To Evaluate and Compare the Effect of Commercially Available Model Hardening Agent on Surface Hardness and Surface Roughness of Refractory Investment Materials: An In-Vitro Study

Ajay Jain[1], Ramdziah Md Nasir[2], Lee Wen Chee[3], Lim Hui Xin[3], Lim Zhi Yeih[3], Sridevi Ugrappa[4], Kasi Marimuthu[5]

[1]Associate Professor, Faculty of Dentistry, AIMST Dental Institute, AIMST University, Semeling 08100 Bedong, Kedah, Malaysia, [2]Professor, Faculty of Engineering, Universiti Sains Malaysia, Nibong Tebal, Malaysia, [3]Dental technology undergraduates, Faculty of Dentistry, AIMST Dental Institute, AIMST University, Semeling 08100 Bedong, Kedah, Malaysia, [4]Senior Lecturer, Faculty of Dentistry, AIMST Dental Institute, AIMST University, Semeling 08100 Bedong, Kedah, Malaysia, [5]Professor, Faculty of Applied Sciences, AIMST University, Semeling 08100 Bedong, Kedah, Malaysia

Abstract

Background: Various studies are conducted to evaluate the effect of hardening agent on surface hardness of refractory investment material but these studies do not evaluate the amount of load and depth at which maximum hardness is anticipated. So, the present study was conducted to evaluate the effect of hardening agent on the surface hardness of dental refractory investment material when treated with hardening agent at varying degree of load and depth. **Materials and methods:** The commercially available phosphate-bonded refractory investment material (Adentatec-Vest PA, Adentatec GmbH, Germany) and hardening agent (Kalthärter cold hardener, Dentaurum GmbH & Co. Deutschland) were used in this study. The specimens were fabricated and divided into two groups: ***Group A:*** specimen without hardening treatment; ***Group B:*** specimen with hardening treatment. Surface hardness test was performed with the use of a nanoindenter (NT Berkovich indenter); while surface roughness test was measured with Surfcom-130A using an electrically operated stylus arm. The data were tabulated and analyzed using t-test for both the surface hardness and surface roughness parameters with *p-value <0.05* was considered significant. **Results:** Both surface roughness *(p=0.043)* and surface hardness *(p= 0.017)* of group A and group B specimens were comparable and have shown with significant difference. **Conclusion:** The hardening treatment is effective on decreasing surface roughness and improving surface hardness of phosphate-bonded refractory investment material.

Keywords: *model hardening agent, surface hardness, surface roughness, refractory investment materials.*

Introduction

Refractory investment is an investment material that can withstand the high temperatures used in soldering or casting[1]. It must be able to reproduce surface details of master cast, must have exceptional surface hardness and must be more resistant to surface abrasion when working with wax pattern on the refractory cast[2].

Phosphate-bonded investments are similar with gypsum-bonded investment; they consist of silica and binder such as magnesium oxide and phosphate to provide high thermal expansion. Mixing of colloidal silica and water will form magnesium ammonium phosphate ($Mg\cdot NH_4\cdot PO_4\cdot 6H_2O$), which increases the setting expansion and strengthens the set material[3,4]. However, they provide drawbacks of a low abrasion

Corresponding Author:
Assoc. Prof. (Dr.) Ajay Jain
Faculty of Dentistry, AIMST Dental Institute, AIMST University, Semeling 08100 Bedong, Kedah, Malaysia.
Email: jainajay9@gmail.com, Mobile: +60109454204

resistance and reduced surface hardness which are difficult to preserve surface detail of the refractory cast model while wax pattern fabrication[5,6].

Hence, to achieve the more desirable properties of surface roughness and surface hardness, refractory investment materials are commonly treated with surface hardening agent, surface coatings or various specific surface treatments. Several reports have been published regarding cast hardening agent that has significantly changed the mechanical and physical properties of various dental investment materials[7-11]. Another recent study conducted by Saji et al[2], application of different hardening agents on two commercially available refractory materials. Surface abrasion, surface hardness and surface detail reproduction were evaluated under appropriate tools and devices. Result showed that hardening agents do improve the surface abrasion resistance and abrasion values of the treated samples, but these agents would mask the surface reproduction of the duplicated refractory model.

