



A novel colored talc filler: Preparation and surface property determination using two distinct methods



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ABSTRACT

A better understanding of filler's surface properties is critical for determining the most effective polymer reinforcement fillers. In this work, a colored talc filler (CTF) was prepared and its surface properties were comparatively studied to those of talc filler (TF), using inverse gas chromatography (IGC) and contact angle methods—Owens–Wendt–Kaelble (OWK), van Oss–Chaudhury–Good (vOCG) and Wu. The results indicated that the dispersive component (γ_s^D) for both samples contributed a major part (68–81%) of the total surface energy (γ_s^T), and displayed a decreasing trend with increasing surface coverage, meaning the fairly energetic surface heterogeneity for them. The γ_s^D values determined by the contact angle methods were consistent, although lower than those using IGC analysis. The specific component (γ_s^{SP}) calculated by the contact angle methods were also consistent and lower than those calculated using IGC. The specific Gibbs free energies of adsorption (ΔG^{SP}) changed with surface coverages, further confirming the heterogeneous nature of both samples. The significantly lower γ_s^T value for CTF could reduce filler particle–particle interactions, allowing a better dispersion in matrix, and thus lead to an improvement in mechanical performance of CTF/polymer composite.

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1. Introduction

Talc is a plate-like layered structure magnesium silicate mineral, in which the octahedral brucite layer is sandwiched between two tetrahedral silica sheets [1,2]. Many reports have indicated an improvement in performance after a polymer matrix was reinforced with talc filler [3–7]. However, it is well known that, the filler nature of talc influences its reinforcement ability depending on the surface activity, particle size, specific surface area and surface functional groups [8,9]. Among these properties, surface activity affects the reinforcement ability the most, as the chemical nature of a particle's surface determines filler–filler and filler–matrix interactions. These interactions in turn affect the filler's dispersion in matrix and thus the final performance of composite [10]. Therefore, a better understanding of a filler's surface properties is critical for determining the most effective polymer reinforcement fillers.

Contact angle measurement is one of the most commonly used techniques for the solid surface characterization. The surface free energy of a solid can be determined by measuring the contact angles of different test liquids (the amount needed depending on the calculation methods) on its surface according to the Young's Equation [11]. There

are numerous methods, such as the Zisman plot [12–14], Fowkes [15], Owens–Wendt–Kaelble (OWK) [16,17], van Oss–Chaudhury–Good (vOCG) [18] and Wu [19] for calculating the surface energy from equilibrium liquid contact angles. However, this method usually involves difficult and time-consuming experiments, as factors such as surface roughness, chemical heterogeneity and bulk penetration of the wetting liquid could result in contact angle hysteresis [20,21]. As an alternative, inverse gas chromatography (IGC) has proven to be a reliable technique for surface characterization, offering advantages of independence from sample morphology and accurate measurement over a wide range of temperatures. However, to the best of our knowledge, there are sparse reports on the surface characterization of talc-derived fillers. Thus, in this work, a colored talc filler (CTF) was prepared and its surface properties were comparatively studied with those of talc filler (TF), using IGC and contact angle measurement—OWK, vOCG and Wu. It aims at probing the CTF as a candidate filler for the masterbatch industry.

2. Basic theories

2.1. Contact angle methods

The key equation used to determine the solid surface energy by contact angle measurement is the Young's equation [22], which was

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Notation

OWK	Owens–Wendt–Kaelble
vOCC	van Oss–Chaudhury–Good
IGC	inverse gas chromatography
TF and CTF	talc filler and colored talc filler
γ_S , γ_L and γ_{SL}	surface free energies of solid, liquid and solid–liquid (mJ/m ²), respectively
θ	contact angle (°)
W_A , W_C	work of adhesion (mJ/m ²) and cohesion (mJ/m ²)
γ_S^{LW} , γ_S^{AB}	Lifshitz–van der Waals and Lewis acid–base component of surface free energy (mJ/m ²)
γ_S^+ , γ_S^-	Lewis acid and Lewis base parameter of surface free energy (mJ/m ²)
γ_S^D	dispersive component of surface free energy (mJ/m ²)
γ_S^{SP}	specific component of surface free energy (mJ/m ²)
γ_S^L	total surface free energy (mJ/m ²)
γ_{50}^D and γ_{50}^{SP}	mean value of dispersive and specific component (mJ/m ²)
ΔG_{sp}	specific Gibbs free energies of adsorption (kJ/mol)
iGC-SEA	iGC surface energy analyzer

derived from the equilibrium condition of forces representing surface tensions at the contact point of three phases: solid, liquid and gas.

$$\gamma_L \cos\theta = \gamma_S - \gamma_{SL} \quad (1)$$

where γ_S , γ_L and γ_{SL} are the surface free energies of solid, liquid and solid–liquid (mJ/m²), respectively. θ is the contact angle between the solid surface and test liquid (°).

