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## Obtaining and Characterization of Polyolefin-Filled Calcium Carbonate Composites Modified with Stearic Acid

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# Obtaining and Characterization of Polyolefin-Filled Calcium Carbonate Composites Modified with Stearic Acid

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**Abstract.** In order to obtain high performance calcium carbonate-reinforced HDPE and PP composites, the dispersibility and compatibility of the inorganic phase in the polymer has been achieved through surface treatment of the amorphous calcium carbonate filler with stearic acid. The surface coating of the inorganic phase has been proved by XRD and FTIR spectroscopy, through forming of an intermediate layer of calcium stearate which acts as a surfactant, efficient in providing an optimum compatibility with the dominantly hydrophobic polymer matrix, as determined from the structural information obtained through samples cross-sections analysing.

## 1. Introduction

Calcium carbonate represents one of the most frequently used fillers for polyolefins, due to its low cost, good chemical stability under plastic processing conditions, fire retardancy, color neutrality and so forth [1,2]. It is a rather ubiquitous material, found in nature as ground mineral (marble, limestone, chalk) or in precipitated form (calcite, aragonite, amorphous calcium carbonate). The latter is usually the most frequently used, accounting for about 60% of all the carbonaceous filler types used in conjunction with thermoplastic matrices [3]. When incorporated into the polyolefin matrix, it can efficiently reduce the overall cost of the material, while providing a series of useful properties, such as high impact resistance, stiffness, good colorability, resistance to environmental stress-cracking and low mold shrinkage [4].

Calcium carbonate loadings, reported to the thermoplastic resin is usually in the range of 20 to 40% wt. range. The hardness impacted by this type of filler to polyethylene is reported in the 70-98 HB unit range. Calcium carbonate filled composites with polyolefin matrix are commonly used as appliances, electrical components, houseware, toys, furniture for indoor and outdoor use, automotive industry, and several utility products [5].

Recently, a lot of research has been conducted in the field of toughening of semicrystalline polymers with the addition of inorganic fillers. It has been reported that both crystalline as well as amorphous calcium carbonate is able to efficiently increase the stiffness of amorphous polypropylene, isotactic polypropylene, or high-density polyethylene due to stress concentration, debonding at the polymer/filler interface and the formation of shear bands, which lead to more efficient energy absorption during deformation [6]. The main condition to achieve an optimum performance for the polyolefin/calcium carbonate composites is to efficiently achieve a good dispersion of the calcium carbonate particles.



Due to the dissimilar nature of polyolefins and calcium carbonate in terms of polarity and surface energy, implicitly, a strong tendency of agglomeration for the inorganic phase has been reported. Many strategies have been employed to address this shortcoming, among which the most frequently employed are the particles surface treatment to reduce their surface energy and in some cases to increase the compatibility with the dominating hydrophobic matrix.

Among the surface functionalization agents,  $\gamma$ -methacryloxypropyl trimethoxy silane or trivinyl ethoxysilane, long alkyl chain organophosphorus coupling agents, non-ionic surfactants and so forth. These types of additives are often expensive, carcinogenic and tend to dissolve only in non-polar volatile solvents, such as toluene, aliphatic hydrocarbons [7, 8], etc.

One of the most economically-efficient functionalization agent is stearic acid (SA). Its loading on the calcium carbonate particles surface usually ranges from 1 to 14 wt.%, and the advantage is that it could be used also in aqueous media (at high temperatures) or in ethanol, a milder volatile solvent [8].

In this study, amorphous calcium carbonate powder has been surface treated with stearic acid by treatment of the particles with different amounts of stearic acid dissolved in ethanol at 60°C. The structure and properties of the obtained surface modified carbonate has been determined by SEM microscopy, XRD and FTIR spectroscopy. The functionalized calcium carbonate has been introduced and blended in high density polyethylene (HDPE) and polypropylene (PP) matrices through thermoforming at 150°C for HDPE and 190°C for PP. The composites cross-section has been examined by electron microscopy, evidencing a good wetting of the functionalized inorganic phase by the polymer.

## 2. Materials and methods

### 2.1. Materials

Amorphous calcium carbonate powder (50-90  $\mu\text{m}$  particle diameter) has been purchased from Sigma-Aldrich. Stearic acid has been purchased from the same company. Both filler and functionalizing agent were used as such.

High density polyethylene (melt flow rate of 3.15 g/10 min) and polypropylene (melt flow rate of 2.65 g/10 min) powder ( $\sim 0.5$  mm sieve) have been used for the forming of the thermoplastic matrix.