Therefore, the present study aims to evaluate and compare the surface hardness and surface roughness of refractory investment material, when treated with hardening solution. It is hypothesized that treated samples would not mask the surface reproduction details of the model and the same time would provide the exceptional surface hardness.

Materials and Methods

This study was conducted in AIMST Dental Centre, AIMST University and Universiti Sains Malaysia, Nibong Tebal, Malaysia after the consent is obtained from the ethical committee.

Procedures of preparing samples

An acrylic round block with the measurement of 30 mm diameter and 12 mm height **(Figure 1)** was prepared using heat cure acrylic material (Kemdent, Kemdent works, Wiltshire, UK). The silicone mold was prepared by using acrylic block with the help of silicone duplicating material (Metrosil, Metrodent Ltd. Huddersfield, England). Two specimens of refractory investment materials were prepared and divided into groups; **group A:** specimen without hardening treatment, and **group B**: specimen after treatment with hardening agent.

The water-powder ratio of the refractory phosphate bonded investment material was mixed according to the manufacturer's instructions. Then, the resulting powder and liquid mixture were poured into a mixing flask with 10 seconds of hand spatulation and 60 seconds of vacuum-mixing process. The mixture was then poured into the silicone mold using a vibrator to prevent trapping of air bubbles. After 60 minutes, both the specimens were removed from the silicone mold **(Figure 1).** Group B specimen was placed in a 4055 model drying oven (CMP Industries, Albany, New York, USA) and heated to 220°C for 40 minutes as recommended by the manufacturer's user manual. It was then dipped into Kalthärter cold model hardening agent for 5 seconds and was placed back in drying oven for 5 minutes as a final drying process.

Testing for surface roughness and surface hardness

Specimens of groups A and B **(Figure 2)** were kept at room temperature for two weeks to ensure complete dryness of the sample[8] and later on, these specimens were tested using Surfcom-130A and Nanoindenter for surface roughness and surface hardness respectively. The specimens were first tested on Surfcom-130A to evaluate their surface roughness. Three randomly selected locations on the specimen surface were electrically operated by a stylus arm to record a desired roughness profile for final measurement of roughness values (R_a in µm). R_a is defined as the arithmetic average of the profile ordinates within the measured section. This can also be called the average height[12].

The stylus tip made of diamond with 2µm/60° with resolution up to 0.001µm was used to evaluate the vertical deviations from nominal surface over a specified length surface of the specimens. A measuring force of 0.75mN was kept constant throughout the operations (DT04800-R001, Tokyo Seimitsu Co. Ltd., Tokyo, Japan). The operated specimens were carefully removed from the machine and the desired data of each location in both groups were recorded in a printout using a high-resolution thermal-printer. The roughness value was calculated and analyzed using SPSS software.

Surface hardness of groups A and B specimens were measured under a Nanoindenter. Both specimens were placed on the travelling stage of a nano-indentation system (Nanotest Vantage, Micro materials, UK). A NT Berkovich indenter with three-sided pyramid and a total included angle of 142.3 degrees and a half angle of 65.27 degrees was employed in the system. Again, three randomly selected locations on the specimen surface were marked as indenting position. The Berkovich indenter was positioned to the starting spot by the program and applied with a constant force of 5mN (0.5g) and progressively increased up to 500mN (50g). The dwell period and loading rate of the indenter was set to 30s and 16mN/s as recommended by the manufacturer's user manual[13].

A total of 10 indentations were measured until a maximum loading force of 500mN (50g). Upon completion of measuring the 10 indentations in that particular location, the Berkovich indenter was moved to the next pre-marked location until all locations were measured. In the end, all values were recorded into the system and further calculated and analyzed using SPSS software.

