparted adhesiveness to gum arabic (Fig 2). In the graph for the control group, there was no peak at 2924 per cm, proving that plain die stone mix did not contain the vinyl group (Fig 5).

SEM

All the four specimens were subjected to 500× magnification under the SEM. Interparticle space in group D (gum arabic/gypsum) was the smallest followed by group A (gypsum). Group C (epoxy resin/gypsum) and group B (cyanoacrylate/gypsum) specimens showed greater and variable inter-particle distance. The large number and greater size of voids in groups C and B rendered them more vulnerable to abrasion (low abrasion resistance). Group D specimens (gum rabic/gypsum) had the highest abrasion resistance, followed by groups A, C, and B.

Discussion

This study was designed to evaluate and compare the abrasion resistance of die stone (Type IV dental stone) mixed with various additives such as cyanoacrylate, epoxy resin, and gum arabic. Thus the binding ability (adhesive property) of these additives could be assessed.

Cylindrical models of die stone were prepared from a split brass mold with internal diameter and length of 1 inch. Four groups were made depending on the adhesives incorporated in the die stone while mixing. The brass mold was lined with petroleum jelly (separating medium), and the junction between the two halves was sealed with modeling wax to prevent the incorporation of air bubbles in the models. Die stone was mixed in a vacuum mixer and poured in the mold. The mix was allowed to set and then taken out of the mold.[4-7]

These models were tested for abrasion resistance by tribology, SEM spectroscopy, and FT-IR. Tribology tests revealed that in models with gum arabic, the depth of penetration of the pointer was shallower than with the other groups. This preliminarily proved that the group with gum arabic as a binder showed higher scratch resistance.

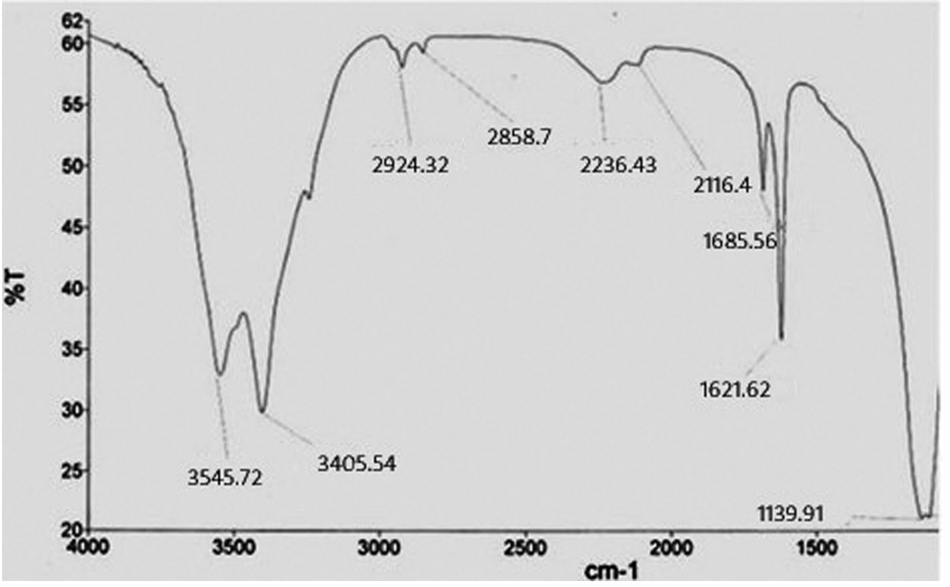

Figure 2 FT-IR graph of gum arabic group.

