# Polypropylene composite material and its rheological and mechanical properties depending on the size of the filler $CaCO_3$

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# Polypropylene composite material and its rheological and mechanical properties depending on the size of the filler $CaCO_3$

**E A Bilalova[1,2], E V Prut[1], O P Kuznetsova[1]**

[1]Department of Chemical Physics,ICP RAS, Moscow, Russia;
[2]MIPT, Dolgoprudny, Russia

E-mail: bilalova@phystech.edu

**Abstract.** The main idea of this paper is to investigate the influence of the filler sizes on the important composite material's properties, such as viscosity, MFR, shear stress, elongation at break and Youngs modulus. Ten compositions of PP/$CaCO_3$ composites were prepared in a two-rotor mixer (Brabender) with calcium carbonate content o 5 - 25 wt. (%). Laser diffraction method was used to analyze sizes of the powder filler, considering it to be approximately spherical. Found out, that with an increase in the concentration of powder filler increases the value of Youngs modulus (E, MPa) and also there is a significant decrease in the elongation at break ($\epsilon$, %), and the strength also decreases, but rather weakly. Viscosity showed different trends for both sizes at the dependence of concentration with constant shear stress.

## 1. Introduction

The problem of the disperse filler in the composite material remains relevant to this day. The main task through the process of developing polymer composite materials (PCM) is to create an optimal structure that meets the requirements for the use of products from PCM. This is achieved by selecting the components of the PCM, their ratios, methods for obtaining and products from it. It is well-known that powder filler, such as $CaCO_3$, makes composite material more durable and reliable, decreases the end cost and has an impact on viscosity. Also, the choice to study PP and $CaCO_3$ is come from the article by Thriveni Thenepalli et al [1], where this combination enhances the mechanical properties of plastic parts used in automobiles.

Many factors of rheological and mechanical properties were studied. Typical yield stress behavior exhibited by suspensions is described in the reviews by YEH WANG [2]. Filling viscous thermoplastics with rigid fillers in an amount of more than 20 % refers to a transition from plastic flow to brittle fracture is observed [3]. In this case, there is a significant reduction in the impact strength, the work of destruction. The modulus of elasticity increases with the amount of filler, but it increases the size and number of cracks, like pores, arising in the loading process when the matrix is peeled off from the dispersed particles at the time of reaching the stresses corresponding to the adhesive strength of the system. Theoretical data shows that by reducing the size of the filler particles and the dispersion of their diameters can significantly reduce the likelihood of large defects.

From the study of AY Goldman, CJ Copsey [4] the toughness of the $CaCO_3$ filled materials

increasing at low temperatures is close to the toughness produced using rubber at 23°C, and even better at lower temperatures.

As known from T. Kaully's et al [5] research, the dominancy of particle size distribution was demonstrated by studying the relative suspensions viscosity as function of shear rate.

Having studied the recent discoveries in the field of filled composite materials, it is found that not much attention is paid to the effect of particle size on the properties, so this issue is relevant.

## 2. Experimental procedure

### *2.1. Materials preparation*

The matrix consists an isotactic PP brand Moplen HP 5008 (Poly Murray, Basel, Corea), melt flow rate MFR=0.6 g/min (5 kg/1900 s), density $\rho$=0.9 g/$cm^3$, melting point T=190 °C. Powder $CaCO_3$ was used of two particle diameters. Particle sizes are ranged from 0.1 $\mu$m to 80 $\mu$m.

Composite with different compositions, viz. PP/ $CaCO_3$ 95/5, 90/10, 85/15, 80/20, 85/25, were prepared by mixing the components in a Plastograph EC (Brabender GmbH & Co. KG, Germany) at 190 °C, 100 rpm for 10 min. The stirring continued until the recorded torque reached equilibrium. To avoid the PP degradation, time of mixture was minimized. For further research, the sheets were prepared by forming the melt of mixed composites in a hot press at 190 °C for 10 min.

### *2.2. Dispersion analysis*

To estimate the size Measurements were carried out on a laser diffraction analyzer of particle size distribution Analysette 22 MicroTec plus with a dispersion unit in the liquid (size range: 0.08 - 2000 $\mu$m). The results are shown in the Figure 1.