Results

The surface roughness for specimens of group A and B were tested and tabulated at three different locations (X, Y, Z) using surfcom-130A as shown in table 1. The surface hardness for specimens of group A and B were tested and tabulated at three different locations using nanoindenter as shown in table 2, 3. A student t-test was used to measure the difference between group A and group B specimens for surface roughness and surface hardness as shown in table 4. Analysis showed that there was a significant difference in the surface roughness (p=0.043) and surface hardness (p=0.017) in the specimens of group A and B (t-value 2.776).

Table 1: Roughness analysis at location X, Y and Z of group A and B specimens

Surface roughness	Group A specimen			Group B specimen		
	Ra (µm)	Rq (µm)	Rz (µm)	Ra (µm)	Rq (µm)	Rz (µm)
Location X	2.774	3.386	15.022	1.922	2.396	12.045
Location Y	2.501	3.210	16.225	1.619	2.024	10.75
Location Z	2.196	2.742	12.488	2.065	2.668	14.637

Table 2: Hardness analysis at location X, Y and Z of Group A specimen

Indent	Location	Max Depth (nm)	Plastic depth (nm)	Max Load (mN)	Hardness (Gpa)
1	X	788.53	721.79	5.00	0.39
	Y	914.42	858.15	5.00	0.27
	Z	1990.65	1930.65	5.00	0.05
2	X	1994.72	1803.17	60.00	0.75
	Y	3010.30	2868.38	60.00	0.29
	Z	3981.38	3831.20	60.00	0.16
3	X	2934.78	2560.49	115.00	0.71
	Y	4181.99	3987.03	115.00	0.29

Cont... Table 2: Hardness analysis at location X, Y and Z of Group A specimen

	Z	3825.13	3632.51	115.00	0.35
4	X	4194.17	3883.65	170.00	0.46
	Y	3915.62	3651.61	170.00	0.52
	Z	4452.66	4167.32	170.00	0.39
5	X	4955.38	4603.98	225.00	0.43
	Y	4627.71	4325.16	225.00	0.49
	Z	4938.20	4566.79	225.00	0.44
6	X	5836.15	5318.32	280.00	0.40
	Y	4473.42	4101.01	280.00	0.67
	Z	4928.26	4560.63	280.00	0.76
7	X	6441.42	6044.34	335.00	0.37
	Y	4981.77	4497.39	335.00	0.67
	Z	6691.90	6320.30	335.00	0.34
8	X	6475.02	6073.44	390.00	0.43
	Y	9354.73	8810.17	390.00	0.20
	Z	4952.00	4567.07	390.00	0. 54
9	X	8891.33	8422.47	445.00	0.25
	Y	7190.09	6701.83	445.00	0.40
	Z	8132.70	7672.55	445.00	0.30
10	X	10499.25	9977.11	500.00	0.20
	Y	7176.70	6629.55	500.00	0.46
	Z	8859.76	8308.31	500.00	0.29

Table 3: Hardness analysis at location X, Y and Z of Group B specimen

Indent	Location	Max Depth (nm)	Plastic depth (nm)	Max Load (mN)	Hardness (Gpa)
1	X	961.22	899.17	5.00	0.25
	Y	2815.56	2760.92	5.00	0.02
	Z	1161.58	1124.40	5.00	0.02
2	X	1848.17	1713.75	60.00	0.83
	Y	2370.24	2249.27	60.00	0.48
	Z	5927.22	5784.03	60.00	0.49
3	X	3497.67	3292.88	115.00	0.43
	Y	3187.13	2955.02	115.00	0.53
	Z	7558.70	7353.56	115.00	0.55
4	X	6346.83	6091.05	170.00	0.18
	Y	6933.98	6718.41	170.00	0.15

