# Microhardness of Gypsum Submitted to Different Methods of Pouring and Storage

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# Abstract

**Statement of Problem:** Whether using different methods of pouring and storage will affect to the surface hardness of gypsum Type IV casts.

**Aim:** To compare the surficial microhardness (Knoop) of gypsum Type IV submitted to different methods of pouring and storage.

Material and Method: Seventy-five specimens were made with gypsum Type IV (Herostone, Coltene) and distributed in 5 groups (n=15). A metal device was fabricated to simulate the tray and standardize the impression procedures. A Polyvinyl Siloxane (PVS. Express XT, 3M ESPE) was manipulated according to the manufacturer's instructions. After the setting time, the impression was removed and cleaned with water, dried, and disinfected with 2% glutaraldehyde. The experimental groups formed were: 1) Control/15-the die was removed from the impression material 15 min after pouring the cast; 2) 4 hour - the die was removed from the impression material 4 h after pouring the cast; 3) 40°C-the die was removed from the impression material, and stored during 30 min at 37°C; 4) sulfate/K- the impression was immersed in 2% potassium sulfate solution during 5 min after the gypsum pouring and removed from the PVS after 15 min; 5) sulfate/Ca- the impression was immersed in 2% Ca sulfate solution during 5 min after gypsum was poured and removed from the PVS after 15 min. The microhardness test Knoop was performed at 3 different points. The average was used for the statistical analysis. The normality of the data was confirmed by Kolmogorov-Smirnov test (P=0.200). Statistical differences between groups were analyzed by one-way ANOVA. The details of the analysis were performed using the Dunnett test and the Tukey HSD test. All tests were performed with a significance level of 5%.

**Results:** The lowest microhardness values were found in the Control group/15<4=40°C=sulfate/K= sulfate/Ca

**Conclusion:** All the experimental treatment methods increased significantly the surface microhardness of the gypsum Type IV when compared to the control.

**Clinical relevance:** Die treatments can improve the hardness of the gypsum die, preventing cast fracture.

Keywords: Impression materials • Knoop microhardness • Gypsum • Microhardness

# Introduction

The demand for aesthetic treatments such as indirect restorations is intertwined with several factors, including diagnosis and planning of restorative treatment. Although all phases of the treatment are essential to obtain a favorable prognosis, it is the impression step that assists in the accuracy of the laboratory processes. The impression procedure, from which the negative imprint of the dental structure is obtained, is intended to accurately reproduce all details of the tooth preparation, as well as the surrounding oral tissues. The impression allows the lab technician to reproduce the characteristics of the oral cavity using a gypsum cast. The casts, constructed with sundry types of dental gypsum materials, especially Type IV gypsum [1] are widely used in laboratory procedures for different purposes, such as the fabrication of temporary restorations, as well as, permanent indirect restorations.

The gypsum casts must present morphological fidelity, dimensional stability, and fine detail reproduction, as well as, they should maintain these characteristics during laboratory management [2, 3]. The surface hardness is a fundamental property for the preservation of the detail fidelity of the cast during the management to fabricate a prosthetic restoration. An inaccurate cast with no wear resistance may cause fracture [4] and fail during abrasive procedures [5] and therefore can lead to a prosthesis that does not fit in the mouth. The accuracy with which the details are reproduced depends on several factors, such as the material selection and its handling during the reproducibility steps [6-8]. The objective of this study was to evaluate the surface microhardness of type IV gypsum in specimens obtained under different conditions. The null hypothesis tested is that there will be no significant differences in microhardness value between the control group and the other pouring and storage methods.

### **Material and Methods**

Seventy-five specimens prepared with gypsum Type IV (Herostone, Coltène, Altstätten, Switzerland) were randomized and distributed into 5 groups (n=15). For the preparation of the specimens, a metal matrix with 2 cm diameter and 1cm height was used to simulate a tray. The impression material was mixed and placed inside the matrix. When set, the impression was removed from the matrix, washed in running water, dried, and disinfected by immersion in 2% glutaraldehyde solution. The impression material was used according to the manufacturer's recommendations.

