How liquid phase composition affects on properties of calcium phosphate biomicroconcretes based on α-TCP?

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INTRODUCTION

Due to the increased requirements for bone tissue substitutes many studies on biomaterials are conducted. The main efforts are focused on the optimization of their parameters, such as: mechanical strength or surgical handiness. Biomicroconcretes possess unique composition, which is inspired by classical concretes, where aggregates in the form of granules provide the satisfactory mechanical properties. Biomicroconcretes based on calcium phosphates are self-setting, bioactive materials for bone tissue engineering with high surgical handiness. The aims of this study were to develop and obtain novel biomaterials for bone substitution and to analyze the influence of the liquid phase composition on the physicochemical properties of the obtained biomicroconcretes. The solid phase of the investigated materials composed of **60 wt%** of highly-reactive **α-TCP** powder and **40 wt%** of **hybrid hydroxyapatite-chitosan (HAp/CTS) granules**. Mixtures of **2 wt%** of **disodium phosphate (Na_2HPO_4)** and **5 wt% citrus pectin (CP**) aqueous solutions were used as the liquid phases of the developed biomicroconcretes.

MATERIALS & METHODS

Setting times of obtained materials

Material	Powder phase (P)	Liquid phase (L)
MC1	α-TCP powder + HAp/CTS granules (3:2)	2 wt% disodium phosphate + 5 wt% CP (ratio 1:0)
MC2		2 wt% disodium phosphate + 5 wt% CP (ratio 3:1)
MC3		2 wt% disodium phosphate + 5 wt% CP (ratio 1:1)
MC4		2 wt% disodium phosphate + 5 wt% CP (ratio 1:3)

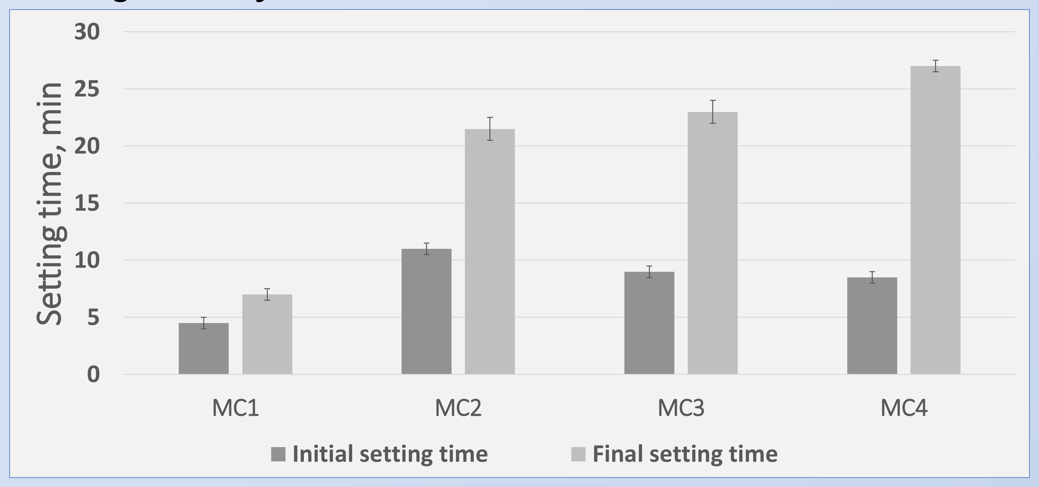
Liquid & powder phase mixing → Samples preparation

