Chapter 2. Literature review

$CaCO_3$ is an exceptional mineral and amongst the most widely occurring natural minerals. Approximately 4% of the Earth's crust consists of $CaCO_3$ in the form of limestone, chalk or marble (Banth et al. 2008). $CaCO_3$ minerals are the building blocks for the skeletons of many marine organisms, for example the shells of molluscs; moreover, it is used in several industrial applications. The natural material is often used without further treatment, but for more demanding applications $CaCO_3$ is purified by precipitation to produce PCC. PCC is also known as synthetic, purified or refined calcium carbonate with the same chemical formula ($CaCO_3$) as the other types of calcium carbonate, such as limestone, marble and chalk.

Worldwide, carbonate rocks cover an area of about 22 million km^2 (Yuan, 1997) with over 90% of rock-forming carbonates consisting of calcite ($CaCO_3$) and dolomite ($CaMg(CO_3)_2$) (Reeder, 1983). The carbonate rocks in South Africa consist essentially of dolomite with only small occurrences of calcite. Figure 2.1 illustrates the distribution of the carbonate rock in SA (Doucet, 2011).

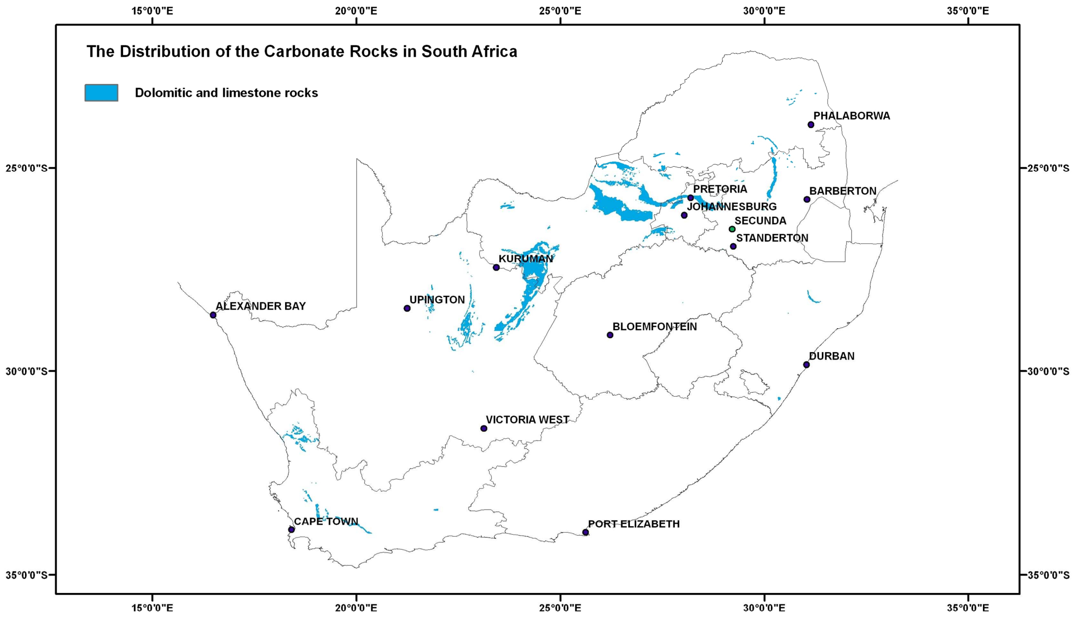
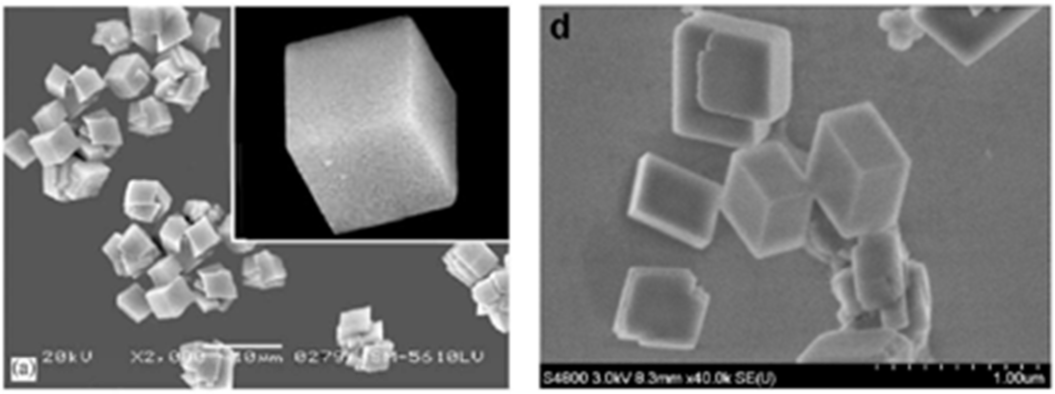


