



## The effect of additives on the linear thermal expansion of Phosphate-bonded investment

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### Abstract

One of the main considerations of the investment materials is their adequacy to expand thermally and by setting expansion to compensate for casting shrinkage, and any investment having zero setting expansion and adequate thermal expansion, by the use of chemical additives and water instead of colloidal silica suspension would eliminate all inaccuracies that arise from distortion of the wax pattern due to setting expansion.

Thus the aim of this study was to evaluate the influence of two alkaline metals as additives  $K_2SiO_3$  and  $Na_2SiO_3$ , to enhance the thermal expansion of phosphate-bonded investment without the use of colloidal silica suspension.

Three mixtures (45 samples) were prepared, mixture I of 15 samples contains  $Na_2SiO_3$ , Mixture II of 15 samples contains  $K_2SiO_3$  in percentages of (0.5, 1.0, 1.5, 2.0, and 2.5) for each three samples of both (mixture I & II), and mixture III of fifteen samples as a control group without additives. The thermal expansion measured by vitreous silica dilatometer from 100 – 950 °C.

The results showed that the highest mean of thermal expansion was in samples that contain  $Na_2SiO_3$ , and the lowest mean of thermal expansion was in the control group samples.

**Key words:** Phosphate-bonded investment, Thermal expansion, Alkaline additives.

### Introduction

Phosphate bonded investment materials have become more popular for casting high melting Nobel-metal (precious and semiprecious) and base metal alloys in fixed prosthodontics<sup>(1, 2)</sup>.

Phosphate bonded investment materials consist essentially of two main groups of ingredients, fillers and binders<sup>(3, 4)</sup>. The fillers are usually quartz and/or cristobalite. The binders are essentially basic MgO and acidic Ammonium – Phosphate hydrate, and they play very important role in the

chemistry of the investment during setting and further thermal reaction<sup>(3)</sup>, in addition to other chemicals as additives<sup>(5)</sup>, particularly soluble silicate, which are systems of an alkali metals, and the most commercially used soluble silicate being those of Sodium and Potassium<sup>(6)</sup>. Other modified phosphate-bonded investment by addition of berlinite ( $AlPO_4$ )<sup>(7)</sup>.

One of the main considerations of the investment materials is their adequacy of investment expansion to compensate for casting shrinkage<sup>(1)</sup>.

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Ideally, the sum of the setting and thermal expansion should be equal the solid thermal contraction of the alloy being used<sup>(8)</sup>.

The thermal expansion of the phosphate-bonded investment materials is the most important expansion for counter balancing shrinkage from the solidus temperature to room temperature of casted alloy than setting expansion or hygroscopic expansion<sup>(9)</sup>.

Hygroscopic expansion of less value especially when the phosphate-bonded investment materials are mixed with water rather than mix with silica solution, however, not all Phosphate bonded investment can expand hygroscopically<sup>(5)</sup>.

The setting reaction of phosphate-bonded investment is exothermic and accompanied by an expansion, which tends to cause a distortion of the invested wax pattern and thereby to affect the precision of the final casting in an adverse way<sup>(10, 11)</sup>. Therefore, the use of investment having zero setting expansion by using water or possibly an aqueous glycerol solution instead of the special liquids would eliminate all inaccuracies arising from distortion of wax pattern, and this investment should be modified to have sufficient thermal expansion to compensate for the thermal shrinkage of the alloys<sup>(12)</sup>.

The thermal expansion of the dental investment could be enhanced by the use of alkali metals as additives, or the use of glycerol when added to the investment mix, particularly the gypsum-bonded investment<sup>(13)</sup>.

Thus, the aim of this study was to determine the influence of two chemical materials as additives on the thermal expansion of phosphate-bonded investment material (without the use of colloidal silica suspension) through calculating the linear thermal expansion of a standard blocks, and heated up to 950 C°.

## Materials and methods

### *Experimental preparation:*

The powder of the experimental phosphate-bonded investment was prepared by mixing of two types of Silica (quartz and cristobalite) as a filler, and MgO and NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> as a binder. Additives studied were Potassium Silicate and Sodium-Silicate in each case (added to the investment powder); water had been used as mixing liquids without colloidal silica suspension.

