Unravelling the effect of thermal treatment on crystallization of sol-gel derived 45S5 bioactive glass

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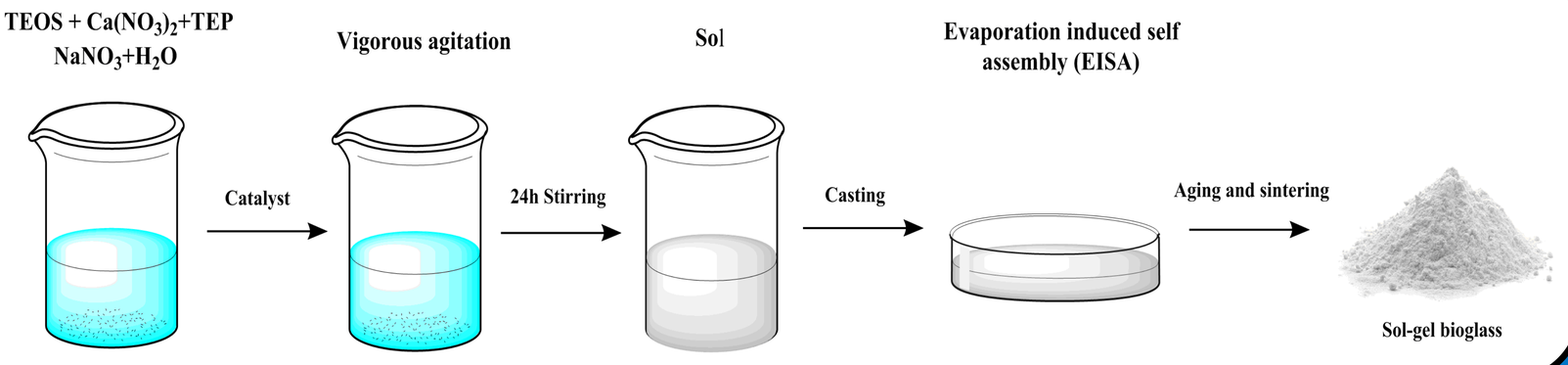
Introduction

The ability to sinter a complex bioactive glass three-dimensional porous structures is of great interest in context of bone regeneration or templates in tissue engineering. Thermal treatment of bioactive glass ceramics dictates many important features such as microstructure, degree of crystallinity, mechanical properties, and biological response. In this context, herein, we investigated the effects of thermal treatments on crystallization of sol-gel derived 45S5 bioactive glass. Temperature and time of the thermal treatment strongly influence the formation of different crystalline phases. Combeite ($Na_2Ca_2Si_3O_9$) crystallizes as the primary crystalline phase along with a calcium silicate-phosphate ($Ca_{15}(PO_4)_2(SiO_4)_6$) phase, which decomposes into rhenanite after prolong thermal treatment. Our results provide a basic platform for tailoring the crystalline phases by controlling the nucleation and growth of crystalline phases via thermal treatments.

Goals

- Synthesis of sol-gel derived 45S5 bioactive glass
- Influence of the sol-gel synthesis method on crystallization
- Influence of thermal treatments on crystallization and identification of formed crystalline phases
- Quantification of amorphous/crystalline fractions via rietveld refinement