***Cont...* Table 3: Hardness analysis at location X, Y and Z of Group B specimen**

	Z	6649.18	6377.90	170.00	0.16
5	X	6109.33	5860.04	225.00	0.26
	Y	8334.63	8061.52	225.00	0.14
	Z	7623.84	7335.24	225.00	0.13
6	X	7025.30	6710.23	280.00	0.25
	Y	3812.51	3484.72	280.00	0.94
	Z	8220.68	7917.50	280.00	0.91
7	X	11011.67	10652.66	335.00	0.12
	Y	6876.72	6482.55	335.00	0.32
	Z	11639.63	11290.40	335.00	0.83
8	X	9567.52	9223.18	390.00	0.18
	Y	7519.71	7162.16	390.00	0.31
	Z	13251.33	12887.24	390.00	0.71
9	X	8468.68	8106.33	445.00	0.27
	Y	9737.03	9391.59	445.00	0.20
	Z	11875.45	11493.15	445.00	0.60
10	X	10784.00	10412.76	500.00	0.18
	Y	8838.03	8436.96	500.00	0.28
	Z	13193.34	12715.27	500.0	0.38

Table 4: Surface roughness and surface hardness description of group A and B specimens

Variables	Surface roughness		Surface hardness	
	Group A (in µm)	Group B (in µm)	Group A (in GPa)	Group B (in GPa)
Location X	2.77	1.92	0.75	0.83
Location Y	2.50	1.61	0.68	0.94
Location Z	2.19	2.06	0.76	0.91
Mean	2.49	1.86	0.732	0.896
Standard Deviation	0.289	0.228	0.046	0.055
p-value	0.043 (<0.05)		0.017 (<0.05)	

