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³¹P solid-state MAS-NMR spectroscopy of the compounds that form in phosphate-bonded dental casting investment materials during setting

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ABSTRACT

Objectives. To use ³¹P solid-state MAS-NMR to determine which compounds form in phosphate-bonded dental casting investment material during setting, when the ambient temperature is altered. To determine whether they differ in material originating at the center of the mix from material that adheres to the mixing bowl wall.

Methods. ¹H high powered decoupled (HPDC) and ¹H cross polarized (CP) ³¹P solid-state MAS-NMR spectroscopy were used at a resonance frequency of 121.4 MHz to determine molecular structure. Four commercial products were examined. Manufacturer's instructions were followed and special liquid used without dilution. Ambient temperature was between 18 and 37 °C.

Results. Molecular structures change with ambient temperature and product. Amorphous Mg₃(PO₄)₂ or struvite dominate with newberyite, cattiite, amorphous Mg₂P₂O₇ and amorphous MgHPO₄ present as minor phases. Exceptionally, amorphous MgHPO₄ dominates. Differences in structure were found in material taken from the center of the mix compared with that scraped from the bowl wall, but the incidence may be specific to the product/mixer combination and not a general effect.

Significance. The formation of compounds in phosphate-bonded investment can be affected by ambient temperature. This effect and the use of material adhering to the bowl wall (instead of that from the center of the mix) are possible causes for the unpredictability of setting expansion measurements between laboratories. There is variation between products. When phosphate-bonded investment is required for casting, a consistent ambient temperature must be used and it would be wise to mix sufficient material to avoid scraping the bowl.

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1. Introduction

Phosphate-bonded investment material is used extensively for casting both precious and base metal dental alloys. A recent investigation into the structure of commercial products using ³¹P solid-state MAS-NMR revealed the presence

of phosphate compounds not previously known to form in phosphate-bonded casting investment material [1]. In the set investment struvite [crystalline MgNH₄PO₄·6H₂O], amorphous Mg₃(PO₄)₂, amorphous MgHPO₄ and newberyite [crystalline MgHPO₄·3H₂O] were found. Earlier investigators used X-ray diffraction spectroscopy to determine which compounds form

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Table 1 – Dental casting investment products studied

Products	Manufacturer	Lot	Sachet mass (g)	Recommended use
Deguvest GF	Degussa Dental GmbH & Co. KG, Frankfurt, DE	100078	60	Precious alloy casting
Optivest	Degussa Dental GmbH & Co. KG, Frankfurt, DE	5000481	400	Chrome casting
Fujivest Super	GC Europe, Leuven, BE	15390414	150	Chrome or precious alloy casting
Wirovest	BEGO GmbH & Co. KG, Bremen, DE	1075311	400	Chrome casting

[2,3]. Struvite was positively identified by Allan and Asgar [2] and its presence was confirmed by Nieman and Sarma [3]. Although they stated that crystalline $Mg_3(PO_4)_2 \cdot 4H_2O$ should form during setting, no experimental evidence was provided to support the claim [2] and Nieman and Sarma did not find it present [3]. The existence of a fine amorphous $Mg_3(PO_4)_2$ structure is consistent with the proposition that a gel forms first during setting and the absence of $Mg_3(PO_4)_2$ peaks in the X-ray powder diffraction spectrum. The application of ^{31}P solid-state MAS-NMR provided positive identification of the amorphous $Mg_3(PO_4)_2$ [1]. Formation of amorphous $MgHPO_4$ and newberyite was attributed to the temperature rise produced by the main setting reaction exotherm creating conditions favourable for their formation [1]. This proposition was supported by the work of Conway-Wagner et al. [4] who demonstrated that an alteration of the temperature rise during setting changes the phosphate compounds formed in related civil engineering cements.

ISO 15912 [5] specifies that the testing of dental casting investment products for compliance with the requirements in the standard should be carried out at a controlled temperature ($23 \pm 1^\circ C$), and that the materials and test equipment is to be held at this temperature for a minimum of 16 h prior to testing. Stringent control over test temperature is considered necessary to obtain reproducible test results, implying that the set structure is affected significantly by variations in ambient laboratory temperature. For the present investigation, the null hypothesis is proposed, that the structure is unaffected by such variations. Insensitivity is a reasonable proposition since the temperature rise produced by the exothermic setting reaction appears to be significantly greater than the expected temperature variation in and between laboratories.

Clearance between the vacuum-mixer paddle and bowl wall (a gap of 1–2 mm) results in part of the mix being pressed onto the wall by the rotating paddle, to remain there without further mixing. (This is most evident with products that have a pourable consistency. After pouring, material with a thicker consistency that does not pour is left coating the walls and base of the bowl.) There is a belief among many technicians that material taken from the center of the mix gives more reproducible results than material scraped from the wall of the bowl and is to be used preferably. Such practice is not research led and the effect could be apocryphal. On the other hand, particular investment product/mixer combinations could produce a significant effect. Technicians who have experienced a problem may assume it to be common knowledge, passed informally around the trade as a technical tip. A second null hypothesis is put, that the structure of the set investment is identical in material originating from the center of the mix and from the mix adhering to the bowl wall.

To test these hypotheses the solid-state ^{31}P MAS-NMR experiments reported in the previous paper [1] were used with the added variables of ambient temperature and sampling site in the mix.

2. Materials and methods

Four commercial dental phosphate casting investment products were investigated (Table 1). For each mix, an unopened powder sachet was used with the appropriate volume of undiluted special liquid recommended in the manufacturer's instructions. The volume of the mixing bowl restricted the mass of investment powder to 100 g. For Fujivest Super, Wirovest and Optivest, the powder was weighed to an accuracy of 1.0 g. For Deguvest GF one complete 60 g sachet was used. Powder and liquid were hand spatulated for 15 s to obtain an even distribution initially. All products were vacuum-mixed for 60 s (Continental Vacuum-mixer, Whip Mix Corp., Louisville, KY, USA) (420 rpm, 85 kPa vacuum, 500 ml bowl). Fujivest Super was an exception. Its manufacturer recommends applying the vacuum for 15 s to degas the mixture before starting mechanical mixing. Specimens were produced in dry-lined stainless steel casting rings 35 mm in length and 30 mm in diameter.

The ambient temperature of the laboratory was set at 18, 21, 28, 33 or $37^\circ C$ and controlled to $\pm 1^\circ C$. Materials and equipment were stored in this environment for 12 h before use.

The vacuum-mixer was sited in a "plaster room" adjacent to the temperature controlled laboratory. The temperature of the plaster room was a constant ($19 \pm 2^\circ C$). Relocation of the mixer to the temperature controlled laboratory was not feasible. After hand mixing in the temperature controlled laboratory, vacuum mixing took place in the plaster room, immediately after which the mix was returned to the temperature controlled laboratory to fill the casting ring. The salient point is whether a significant change in the temperature of the mix resulted from the transient change in ambient temperature during mechanical mixing. To investigate this, the temperature of the material was measured (to an accuracy of $0.1^\circ C$) from storage to the completion of setting at an ambient temperature of $34^\circ C$. During this time the hand mixed material was transferred to an ambient temperature of $20.5 \pm 1.5^\circ C$, held for 60 s and then returned to the higher ambient temperature environment. No mechanical mixing took place since it was impossible to record mix temperature during vacuum mixing.

In a second series of tests, a constant ambient temperature existed at all times (while the temperature of the material was being measured) to provide information on the effect of the heat of solution and temperature change in the absence

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