Figure 2.1 The distribution of carbonate rocks in South Africa. (Doucet 2011)

2.1 $CaCO_3$ polymorphs, morphology and physicochemical properties

$CaCO_3$ has several different schemes of atomic arrangements, called polymorphs (Latin for *"many shapes"*), meaning they all have the same chemical composition, but different crystal structures and symmetries. The polymorphs of $CaCO_3$ include three anhydrous (calcite, aragonite and vaterite) and two hydrated (monohydrocalcite, $CaCO_3.H_2O$ and ikaite, $CaCO_3.6H_2O$) crystalline phases, and amorphous calcium carbonate (ACC) (Oates 1998). Generally, the hydrated and amorphous forms are metastable and show the tendency towards rapid transformation into calcite, aragonite, or vaterite. The thermodynamic stability of the crystalline anhydrous polymorphs at ambient conditions decreases in the order of calcite, aragonite, and vaterite (Brecevic & Nielsen 1989), whilst their typical morphologies are classified as rhombohedral, needle-like and spherical, respectively (Shen et al. 2006).

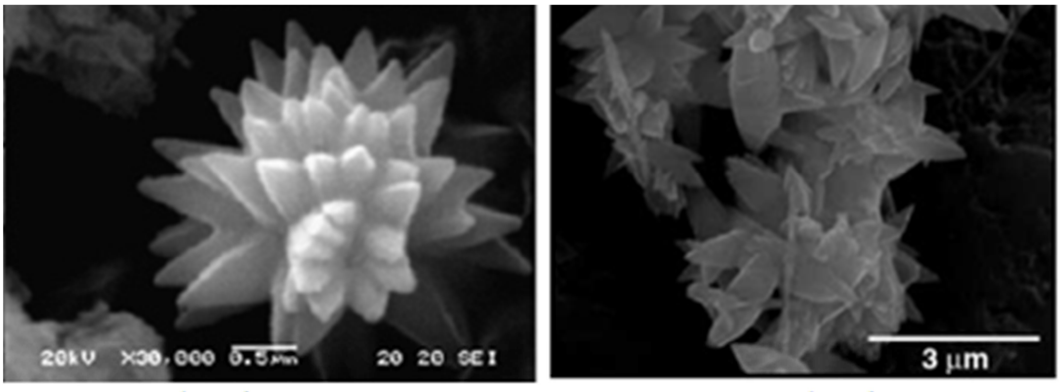
Calcite has trigonal crystal symmetry with a density of 2.71 g.cm^{-3} (Plummer & Busenberg 1982). Calcite is the thermodynamical equilibrium phase of $CaCO_3$ at ambient temperature and pressure (Tai & Chen 1998). The calcite crystal system appears in a range of morphologies with the most common being the rhombohedral, often shaped as cubes (Figure 2.2), and the scalenohedral (Figure 2.3) forms (Vecht & Ireland 2000).



Guo, H. (2006)

Ibrahim, A. (2012)

Figure 2.2 SEM images of rhombohedral calcite particles (Ibrahim et al. 2012; Guo et al. 2006)

