

# Determination of Surface Hardness of Refractory Cast Investment Materials Before and After Dipping in Wax Hardening Agents

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## Abstract

**Background:** Phosphate-bonded investments resulted in a brittle cast model with a pores surface. Dental Surface hardening agents are then applied to refractory investment materials to enhance its properties.

**Objective of the study:** evaluate the effects of different hardening agents on surface hardness of refractory model investment materials.

**Material and Methods:** Thirty specimens prepared from commercially available phosphate bonded investment material, specimens were evaluated for hardness test before dipping in hardening wax and then divided into three groups according to the type of wax in which they were immersed (Bee's wax, Carnauba wax, and Ceresin wax). (Each group have 10 specimens). Surface hardness test was done by a Micro Vickers hardness device and three readings were taken, the mean was evaluated for the hardness value for all specimens before and after dipping in waxes.

**Results:** The hardness value increased in all specimens after dipping in different waxes, (P value < 0.001 HS highly significant).

**Conclusion:** Conclusion was derived that hardening agents' addition significantly affects hardness of the specimen surface of refractory investment materials.

**Key words:** Investment's materials; hardening agents; Ceresin wax; hardness

## Introduction

The refractory models are dipped in dental hardeners agents to obtain a harder surface and desirable properties <sup>(1,2)</sup> The dipping procedure with hardeners agents facilitates wax pattern adherence and makes the models adaptable also increase the hardness of refractory models and seals the pores that present on the surface models <sup>(3,4)</sup>

Bee's wax is a popular type being used in the hardening process of investment materials, other types of wax alternative to bees wax that can be used as hardening agents in dipping methods, may be plant such as carnauba wax or mineral such as ceresin wax <sup>(5)</sup> Bees wax is a natural wax of animal origin extruded by wax glands in honey bee hives, it is composed primarily of fatty acid esters, hydrocarbons, and multiple long chain alcohols <sup>(6)</sup> It used in dental casting process and for its antibacterial characteristics, it used in medicine coating pills and preservation of natural foods <sup>(7)</sup>

Natural wax made from palm leaves is Carnauba wax also can be used as hardeners agents, it's made entirely of esters and saturated long-chain alcohols, usually in the form of shiny hard flakes that range in hue from yellow to light brown. Carnauba wax is a healthy and inert chemically that is utilized food products and in the pharmaceutical sector as a tablet-coating agent, assisting patients in swallowing tablets <sup>(8)</sup> Ceresin wax, which is categorized as a natural wax with mineral origin from natural-mineral petroleum or lignite refining, is another type that can be applied as hardening agents. Because of larger molecular weight, it has high hardness <sup>(9)</sup>

Hardness is one of mechanical property that is defined as a resistance of materials to permanent deformation such as penetration or scratching the surface which is typically produced by an indentation force, the hardness test is a mechanical test for material properties which are used in dental applications, the principal purpose of it to determine the suitability of a material or the particular treatment to which the material has been subjected <sup>(10)</sup>

Scanning Electron Microscope test (SEM): Is a test process that scans a specimen with an electron beam performed at high magnifications to produce a magnified image for analysis, used very effectively in microanalysis of solid inorganic materials and precisely measures very small features and objects <sup>(11,12)</sup> This study was designed to determine the effects of different hardening wax agents on surface hardness of refractory model investment materials.

## Materials and Method

### Specimens grouping:

A total of (30 specimens) were prepared from commercially brand of Phosphate bonded investment material has its powder and special liquid which is: Easy Dental group (Bulgaria). Specimens were divided into three main groups according to the type of wax (bees wax, carnauba wax and ceresin wax) immersed in. (10 specimens in each group).

