



An In-Vitro Study Effect of Adding Silica Gel on Surface Hardness of Phosphate-Bonded Investment Material

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Received 29/02/2024

Accepted in revised form 03/06/2024

Published 30/06/2024

Abstract

Background: Few studies have been undertaken to assess the influence of nanoparticles on the surface hardness of thermal investment materials. As a result, the current investigation was suggested to assess the influence of silica gel agents on the mechanical surface hardness of phosphate-bonded investment material at various percentages. **Materials and methods:** In this investigation, commercially available phosphate-bonded refractory investment material (Zetavest fine) and silica gel powder (Himedia-India) were used. The specimens were manufactured and split into four groups (n=16): specimens without silica gel substance; specimens with 1% silica gel material; specimens with 2% silica gel material; and specimens with 3% silica gel material. A mechanical surface hardness property test was completed with the use of an indenter durometer (Shore D). Surface hardness characteristics were analysed using a one-way ANOVA and post-hoc (Tukey) tests, with a p-value of ≤ 0.05 indicating significance. **Results:** The surface hardness of investment specimens with 1%, 2%, and 3% silica gel powder before heating were comparable and have shown substantial differences from the specimens with non-additive. **Conclusion:** Silica gel of 1, 2, and 3% can improve the phosphate-bonded surface hardness.



Keywords: Silica gel, hardness, phosphate-bonded, refractory materials, investment.

Introduction

For many years, dental application, investments have resulted in becoming a familiar and diverse class of laboratory materials. They are commonly used increasingly in the construction of cobalt-chromium frameworks. Their applications in a variety of dental restorations are now customarily for the precision casting of high-fusing dental alloys ranging from multi-unit bridgework substructures to removable partial dentures [1],[2].

The investment applied for moulding the patterns for the partial denture framework must be hard enough to prevent the mould from chipping or fracturing during heating and casting. As a result, an investment should have appropriate strength [3].

Dental investment materials are essential in the fabrication of precision dental castings [4]. In general, gypsum-bonded and phosphate-bonded investment materials are two types commonly applied in dentistry depending on the melting range of the alloy. The phosphate-bonded investment materials tend to decompose at high temperatures and, since then have been used in the fabrication of dental castings using high melting temperature dental alloys (1200 ± 1300 °C) [5],[6],[7]. However, this material is a brittle cementitious solid with fracture behaviour very similar to heterogeneous materials.

Refractory investment is a substance that can resist a high temperature as in soldering and casting [8]. Such materials should be able to replicate the surface details of the master cast. In addition, it provides significant resistance to indentation and is more abrasion tolerant during the application of wax patterns on the refractory cast [9] [10].

Phosphate-bonded refractory materials are similar to gypsum-bonded investments, silica plus magnesium oxide or phosphate as a binder must be included, which gives significant thermal expansion. Magnesium ammonium phosphate ($Mg\cdot NH_4\cdot PO_4\cdot 6H_2O$) is formed as soon as colloidal silica and water are mixed which expands and reinforces the set material [11] [12]. Nevertheless, they have the disadvantages of limited abrasion resistance and reduced surface hardness, making it challenging to reserve the features of the refractory cast model surface during the manufacturing of wax patterns [3] [13].

Investment casting (also known as lost wax casting) is a generic production procedure for jewelry metals and alloys [14]. An investment casting is used to form complex internal designs of castings [15],[16]. Thermal moulds of phosphate-bonded investments have been commonly used over six decades for casting alloys of high melting points such as Co-Cr alloys and alloys for porcelain-fused-to-metal restorations

[17]. A complicated chemical procedure produces phosphate-bonded investment materials. An equimolar mixture of setting agents, mono ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) and magnesium oxide (MgO) combines with water to generate ammonium magnesium phosphate hexahydrate ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$), which clusters around excess unreacted MgO and fillers [18]. Heat causes the mixed phosphate to degrade. At 800°C , the single-phase $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ dehydrates and converts to magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) [19]. The thermal process is a difficult reaction since it creates a combination of magnesium metaphosphate ($\text{Mg}(\text{PO}_3)_2$) and farringtonite ($\text{Mg}_3(\text{PO}_4)_2$) [20]. Chemical reactions may also occur between phosphate refractory cristobalite and quartz components of silica, MgO , and additives such as zircon (ZrSiO_4) [21].

