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Understanding the early stages of crystallisation of $CaCO_3$ using advanced Electron Microscopy techniques.

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Calcium carbonate ($CaCO_3$) crystallisation occurs in a variety of processes such as bio-mineralisation in shellfish and production of personal health products, and construction materials. Equally important is the prevention of unwanted deposition of $CaCO_3$ in the form of mineral scales in the oil, and water industries causing major operational difficulties from clogging pipes to increasing the amount of energy needed to remove them. Despite numerous studies carried out on the mechanisms of crystallization, still little is known about this fundamental phenomenon because crystallisation occurs at the nano-scale and significant changes can occur over extremely short time which is very difficult to study experimentally. Understanding the early stages of $CaCO_3$ crystallisation not only allows the ability to control undesirable scale formation, but also enables us to mimic biocompatible structures for bio-applications such as bone replacement implants.

Hence, the purpose of this current study is to understand the early stages of crystallisation of $CaCO_3$. Both conventional scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques were used to investigate the presence of polymorphs including the amorphous state of $CaCO_3$ which were formed at different degrees of supersaturation.

The crystallisation of $CaCO_3$ was carried out in bulk solution at 25±1 ºC. Supersaturated solutions of calcium carbonate were initially prepared by mixing identical molar ratios and volumes of $CaCl_2$ and Na_2CO_3 leading to the final $CaCO_3$ concentrations of 0.5, 1.0, 2.5 and 4.5 mM. Also, the morphological properties of the particles as a function of changes in molar ratio of $CaCl_2/Na_2CO_3$ were investigated.

During crystallisation of calcium carbonate solutions (0.5, 1.0, 2.5 and 4.5 mM) containing equal molar ratios of $CaCl_2$ and Na_2CO_3, a variety of calcium carbonate particles including amorphous calcium carbonate (ACC) particles, hexagonal vaterite and rhombohedral calcite were observed, but no evidence was found for the presence of aragonite. However, a considerably different molar ratio of $CaCl_2$ and Na_2CO_3 was found to give rise to the formation of aragonite in the solution at 25º C, despite the fact that aragonite is an abundant polymorph at temperatures between 60°C to 80 °C.