### Preparation of Mold:

Silicone mold (rectangular in shape) was prepared by using block of modeling wax material (Renfert , Germany) with a dimension of (5cm length, 2cm width, 1cm)

### Mixing procedure:

Powder- Liquid ratio of the phosphate bonded investment material (100g: 22ml), was mixed according to the manufacturer's instructions of Easy Dental group company. The mixture was then poured into the silicone mold using a vibrator device to height) and then duplicating by addition silicon martial (Dublislil, Germany) Fig.1 The mold is used for pouring (30 specimen) with phosphate bonded investment Martials, specimens' preparation for hardness test before and after dipping in waxes <sup>(13)</sup>



Fig 1 Mold for hardness test

prevent trapping of air bubbles, after 60 minutes the specimens were separated from the silicone molds <sup>(14)</sup> Specimens were subjected in a furnace for hardening treatment at 220 C° for 20 minute to ensure drying them and derives off moisture to get dense surface <sup>(15)</sup>

### Procedure for hardness test:

The Micro Vickers hardness device was used to measure the hardness values for all specimens, it consists of indenting the specimens test with a diamond indenter in the form of a right pyramid with square base, and angle of 136° degrees between opposite faces, the load of 50 g at 15 second was used <sup>(16)</sup> Each tested specimen was mounted in the horizontal stage of the tester machine then applying a load on the specimen surface for 15 second by diamond Vickers indenter, the specimens were tested three times (in the middle , right and left side) and the tester machine can automatically get the hardness value of each specimen and show on the large screen as in Fig.2



Fig 2 Hardness device

each type of wax that has a specific melting point and melting time than the other types of waxes <sup>(17)</sup> The melting point of bees wax is around 64 C°, after 10 minutes the wax softens and becomes a yellow fluid, ready to dip the specimen within, the inside the thermal melting device with a medium melting range about 78 C°, it takes about 15 minutes until we get a yellow-brown waxy liquid with a high viscosity <sup>(20)</sup>



Fig 3 (A) Specimens before dipping



Fig 3 (B) Specimens after dipping

**Results and Discussion**

Descriptive statistics are shown in table (1) and Fig .4for values of surface hardness test which included (mean value, SE and SD). The result of Easy Dental Group Company revealed that the highest mean value (47.8370) after dipping in Ceresin wax, while the lowest mean value (28.6150) before dipping in Carnauba wax.

Table (1): Summary Statistics concerning Hardness values test before and after dipping in ( Bess wax, Carnauba wax and Ceresin wax)

	N	Mean	SE	SD
Before dipping in Bees wax.	10	29.6410	1.28469	4.06255
After dipping in Bees wax.	10	46.4200	.76414	2.41642
Before dipping in Carnauba wax.	10	28.6150	1.08755	3.43914
After dipping in Carnauba wax.	10	47.6400	.45776	1.44757
Before dipping in Ceresin wax.	10	28.6360	1.13930	3.60279
After dipping in Ceresin wax.	10	47.8370	.44561	1.40913

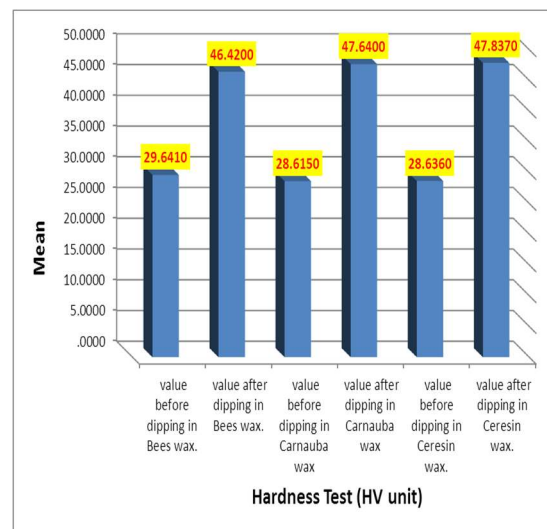


Fig 4 Bar chart the mean value of hardness test of specimens before and after dipping in (Bess wax, Carnauba wax and Ceresin wax)

**Statistical Methods**

IBM SPSS statistical program Version 24 used for doing the statistical analysis of the current study and Microsoft Excel 2010 was used for graphics presentation. The usual statistical methods were used in order to assess and analyze the results; these include Descriptive statistics (mean, standard deviation, minimum, maximum) and Inferential statistics (Student test (t-test).