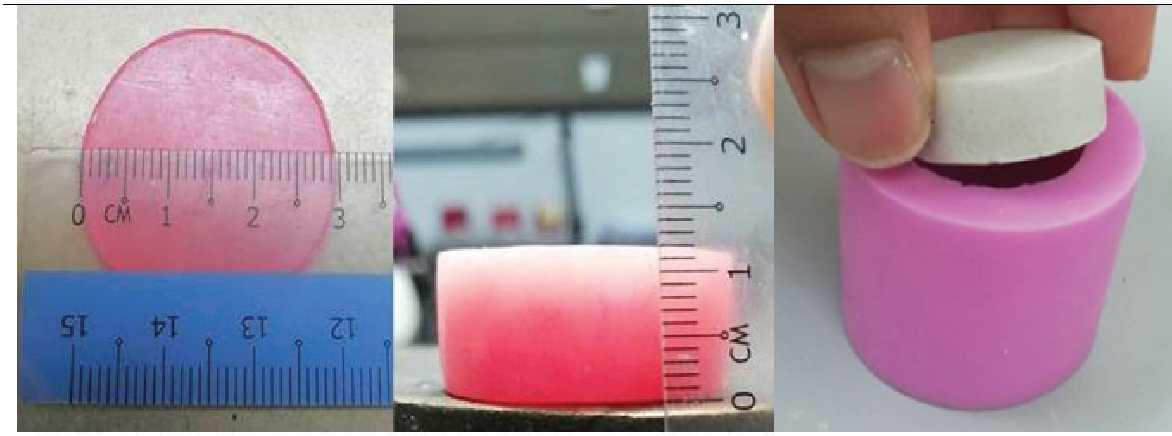

Figure 1: Acrylic round block fabrication and sample removal from silicone mold

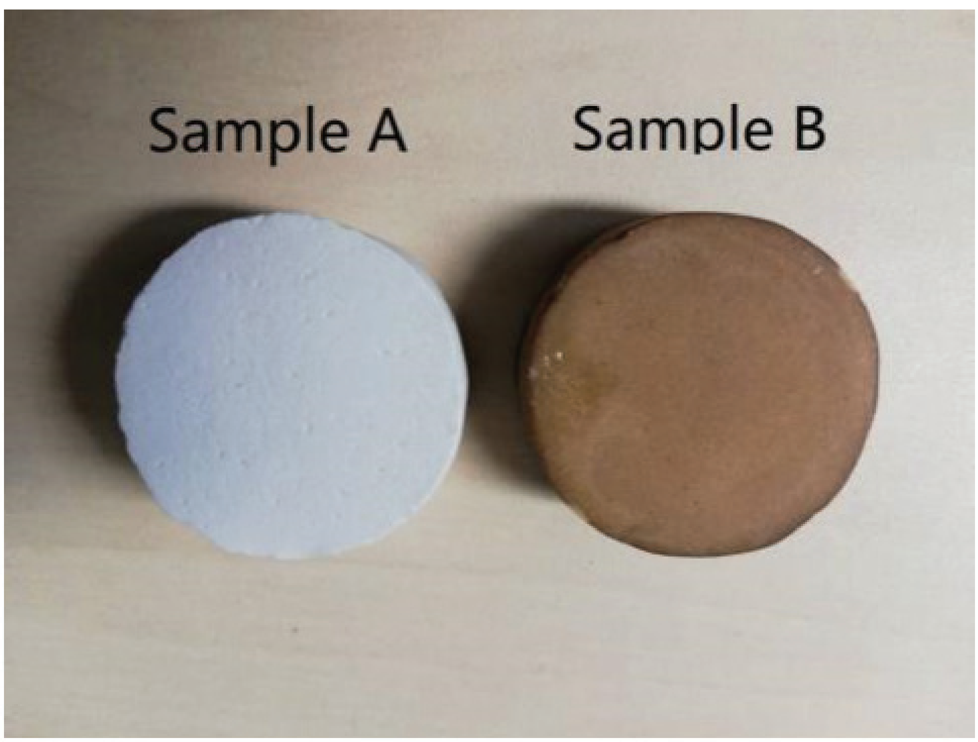

Figure 2: Specimens of group A and group B are kept at room temperature

Discussion

Phosphate bonded refractory investment material was used to test the surface hardness and surface roughness in this study. Due to high brittleness and low abrasion resistance of the material, the surface of the samples were protected with soft material during the preparation[2,14-15] and stored at room temperature to avoid alteration of material properties.[16] Hardness can be affected by a high drying temperature, long drying period or a hardener with an overdue shelf-life.

The present study investigated surface roughness of refractory investment materials and showed that samples treated with hardening agent have lower surface roughness. The hardening agent is found to be significantly effective on improving surface roughness of cast refractory investment materials. This result can be explained by the findings from Sanad et al, in which a dental hardener (sulfonated fatty acid), can react with calcium sulphate to form the calcium salt of the fatty acid. The particles of this salt can block or partially block and penetrate and fill the superficial pores of the investment material.[20]

Lindquist et al measured three different commercial products of type IV and one commercial product of type V gypsum material with application of two different surface die hardeners. The abrasion resistance was evaluated using the application of stylus on abrasion device to each group of specimens. Singh NSK et al used a similar method with Linquist et al by using stylus-based abrasion device but with three different products of type IV gypsum material and two different brands of hardening agent. They have concluded that abrasion resistance would vary with different applications of dental hardeners. [7,11]

The present study has shown that model hardening agent has effective role in providing surface hardness which is statistically significant. Saji P et al conducted a study, in which the hardness value of Mohs' scale of treated sample was higher when compared to untreated samples.[2] However, Mohs' scales which do not give a linear result can lead to technical weakness, hence indentation hardness tests such as nanoindentation were developed for improvement of accuracy and consistency [21-22], as employed in this study. From the study of Harris et al[6], the micro-hardness of type III and type IV gypsum material does not vary with each other with the use of cyanoacrylate die hardener, but observed with reduction of surface hardness. Therefore, they concluded that hardeners do not give influence on gypsum material instead it leads to reduction in hardness of gypsum investment, which contradicts the result of our present study. However, Khan et al[8] show that cyanoacrylate increases the surface coating on type IV gypsum material and improves the surface abrasion resistant. Hence, they claimed that cyanoacrylate is suitable to be used as hardening application, which is in accordance with the result of our present study.

Conclusion

Based on the result analysis, there is a significant difference for both surface roughness and surface hardness when treated with model hardening agent. Hence it can be concluded that model hardening agent is significantly effective on decreasing surface roughness and improving surface hardness of phosphate-bonded refractory investment material and it should be used meticulously to achieve the successful restoration.

Conflict of Interest: Nil

Source of Funding: This student research was funded by AIMST University.

Ethical Clearance: AIMST University ethical clearance was obtained.