In the Young's equation, both θ and γ_L are measurable. In order to obtain γ_S and γ_{SL} by solving this equation, an additional relationship between these quantities has to be made. An understanding of the different methods requires an explanation of work of adhesion (W_A). The equation for W_A can be written as:

$$W_A = \gamma_A + \gamma_B - \gamma_{AB} \quad (2)$$

where γ_A and γ_B represent the surface tensions of phases A and B; and γ_{AB} represents the interfacial tension between the two phases. For the solid–liquid system, the equation can be written as:

$$W_A = \gamma_S + \gamma_L - \gamma_{SL} \quad (3)$$

Combining it with the Young's equation yields:

$$W_A = \gamma_L(1 + \cos\theta). \quad (4)$$

In a similar way, the work of cohesion (W_C) of one substance (e.g., A) can be defined as:

$$W_C = \gamma_A + \gamma_A - 0 = 2\gamma_A. \quad (5)$$

Berthelot [23] stabilized the direction to surface energy calculations and assumed that W_A between the solid and liquid equals the geometric mean of individual cohesion work.

$$W_A = \sqrt{W_{SS}W_{LL}}. \quad (6)$$

Combining it with Eqs. (4) and (5) yields:

$$W_A = \sqrt{W_{SS}W_{LL}} = 2\sqrt{\gamma_S\gamma_L} = \gamma_L(1 + \cos\theta). \quad (7)$$

2.1.1. OWK method

Owen and Wendt [17] extended Fowkes' idea [24,25] and proposed the following form for W_A :

$$W_A = 2\sqrt{\gamma_S^D\gamma_L^D} + 2\sqrt{\gamma_S^{SP}\gamma_L^{SP}}. \quad (8)$$

Combining it with the Young's equation leads to Eq. (9):

$$\gamma_L(1 + \cos\theta) = 2\sqrt{\gamma_S^D\gamma_L^D} + 2\sqrt{\gamma_S^{SP}\gamma_L^{SP}} \quad (9)$$

There are two unknowns γ_S^D and γ_S^{SP} in Eq. (9), thus at least two liquids with known dispersive and specific components are needed to solve it. In this work, the surface energy parameters for TF and CTF were determined using formamide and diodomethane as the test liquids.

2.1.2. vOCC method.

Van Oss et al. [26–28] followed Fowkes' theory and treated surface energy as a sum of Lifshitz–van der Waals component (γ_S^{LW}) and acid–base component (γ_S^{AB}). The interfacial tension was postulated for solid–liquid systems as:

$$\gamma_{SL} = \gamma_S + \gamma_L - 2\left[\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + \sqrt{\gamma_S^+\gamma_L^-} + \sqrt{\gamma_S^-\gamma_L^+}\right]. \quad (10)$$

Combining it with the Young's equation, we can obtain:

$$\gamma_L(1 + \cos\theta) = 2\sqrt{\gamma_S^{LW}\gamma_L^{LW}} + 2\sqrt{\gamma_S^+\gamma_L^-} + 2\sqrt{\gamma_S^-\gamma_L^+}. \quad (11)$$

Since there are three unknowns γ_S^{LW} , γ_S^+ and γ_S^- in Eq. (11), at least three test liquids are needed to solve it. In this study, the distilled water, formamide and diodomethane were used.

2.1.3. Wu method

In addition to the OWK method, the other two liquid method for considering the harmonic mean relationship was proposed by Wu [19, 29]. He discerned between dispersive and specific components of the surface energy, but instead of using the geometric mean as in Eq. (9), he uses a harmonic mean in the expression for γ_{SL} .

$$\gamma_{SL} = \gamma_S + \gamma_L - 4\left[\frac{\gamma_S^D\gamma_L^D}{\gamma_S^D + \gamma_L^D} + \frac{\gamma_S^{SP}\gamma_L^{SP}}{\gamma_S^{SP} + \gamma_L^{SP}}\right]. \quad (12)$$

In combination with the Young's equation, Wu's equation can be written as:

$$\gamma_L(1 + \cos\theta) = \frac{4\gamma_S^D\gamma_L^D}{\gamma_S^D + \gamma_L^D} + \frac{4\gamma_S^{SP}\gamma_L^{SP}}{\gamma_S^{SP} + \gamma_L^{SP}}. \quad (13)$$

As in the OWK method, Wu's method also requires the use of at least two liquids; thus the formamide and diodomethane were applied.

2.2. IGC method**2.2.1. Surface energy**

The basic theory of IGC is presented here and more details can be found elsewhere [30,31]. In IGC, the term “inverse” indicates that the solids to be examined are packed into the column and this material is probed by gas mixtures injected into the column. The injection of known molecule probes, including both nonpolar and polar probes, enables us to characterize the surface properties of the packed materials. Stationary-phase characterization can be achieved by partitioning the sample between the mobile phase and the stationary phase, indicated by the time taken to elute the samples. In IGC analysis, the surface energy has traditionally been determined at infinite dilution (ID-IGC), where

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