Hydrothermal synthesis of chrysotile nanotubes and lizardite nanoflakes and investigation of their behaviour in microwave assisted carbonation

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It is widely known that serpentine minerals and other Mgbearing silicates (olivine, orthopyroxenes and clinopyroxenes) are targeted as potential naturally occurring phases for carbon capture and storage (CCS) by mineral carbonation. This reaction consists in the combination of CaO and MgO with CO_2 with spontaneous and exothermic formation of dolomite and magnesite. However, several kinetic barriers pose great limitations for the exploitation of this reaction.

One possible way to achieve a better carbonation reaction of serpentine is the innovative microwave (MW)-assisted process that can be employed to catalyze the reaction through thermal and non-thermal mechanisms. The aim of this work is to test the carbonation reaction on model samples of serpentine. In the first phase of the work, we synthesized serpentine nanocrystals in hydrothermal conditions with autoclave at 250 °C. Two stoichiometric phases of Mg-serpentine (chrysotile) and Niserpentine (nepouite) were produced and analyzed by powder X-ray diffraction (PXRD) and transmission electron microscopy. These analyses were exploited to deduce the crystallization dynamics and to understand the influence of the presence of Mg or Ni on the synthesis process and on the final morphology of the nanocrystals.

In a second phase of the work, MW-assisted carbonation tests were carried out in aqueous solution, between 70-200°C and 6-12 bar ($CO_2 + N_2$) with a SynthWave (Milestone) apparatus on samples of chrysotile and nepoute previously obtained and characterized.

The results show that MW-assisted carbonation in aqueous solution can be used to achieve the dissolution of SiO_2 layers and the reaction of the Mg(OH)₂ and Ni(OH)₂ layers with CO₂ to form hydroxy-carbonate phases. At 100°C, amorphized serpentine and probably amorphous silica are present in supernatants recovered after 1-h treatments. In experiments conducted at 200 °C with precipitates from the 100 °C tests, we achieved a minor dissolution of SiO₂ and PXRD analysis did not reveal the presence of other phases. These results can be attributed to the particular structure of chrysotile, in which the brucitic reactive layers are embedded between inert sialic layers. In order to enhance the effect of MW, longer treatments or pretreatments of starting materials with MW-active species may be necessary.