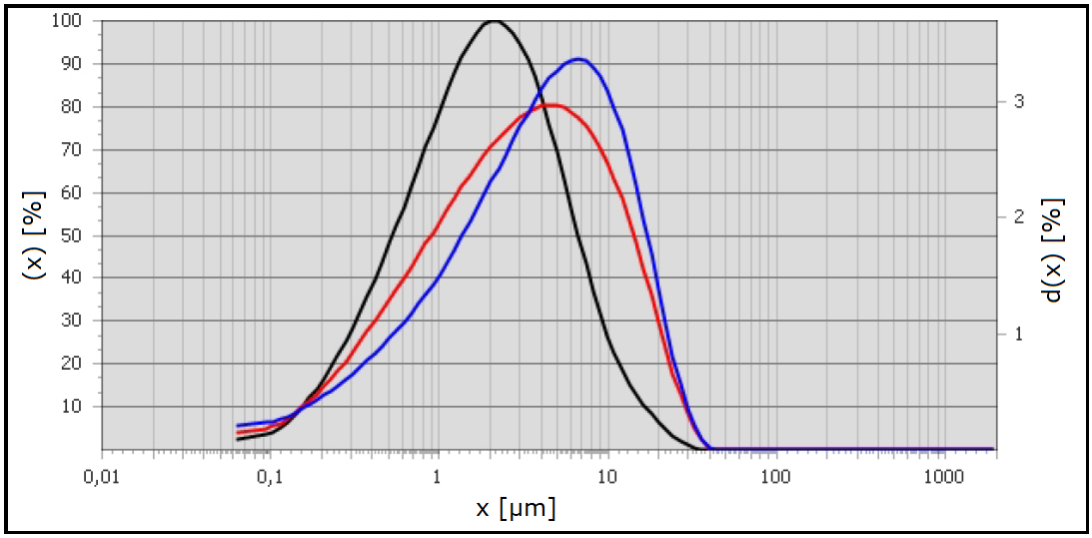


**Figure 1.** The dependence of the integral and differential curves on the size of the first $CaCO_3$ powder in the sample.

### *2.3. Rheological properties*

Rheological tests were carried out on the viscometer IIRT-5 at a temperature of 190 °C and a load of 1.905 kg to 14.005 kg in increments of 0.5 kg, 1 kg and 1.5 kg to obtain average values, 3 samples were tested at each load. The test for MFR was performed with the load varied

according to GOST by 2.16 kg, 5 kg and 10 kg at measurement times - 10 min, 2 min, 1 min and 30 sec (for 10 kg), respectively.

Results are shown in Figure 2 (a) and (b).

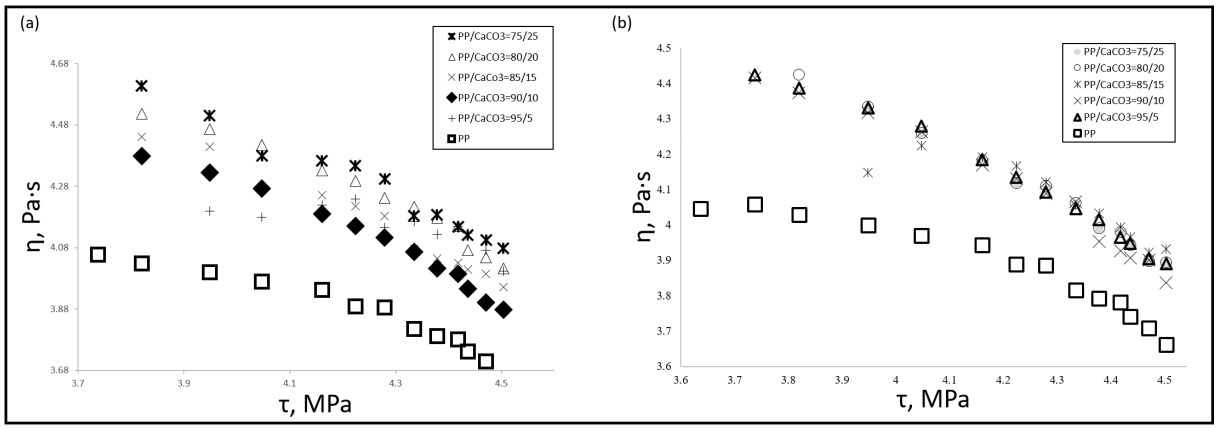


**Figure 2.** Plots of $\log|\eta|$ versus shear stress at 190 °C for (a) $d_1$ and (b) $d_2$ sizes of the filler.

*2.4. Mechanical properties*

The tensile test was carried out on a universal testing machine Instron, model 1122 of the company "INSTRON Ltd."(England.) For testing samples were prepared in the form of two-sided blades with a length of the working part l = 35 mm, a width a = 5 mm and a thickness b = 0.5 mm. The tests were carried out at a relative tensile speed of 50 mm/min at room temperature. The load-deformation diagrams obtained were used to calculate the main deformation-strength properties of materials: modulus of elasticity, deformation at yield point, strength and elongation at break. To obtain average values, 5 samples of each composition of different filler concentrations were tested.

## 3. Results and discussion

*3.1. Dispersion analysis*

Received results on Figure 3 (a) and (b) show us unimodal distribution and that most of the particles in the $CaCO_3$ filler have the following average sizes: $d_1 = 4.1$ $\mu$m and $d_2 = 3.3$ $\mu$m.