**Results**

Descriptive statistics of surface roughness were obtained which include the minimum, maximum, mean and standard deviation for Harvard-temp and Charm-temp of the two groups – dry and aging – in artificial saliva. Fig. 5 shows the highest mean value observed in the Harvard-temp groups.

Comparison between the two groups – dry and aging – in artificial saliva of Harvard- temp using Paired t-test for the surface

## Scanning Electron Microscope (SEM):

The morphological characterization of specimens was explored by SEM analysis that was taken for (4 specimens) prepare by phosphate-bonded investment material both before and after dipping in wax, cutting a small piece from specimens and place wax, Carnauba inside Scanning microscopy device, the SEM photomicrographs was done under power magnification (1600X) <sup>(11,12)</sup>

In table (2), the results of T-test were recorded high significant different  $p < 0.001$  in surface Hardness test before and after dipping in three different waxes.

Table (2): T-test between before and after dipping in (Bess wax and Ceresin wax)

		t-test	P-value
Hardness Test.	Before and after dipping in Bess wax.	22.159	$P < 0.001$
	Before and after dipping in Carnauba wax.	24.509	$P < 0.001$
	Before and after dipping in Ceresin wax.	26.395	$P < 0.001$

\* $P < 0.001$  High significant

The result of ANOVA (F-test) and LSD shows that High-significant difference among all tested groups as in table (3, 4).

Table (3): ANOVA test among tested groups for hardness value

		F-test	P-value	Sig
Hardness Test	After dipping in Bees wax	3567.8	$P < 0.001$	HS
	After dipping in Carnauba wax	10505.1	$P < 0.001$	HS
	After dipping in Ceresin wax	11129.1	$P < 0.001$	HS

Table (4): LSD of Hardness value after dipping in (Bess wax, Carnauba wax and Ceresin wax)

			Mean	Std. Error	P-value
Hardness Test.	After dipping in Bess wax.	After dipping in Carnauba wax.	46.41999	.62757	.000
	After dipping in Carnauba wax.	After dipping in Ceresin wax.	47.63999	.37605	.000
	After dipping in Ceresin wax.	After dipping in Bess wax.	47.83699	.36686	.000

SEM for control specimen of phosphate investment material before dipping in wax noticed the surface is extremely rough and contains clear pores as in Fig.5

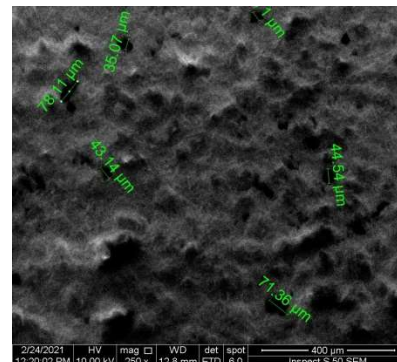


Fig 5 SEM for control group

While for three other specimens of phosphate investment material were dipped in three types of waxes, noticed that its surface became smoother and pores disappear as in Fig.6