The conditions for obtaining the specimens from each group were established immediately after the removal of the template from the metal matrix. For the control group /15 the gypsum model was separated from the impression15 min after pouring the cast into the mold. In the group 4 h, the separation of the model was performed 4 hours after pouring. In the 40°C group, after impression and cast separation, the gypsum model was submitted to a 40°C oven temperature for 30 min. In the Sulphate/K group, after immersion of the mold in 2% Potassium Sulphate solution for 5 min, the impression was poured and the separation was carried out 15 min after pouring. For the sulphate/Ca group, after immersion of the impression in 2% Calcium sulphate solution for 5 min the impression was poured and the separation of the cast from the impression occurred 15 min after pouring. All materials were manipulated according to the manufacturer's instructions.

The experiments were conducted by measuring the Knoop microhardness of each specimen after treatment. The evaluation of the superficial microhardness was performed using a load of 20 kgf for 15sec (Shimadzu series HMV-2/Kyoto-Japan).

The normality of the data was confirmed by the Kolmogorov-Smirnov test (P=0.200). Statistical differences between groups were analyzed by one-way ANOVA. The details of the analysis were performed using the Dunnett test and the Tukey HSD test. All tests were performed with a significance level of 5%. The analyses were performed with the assistance of Microsoft Excel 2007 (Microsoft Office system 2007) and SPSS 17 (SPSS Inc., Chicago, IL, USA) software.

# **Results**

The data are presented in Table 1. The one-way ANOVA test suggested

Table 1.	KHN, standard	deviation by	the Dunnett	and Tukey	HSD tests.
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Groups	KHN (SD)	
Control/15	38,93 (7,43) a	
4h	58,80 (8,32) b	
40°C	66,80 (10,23) b	
Sulfate/K	69,40 (16,79) b	
Sulfate/Ca	70,27 (18,11) b	

a statistical difference between the groups evaluated (p<0.001). The lowest values of Knoop Hardness Number (KHN) were observed in the control group(38,93). The comparison between the other groups showed no statistically significant difference among them (58,80-70,27) (Table 1).

Equal lowercase letters indicate no statistical difference between Tukey HSD test, at a significance level of 5%.

### Discussion

Based on the results of this study, the null hypothesis was rejected. For the microhardness, higher values were obtained using different methods of pouring and storage, when compared to the control group. In the present study, the surface microhardness of the Type IV gypsum was investigated by the Knoop microhardness test. The Knoop hardness is considered a simple and safe test for the analysis of very hard materials, such as gypsum [9,10].

Different pouring and storage methods are suggested in the literature aiming to improve the hardness of the gypsum casts during their fabrication process, including handling, pouring, and trimming the material [11-13]. Some treatments involve the immersion of the gypsum cast in a container with slurry water, to provide a saturated solution of calcium sulphate [10].

The results of this study showed that the gypsum hardness could be improved by the pouring and storage methods proposed. The highest hardness values were obtained with 2% Calcium Sulphate however; there was no statistical difference among the groups (4 h, 40°C, and Sulfate/K). The increase in gypsum hardness allows improvement of the handling during the fabrication of a prosthodontic restoration. Although some studies show an increase in gypsum cast hardness, [10-13]. What is in agreement with the results of this study, others presented no improvement in the surface hardness of dental stones. In a previous study, the hardness of type IV gypsum was not improved by the treatments performed [14]. It can be speculated that the different results can be associated with different methodologies and materials.

Plaster and stone products used in Dentistry are made by calcining calcium sulphate dehydrate [10, 14]. The main constituent of gypsumbased products is calcium sulphate hemihydrate, which is  $CaSO_4$ .  $\frac{1}{2}H_2O$ . Gypsum products are classified by ANSI/ADA Specification No. 25, into 5 types: 1 Type I - impression plaster; Type II - model plaster; Type III - stone; Type IV - high strength, low expansion stone; Type V-high strength, high expansion stone. Among these, Types II and III are usually used for diagnostic casts [10, 14].

Although an increase in the microhardness of the gypsum surface was observed, the accuracy in dimension stability was not evaluated. Further research is suggested to analyze the accuracy and reproducibility of the model after the pouring and storage with the methods proposed.

# Conclusion

Under the conditions of the present study, it can be concluded that:

 The hardness of the type IV gypsum tested was significantly influenced by the gypsum pouring method. The treatment with 2% Potassium sulphate and 2% Calcium sulphate, as well as, the delay in the removal of gypsum for a period of 4 h and storage in an oven at 37°C, may be recommended to obtain improvement in the hardness of a die.

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