### 2.2. Methods

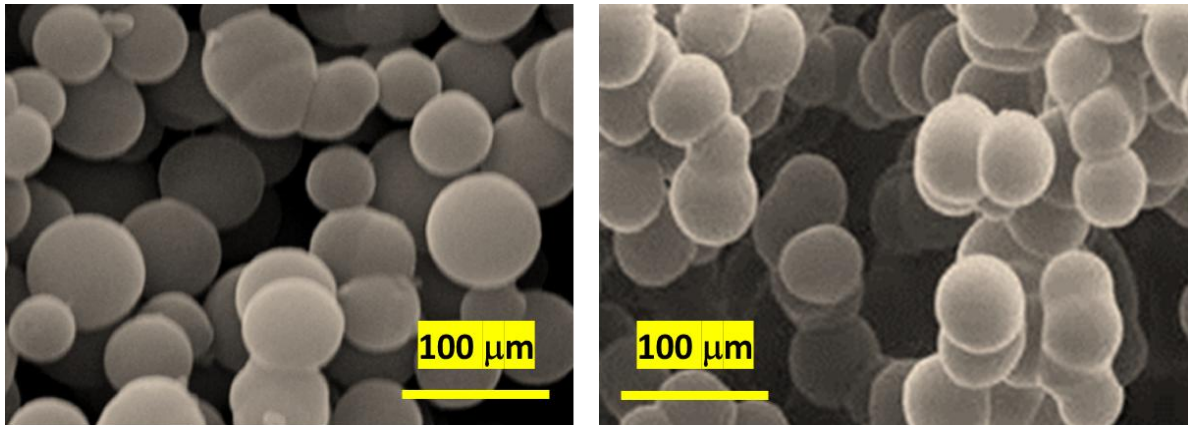
**2.2.1. Calcium carbonate powder SA surface treatment.** A concentration of 10% stearic acid in 98% ethanol has been prepared. A determined amount of the calcium carbonate powder (10g) has been immersed in 50 mL of the SA ethanol solution and magnetically stirred at 60°C under reflux for 1 hour. After the surface functionalization, the solution has been filtered, washed several times with distilled water and dried in a vacuum drying stove at 40°C and 20 mbar.

**2.2.2. Composites obtaining through thermoforming.** PP and HDPE powders were mixed with the surface treated stearic acid calcium carbonate (10% wt. reported to the polymer), placed in a stainless-steel matrix, and pressed at 100 atm for 30 s. After pressing, the metal matrix has been introduced in an oven at 150°C for HDPE and 190°C for PP for 1 hour, followed by cooling to room temperature. The nominal dimensions of the obtained composites are 100 mm x 10 mm x 4 mm (length x width x thickness).

**2.2.3. Characterization methods.** To evidence the surface functionalization of the amorphous calcium carbonate with SA, XRD spectra have been obtained, with a Bruker Discover diffractometer ( $K_{\alpha 1} = 0.154$  nm, scan speed 5 degrees/min). To characterize the surface chemistry of the calcite particles and the composites, FTIR spectra have been acquired in the 4000-600  $\text{cm}^{-1}$  interval (Bruker VERTEX 70 spectrophotometer working in reflectance mode). A QUANTA 200 scanning electron microscope was used to study the morphological appearance of the functionalized powder and composites cross-sections.

### 3. Results and discussion

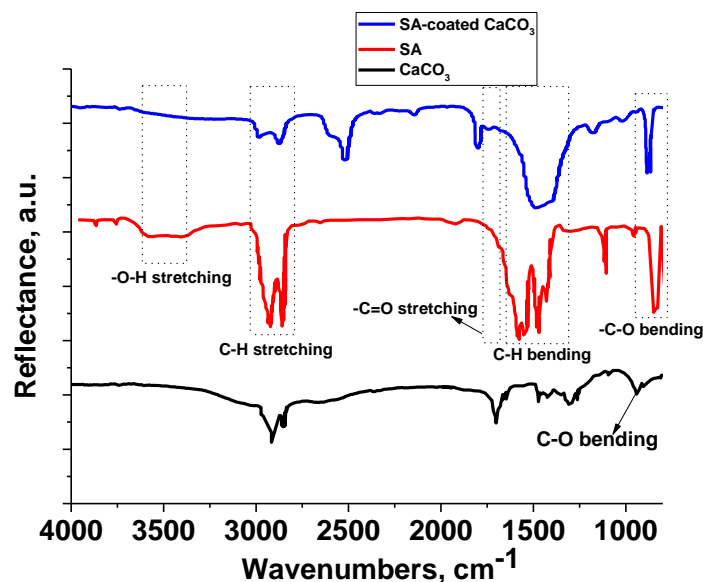
As it could be observed from figure 1, the amorphous calcium carbonate powder presents a spherical morphology, with a mean particle diameter of 70  $\mu\text{m}$ .