Silica gel powder is a type of absorbent substance that primarily absorbs moisture from its surroundings. It is typically made up of fine grains of gelatinous silica, sometimes known as "silica gel". This powder's capacity to effectively absorb moisture makes it beneficial in a variety of applications, including protecting delicate products from moisture damage, drying materials, and maintaining dryness in closed spaces [22]. Silica gel was found in the nineteenth century, but its origins date back to 1640. However, they were not employed commercially until the early twentieth century. Initially, silica gel was utilized to dry industrial manufacturing air by extracting

moisture. It has evolved into an excellent method of preserving fragile objects and drying out materials [23]. Because of technical advancements and the unique qualities of silica gel, this substance has become widely used in fields such as sensitive material packaging and storage, the pharmaceutical industry, and even the food business [24].

This study aims to add the silica gel to produce a high-fired strength phosphate-bonded investment mould to withstand the surface burn-out process and is strong enough to withstand the pressure of centrifugally cast molten alloy. The hypothesis suggests that the addition of silica gel in different percentages affects the surface hardness of the phosphate-bonded investment mould. Therefore, the objective is to investigate the effect of the addition of 1%, 2%, and 3% silica gel on the surface hardness of phosphate-bonded investment materials.

Materials and Methods

A 1:1 ratio of silicone (WAGNERSIL 17 N Premium, Germany) was prepared in a vacuum mixer (Koala", MESTRA, Italy), and put into a plastic container with dies measuring $30 \times 25\text{mm}$ in length and diameter [25]. The mould-containing dies are allowed to polymerize completely at room temperature for 48 hours. Later, the dies were removed with a jet air spray.

Investment specimens were prepared by pouring investment material (Zetavest fine, Italy) of particle size $425\text{-}445\mu\text{m}$ into a silicone mould with cylindrical specimens dimensions of 30mm height

and a diameter of 25mm according to ADA specification [26],[27], [25].

A total sample group of sixty-four specimens were prepared with each group variable having sixteen specimens (n=16). The silica gel powder (Himedia-India) with particle sizes ranging from 125 to 250 μ m was weighed using a sensitive electronic balance (Ricerca, Italy). The investment powder to special liquid was measured according to the manufacturer's instructions. By using a vacuum mixture unit, each ingredient was mixed with the appropriate mixing liquid [3],[28].

The phosphate-bonded investment material used in this study was precisely weighed following the manufacturer's instructions for a liquid/powder ratio of 60ml/400gm. The addition of silica gel powder of 1%, 2%, and 3% were added to the investment powder for each identified group and dispensed for 5 h.

The manufacturer's recommendations were followed for mixing the precise ratio of liquid-to-powder refractory phosphate bonded investment material. The resulting mixture was then transferred to a mixing flask and vacuum-mixed for 60 seconds after 10 seconds of manual spatulation. To prevent air bubbles from becoming trapped, the vibrator was used during the mixture pouring into the silicone mould. After 1h, the specimens were removed from the silicone mould. All specimens were placed in a model drying oven (BEGO, Miditherm TH, Germany) and heated up to 850°C for 1h, as specified

by the manufacturer's user manual, Figure (1).

Testing for surface hardness

To guarantee full dryness, all specimens were maintained at room temperature for 24 hours before being examined with a Shore D dumeter surface hardness tester. The specimens were initially evaluated before heat treatment and subsequently after heat treatment, Figure(2).

The arithmetic average of the profile coordinates inside the measured segment is also known as the average height [29]. The diamond stylus tip with 0.8 μ m/60° resolution (up to 0.001 μ m) was used to evaluate vertical indentation from the nominal surface across the specimen surface (indenter running 0-2.5mm). A measuring force of 5N remained constant throughout the operations (560-10Dm, Gain Express Holdings Ltd., China). The data for all groups was recorded after three readings for each specimen. The surface hardness value for the addition of 1%, 2%, and 3% silica gel to phosphate-bonded investment material was computed and analyzed using SPSS software (V, 22). The data were tested for homogeneity by Levene's test (non-significant) and analyzed using One-way ANOVA with post-hoc (Tukey-test) with a p-value of ≤ 0.05 indicating significance.

