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Hollow spheres as nanocomposite fillers for aerospace and automotive composite materials applications

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A B S T R A C T

There were studied four types of powder filler materials for polyolefin composite parts production for automotive and aerospace industry. There was confirmed, that the particle shape has a strong effect on the acoustic and mechanical properties of the powder bed as influenced by the varying packing density. The calcium carbonate spherical hollow particles exhibited the best aerodynamic performance when aerated and were completely fluidised. Simultaneously they were exhibiting the easy flowing behaviour as reflected in the observed flowability of 4.71. In contrary to this, the flat lamellar geometry of the precipitated calcium carbonate resulted in the worst fluidisation behaviour, as the aeration energy was 2.5× higher in comparison to the spherical particles. Remaining samples under study, i.e. flash calcined kaolin and dolomite powder, exhibited cohesive rheological behaviour as reflected in the observed flowability. There was found a clear correlation between powder rheological and electrostatic charge data with the observed acoustic performance as reflected in the frequency dependence of the normal incident sound damping coefficient. This was demonstrated by a relatively high increase in the damping efficiency with increasing porosity of the powder bed as reflected in the decreasing packing density. However the best fit was found between the absolute value of the electrostatic charge values and the sound damping properties.

1. Introduction

At present there is an increased demand for the application of synthetic polymers in the automotive and aeronautic industries. It is mainly targeted for utilisation of poly(ethylene), poly(propylene), polycarbonate and polyamide components of the interior, exterior or other functional parts of vehicles [1,2]. Minerals first served as additives in polymer systems as a cost reducing technology [3] and due to the technological improvements in minerals processing and polymer chemistry, these materials are now used as functional additives [4] bringing specific mechanical and functional properties to the final composite products [5,6]. For example, hollow particle filled composites, called syntactic foams, are used in applications requiring high damage tolerance and low density, e.g. in aerospace and marine engineering structural applications. It was found that the presence of stiff hollow inclusions can enhance the composite elastic modulus in comparison to neat resin. Moreover, with the latter elastic modulus enhancement, an increase in energy absorption under compressive load was found due to the hollow fillers progressively crushing [6].

The recent review article by Wang et al. [7] described the present state of the art chemical synthesis routes and strategies for the synthesis of hollow micro/nano structured materials. The synthetic strategies were grouped into three major categories; hard templating, soft templating, and self-templating synthesis. Hollow

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spheres have a wide range of applications due to their regular uniform shape, meaning that they have the same properties, regardless of their orientation. Nano/micro scale hollow spheres have been produced through a variety of different methods [8–15] and have the benefit of being a colloidal particle that can flow, and remain untangled with other particles when put into a complex formulation [16]. Hollow latex spheres have been made commercially since the 1980's [17,18], with the primary production method being osmotic swelling [19]. Hollow latex spheres however have the disadvantage of being easily squashed or ruptured, so sturdier mineral structures are often preferred. This can be achieved by armour plating hollow latex spheres [20,21], or by creating hollow spheres directly from mineral structures, which has the benefit of fewer production steps.

Creating hollow spheres made of calcium carbonate has proved to be a success, with [22], and [23] making them using a carbon dioxide bubbles as a template, whilst [24,25] used an emulsification process. Consequently, hollow $CaCO_3$ spheres have proved to be a success, and the understanding of their properties, and their possible applications continue to increase.

Another route for the production of the perfectly formed dense agglomerates and the formation of deformed or hollow agglomerates is affected by particle consolidation during spray drying evaporation, particle rearrangement during the consolidation period and by the inter-particle potential. In general, the consolidation can proceed in the thermodynamic state, where the inter-particle potential is either repulsive or attractive. In the dispersed slurry, the long-range inter-particle potential allows the particles to repel one another. When the particles are repulsive, the meniscus that separates the two fluids exerts a capillary pressure on the particles at the surface, forcing them together as a dense agglomerates. In the case of the mechanism, where particles were not allowed to rearrange after a short period of evaporation, the agglomerate becomes hollow and deformed. Whereas in the case of the attractive particle network, if the capillary pressure exceeds the yield stress, the spherical agglomerates with uniform density are formed [26].

