Evaluation of the Various Drying Methods on Surface Hardness of Type IV Dental Stone
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Introduction
Die materials play an important role during the fabrication of indirect dental restorations and prostheses. Indirect method of fabrication of inlays, crowns and bridges demand die materials that are of the highest quality with respect to accuracy and strength. A die should be accurate in every respect, i.e. dimensionally stable over time, minimal setting expansion, ease and efficiency of manipulation, compatible with impression materials, hard enough to withstand the fabrication process, resistant to the inadvertent abrasions caused during fabrication, good transverse strength and a stable shelf life.¹ Dentists and laboratory technicians depend on these characteristics to predictably fabricate accurate prostheses.

Surface hardness serves as a satisfactory criterion of the surface condition of the material.²–⁴ To increase the surface hardness of Type IV dental stone, various methods have been tried such as air drying for 24-48 h, micro oven drying to reduce the waiting period, incorporating some additives into die stone, application of die hardener etc.⁵⁻⁷ Studies regarding the effect of these methods on surface hardness of Type IV dental stone are not conclusive.

Hence, this study was undertaken to investigate the effect of these methods on surface hardness of Type IV dental stone and to find out the most effective method to increase the surface hardness.

Materials and Methods
Four stainless steel master dies were prepared with a height of 40 mm and a diameter of 20 mm according to ANSI/ADA specification products⁶ (Figure 1). Polyvinyl siloxane impression material was used to make the impressions of metal die. The molds were poured with two types of Type IV die materials and grouped as Group A (Kalrock, Kalabhai, Mumbai) and Group B (Pearl Stone, Asian Paints, Gujarat) of 60 specimens each. These specimens were further subdivided into groups of 15 specimen each. All the 15 specimens of each group were evaluated for surface hardness after the following.

- 1 h of mixing (1 h)
- 1 h from the time of removal from the mold and placing in the micro oven (MV) for 10 min at low power, (1 h/MV) (Figure 2)
- 24 h of air drying (24 h/AD) and
- Application of the die hardener after air drying the specimens for 24 h (24 h/DH) (Figure 3).

Results of the study showed that Group A showed better results than Group B at all times. Application of the die hardener showed highest hardness values followed in the order by 24 h air drying, microwave oven drying and 1 h air drying in both groups. The study showed that air drying the dies for 24 h followed by application of a single layer of the die hardener produced the best surface hardness and is recommended to be followed in practice.

Key Words: Air drying, microwave oven drying, surface hardness, Type IV dental stone
Evaluation of knoop hardness

Knoop microhardness test was performed using a hardness tester, equipped with knoop diamond indenter, in accordance with ADA specification No. 25 for dental gypsum products. Three indentations were obtained for each specimen using 300 g (3N) load for 20 s and average Knoop hardness number (KHN) of the three readings for each specimen was recorded. The obtained data was tabulated and subjected to statistical analysis.

Results

The hardness of Group A specimens was 24.6 ± 0.4 after 1 h air drying, 58.2 ± 0.88 after microwave oven drying, 60.47 ± 0.41 KHN after 24 h air drying and 64 ± 0.54 KHN after the application of die hardener. The hardness of Group B specimens was 19.91 ± 0.64 KHN after 1 h air drying, 38.28 ± 0.55 KHN after microwave oven drying, 40.2 ± 0.63 KHN after 24 h air drying and 45.59 ± 0.63 KHN after the application of die hardener.

The hardness in both the groups was higher after application of the die hardener, followed by 24 h air drying, microwave oven drying and 1 h air drying (Graph 1 and Tables 1 and 2). A Tukey post-hoc test revealed that the hardness was significantly higher at each level.

There was a statistically significant difference between the groups A and B as determined by analysis of variance test (ANOVA). Group A (Kalrock) was better than Group B (Pearl.

Table 1: Mean hardness (KHN) of Group A (kalrock) at various times.

| Time intervals | n  | Mean (hardness) | SD      | Standard error |
|----------------|----|-----------------|---------|----------------|
| 1 h            | 15 | 24.6133         | 0.40685 | 0.10505        |
| 1 h/MV         | 15 | 58.2267         | 0.88678 | 0.22897        |
| 24 h/AD        | 15 | 60.4733         | 0.57998 | 0.14975        |
| 24 h/DH        | 15 | 64.0067         | 0.54178 | 0.13989        |
| Total          | 60 | 51.8300         | 15.99353| 2.06476        |

KHN: Knoop hardness number, SD: Standard deviation

Table 2: Mean hardness (KHN) of Group B (pearl stone) at various times.

| Time intervals | n  | Mean (hardness) | SD      | Standard error |
|----------------|----|-----------------|---------|----------------|
| 1 h            | 15 | 19.9133         | 0.64128 | 0.16558        |
| 1 h/MV         | 15 | 38.2800         | 0.54929 | 0.14182        |
| 24 h/AD        | 15 | 40.2133         | 0.63343 | 0.16355        |
| 24 h/DH        | 15 | 45.5933         | 0.62845 | 0.16227        |
| Total          | 60 | 36.0000         | 9.76641 | 1.26084        |

KHN: Knoop hardness number, SD: Standard deviation

Graph 1: Comparison of mean surface hardness of Type IV dental stone after various treatments.

