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# Investigation of advanced mica powder nanocomposite filler materials: Surface energy analysis, powder rheology and sound absorption performance

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## ABSTRACT

Two types of nano/micro sized mica powders for polymer composites, muscovite and phlogopite, were tested for their sound absorption capabilities. Acoustical performance was correlated to surface energy analysis and powder rheology testing. Inverse gas chromatography (iGC) was used to determine the surface energy, with the dominant component being the dispersive component. This reflected the non-polar, hydrophobic, character of the micas. The determined yield locus and Mohr's circles indicated that the material with the highest packing density exhibited more free flowing powder characteristics, compared with the lower packing density materials, which exhibited a greater cohesive powder flow behaviour. All tested mica powders were sensitive to aeration and become fluidised. Based on the acoustical measurements the worst sound absorption performance was found for the highest packing density material exhibiting the highest magnitude of the longitudinal elastic coefficient.

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# 1. Introduction

There is a rapidly increasing application of nano particles in most industrial sectors across the globe. Their use is significant benefits to many products, and a large number of research and industrial organisations are attempting to create "nano material" products. The manufacture of large quantities of nano materials, especially in the composite materials market has become a reality and wide range of commercial products can be provided for the global market. Similarly, the surface coatings and paint industries have also started to use available nano materials. Investigations have shown that the size of nano-particle fillers is likely to be the

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reason behind the performance increase when compared to micrometer sized particles [1,2].

Paint and low temperature coating technologies have moved from a basic chemical process to one where it is now a combination of metallurgy, chemistry and physics, with chemistry at the molecular level approach being now of greatest interest. Typically, colloidal slurry coatings are manufactured for a wide variety of applications, and are used for hard clear coats for supersonic plastic wind screens, intumescent paints to protect composites, metals and parts such as fuel pumps from fire and erosion coatings for leading edges of wings on sub sonic aircraft. These coatings are also used on: wind turbines, ensuring the longevity of the aerofoil shape; erosion resistant coatings for the leading edges of wings on supersonic aircraft and missiles; erosion coatings on both land based and aeronautical gas turbine parts; in the steel industry for extreme environment protection in the moulds of steel continuous casters: and coatings to down-hole directional drilling mud rotors in the oil industry. High temperature oxidation slurry coatings such







as those based on alumina and aluminides are used as overlay barrier coatings in high temperature oxidation applications, dry film lubricants for a wide range of applications, chemical resistant coatings, semiconducting coatings and others. In the last few years, silicate minerals were used in many composite materials technical applications [3]. Incorporating inorganic fillers into thermoplastic/ thermosetting polymer networks result in improved physicochemical, mechanical, and electrical insulation characteristics. For this reason the research and development activities focused on composites containing inorganic filler is of increasing importance. Composite materials matrix modification, its degree of crystallinity, type of reinforcement, quality of adhesion between filler and matrix, filler particles size, size distribution and shape [4,5] as well as the addition of coupling agents enhancing filler/matrix adhesion affects both, the physico-chemical and thermo-mechanical properties, as well as the internal structure and strength of final composites.

The mica group of sheet silicate (phyllosilicate) minerals includes several closely related materials with close to perfect basal cleavage. All are monoclinic, with a tendency towards pseudohexagonal crystals, and are similar in chemical composition. The nearly perfect cleavage, which is the most prominent characteristic of mica, is explained by the hexagonal sheet-like arrangement of its atoms. Chemically, micas can be given the general formula  $X_2Y_{4-6}Z_8O_{20}(OH,F)_4$  in which X is K, Na, or Ca or less commonly Ba, Rb, or Cs; Y is Al, Mg, or Fe or less commonly Mn, Cr, Ti, Li, etc.; Z is chiefly Si or Al, but also may include Fe<sup>3+</sup> or Ti. As mentioned above, the micas have laver structures in which silicate sheets are combined with aluminate units: the aluminum ions can be octahedrally as well as tetrahedrally coordinated. For example, the mica muscovite KAl<sub>2</sub>(OH)<sub>2</sub>(Si<sub>3</sub>AlO<sub>10</sub>) contains both octahedral and tetrahedral Al<sup>3+</sup> cations. The potassium ions are located between the flat aluminosilicate sheets. Phlogopite, KMg<sub>3</sub>(OH)<sub>2</sub>(Si<sub>3</sub>AlO<sub>10</sub>), has a similar structure but with Mg<sup>2+</sup> in octahedral environments instead of Al<sup>3+</sup>. The structure and microtopography of mineral filler surfaces are very important because they directly affect surface chemical reaction rates and mechanisms. In general, the surface structure of a mineral differs from the internal structure, however the structure of mineral surfaces may relax due to slight displacements of surface atoms in response to the force asymmetries at the surface or may reconstruct to a surface unit cell of different size and symmetry from that of the equivalent bulk plane [6]. The observed degree of surface relaxation, a consequence of rotation and tilting of SiO<sub>4</sub> tetrahedral after cleaving, is larger in muscovite than in phlogopite. A more tetrahedral rotation after removal of the interlayer K is observed in muscovite. This is due to the greater limitation in the amount of tetrahedral rotation in the internal structure by the interlayer K in muscovite than in phlogopite [6].