As a composite system matrix, polyolefin polymers are widely used, such as poly(ethylene) or poly(propylene), with different grades, types and qualities [3]. There was found to be an increase in tested polyolefin melts viscosity, a decrease of the elasticity with increasing filler loading and the presence of yield values in the flow curves depending on the filler particles volume loading and particles size [27]. When comparing the acoustic properties of the composite materials, the process of the interaction of the mechanical acoustic wave and the material structure is based on the assumption that an incoming wave is reflected at the boundary between two acoustically different materials due to the differences in the acoustic impedance of the involved materials [28–31].

This paper is focused on the application of powder rheology, acoustic performance testing and electrostatic charge measurements on the evaluation of hollow spheres and lamellar mineral powder materials as prospective fillers for polyolefin composite materials applications.

2. Theoretical background

Sound is an acoustic wave with frequencies ranging from 10 Hz to 16 kHz, with sonic waves spreading in all directions from the source. On the basis of different points of view on the problem of noise attenuation it is possible to distinguish the following methods of sound and vibration damping [32]:

- Reduction method – attenuation at the noise source, e.g. during the machinery construction stage.
- Sound isolation method – covering the sound source by material with high airborne sound insulation characteristics.
- Sound absorption method – endeavour to minimise sound reflections e.g. to absorb the maximum of the incident acoustic energy.

In the matrix of the sound/vibration attenuating material, dissipation of the sonic wave to mechanical energy and heat takes place. This proceeds by the combination of the following processes:

- By friction of the vibrating air particles on the walls during their penetration into the pores of the sound absorbing material. This lowers the kinetic energy of the incident sound field. Effectiveness of this process increases with growing porosity of the absorption material.
- By decreasing the potential energy of the sonic wave penetrating into the material. This lowers the acoustic pressure due to the heat exchange between air and the skeleton of the absorbing material during periodic pressure changes.
- By non-elastic deformation of the absorbing material body. At the specifically aimed construction of the vibration or noise–isolation material it is therefore possible to utilise all of the above mentioned processes for their synergistic effect in obtaining maximum effectiveness of attenuation. This is possible by modelling the geometry of the damping material body as well as by proper selection of the main material matrix and adhesive system.

2.1. Sound absorption measurements

Sound absorption properties express a material's ability to absorb incident acoustic energy and is described by the sound absorption coefficient (α) which is defined by the ratio of dissipated power in a tested material and incident power. Sound absorption of a given material depends on many factors, including; excitation frequency, thickness, structure, temperature, density and humidity [28,32]. The effect of the excitation frequency on the sound absorption coefficient is expressed by the noise reduction coefficient (NRC), which is defined as the arithmetic mean of the sound absorption coefficients of a given material at the excitation frequencies of 250, 500, 1000 and 2000 Hz [33]. On the basis of the primary absorption peak frequency (f_{p1}), it is possible to determine the speed of sound (c) of an elastic wave through a powder bed and the longitudinal elastic coefficient (K) of the power bed as follows, where h is the height of a given powder bed and ρ_b is the bulk density of the powder bed [34].:

$$\boldsymbol{c} = 4\boldsymbol{f}_{\boldsymbol{p}1}\boldsymbol{h} \tag{1}$$

$$\boldsymbol{K} = \boldsymbol{c}^2\boldsymbol{\rho_b} = 16\boldsymbol{f}^2_{\boldsymbol{p}1}\boldsymbol{h}^2\boldsymbol{\rho_b} \tag{2}$$

3. Materials

The materials studied were commercially available mineral filler powders and are described in Table 1. Four samples were analysed; formulated calcium carbonate spheres (process route developed at The University of Birmingham, UK), flash calcined kaolin, dolomite and calcined kaolin. Samples 1 was the hollow calcium carbonate spheres, sample 2 was flash calcined kaolin based filler (Imerys, UK), sample 3 was dolomite powder (CaO (30.3 wt%), MgO (21.6 wt %), Fe_2O_3 (0.08 wt%)) (Omya, Switzerland) and sample 4 was a