Synthesis Route



XRD analysis

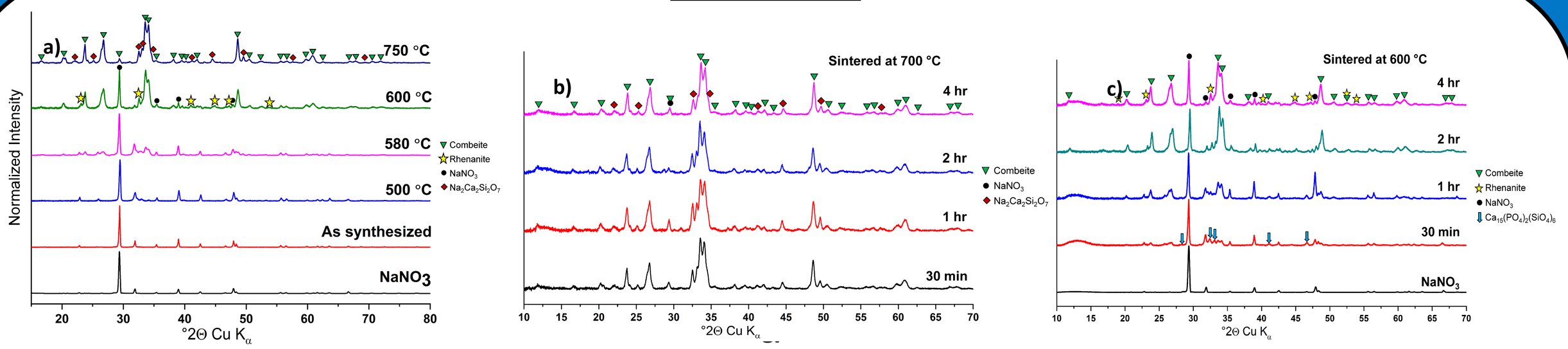


Fig: (a) XRD analysis of 45S5 Bioactive glass powder thermally treated at different temperatures , (b) 700 °C and (c) 600 °C for different time points (taken from previous open access publication) [1].

Table: Evolution of crystalline phases as a function of time obtained by Rietveld refinement of powder treated at 600 °C and 700°C (taken from previous open access publication) [1] .

700 °C	Relative amount (wt. %)		
	Combeite	$NaNO_3$	$Na_2Ca_2Si_2O_7$
30 min	75.8	2.6	21.5
1 h	73.3	2.5	24.2
2 h	77.0	1.2	21.9
4 h	81.3	1.5	17.2

600 °C	Relative amount (wt. %)			
	Combeite	Rhenanite	$NaNO_3$	$Ca_{15}(PO_4)_2(SiO_4)_6$
30 min	30.6	-	39.2	30.2
1 h	44.6	-	37.3	18.1
2 h	74.2	11.3	14.5	-
4 h	72.5	14.3	13.2	-