The composition of the experimental investment powder was identical in all cases (30% cristobalite, 50% quartz, 10% MgO, 10% NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>) weight ratio, and was prepared by mixing all ingredients in laboratory mixer for 60 minutes. While additives used in different percentages (0.5, 1.0, 1.5, 2.0, 2.5) for both Sodium-Silicate and Potassium-Silicate.

All ingredients were mixed in laboratory mixer (Zuma auto-mixing machine\ Italy) for 60 minutes.

### *Sample preparation;*

Cylindrical mold of 100 mm length, with 10 mm diameter (according to BSI 5189: 1975)<sup>(14)</sup> was used for the preparation of forty five samples. Fifteen samples with Na<sub>2</sub>SiO<sub>3</sub> used different percentages, (3 samples for each percentage of 0.5, 1.0, 1.5, 2.0 and 2.5), the other fifteen samples used K<sub>2</sub>SiO<sub>3</sub>, at different percentages, (3 samples for each percentage of 0.5, 1.0, 1.5, 2.0 and 2.5), and the last fifteen samples without any additives (the control group). The mixing of powder\liquid ratio was determined based on ISO 9694: 1996<sup>(15)</sup> Samples were allowed to set at room temperature in air for one hour.

### *Thermal Expansion*

The apparatus for measurement of the thermal expansion was vitreous silica dilatometer fig. (3) <sup>(16)</sup> which comprises the following:

- 1-Silica tube ST located in the hot zone of a tube furnace F.
- 2-Silica rod SR which rest on the specimen contain within the tube.
- 3- Displacement transducer DT, the core of which is directly connected to the rod SR (the maximum restraint on the specimen not exceed  $(1\text{KN} \setminus \text{M}^2)$ ).

The (dial gauge equipment) capable of measuring the change in length to the nearest 0.001mm (one micrometer), on heating from room temperature up to 950 °C.

The samples were placed inside the test apparatus with the tube furnace at room temperature, then heated to 100 °C (holding at this temperature for one hour) the temperature then raised at rate of 5 °C \ min. up to 950 °C.

The reading of the dial gauge had been recorded at intervals of 50 °C, so 19 readings had been obtained for each sample. The thermal expansion was calculated as a percentage of the initial length.

Analysis of variance (ANOVA) considering the effect of the additives on the linear thermal expansion was done to evaluate the possible main and interactive effects of these basic parameters in addition to paired test to compare between groups.

## Results

The mean curves depicting the effect of additives on the linear thermal expansion of phosphate-bonded investment for the three groups are presented in (figure 1).

(figure 2) shows that phosphate-bonded investment without additives achieve maximum total expansion of approximately 0.84 % at 650 °C , phosphate-bonded investment with

K<sub>2</sub>SiO<sub>3</sub> additives achieve maximum total expansion of approximately 1.06 % at 700 °C ,while phosphate-bonded investment with Na<sub>2</sub>SiO<sub>3</sub> additives achieve maximum total expansion of approximately 1.12 % at 760 °C.

The results were analyzed using one-Way ANOVA test and paired sample statistics.

The mean linear thermal expansion for phosphate-bonded investment without additives, with K<sub>2</sub>SiO<sub>3</sub> additives and with Na<sub>2</sub>SiO<sub>3</sub> additives were 0.84, 1.06 and 1.12 respectively as shown in table (1).

One-Way ANOVA test (table 2) shows that there is a significant difference among different concentrations of K<sub>2</sub>SiO<sub>3</sub> on thermal expansion, and there is a highly significant difference among different concentrations of Na<sub>2</sub>SiO<sub>3</sub> on thermal expansion

Paired sampled test revealed that there was a highly significant differences ( $P < .001$ ) between phosphate-bonded investment with Na<sub>2</sub>SiO<sub>3</sub> additives group and control group, and between phosphate-bonded investment with K<sub>2</sub>SiO<sub>3</sub> additives group and control group, and also between phosphate-bonded investment with Na<sub>2</sub>SiO<sub>3</sub> additives group and K<sub>2</sub>SiO<sub>3</sub> additives group (table 3).

## Discussion

In general the powder of the phosphate-bonded investment consists of SiO<sub>2</sub> as refractory fillers, MgO and NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> as binder, and other additives that could modify the properties of the investment which the manufacturers not announce about them.