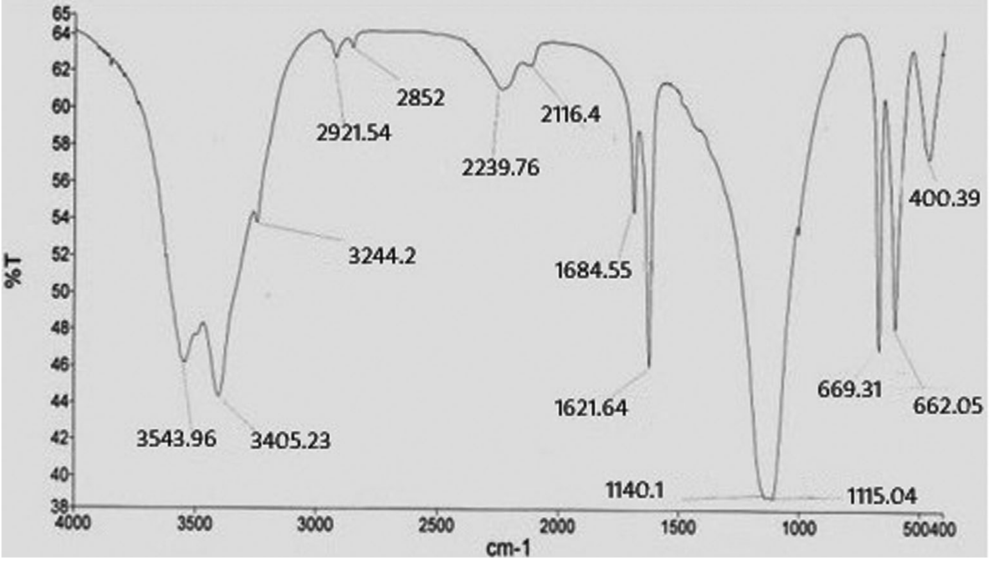

Figure 3 FT-IR graph of epoxy resin group.

To further validate the results, FT-IR was performed on all groups to check the chemical identities and find out why gum arabic performed better. The FT-IR graphs for gum arabic models showed the presence of CH_2, which is responsible for its binding activity. Earlier studies[1,2,4-6,8] evaluated physical and mechanical properties. This study probed the chemical basis of strong bonding of gypsum and gum arabic.

SEM revealed that the irregular particles of gum arabic display an interlocking arrangement. This jigsaw puzzle pattern results in stronger physical bond formation. Thus, the extent of chemical and physical bonding is greater in specimens with gum arabic than the other groups.

Earlier experiments performed to estimate scratch resistance of die stone used these adhesives as a surface paint on the models.[9] This increased the dimension of the models. Applying a layer of adhesive on the surface of a die stone cast would change the dimensions. In cases of abutment prepared casts, painting a layer of surface adhesive would mean changing the dimensions of the abutment minimally, which would result in incorrect fit of the prosthesis.

Sanad et al[10] used only epoxy resin as a surface hardener while Nitasha et al[3] used gum arabic. Cyanoacrylate was used by Muhammad et al as a surface hardener.[2,17] Application of the surface hardener was a method used by all earlier research

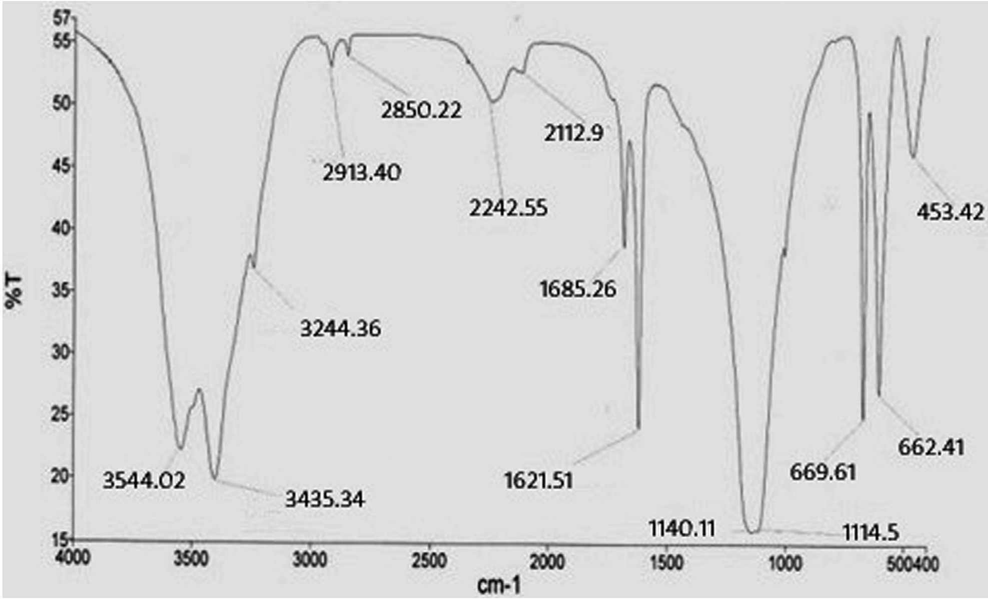

Figure 4 FT-IR graph of cyanoacrylate group.

