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Synthesis and rapid quantification of phase-pure vaterite for industrial waste valorisation and CO2 emission mitigation

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Calcium carbonate particles are widely applied due to their morphology, porosity, high surface area, and non-toxicity. Among synthesis methods, CO_2 mineralization is one of the most eco-friendly, as it mitigates emissions by fixing CO_2 from industrial flue gases into valuable products. The reuse of $Ca(OH)_2$-rich industrial waste, such as air pollution control residues, further lowers the environmental impact of the process. Calcium carbonate occurs in three crystalline forms; among them, vaterite is metastable but exhibits excellent binding properties, making it a promising clinker substitute in cements and mortars. Its formation is favoured by additives able to stabilize its crystal faces. Two additives were investigated: sodium tripolyphosphate (STPP) and polyacrylic acid (PAA). Their ability to steer precipitation towards vaterite at different temperatures was evaluated through batch carbonation reactions, within the framework of the PR FESR Program – CCS4CER Project (CUP: E87G22000550007). Attenuated Total Reflectance Infrared Spectroscopy (ATR-IR) was employed to quantify calcium carbonate polymorphs, enabling reliable quantification based on band intensity. An ATR-IR calibration curve was constructed using binary mixtures of phase-pure calcite and vaterite. The ratio between the vaterite peak area and the sum of calcite and vaterite peak areas was calculated and employed as a predictor for vaterite molar fraction. Carbonation reactions were carried out in an autoclave by loading 10 g of ceramic waste (72wt% $Ca(OH)_2$) with either STPP or PAA added at 5mol%, suspended in water to obtain a 10 wt% slurry. After nitrogen flushing (15 min), the reactor was heated (25–100 °C) and pressurized with 7 bar of CO_2 for 5 minutes. The calibration curve showed a linear correlation coefficient close to one, with good agreement between real and predicted vaterite content, including validation samples. Optimization of reaction conditions and additive selection enabled a maximum vaterite yield of 61% at 50 °C using STPP. When PAA was used, the maximum vaterite yield obtained was 32% at 25 °C. The accurate prediction of the ATR-IR method on the validation samples confirmed its reliability; it was then used to expedite the study on the real samples. The addition of additives during carbonation is crucial for obtaining significant amounts of vaterite. The obtained materials show promising features as secondary raw materials for the cement industry, with vaterite potentially replacing part of the costly and environmentally impactful clinker, increasing the sustainability of the cement while promoting the valorisation of materials commonly regarded as waste.