To obtain general result, we use two types of fillers (quartz and cristobalite) in a percentage of 30: 50. The weight ratio of MgO and NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> was 10%: 10%. The

increase or alteration of these percentages to 6:14 (which used by some manufacturers) gives more setting expansion, and as a consequence more wax pattern distortion, (which we try to avoid), obtaining that investment of lesser setting expansion and more thermal expansion.

Although colloidal Silica suspension increase thermal expansion, but it also increase the setting expansion, thus it had not been used in this study. <sup>(17)</sup>

Based on the types of additives, three mixtures were obtained:

- 1-Mixture I: that contain Na<sub>2</sub>SiO<sub>3</sub> in different percentages (0.5, 1.0, 1.5, 2.0 and 2.5) we find that the increase in the percentage of the Na<sub>2</sub>SiO<sub>3</sub> increase the thermal expansion till concentration 2% above which very little expansion had been obtained.
- 2-Mixture II: that contain K<sub>2</sub>SiO<sub>3</sub> in different percentages (0.5, 1.0, 1.5, 2.0 and 2.5) in which the increase in the thermal expansion continuous till concentration 2.5%.
- 3-Mixture III: that contains no additives as a control group.

The three mixtures of phosphate-bonded investment on heating they start to expand at about 100 °C, at 170 – 230 °C the control group show no expansion, while the K<sub>2</sub>SiO<sub>3</sub> group at 270 – 330 °C and the Na<sub>2</sub>SiO<sub>3</sub> group at 330 – 380 °C show no expansions figure 1 and figure 2 .

All the three groups show great expansion between 400 – 600 °C, and the maximum expansion at 650 °C for the control group, after this temperature a very little expansion till 950 °C. While the other two groups K<sub>2</sub>SiO<sub>3</sub> and Na<sub>2</sub>SiO<sub>3</sub> continuous till 700 °C and 760 °C respectively, further heating up to 950 °C give no more expansion.

The results of paired sample test show that the differences was highly

significant among the three groups and indicate the efficiency of these two chemical additives in enhancement of the phosphate – bonded investment thermal expansion, with the Na<sub>2</sub>SiO<sub>3</sub> was better than K<sub>2</sub>SiO<sub>3</sub> in its result, and further investigations may be required to evaluate the effect of these additives on the setting expansion of the phosphate-bonded investment.

## Conclusion

Based on findings of the present study; the use of alkaline metals Na<sub>2</sub>SiO<sub>3</sub> and K<sub>2</sub>SiO<sub>3</sub> enhance the thermal expansion of the phosphate-bonded investment, especially the Na<sub>2</sub>SiO<sub>3</sub>. But still this value of thermal expansion alone (without the setting expansion) not enough for counter-balancing the shrinkage from the solidus temperature to room temperature of the casted (non-precious) alloy.

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## Appendix

Table (1) Descriptive Statistics.

	N	Minimum	Maximum	Mean	Std. Deviation
Control	15	0.75	0.92	0.8400	0.05695
Na <sub>2</sub> SiO <sub>3</sub>	15	1.06	1.19	1.1200	0.04226
K <sub>2</sub> SiO <sub>3</sub>	15	1.01	1.14	1.0600	0.03606
Valid N (listwise)	15				

Table (2) ANOVA

	Sum of Squares	df	Mean Square	F	Sig.
K <sub>2</sub> SiO <sub>3</sub> between Groups	0.012	4	0.003	4.684	0.022
Within groups	0.006	10	0.001		
Total	0.018	14			
Na <sub>2</sub> SiO <sub>3</sub> between Groups	0.021	4	0.005	12.621	0.001
Within groups	0.004	10	0.000		
Total	0.025	14			

Table (3) Paired Samples Test

	Paired Differences					t	df	Sig. (2-taild)
	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the differences				
				Lower	Upper			
Pair 1 K2SiO <sub>3</sub> - Control	0.2200	0.03910	0.01009	0.19835	0.24165	21.793	14	0.000
Pair 2 Control- Na2SiO <sub>3</sub>	-0.2800	0.06793	0.01754	-0.31762	-0.24238	-15.964	14	0.000
Pair 3 K2SiO <sub>3</sub> - Na2SiO <sub>3</sub>	-0.6000	0.04826	0.01264	-0.08672	-0.03328	-4.8160	14	0.000