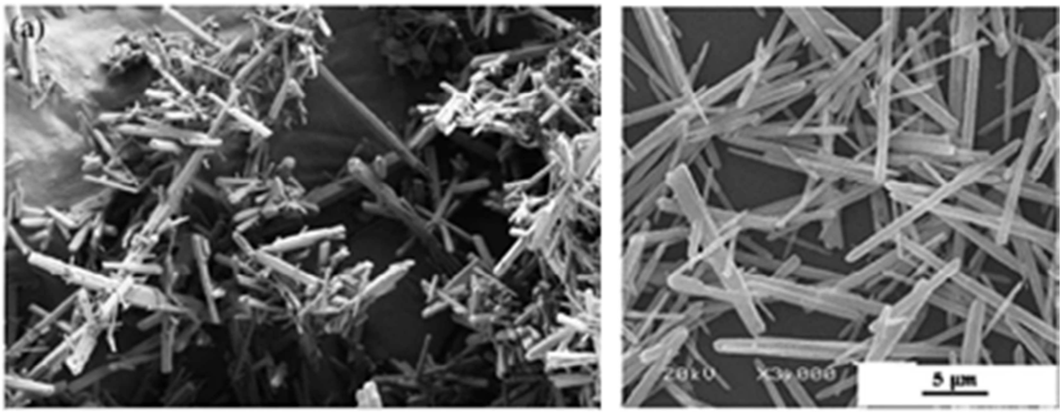


Kosma, V.A. (2013)

Garcia Carmona, J. (2003)

Figure 2.3 SEM images of scalenohedral calcite particles (Kosma & Beltsios 2013; García-Carmona et al. 2003)

Aragonite has the same chemical composition as calcite but a different structure, and more importantly, different symmetry and crystal shape. Aragonite belongs to the orthorhombic crystal system and has a density of 2.94 g.cm^{-3} (Plummer & Busenberg 1982). It usually forms needle-like orthorhombic crystals (Figure 2.4) and its formation is favoured at higher temperatures and pressures (Chen & Xiang 2009; Medeiros et al. 2006). Aragonite is metastable and converts slowly to calcite in aqueous solutions. Other morphologies of the aragonite polymorph, such as multi-layered (Wang et al. 2010), pseudo-hexagonal (Liu et al. 2010), lamellar-like (Wang et al. 2009), rod-like (Hu et al. 2009), and dendrite-like (Zhao et al. 2009) have also been synthesized under various experimental conditions.

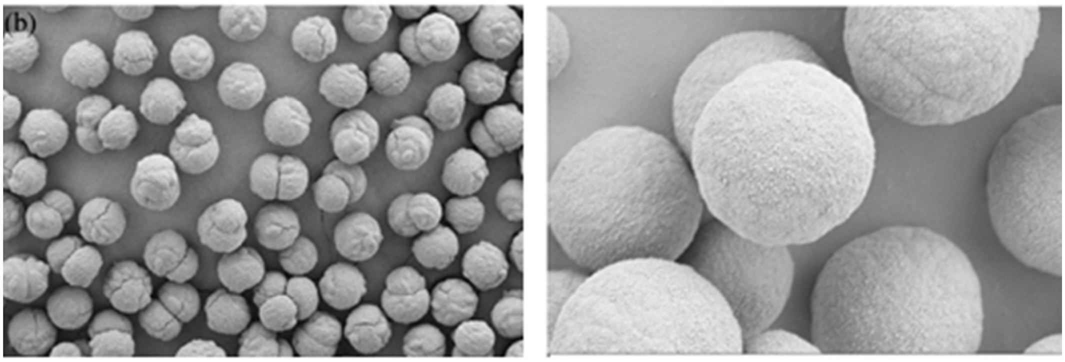


Beck, R. (2012)

(Xu, X. (2010)

Figure 2.4 SEM images of needle-shaped aragonite particles (Beck & Andreassen 2012; Xu et al. 2011)

Vaterite is thermodynamically the least stable of the three crystalline polymorphs. It belongs to the hexagonal crystal system and has a density of 2.54 g.cm^{-3} (Plummer & Busenberg 1982). It has the highest solubility of the three polymorphs and transforms by dissolution and re-crystallization into a more stable modification over time when in contact with water (Andreassen 2005). The hexagonal structure and the crystals typically form polycrystalline spherical particles (Figure 2.5) that are microns in diameter, often referred to as spherulites (Andreassen & Hounslow 2004). Although rare in nature due to its instability, vaterite can be artificially synthesized (Udrea et al. 2012; Beck & Andreassen 2010; Mori et al. 2009). Vaterite is expected to have potential applications for various purposes due to its special properties such as high solubility, high dispersion, high specific surface area and lower specific gravity compared with the other two crystalline forms, calcite and aragonite (Naka et al. 2002).