Figure 1: Stainless steel cylinder and mold space in Poly vinyl siloxane impression material.

Figure 2: Specimens placed in micro oven after 1h for 10 min.

Figure 3: Application of die hardener.
stone) at all times. Drying by micro oven was better when compared with 1 h air drying and comparable to 24 h air drying. 24 h air drying followed by application of the die hardener was the best in hardness for both the groups.

Discussion

In general sense, hardness refers to “resistance to indentation or scratching.” The surface hardness and abrasion resistance is directly related to compressive strength when the gypsum material is in dry condition. Wet die hardness is half of the total hardness of dry die. Water requirement of each type of the gypsum product will be different due to the difference in density of the powder. Density in turn depends on the adhesiveness of particles in the dry powder state, which persists even when they are suspended in water. Dental stone requires less water than plaster as the dental stone particles are denser than plaster. While setting, some of the excess water, which does not react, is trapped in the mass. The presence of excess water has significant consequence on the strength and hardness of the material. Therefore, this trapped excess water is to be removed to improve their properties.

The property, which is measured for the hardness of die materials, is their surface hardness and since compressive strength indicates the condition of the entire specimen, it was found that the hardness increases faster than the compressive strength. The surface being drier than the center is a necessary condition for the diffusion process. In practice, as these materials rarely fracture or break, compressive strength appears undesirable criteria for material condition. Surface hardness has been established as the most important property as the surface of the die is constantly worked and should withstand indentation and scratching.

Knoop hardness has been used to evaluate very hard and brittle materials with low modulus of elasticity like enamel, amalgam, gypsum, and porcelain. Various methods have been followed to improve the surface hardness such as surface coatings or treatments like soaking the dies in oil or boiling water.

In the present study, a total of 120 specimens were obtained from the mold (60 for each product). These specimens were tested in Knoop hardness testing instrument after subjecting them to air drying for 1 h and 24 h, micro oven drying after 1 h and application of die hardener after 24 h.

The results of the present study indicate that the hardness of die stone increased as a function of time. The surface hardness value of both groups was significantly higher if the die hardener was applied after 24 h of air drying. The possible reason could be that the application of die hardener after removal from the mold following air drying the specimens for 24 h would have allowed the solution to penetrate to the greatest depth and resulted in the formation of the protective layer that may hold the surface particles together. This increase in the surface hardness is also beneficial for abrasion resistance. Previous studies have differed in the application of number of layers of die hardener and application of the load. Application of 3-8 layers of the die hardener may affect the surface topography and decrease the surface hardness. Hence, in this study, a single layer of the die hardener was applied to produce a smooth and shiny surface.

A 24 h air drying without the die hardener was significantly higher in hardness than at 1 h and was comparable to specimens dried in micro oven. In this study, drying the specimen by micro oven for 10 min at low power was used to minimize the rapid steam expulsion from the specimen. Although micro oven drying reduces the waiting period as compared to 24 h air drying, the hardness values obtained by micro oven drying was less than 24 h air drying for both product A and B and it seemed to produce rough and porous surface. The rapid removal of water may not allow the fine crystals to precipitate to anchor the larger crystals of dihydrate formation. In air-dried samples, as the last traces of water leave, fine crystals of gypsum precipitate to anchor the larger crystals of dihydrate formation.

At 1 h, the hardness value was significantly lower when compared with other drying methods for both the products. This may be attributed to the presence of residual water, which weakens the specimen. As the time progresses, there is an increase in the hardness due to loss of residual water.

On comparing the two types of products, it was observed that hardness value of Group A (Kalrock stone) was higher at all times than Group B (Pearl stone).

While microwave oven drying seemed to produce a rough surface, the 24 h air-dried specimens looked smooth and shiny. Further studies are needed to be undertaken to study these surface characteristics of die stones after various treatments. Furthermore, nano-indentation hardness tests may be used to test the surface hardness of die materials.

Conclusion

The following conclusions were drawn from this study:

1. The specimens placed in micro oven for 10 min at low power improved the surface hardness for both products. 1 h/MV specimens were markedly superior to 1 h specimens but drying the specimens in micro oven produced visibly rough surface
2. 24 h/AD specimens exhibited higher hardness values than 1 h and 1 h/MV specimens but lesser values than 24 h/DH. The application of a single layer of the air thinned die hardener after 24 h of air drying showed greatest hardness values for both Kalrock and Pearl stone. This also seems to produce smooth and shiny surface
3. Kalrock stone was better than Pearl stone at all treatments.
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