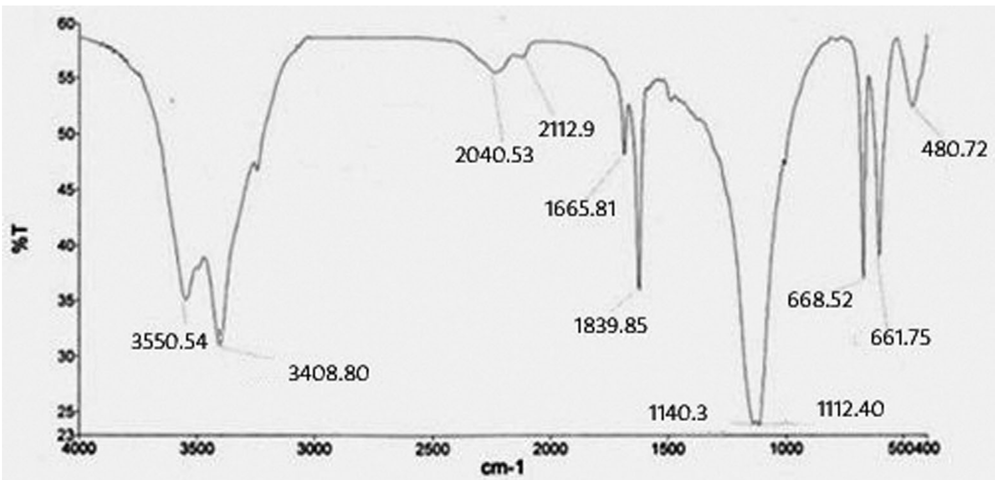

Figure 5 FT-IR graph of control group.

groups. In the present study all three additives were used while mixing to enhance toughness without altering the surface dimensions. Incorporating the adhesives in the mix was directed at minimizing the laboratory error, thus preventing clinical errors, while enhancing the abrasion resistance of the die stone cast.

Sanad et al[10] standardized the incorporation of 1% gum arabic for types II and III gypsum. They proposed that this was the most efficient concentration to enhance abrasion resistance and strength of types II and III gypsum.[14,15] No research group has formulated an appropriate concentration for incorporating gum arabic in type IV gypsum. This study found that incorporating 2% gum arabic in type IV gypsum resulted in optimum abrasion resistance in the gypsum model (Table 4).

Conclusion

Observations obtained from the above tests show that the addition of gum arabic increases resistance to abrasion in type IV gypsum. Chemically stable hydrogels are formed by the bonding of vinyl groups to the backbone of polysaccharides. Physically, the irregularly shaped particles of die stone interlock with the particles of gum arabic, resulting in a stronger binding.

Cyanoacrylates are good adhesives as well, but a major drawback is that they have very low resistance to chemical action with water and physical actions such as sunlight. The medium of mixing die stone is water. There is a rapid anionic polymerization reaction on exposure to water, and the cyanoacrylate

Table 4 Abrasion resistance with different concentrations of gum arabic

Group	Weight before testing (g)	Weight after testing (g)	Difference in weight (g)	Wear volume (mm^3)	Wear rate (mm^3/Nm)
1%	18.0262	18.0232	0.0030	4.23	3.07×10^{-2}
1.5%	18.1542	18.1510	0.0032	4.23	3.07×10^{-2}
2%	18.2622	18.2593	0.0029	4.23	3.07×10^{-2}
2.5%	18.3712	18.3681	0.0031	4.23	3.07×10^{-2}
3%	18.4882	18.4846	0.0036	4.23	3.07×10^{-2}

does not spread homogeneously. Hence, it is not a suitable additive to increase the abrasion resistance of die stone. Epoxy resins are powerful adhesives that attain their full efficiency when cured with heat; however, if the mix containing die stone and epoxy is heated during manipulation, then the die stone would set faster, which would prevent the epoxy resin from spreading homogeneously within the gypsum mix.

