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Synthesis of a glass-ceramic nano-material in the ternary system SiO-CaO-MgO-CuO: effect of ball milling on the particle size, morphology and bioactive behavior

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Various studies have reported that, Si-, Ca-, Mg- and Cu containing glasses, are highly bioactive and could be used in biomedical applications such as bone tissue regeneration [1]. Moreover, Cu is a trace element in the human body, known to play a significant role in angiogenesis [1]. Nanotechnology has a great potential to improve biomaterials used in tissue engineering [2]. Nano-bioceramics are preferable compared to their micro-scale counterparts, because of their increased surface area which can improve both mechanical properties and apatite formation ability due to the increased nucleation sites provided [3]. The aim of this study was to produce nano-sized glass-ceramics of the ternary system SiO-CaO-MgO-CuO.
Sol-gel derived bioactive glasses with composition in wt%: 60 SiO_2, 30 CaO, 7.5 MgO, 2.5 CuO were produced by the hydrolysis of TEOS in a mixture of d.d. H_2O, ethanol and HNO_3. Afterwards Ca, Mg, Cu were added as nitrate salts while ammonia solution was inserted dropwise under stirring in an ultrasonic bath [4]. The synthesized glass-ceramics were inserted in a planetary system (Retsch S100) for 6h with different rpm (300, 400 and 500). Bioactivity evaluation was conducted in SBF, with a ratio of 1.5 mg/mL for 1, 3, 5, 15 and 20 days under renewal conditions [5]. FTIR, TEM, XRD and SEM/EDS were used for the characterization of the samples.

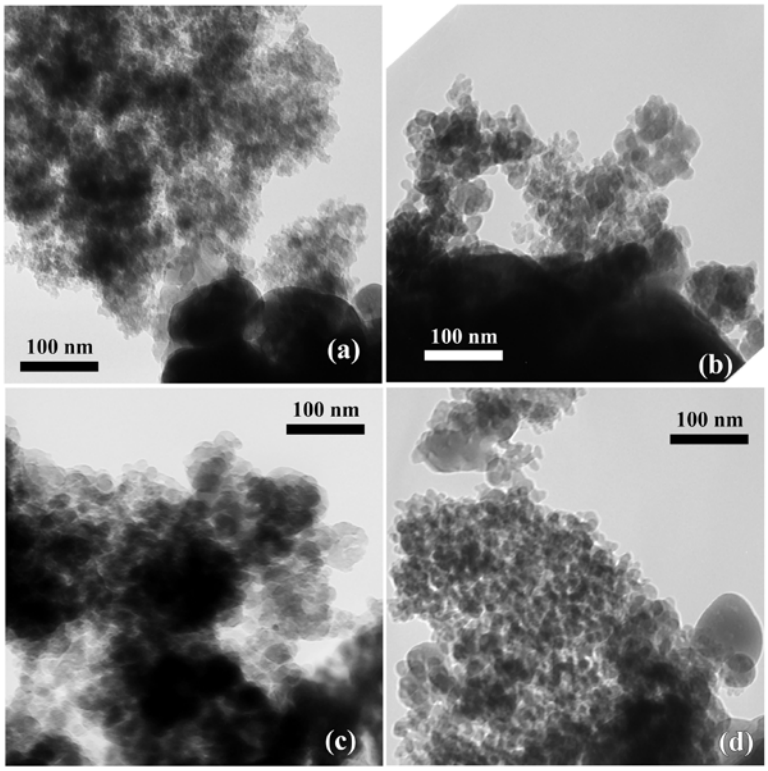

Fig. 1: TEM microphotographs of glass ceramic material before (a) and after ball milling treatment with different rpm: (b) 300 rpm, (c) 400 rpm and (d) 500 rpm. (The bar of 100nm stands for all images)

TEM microphotographs of pristine and milled materials show the coexistence of both nano-crystalline and amorphous phases (Fig.1).
The size of the nano-agglomerates decreases as the rpm are increased. Simultaneously, the amorphous proportion of the glass-ceramic material is increased. Finally, the bioactivity test revealed the formation of apatite after 5 days of immersion. Thus, a bioactive glass-ceramic nano-material was successfully synthesized and the milling process had no significant effect on the agglomerate size although a slight increase in the amorphous phase was observed.

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