Beck, R. (2012)

Beck, R. (2010)

Figure 2.5 SEM images of spherical vaterite particles (Beck & Andreassen 2012; Beck & Andreassen 2010)

In recent years, synthetic vaterite of various morphologies have been produced using different additives (Leng et al. 2010; Aziz et al. 2011; Ahmed et al. 2009; Xu et al. 2006). For instance, sub-micron-sized vaterite tubes were formed through nanobubble-templated crystal growth (Rodriguez-Navarro et al. 2007; Fan & Wang 2005), whilst vaterite nanowires were obtained through the interaction of a polyelectrolyte with a self-assembled organic monolayer (Balz et al. 2005). Nanorods have also been synthesized by the micro-emulsion method using cetyl trimethylammonium bromide (CTAB) as cationic surfactant (Ahmed et al. 2009), and dagger-like vaterite particles by a fast microwave-assisted method (Qi & Zhu 2006). Unfortunately, none of these methods is suitable for the production of the above mentioned morphologies on large scale. In addition, the synthesized vaterite usually coexists with either one or both of the other two polymorphs, calcite and aragonite (Vogel et al. 2009). A major challenge remains, to synthesize a pure vaterite product in aqueous solution (Udrea et al. 2012).

2.2 Existing precipitated calcium carbonate production routes

The precipitation of $CaCO_3$ to produce PCC can be carried out via two synthetic routes, namely, the carbonation method (the $Ca(OH)_2$-H_2O-CO_2 reaction system), and the solution or double decomposition method (the Ca^{2+}-H_2O-CO_3^{2-} reaction system) (Feng et al. 2007). In the former, CO_2

gas is supplied as the source of carbonate ions, whereas in the latter, sources of both the calcium and carbonate ions are supplied in the form of soluble reagents (Kirboga & Oner 2013; Udrea et al. 2012; Zhang et al. 2008; Kitamura 2002; Uebo et al. 1992).

Extensive and continuous studies have indicated that, although the different $CaCO_3$ polymorphs (calcite, aragonite and vaterite) and different morphologies, i.e., rhombohedral or cubic, (Ibrahim et al. 2012; García Carmona et al. 2003; Uebo et al. 1992), spindle (Zhou et al. 2010), rod-like (Gantenbein et al. 2011), needle-like (Ma et al. 2011; Hu et al. 2009), chain-like (Carmona et al. 2004; García-Carmona et al. 2003) or some super structures (Fried & Mastai 2012; Menahem & Mastai 2008; Gehrke et al. 2005), can be obtained with and without the assistance of additives, the single calcite phase is generally formed in the $Ca(OH)_2$–H_2O–CO_2 system without additives.

However, most research has been directed toward the method of mixing solutions containing calcium and carbonate ions. Especially in the sodium carbonate and calcium chloride systems, the precipitation behaviour of the $CaCO_3$ polymorphs is well understood and experimentally researched. It is known that the crystallization behaviour of calcium carbonate polymorphs is influenced by various operational conditions (Kirboga & Oner 2013; Aziz et al. 2011; Martos et al. 2010; Zhou et al. 2010). In the Ca^{2+}-H_2O-CO_3^{2-} system, calcite and/or aragonite are usually obtained (Kawano et al. 2009) although the precipitation of vaterite has also been reported (Chen et al. 2010; Mori et al. 2009; Naka et al. 2002). The morphology of the precipitated calcite in the $Ca(OH)_2$-H_2O-CO_2 reaction system, at typical temperatures of the industrial process (between 30° and 70°C), is normally the scalenohedral form. The rhombohedral morphology is more often precipitated by using solution routes, but rarely by the $Ca(OH)_2$-H_2O-CO_2 industrial process (Ibrahim et al. 2012; García Carmona et al. 2003; Uebo et al. 1992).

PCC is widely used in industrial processes and consequently its existing production routes on large scale are well known. It is currently produced by three different processes, namely i) the caustic soda production process, ii) the calcium chloride process in the Ca^{2+}-H_2O-CO_3^{2-} reaction system, and iii) the conventional carbonation process in the $Ca(OH)_2$-H_2O-CO_2 reaction system (Mark et al. 1978).