References

1. Keith JF. The Glossary of Prosthodontic Terms. J Prosthet Dent 2017; 117(5):e75.
2. Saji P, Mohamed R, Noufal PK, Reddy PS, Jain AR, Gounder R. Comparative evaluation of the influence of cast hardening agents on surface abrasion, surface hardness and surface detail reproduction properties of refractory investment materials. Biomed & Pharmacol J 2017; 10(3), 1517-24.
3. McCabe JF, Walls AWG. "Investment and refractory die." Applied Dental Materials, 2008; 46-52.
4. Anusavice KJ. Casting investment and procedure. Philip science of dental materials, 2003; p.296-311.
5. Abdullah MA. Surface detail, compressive strength, and dimensional accuracy of gypsum casts after repeated immersion in hypochlorite solution. J Prosthet Dent 2016; 95: 462-8.
6. Harris PE, Hoyer S, Lindquist TJ, Stanford CM. Alterations of surface hardness with gypsum die hardeners. J Prosthet Dent 2004; 92 (1):35-38.
7. Lindquist TJ, Stanford CM, Knox E. Influence of surface hardener on gypsum abrasion resistance and water sorption. J Prosthet Dent 2003; 90(5): 441-446.
8. Khan MF, Dall AQ, Sajid K. Effect of cyanoacrylate as surface hardener on gypsum die material. J Liaquat Univ of Med and Health Scie 2012; 11(3):185-89.

9. Peyton FA, Leibold JP, Ridgley GV. Surface hardness, compressive strength, and abrasion resistance of indirect die stones. J Prosthet Dent 1952; 2(3): 381-89.

10. Dootz ER, Graig RG, Peyton FA. Influence of investments and duplicating procedures on the accuracy of partial denture castings. J Prosthet Dent 1965; 15(4): 679-690.

11. Singh NSK, Singh NS, Thangjam P, Devi NR. Effect of surface hardeners on physical property of Type IV gypsum products. Ann of Int Med and Dent Res 2018; 4(4):DE01-DE05.

12. BS EN ISO 4287: 2000 Geometrical product specification (GPS). Surface texture: profile method-terms, definitions and surface texture parameters.

13. Tarefder, Rafiqul & Faisal, Hasan. (2013). Effects of dwell time and loading rate on the nanoindentation behavior of asphaltic materials. J Nanomechanics and Micromechanics 2013; 3: 17-23.

14. Habib H, Mannezhad G, Mohammed SE, Steward GP, Weinberg R. Effects of cyanoacrylates on die stones. J. Prosthet Dent 1983; 49(5):639-46.

15. Fukui H, Lacy AM, Jendresen MD. : Effectiveness of hardening films on die stones. J Prosthet Dent 1980; 44(1): 57-63.

16. Razzak NHA, Mustafa MA, Al-Nuzal SMD, Ali FH, Lafta BA. Preparation of dental phosphate-bonded investment to cast nickel chromium alloy and the evaluation of its mechanical and physical properties. World Journal of Pharmaceutical Research 2014;3(9):173-175.

17. Karda B, Jindal R, Mahajan S, Sandhu S, Sharma S, Kaur R. To analyse the erosive potential of commercially available drink on dental enamel and various tooth coloured restorative materials - An in-vitro study. J Clin Diagn Res. 2016 May;10 (5):ZC117-21.

18. Rashid H. Evaluation of the surface roughness of a standard abraded dental porcelain following different polishing techniques. J Dent Sci 2012; 7(2):184–189.

19. Attin T, Koidl U, Buchalla W, Schaller HG, Keilbassa AM, Hellwig E. Correlation of microhardness and wear in differently eroded bovine dental enamel. Arch Oral Biol. 1997;42 (3): 243–250.

20. Sanad MEE, Combe EC, Grant AA. The effect of model sealant solutions on the properties of gypsum. J Dent 1980; 8(2):152–157.

21. Bradt RC. Ceramic Crystals and Polycrystals, Hardness of. Encyclopedia of Materials: Science and Technology 2001; 1045-1051.

22. Sundararajan G, RoyM. Hardness Testing. Encyclopedia of Materials: Science and Technology 2001; 3728–3736.