This paper focuses on the application of powder rheology, surface energy analysis and acoustical performance testing to evaluate mica powder materials as effective fillers for composite materials applications.

## 2. Theoretical background

#### 2.1. Surface energy analysis

The surface free energy of a solid can be described as the sum of the dispersive and specific contributions. Dispersive (apolar) interactions, also known as Lifshitz-van der Waals interactions, consist of London interactions which originate from electron density changes but may include both Keesom and Debye interactions [7–9]. The dispersive component of the surface energy ( $\gamma_S^D$ ) can be calculated from the retention time of a series of n-alkane probes injected at infinite dilution as measured using inverse gas

chromatography (the probe concentration falls within the Henry's Law portion of the adsorption isotherm) [8,10,11]. There are two methods of determining the surface energy from inverse gas chromatography: the first one is according to Schultz et al. [12] (Equation (1)); and the second one according to Dorris and Gray [13] (Equation (2)):

$$RT \ln V_N = a \left(\gamma_L^D\right)^{1/2} 2N_A \left(\gamma_S^D\right)^{1/2} + C \tag{1}$$

where *R* is the universal gas constant,  $N_A$  is Avogadro's number,  $\gamma_L^D$  is the dispersive component of surface free energy of the liquid probe,  $\gamma_S^D$  is the dispersive component of the surface free energy of the solid and *C* is a constant, and

$$\gamma_{S}^{D} = \frac{\left[RT \ln\left(V_{N(C_{n+1}H_{2n+4})}\right) / V_{N(C_{n}H_{2n+2})}\right]}{4N_{A}^{2}a_{CH_{2}}^{2}\gamma_{CH_{2}}}$$
(2)

where  $a_{CH_2}$  is the surface area of CH<sub>2</sub> (~0.06 nm<sup>2</sup>) and  $\gamma_{CH_2}$  is the free energy of CH<sub>2</sub> (approximately 35.6 mJ/m<sup>2</sup>).

### 2.2. Sound absorption measurements

The ability of a material to absorb incident sound is described by the sound absorption coefficient  $\alpha$ , which is given by the ratio [14]:

$$\alpha = \frac{P_d}{P_i} \tag{3}$$

where  $P_d$  is the dissipated power in the tested material, and  $P_i$  is the incident power. Sound absorption is influenced by many factors, including: excitation frequency; material thickness; porosity; density; and design [15,16]. Frequency dependencies of the sound absorption coefficient of materials can be determined by the transfer function method ISO 10534-2 [17–19]. The complex acoustic transfer function  $H_{12}$  is expressed by the formula:

$$H_{12} = \frac{p_2}{p_1} = \frac{e^{k_0 \cdot x_2 i} + r \cdot e^{-k_0 \cdot x_2 i}}{e^{k_0 \cdot x_1 i} + r \cdot e^{-k_0 \cdot x_1 i}}$$
(4)

where  $p_1$  and  $p_2$  are the complex acoustic pressures at two microphone positions,  $k_0$  is the complex wave number,  $x_1$  and  $x_2$  are the distances between two microphones and the material sample, r is the normal incidence reflection factor [19], expressed by the following equation:

$$r = r_r + ir_i = \frac{H_{12} - H_I}{H_R - H_{12}} \cdot e^{2k_0 \cdot x_1 i}$$
(5)

where  $r_r$  and  $r_i$  are the real and imaginary parts of the normal incidence reflection factor,  $H_l$  is the transfer function for the incident wave, and  $H_R$  is the transfer function for the reflection wave. The transfer functions  $H_l$  and  $H_R$  are defined as follows:

$$H_{I} = e^{-k_{0} \cdot (x_{1} - x_{2})i} \tag{6}$$

$$H_R = e^{k_0 \cdot (x_1 - x_2)i}$$
(7)

The sound absorption coefficient  $\alpha$  is then defined by Equation (8):

$$\alpha = 1 - |r|^2 = 1 - r_r^2 - r_i^2 \tag{8}$$

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