In the caustic soda production process, calcium hydroxide is reacted with sodium carbonate to produce a sodium hydroxide solution from which the calcium carbonate is precipitated. This process is commonly used by alkali manufacturers and the paper industry for which sodium hydroxide recovery is the main objective and the coarse PCC produced is only a by-product (Mark et al. 1978). This $CaCO_3$ by-product from the paper industry is widely used as a source of alkali for the neutralization of acid mine water (Maree et al. 2004; Maree et al. 2004). Other uses may include the re-burn in rotary kilns to recover lime (CaO), its application as one of the raw materials of cement, or it may otherwise be disposed of as a waste when it cannot be sold or used.

In the calcium chloride process, calcium hydroxide is reacted with ammonium chloride, forming ammonia gas and a calcium chloride solution. The calcium chloride solution is reacted with sodium carbonate to form a calcium carbonate precipitate and a sodium chloride solution. This process is the simplest of the three but requires a low-cost source of calcium chloride to be economical (Mark et al. 1978).

However, the most prevalent route followed for industrial-scale PCC synthesis is through direct carbonation (Uebo et al. 1992), mainly due to the availability and low costs of the raw materials required. This procedure consists of reacting CO_2 gas with an aqueous calcium hydroxide suspension in a batch reactor (Kawano et al. 2009; Kemperl & Macek 2009; Uebo et al. 1992; Mark et al. 1978).

The conventional carbonation process for PCC production uses mined, crushed limestone as raw material. Crushed limestone is burnt in a lime kiln at about 1000°C where it decomposes ("calcines") into calcium oxide (CaO) and CO_2:

$$CaCO_3\,(s) + \text{heat} \rightarrow CaO\,(s) + CO_2\,(g) \qquad (2.1)$$

CaO is subsequently slaked (hydrated) with water at temperatures of 30–50°C to produce a calcium hydroxide ($Ca(OH)_2$) slurry.

$$CaO\,(s) + H_2O\,(l) \rightarrow Ca(OH)_2\,(aq) \qquad (2.2)$$

$CaCO_3$ is precipitated through a gas–liquid–solid carbonation route which consists of bubbling CO_2 through the aqueous slurry of $Ca(OH)_2$. The overall reaction of this three-phase system is:

$$Ca(OH)_2\,(aq) + CO_2\,(g) \rightarrow CaCO_3\,(s) + H_2O\,(l) \qquad (2.3)$$

However, the control of $CaCO_3$ polymorphism and morphology remain an important on-going research area because the application of $CaCO_3$ powder is determined by a great number of defined parameters, of which polymorphism and morphology are considered to be the most important (Jiang et al. 2012). Industrial PCC production aims at controlling the phase, morphology, and physical properties (i.e., particle size, aspect ratio, and specific surface area) by controlling the reaction temperature, CO_2 partial pressure, flow-rate of CO_2, lime slurry concentration, and agitator speed (Cizer et al. 2012; Ruiz-Agudo et al. 2011; Ukrainczyk et al. 2007; Domingo et al. 2006; Domingo et al. 2004; Carmona et al. 2004).

2.3 Industrial solid wastes as alternative feedstock for PCC production

Limestone rock is a natural resource and is the preferred raw material for the production of PCC on industrial scale. The replacement of natural limestone by waste-derived products as feedstock for PCC production would contribute towards a more sustainable use of a country's natural resource of limestone.

Modern society uses vast amounts of resources and produces equivalent amounts of waste that must be managed to avoid environmental problems. For instance, many industrial processes co-produce large quantities of currently unmarketable materials or industrial solid wastes of which the majority is discarded to dumps or landfills. Escalating costs and the enforcement of more stringent regulations worldwide highlight an increasing need for reduction and re-use of industrial wastes. The reduction and re-use of waste has therefore become more financially viable, particularly through the development and/or application of innovative solutions and emerging technologies. Mineral carbonation, on the other hand, is one of the carbon capture and storage (CCS) technologies that has received increasing attention because of its safe and essentially permanent chemical conversion of CO_2 into stable carbonate minerals (Pires et al. 2011; Zevenhoven et al. 2011; Prigiobbe et al. 2009; Lackner et al. 1995). Aqueous mineral carbonation involves the reaction of CO_2 with calcium and/or magnesium-bearing minerals in an aqueous suspension (Sipilä et al. 2008).