Results

The surface hardness for all specimens was tested and tabulated at three different locations. The data were normally distributed among all groups,

therefore, one-way ANOVA with post-hoc (Tukey) tests were conducted to measure the difference in surface hardness between the studied groups as shown in Tables (1), (2), (3), and Figure 3. The analysis revealed that there was a considerable difference in the surface hardness of invested specimens before heating for casting purposes and with all added percentages of 1% ($p=0.005$); 2% and 3% ($p\leq 0.001$) silica gel compared to non-additive specimens. However, after heating the investment to casting metal temperature of 800°C for one hour and after complete cooling on-bench a non-significant difference in the surface hardness was reported in invested specimens after the heating process for all experimental percentages.

Discussion

In this work, the surface hardness of phosphate-bonded investment material was assessed after the addition of silica gel powder. Additives, high drying temperatures, and long drying periods may all have an impact on hardness [30], [9]. The current study evaluated the surface hardness of refractory investment materials after the addition of silica gel powder and showed that the specimens with no additive silica gel agent have reduced surface hardness. The silica gel powder was shown to be expressively operative for improving the surface hardness of refractory mould before the heating process for casting metal. This outcome can be explained by evidence from McCabe and Walls in 2013 and later by Beeley and Smart in 2023. This may related to the presence

of additional silica gel powder in phosphate-bonded investments that already contain some silica and the combining of colloidal silica and water created the extra magnesium ammonium phosphate that enhances the setting and reinforces the set material [11], [12].

However, after heating, a non-significant difference was reported in the surface hardness of cast refractory investment materials. This may be in agreement with Phumying (2019). According to Zheng, a mono-ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$) and magnesium oxide (MgO) in the phosphate-bonded investment materials react with water to produce ammonium magnesium phosphate hexahydrate ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$) that aggregates around excess unreacted MgO and fillers [18]. Yet, Phumying stated that during heating up to 800°C, the single-phase phosphate mixture represented by $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ dehydrates decomposes to convert to magnesium pyrophosphate ($\text{Mg}_2\text{P}_2\text{O}_7$) [19].

Conclusion

According to the analysis of the results, there is a considerable difference in surface hardness when a silica gel powder of 1%, 2%, and 3% is added to phosphate-bonded investment materials.

As a result, it can be stated that model surface strengthening using silica gel powder is significantly beneficial in increasing the surface hardness of phosphate-bonded investment material, which must be applied precisely to accomplish successful restoration.

Conflict of interest

The authors reported that they have no conflicts of interest.

References

- Oh, K.C., J. Jeon, and J.-H. Kim, *Fabrication of a removable partial denture combining conventional and digital techniques*. The Journal of Prosthetic Dentistry, 2021. **125**(4): p. 588-591.
- Akl, M.A. and C.G. Stendahl, *Removable partial denture frameworks in the age of digital dentistry: A review of the literature*. Prosthesis, 2022. **4**(2): p. 184-201.
- Jain, A., et al., *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*. Indian Journal of Public Health Research & Development, 2020. **11**(8): p. 11-18.
- Kareem Rasheed, R. and N.S. Mansoor, *Bond Strength of Porcelain Fused to Casting and Selective Laser Base Metal Alloy with a Bonding Agent*. Journal of Techniques, 2022. **4**(3).
- Shen, C., H.R. Rawls, and J.F. Esquivel-Upshaw, *Phillips' Science of Dental Materials E-Book*. 2021: Elsevier Health Sciences.
- Shen, C., R. Rawls, and J.F. Esquivel-Upshaw, *Phillips' science of dental materials*. (No Title), 2022.
- Muhsin, S.A. and B.A. Abd Allah, *Modified CAD/CAM Wax: The Dimension Accuracy of Wax/Metal Copy Patterns*. Mustansiria Dental Journal, 2022. **18**(1).
- Morgano, S.M., et al., *The history of the glossary of prosthodontic terms*. Journal of Prosthetic Dentistry, 2018. **119**(3): p. 311-312.
- Saji, P., et al., *Comparative evaluation of the influence of cast hardening agents on surface abrasion, surface hardness and surface detail reproduction properties of refractory investment materials*. Biomedical and Pharmacology Journal, 2017. **10**(3): p. 1517-1524.
- Hummudi, I.M., *Influence of different Wax Hardening Agents on Surface Roughness of Refractory Cast Investment Materials*. Journal of Techniques, 2021. **3**(3).
- McCabe, J.F. and A.W. Walls, *Applied dental materials*. 2013: John Wiley & Sons.
- Beeley, P.R. and R.F. Smart, *Investment casting*. 2023: CRC Press.
- Yassen, Z.N. and I.M. Hummudi, *Influence of Different Wax Hardening Agents on Surface Roughness of Refractory Cast Investment Materials: surface roughness of refractory investment material*. Journal of Techniques, 2021. **3**(3): p. 91-95.
- Chomsaeng, N., P. Chidnudsadeegul, and S. Niyomsoan, *Characterisation of phosphate-bonded investments for platinum casting*. Suranaree Journal of Science and Technology, 2021. **28**(6).
- Kazemi, A., M. Faghihi-Sani, and H. Alizadeh, *Investigation on cristobalite crystallization in silica-based ceramic cores for investment casting*. Journal of the European Ceramic Society, 2013. **33**(15-16): p. 3397-3402.
- Barroso, G., et al., *Polymeric and ceramic silicon-based coatings—a review*. Journal of materials chemistry A, 2019. **7**(5): p. 1936-1963.
- Çeker, E., *Effect of Processing Conditions on the Properties of Dental Porcelain*. 2019, Marmara Universitesi (Turkey).
- Zheng, B., *Development of Novel Slow-Release Micronutrient Fertilizers Using Mechanochemical Synthesis*. 2023.
- Phumying, S., *Synthesis and structural properties of ammonium metal*