**Figure 1.** Initial  $\text{CaCO}_3$  powder (left) and SA-treated  $\text{CaCO}_3$  powder (right).

Stearic acid functionalization preserves the initial morphology of the carbonate particles. No significant modifications in mean diameter occurs, probably due to the adsorption of the organic molecule in a monolayer on the particles surface, as the reference literature suggests [8].

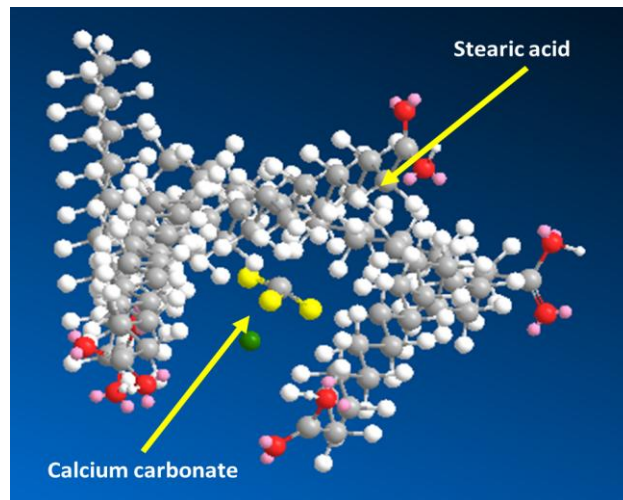
Stearic acid can efficiently coat the amorphous calcium carbonate powder, as determined from the infrared spectra of the functionalized and neat powders (figure 2). The coated calcium carbonate presents several structural features, common of both components, namely stearic acid, and calcium carbonate.



**Figure 2.** FTIR spectra of the neat and SA-coated calcium carbonate particles.

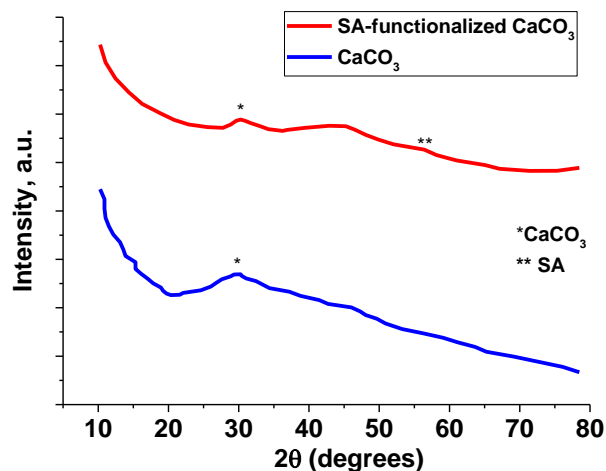
The band centred at 3400  $\text{cm}^{-1}$  could be ascribed to in plane -O-H stretching vibration from the carboxyl groups of the stearic acid or from the weakly adsorbed water on the surface of the carbonate particles [9]. Also, alkyl groups vibration (hydrocarbonate chain of stearic acid) could be noted on the

FTIR spectra of the functionalized particles, as well as C-O bending vibration in carbonates ( $946\text{ cm}^{-1}$ ) [10]. The mechanism responsible for carbonate particles functionalization employs in a first step, the monolayer adsorption of SA molecules on  $\text{CaCO}_3$  particles surface (figure 3), followed by their reaction with the carbonate, ultimately forming a layer of calcium stearate, which acts as a surfactant, improving the dispersibility of the inorganic phase in the selected polymer matrices and lowering their surface energy.



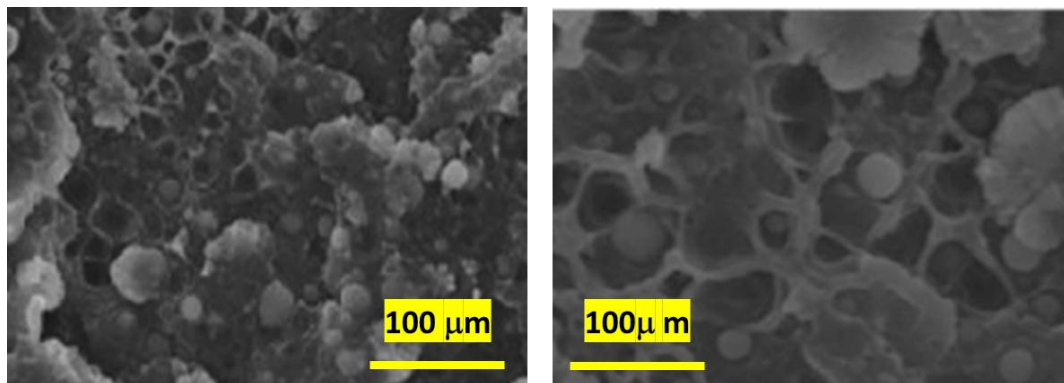
**Figure 3.** Mechanism of SA interaction with the calcium carbonate particles.