Industrial solid wastes that contain large mass fractions of calcium (Ca) are of great interest for the mineral carbonation process as it can combine the economic benefits of producing a valuable carbonate material (PCC) whilst improving the quality of the industrial residue for disposal or re-use as well as to reduce the overall quantity of CO_2 emitted (Bobicki et al. 2012; Kunzler et al. 2011; Zevenhoven et al. 2006). The aqueous carbonation of alkaline industrial solid wastes can be performed in two different ways, namely the direct and indirect mineral carbonation routes. Direct aqueous mineral carbonation involves the reaction of CO_2 with solid calcium-bearing minerals in an aqueous suspension (Sipilä et al. 2008). The indirect mineral carbonation process route involves two separate steps, in which the calcium is first selectively leached from the waste matrix by a leaching medium, followed by carbonation in a separate step for the production of PCC of high quality and purity. The industrial solid waste residues of interest, studied to date for the production of PCC include steelmaking slag (Eloneva et al. 2012; Mattila et al. 2012; Chang et al. 2011; Sun et al. 2011; Zhu & Yang 2011; Bao et al. 2010; Doucet 2010; Eloneva et al. 2009; Lu et al. 2009; Bonenfant et al. 2008; Lekakh et al. 2008; Teir et al. 2007; Huijgen et al. 2005), red mud (Johnston et al. 2010; Sahu et al. 2010; Yadav et al. 2010; Bonenfant et al. 2008), fly ash (Muriithi et al. 2011; Uliasz-Bocheńczyk & Pomykała 2011; Wang et al. 2010; Montes-Hernandez et al. 2009; Soong et al. 2006), oil shale ash (Uibu et al. 2010), flue gas desulphurization (FGD) and gypsum (Lee et al. 2012; Song et al. 2012).

2.4 PCC needs and applications

PCC has long been recognized as a versatile additive and its importance as filler is well known (Donate-Robles & Martín-Martínez 2011; Shen et al. 2010; Gao et al. 2009; Gorna et al. 2008; Shen et al. 2008; Gliese 2005; Lazzeri et al. 2005; Li et al. 2002). PCC is also a material of great interest due to its large range of other industrial applications: e.g. as polymer composite; filler for rubber and ceramics; additives for plastics, paper, ink, cosmetics, toothpastes and detergents; a component of pharmaceuticals and foodstuffs as well as numerous uses in electronics and catalysis.

The PCC used in industry as ordinary passive fillers or pigments is most often in the form of calcite due to its easy production routes. However, the different polymorphs of $CaCO_3$ (i.e. calcite, aragonite and vaterite) can have different functions as additives to act as functional active fillers. Owing to its larger specific surface and higher surface activity, vaterite can be used as filler for the improvement of mechanical properties of materials (Gao et al. 2009; Chen et al. 2006). Polyvinyl alcohol or polypropylene composites with aragonite filler showed improved tensile strength, impact strength, glass temperature and decomposition temperature (Yang et al. 2006; Mishra et al. 2005), while paper coating containing aragonite benefits from improved brightness, opacity, strength and printability (Antunes et al. 2008; Gliese 2005). Also, dispersion can be increased if cubic calcite crystals are added to paint formulations (Kontrec et al. 2008). Acicular or needle-like aragonite crystals have a reinforcing effect on rubber and plastics (Donate-Robles & Martín-Martínez 2011); and spherical vaterite particles have a significant impact on the brightness and transparency of ink (Kettle et al. 2010). Therefore, controlling the structure and morphology of $CaCO_3$ particles is an important subject for research and development.

2.5 Effect of process conditions on the physicochemical properties of $CaCO_3$

Polymorphism has tremendous technological importance due to the dependence of material properties like dissolution kinetics, solubility, hardness, density, optical properties and morphology on the solid-state structure. The control of polymorphism in crystalline $CaCO_3$ has been the focus area of research for many years but the ability to predict and control the polymorphism, the particle size and particle size distribution during crystallization remains one of the key challenges.