- phosphate $NH_4MPO_4 \cdot H_2O$ ($M = Mg, Co, Fe, Mn, Ni, Zn, Cu$). 2019, School of Physics Institute of Science Suranaree University of Technology.
20. Saran, D., et al., *Review on the phosphate-based conversion coatings of magnesium and its alloys*. International Journal of Minerals, Metallurgy and Materials, 2022. **29**(7): p. 1435-1452.
 21. Sengupta, P., *Refractories for the Chemical Industries*. 2020: Springer.
 22. Chen, X., et al., *High strength silica-based ceramics material for investment casting applications: Effects of adding nanosized alumina coatings*. Ceramics International, 2020. **46**(1): p. 196-203.
 23. Bazhenov, V., et al., *Analysis of the slurry and ceramic properties for investment casting obtained with domestic colloidal silica binders*. NON-FERROUS METALLURGY, 2023. **29**(2): p. 15-28.
 24. Fedorov, K., C. Ravindran, and K. Fayazbakhsh, *Effects of process parameters on friability and surface quality in the rapid investment casting process*. The International Journal of Advanced Manufacturing Technology, 2023. **125**(1): p. 731-742.
 25. Hao, Y., et al., *Effects of mold materials on the interfacial reaction between magnesium alloy and ceramic shell mold during investment casting*. Metals, 2020. **10**(8): p. 991.
 26. Canay, Ş., et al., *Comparision of diametral tensile strength of microwave and oven-dried investment materials*. The Journal of prosthetic dentistry, 1999. **82**(3): p. 286-290.
 27. Serra-Prat, J., et al., *Adhesion of dental porcelain to cast, milled, and laser-sintered cobalt-chromium alloys: shear bond strength and sensitivity to thermocycling*. The Journal of prosthetic dentistry, 2014. **112**(3): p. 600-605.
 28. Nagasawa, Y., et al., *Characteristics of a new dental stone mixed by shaking*. Dental Materials Journal, 2020. **39**(3): p. 355-366.
 29. Kacalak, W., et al., *Assessment of the classification ability of parameters characterizing surface topography formed in manufacturing and operation processes*. Measurement, 2021. **170**: p. 108715.
 30. Sudhakar, A., et al., *Evaluation of the various drying methods on surface hardness of type IV dental stone*. Journal of International Oral Health: JIOH, 2015. **7**(6): p. 103.