The particles functionalization has been also evidenced from the XRD diffractograms (figure 4), albeit, considering the amorphous character of the inorganic phase and the low crystallinity of SA, the diffraction peaks are broad and less characteristic.



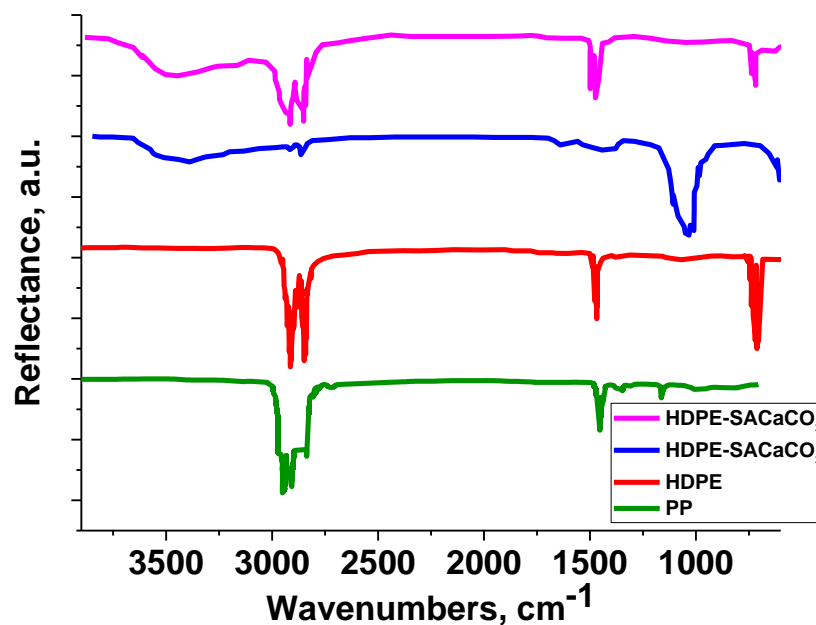
**Figure 4.** XRD diffractograms of neat calcium carbonate powder and SA-treated calcium carbonate powder.

The cross-sections of the composites obtained through functionalized carbonate particles inclusion in HDPE and respectively PP reveal a satisfactory dispersibility of the inorganic phase throughout the observed area (figure 5).



**Figure 5.** FTIR spectra of neat PP, HDPE and stearic acid coated calcium carbonate filled PP and HDPE.

Also, a good “wetting” of the inorganic phase by the thermoplastic matrix has been achieved, as determined from the continuity of the material in the interphase zone, determined by SEM micrographs image analysis.



**Figure 6.** FTIR spectra of neat PP, HDPE and stearic acid coated calcium carbonate filled PP and HDPE.

The FTIR spectra of neat polymers (HDPE and PP) reveal the presence of the usual absorption bands, such as methyl group  $-CH_3$  centered at  $\sim 2970\text{ cm}^{-1}$ , vibration of methylene group  $-CH_2-$  at  $\sim 2870\text{ cm}^{-1}$  (methylene rocking vibration  $\sim 1460\text{ cm}^{-1}$ , methyl out-of-plane vibration  $\sim 1370\text{ cm}^{-1}$ ). Supplementary from PP, HDPE presents a strong absorption at  $\sim 1483$  and  $1493\text{ cm}^{-1}$ , ascribed to collective bending vibration overtones of the alkyl groups. It could be seen that the obtained composites present several features common to stearic acid, such as the  $-OH$  groups stretching vibration [11].

#### 4. Conclusion

Amorphous calcium carbonate with spherical particle morphology has been coated by stearic acid to improve its dispersibility and compatibility with several hydrophobic thermoplastic polymer matrices, such as high-density polyethylene and polypropylene. An efficient hydrophobization of the powder has been achieved, determined from the scanning electron micrographs of the obtained hybrid organic/inorganic composites.

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