Cyanoacrylate and epoxy resin displayed poor physical bonding, primarily because of inhomogeneity. Bonding of heterogenous particles of these two synthetic molecules with gypsum varied in different areas of the tested specimens. Interparticulate distance was both variable and large in certain areas, as visible on SEM examination of specimens of groups B (cyanoacrylate/gypsum) and C (epoxy resin/gypsum). Group D specimens (gum arabic/gypsum) showed closely adhered particles of the two components and a regular interparticulate distance. The results showed a direct correlation of abrasion resistance with particulate adherence in the different specimens. Therefore, it is the physico-chemical bond of gum arabic with die stone that assigns better abrasion resistance to the set mix.

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References

1. Skinner, Gordon CC: Some experiments on the surface hardness of dental stones. J Prosthet Dent 1956;6:94-100
2. Muhammad FK, Abdul QD, Khizran S: Effect of cyanoacrylate as surface hardener on gypsum die material. JLUMHS 2012;11:185-189
3. Nitasha G, Rajshekhar S, Dayakare HR, et al: Effects of gum Arabic and calcium hydroxide on the surface hardness of Type I, Type II and Type III gypsum products- a comparative study. Ind J Dent Sci 2013;5:18-20
4. Schneider RL, Taylor TD: Compressive strength and surface hardness of type IV die stone when mixed with water substitutes. J Prosthet Dent 1984;52:510-514
5. Alsadi S, Combe EC, Cheng YS: Properties of gypsum with the addition of gum Arabic and calcium hydroxide. J Prosthet Dent 1996;76:530-534
6. Lindquist TJ, Stanford CM, Knox E: Influence of surface hardener on gypsum abrasion resistance and water sorption. J Prosthet Dent 2003;90:441-446
7. Abdelaziz KM, Attia A, Combe EC: Evaluation of disinfected casts poured in gypsum with gum Arabic and calcium hydroxide additives. J Prosthet Dent 2004;92:27-34
8. Roy SM, Sridevi J, Kalavathy N: An evaluation of the mechanical properties of type III and type IV gypsum mixed with two disinfectant solutions. Indian J Dent Res 2010;21:374-379
9. Wankhade SV, Sanghavi KV, Rajguru V, et al: A comparative evaluation of six commonly used types of die materials for the property of abrasion resistance, at two time intervals: An in vitro study. Int J Prosthodont Rest Dent 2013;3:1-6
10. Sanad ME, Coombe EC, Grant AA: Hardening of model and die materials by an epoxy resin. J Dent 1980;8:158-162
11. Earnshaw R, Smith DC: The tensile and compressive strength of plaster and stone. Aus Dent J 1966;11:415-422
12. Fukui H, Lacy AM, Jenderson MD: Effectiveness of hardening films on die stone. J Prosthet Dent 1980;44:57-63
13. Fan PL, Powers JM, Reid BC: Surface mechanical properties of stone, resin and metal dies. J Am Dent Assoc 1981;103:408-412
14. Winkler MM, Monaghan P, Gilbert JL, et al: Comparison of four techniques for monitoring the setting kinetics of gypsum. J Prosthet Dent 1998;79:532-536
15. Canay S. Hersek N, Ciftci Y, et al: Comparison of diametral tensile strength of microwave and oven-dried investment materials. J Prosthet Dent 1999;82:286-290
16. Yap AU, Yap SH, Teo JC, et al: Microwave drying of high strength dental stone: effects on dimensional accuracy. Oper Dent 2003;28:193-199
17. Sabooni MR, Ghanbarzadeh J: Comparison of tap water, distilled water and slurry water on surface hardness of gypsum die. J Med Sci 2007;7:1350-1353
18. He LH, van Vuuren LJ, Planitz N, et al: A micromechanical evaluation of the effects of die hardener on die stone. Dent Mater J 2010;29:433-437