Figure 1: Investment specimens heated using model drying oven



Figure 2: Investment specimen under surface hardness Shore D durometer unit

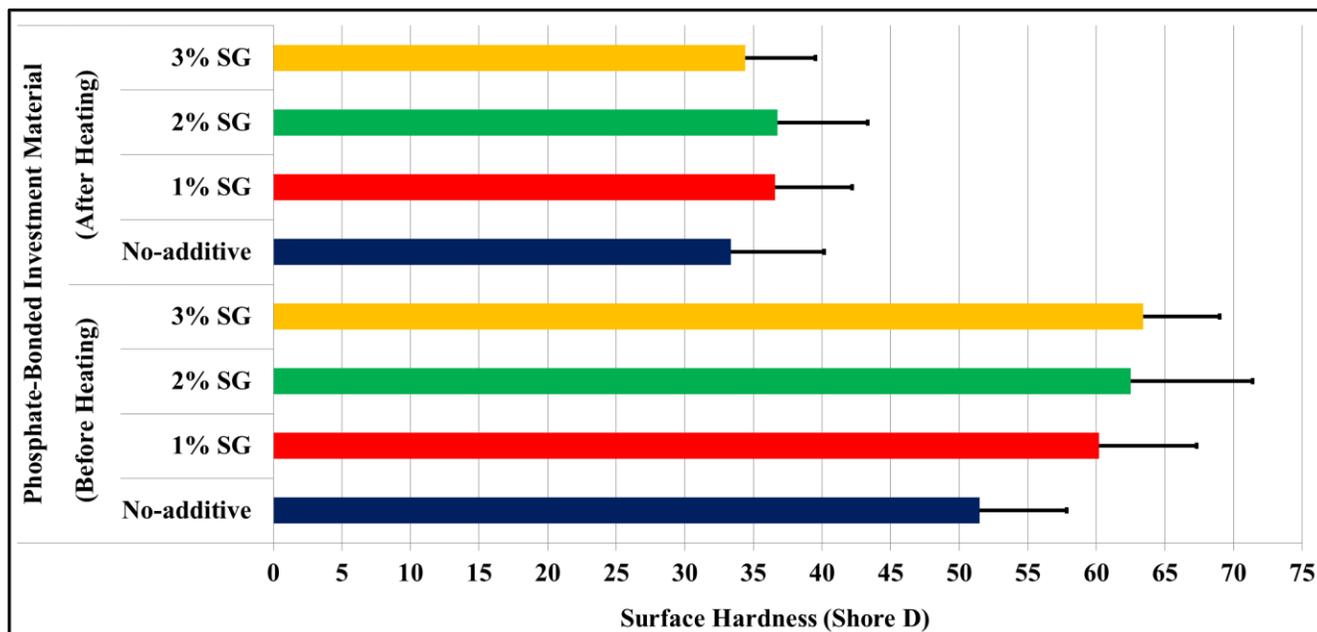


Figure 3: Bar-chart of studied phosphate-bonded investment material after the addition of silica gel before and after heat treatment

Table 1: Analysis of variance (ANOVA) for surface hardness between investment-tested materials before dry heating

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1421.687 ^a	3	473.896	9.441	.000*
Intercept	225848.305	1	225848.305	4499.412	.000*
BGroupsSR	1421.687	3	473.896	9.441	.000*
Error	3011.704	60	50.195		
Total	230281.696	64			
Corrected Total	4433.391	63			

a. R Squared = .321 (Adjusted R Squared = .287)

* Indicate a significant difference ≤ 0.05

Table 2: Multiple comparisons (Tukey HSD test) of Surface hardness between investment-tested materials before dry heating

Groups		Mean Difference	Std. Error	P-Value	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
Control	1%SG	-8.6769*	2.50487	.005*	S	-15.2961	-2.0577
	2%SG	-11.0300*	2.50487	.000*	S	-17.6492	-4.4108
	3%SG	-11.9056*	2.50487	.000*	S	-18.5248	-5.2864
1%SG	2%SG	-2.3531**	2.50487	.784	NS	-8.9723	4.2661
	3%SG	-3.2287**	2.50487	.573	NS	-9.8479	3.3904
2%SG	3%SG	-.8756**	2.50487	.985	NS	-7.4948	5.7436

* Indicate a significant difference ≤ 0.05

Table 3: Analysis of variance (ANOVA) for surface hardness between investment-tested materials after dry heating

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	133.919 ^a	3	44.640	1.208	.314
Intercept	79605.096	1	79605.096	2154.822	.000*
Groups	133.919	3	44.640	1.208	.314
Error	2216.566	60	36.943		
Total	81955.580	64			
Corrected Total	2350.485	63			

a. R Squared = .321 (Adjusted R Squared = .287)

* Indicate a significant difference ≤ 0.05