L/P= 0.5 g/g

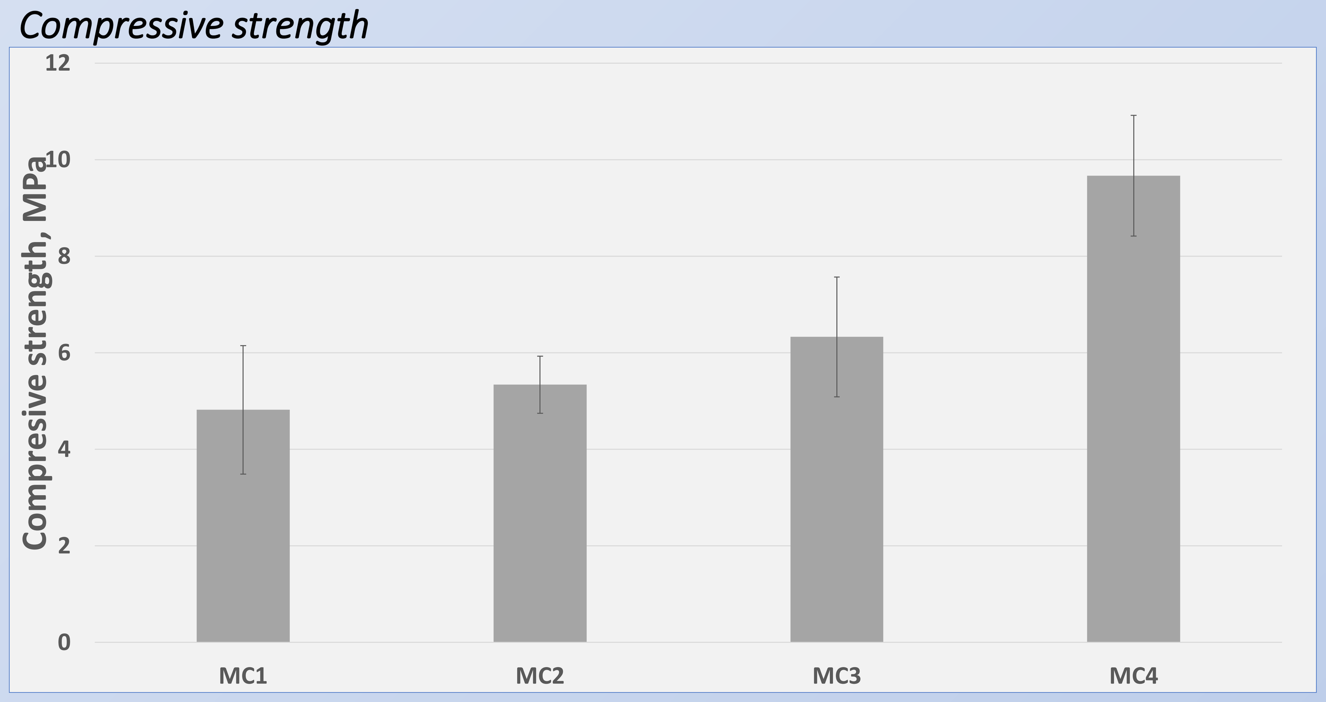
METHODS
- **Setting time** (Gillmore apparatus)
- **Phase composition** (D2 Phaser, Bruker)
- **Compressive strength** (Instron 3345)
- **SEM observations** (Phenom Pure, Thermo Fisher)