The control of polymorphism is a complex interplay between thermodynamic and kinetic factors. Traditional strategies for selection of polymorphs often involve changing parameters, such as initial supersaturation, mixing or stirring conditions, temperature, pH, solvents, and the use of organic or inorganic additives. In aqueous solutions, crystals of $CaCO_3$ can be synthesised as either cube-like or plate-like crystals of calcite, needle-like crystals of aragonite and spherical polycrystalline particles of vaterite, depending on the reaction conditions. Supersaturation (SS) is known to affect the nucleation and growth steps of $CaCO_3$. Higher supersaturation conditions (SS > 1) generally favour calcite formation (Schlomach et al. 2006; Kitamura 2002), whereas a low degree of supersaturation (SS << 1) appears to promote aragonite formation (Kirboga & Oner 2013). Hu & Deng (2003) also attributed the formation of aragonite crystals at higher temperatures (> 60°C) to a decrease in supersaturation conditions. The fundamental experimental conditions for obtaining rhombohedral calcite consist of relatively low-concentration solutions of both CO_3^{2-} and Ca^{2+} ions. These conditions, however, are not acceptable on industrial scale. Chakraborty et al. (1996) reported that the type of crystalline form and the particle size distribution are dependent on the supersaturation level and ionic ratio of $[Ca^{2+}]/[CO_3^{2-}]$ in solution. In terms of temperature, it is reported that calcite usually precipitates at lower temperatures, whereas aragonite often dominates at higher temperatures (Flaten et al. 2009).

The use of additives is often crucial for tailoring the crystal habit to produce particles with unusual morphologies (Beck & Andreassen 2012; Cizer et al. 2012; Rodriguez-Navarro et al. 2007; Han et al. 2005). In this regard, the different polymorphs of $CaCO_3$ have been synthesized in the presence of different agents, such as metal ions (Beck & Andreassen 2012; Rodriguez-Blanco et al. 2012; Ma et al. 2011; Hu et al. 2009; Hu et al. 2009; Xiang et al. 2006; Zhang & Dawe 2000; Wada et al. 1995), polyacrylamide (Wang et al. 2006; Yu et al. 2006), amino acids (Njegić-Džakula et al. 2010; Xie et al. 2005), organic acids (Ukrainczyk et al. 2013; Ukrainczyk et al. 2012; Zhou et al. 2011), surfactants (El-Sheikh et al. 2013; Chen & Nan 2011; Chen et al. 2010), polyvinyl alcohol (Malkaj et al. 2007), and polyethylene glycol (PEG) (Jiang et al. 2012; Konopacka-łyskawa & Lackowski 2011; Flaten et al. 2009), to effectively control the crystal growth of $CaCO_3$. The use of additives does not only influence the polymorphism of $CaCO_3$ (Škapin & Sondi 2010; Zhang et al. 2010; Kitamura 2009), but it can also alter the morphology of the polymorphs (Pouget et al. 2010; Zhang et al. 2010; Nan et al. 2008; Lei et al. 2006). For example in the case of the vaterite polymorph, not only spherical particles but also sponge-like, lens-like, cauliflower-shaped and raspberry-like shapes had been reported.

Several methods or different experimental techniques have also been developed or tested for controlling the growth of $CaCO_3$, including slow gas diffusion techniques (Xu et al. 2008; Xu et al. 2006), micro emulsion (Chen & Tai 2010; Tai & Chen 2008; Shen et al. 2007), microwave-assisted methods (Qi & Zhu 2006) and the effect of ultrasound (Boels et al. 2010; Sonawane et al. 2008). Gehrke et al. (2005) found that the formation of hexagonal, lens and rosette-shaped $CaCO_3$ superstructures can be obtained by the vapour diffusion method in the absence of any additives. Matsumoto et al. (2010) used the minute gas–liquid interface around CO_2/NH_3 micro bubbles as a reaction field to promote aragonite nucleation.

The control of operating variables and the use of additive or special techniques during the crystallization process are crucial for tailoring the crystal form (polymorphism) and shape (morphology), which are the most important parameters to the properties and performance of the PCC products if further processing is to be done.

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