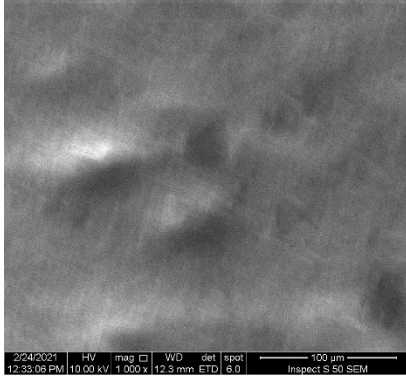


Fig 6 SEM for experimental groups (A) for Bees wax

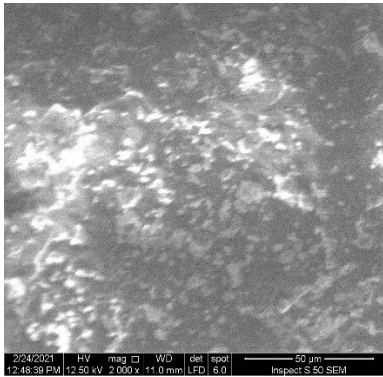


Fig 6 SEM for experimental groups (B) for Carnauba wax

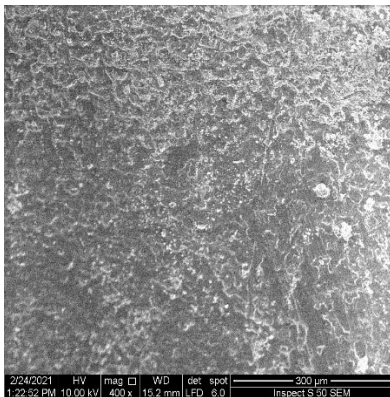


Fig 6 SEM for experimental groups (C) for Ceresin wax

## Discussion

Phosphate-bonded investment material consisting of silica with a binder such as magnesium oxide and phosphate, when colloidal silica and liquid are mixed, magnesium ammonium phosphate ( $MgNH_4PO_4 \cdot H_2O$ ) is produced, which expands the setting and reinforces the set material<sup>(2)</sup> However, it gives undesirable qualities such as brittleness, little hardness of the material, and insufficient precision in details<sup>(1)</sup> Hence, refractory investment materials are

frequently treated with surface coatings hardening agents' treatment to acquire better acceptable qualities<sup>(3)</sup>

In this study, all phosphate-bonded investment specimens were dipped in dental hardeners agents to improve surface hardness and provide a stronger working surface, as shown in Fig. 4 the mean values of hardness test of specimens were increase after dipping in waxes. Dental agents used in this study basically are natural waxes, which are thermoplastic materials when melted by heat will turn into a liquid, usually materials that contain pores on their surface when immersed in liquids, all its pores were filled with liquid and its surface was covered meaning another substance was added to it, which increased its weight and strength, for this reason the hardness improved<sup>(5,6)</sup> The elements contained in bees wax (calcium, sodium, carbon, magnesium, fluorine, oxygen, and nitrogen) which are all elements found in nature, if they are added to any substance they increase the hardness and strength of the material<sup>(18)</sup> While the elements contained in carnauba wax which are the same elements that bees wax contains, but with added iron (Fe) due to this type of wax is plant origin<sup>(19)</sup> Ceresin wax also contained natural which are calcium, sodium, carbon, magnesium, oxygen, and nitrogen<sup>(20)</sup> These result is agree with the studies in 2020 by Jain et al., and 2017 by saji et al., that concluded the hardening treatment is effective on improving surface hardness of phosphate-bonded refractory investment material. Aljbori et al., 2020 demonstrate in their studies that dental gypsum which usually used as an investing and molding material for prosthodontics restoration must be hard enough to resist any deformation caused by forces applied on their surface, also they emphasize the importance of hardness of the material that measured by using a Vickers device for complicated prosthodontics procedures with desired strength and fine details reproduction.

According to the result of SEM of specimen prepare by phosphate-bonded investment material before dipping in wax, revealed the surface of this material is extremely rough and contains clear pores, as in Fig. 5 While SEM for three other specimens of phosphate investment material were dipped in three types of waxes, observes that its surface became smoother and pores disappear as in Fig. 6

## Conclusions

Based on the results and the values in this study, we concluded the following:

1. Hardening gents are natural waxes that contain natural elements, if they are added to another material that increases its strength, therefore hardness improved.
2. The liquid wax covered the surface of the specimen entirely and closed all the pores on its surface, thus the surface became smoother, solid, and with good properties desirable for working.

## RECOMPNDATION

Determination of surface hardness of refractory cast investment materials before and after dipping in wax hardening agents at 850 °C.

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