EFFECT OF PHOSPHATE PRECURSORS ON SOL-GEL PHOSPHATE GLASS
ELUCIDATION ON THE MECHANISM

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The aim of this study is to make a comparison between the two main phosphate sol-gel precursors, n-butyl phosphate (mixture of mono and di butyl) and ethyl phosphate (mono and di ester mixture) in the preparation of P-Ca-Na sol-gels. For the first time a possible mechanism is proposed.

The ultimate goal of this work is the development of porous, soluble sol-gel glasses capable of holding growth factors and dissolving overtime for an enhanced bioactivity/ bio-remineralisation by stimulating odontoblasts to deposit a new hydroxyapatite layer.

MATERIALS & METHODS

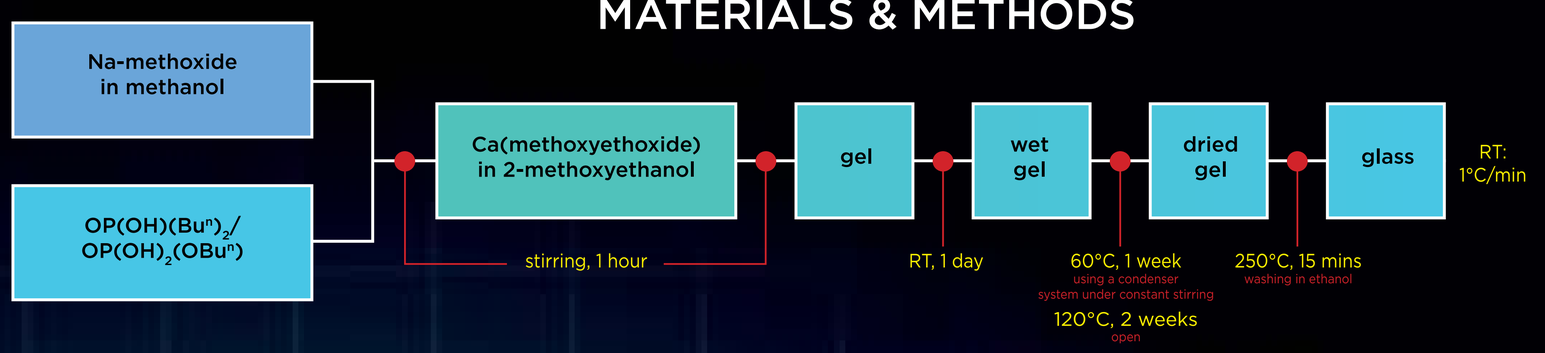

Fig.1 Flow diagram of the sol-gel preparation

Table 1 BET analysis on the sol-gel derived glass particles

Glasses	Pore volume [cms3 g-1]	Pore size [nm]	Surface area [m2 g-1]	pH
Bu/PBG-C24	0,014651	10,537	5,5619	3.27
Et/PBG-C24	0,047313	18,711	10,115	3.20

Table 2 Actual and theoretical sol-gel derived glass formulation given by XRF analysis

Mol%	P205 (53%)	Ca0 (24%)	Na20 (23%)
Bu/PBG-C24	49%	23%	27%
Et/PBG-C24	56%	24%	20%

RESULTS

After calcination, the ethyl-derived glass powder appeared more homogenous and the yield was 5% more than the butyl one. The XRD shows for butyl source a 98.9wt% amorphous glassy phase (**Fig. 2**), 100wt% for the ethyl one (**Fig. 3**). The XRF indicates the following compositions P2O549%-CaO23%-Na2O27% and P2O556%- CaO24%-Na2O20% for the butyl (Bt/PBG) and ethyl (Et/PBG) phosphate respectively (**Table 2**). 1mg/mL of each powder was incubated in BME (Basal Medium Eagle) at 37 °C. The pH was measured after 24 hours giving 3.27±0.02 and 3.20±0.02 for the butyl and ethyl phosphate respectively (**Table 1**). The BET analysis shows that the surface area (S.a.), pore size (P.s.) and pore volume (P.v.) are respectively 45%, 44% and 69% bigger for the ethyl than for the butyl glass (**Table 1**).

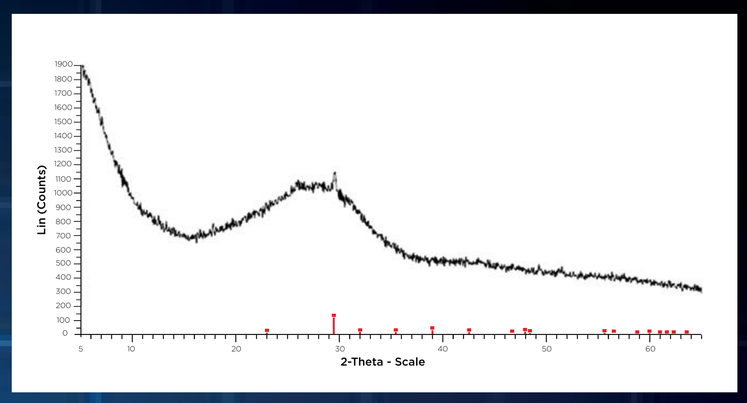

Fig.2 XRD analysis on the PBG-C24 formulation

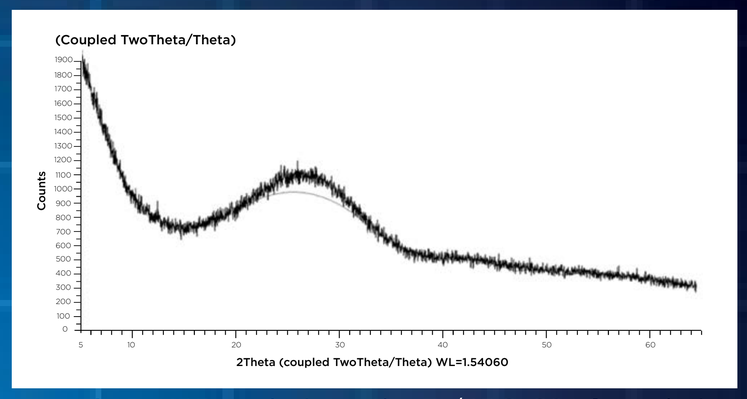

Fig.3 XRD analysis on the Et/PBG-C24 formulation

CONCLUSIONS

The reported data shows that the different phosphate precursors affect the actual composition and the physical parameters of the glass particles. The ethyl precursor leads to final compositions that are much closer to the target stoichiometry (**Table 2**) compared to the butyl one. All the physical parameters, desirable for the stated dental application, are enhanced via Et/PBG (**Table 1**); whereas, no difference is detectable concerning the pH (**Table 1**).

Therefore, here we show how the use of different phosphate sol-gel precursors reveals differences in the properties of the phosphate glass products and the kinetics of the sol-gel reactions taking place. It is suggested that these differences are caused by steric variations between the ethyl and butyl substituents. A possible reaction mechanism is suggested where the butyl is displaced and replaced faster than the ethyl. In addition, due to the smaller size, the ethyl remains trapped easily into the network during the formation, giving an explanation for the bigger values founded (**Table 1**).

In order to improve both the glass formulation and elaboration, further doping will be tested using some inorganic therapeutic ions and less toxic precursors will be employed. Assessment of powders will cover cell response and rate of ion/therapeutics dissolution into aqueous media.

ACKNOWLEDGMENTS

The research leading to this poster has received funding from the European Union Seventh Framework Program (FP7/2007-2013) under grant agreement n°608197