TEM and nano-CT

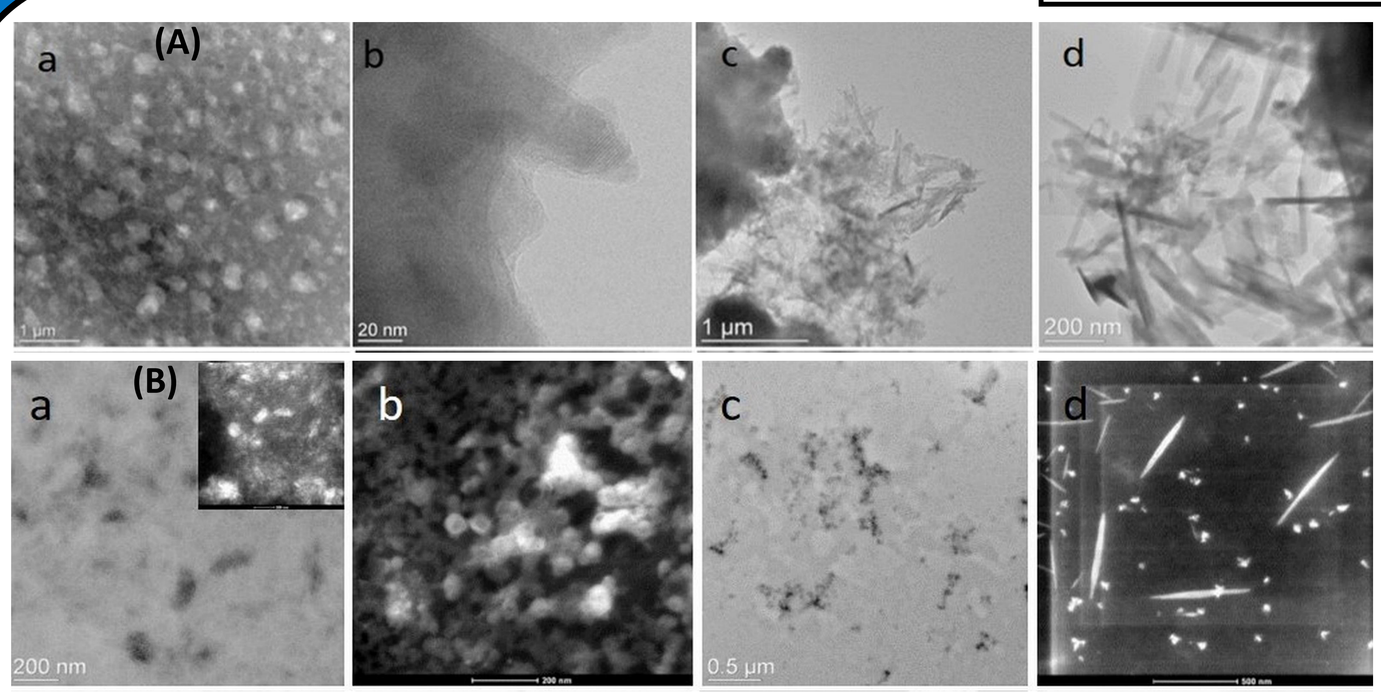


Fig: TEM micrographs of the samples obtained at 600°C (A) and 700°C (B) respectively , during 30 min (a,b), 1 h (c,d) (taken from previous open access publication) [1].

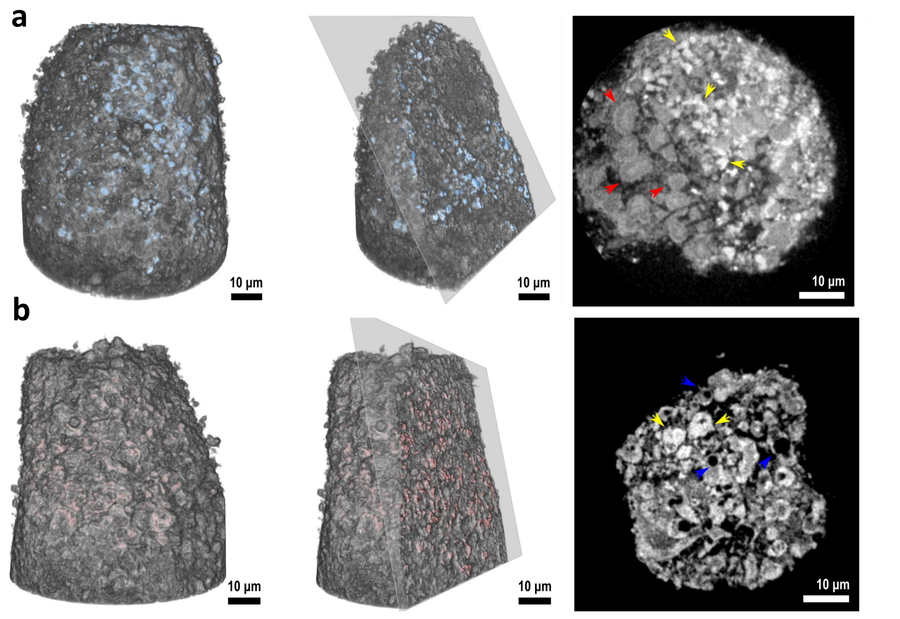


Fig: Nano-CT images of the heat-treated powders (a) at 600°C for 4 h, and (b) at 700°C for 4 h. Left: volumetric representation; center: volumetric representation with a virtual cut; and right: tomograms. Arrows highlight different morphological features in the samples (taken from previous open access publication) [1].

Conclusions

- 45S5 bioactive glass ceramic was successfully synthesized via sol-gel route;
- The unreacted precursors i.e nitrates are decomposed after sintering at 600 °C;
- At 600°C combeite crystallized as the main phase along with a calcium silicate-phosphate phase, which decomposes into rhenanite at 700 °C, and a new sodium calcium silicate ($Na_2Ca_2Si_2O_7$) crystalline phase formed.

Acknowledgments

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Reference

[1] Q. Nawaz, A. de Pablos-Martín, J.M. de S. e Silva, K. Hurle, A.C. Jaimes, D.S. Brauer, A.R. Boccaccini, New insights into the crystallization process of sol-gel derived 45S5 bioactive glass, J. Am. Ceram. Soc. 103 (2020) 4234–4247.

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