RESULTS

Setting times of obtained materials



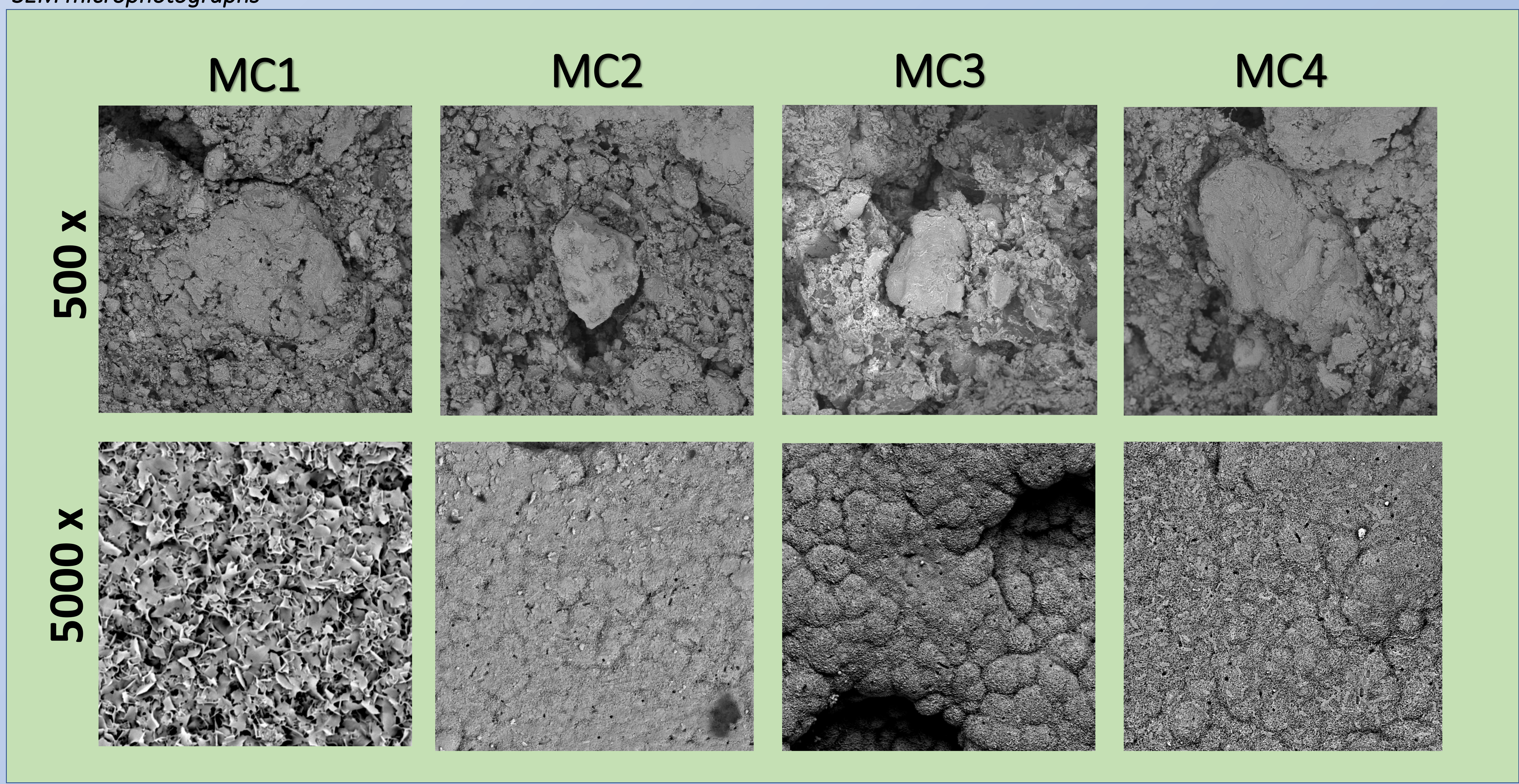
Compressive strength



Phase composition

Material	After 7-days setting and hardening (in air)		After 28-days incubation in SBF	
	α-TCP, wt%	Hydroxyapatite, wt%	α-TCP, wt%	Hydroxyapatite, wt%
MC1	59.0	41.0	2.0	98.0
MC2	58.0	42.0	1.0	99.0
MC3	62.0	38.0	1.0	99.0
MC4	65.0	35.0	3.0	97.0

SEM microphotographs



SUMMARY

- The biomicroconcretes composed of highly-reactive α-TCP powder, hybrid hydroxyapatite-chitosan (HAp/CTS) granules and citrus pectin were developed
- The studies have shown that, due to the ***dual setting system*** originated from hydrolysis of α-TCP to non-stoichiometric hydroxyapatite and interactions between polycationic chitosan in hybrid granules and polyanionic pectin in liquid phase, the obtained bone substitutes were characterized by unique properties
- It was observed that liquid phase in the form of solely pectin solution prevented biomicroconcrete from set. The rest of materials were characterized by self-setting properties. The presence of disodium phosphate as a setting accelerator allowed to obtain acceptable setting times (**4.5 – 11.0** min for initial, **7.0 - 27.0** min for final setting time).
- The compressive strength of obtained materials increased with increasing ratio of citrus pectin in liquid phase (**4.8 – 9.7 MPa**) and was suitable for low-load bringing applications.
- Developed biomicroconcretes can act as an alternative to commercially used sintered ceramic implants with predefined shape and size. The surgically handy and self-setting biomicroconcretes are potential candidates for bone tissue engineering, supporting its regeneration. Further biological studies are necessary.

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ACKNOWLEDGMENTS

Research funded by the Faculty of Materials Science and Ceramics AGH UST – University of Science and Technology, Kraków, Poland, Project No. 16.16.160.557 (2021). Supported by the National Science Centre, Poland Grant No. 2017/27/B/ST8/01173