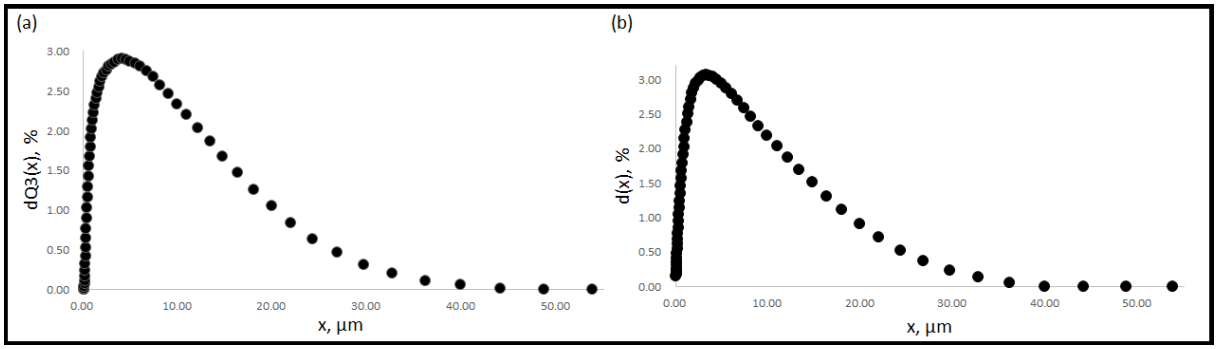


**Figure 3.** Unimodal volume distribution of the particles sizes for (a) $d_1 = 4.1$ $\mu$m and (b) $d_2 = 3.3$ $\mu$m, different colour corresponds to 3 measurements.

*3.2. Rheological properties*

In the studied shear range on Figures 2 (a) and (b), the viscosity of both PP and composites decreases monotonically with increasing shear stress, which is typical for non-Newtonian flow. At the same time, with the growth of the filler content, there is a slight deviation from the Newtonian flow. It can be assumed that the rheological behavior depends significantly on the agglomeration of the filler particles. However, having built the dependence of viscosity on the filler concentration at a constant shear stress, new results arise on Figures 4 (a) and (b).

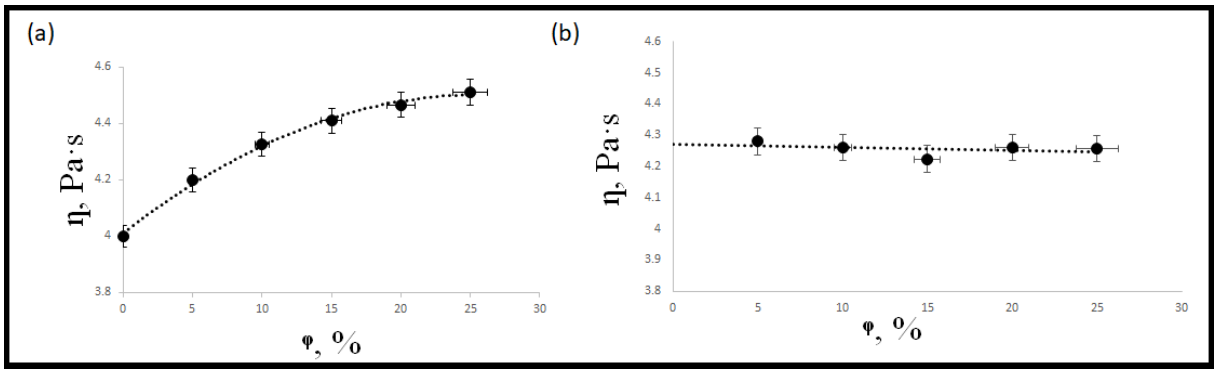


**Figure 4.** Plots of $\log|\eta|$ versus concentration of the filler $CaCO_3$ [wt.%] at constant shear stress $\tau = 4MPa$ for (a) $d_1 = 4.1\mu$m and (b) $d_2 = 3.3$ $\mu$m .

It is generally accepted [6] that the simplest dependence describing the viscosity of the filled composite is the Einstein equation (1):

$$\eta = \eta_0 (1 + \alpha\varphi)$$

where $\eta_0$ - viscosity of the matrix, $\varphi$ - volume fraction of the filler, $\alpha$ - constant,
and Guth equation (2)

$$\eta = \eta_0 \left(1 + \beta\varphi + \delta\varphi^2\right)$$

where $\beta$ and $\delta$ are constants.

Equations (1) and (2) can describe the dependence on Figure 4 (a) on equal terms. For compositions on Figure 4 (b) with sizes of the filler $d_2$=3.3$\mu$m no dependencies are appeared. That can be explained by variations in the amount of $CaCO_3$, which could change little the morphological state of PP/$CaCO_3$ compositions [7].

*3.3. Mechanical properties*

Received mechanical characteristics show us typical deformation dependencies for filled polymers compositions. In the study of deformation stretching of samples, the formation of a 'neck' is observed. On the stress-elongation curves, yield stress is observed .

Found out, that with an increase in the concentration of powder filler goes up the value of Youngs modulus (E, MPa) and also there is a significant decrease in the elongation at break ($\epsilon$, %), and the strength also decreases, but rather weakly.

Hence, amount of the filler and its size have a significant influence on rheological and mechanical properties, even if the diameters of particles are different on 20 %.

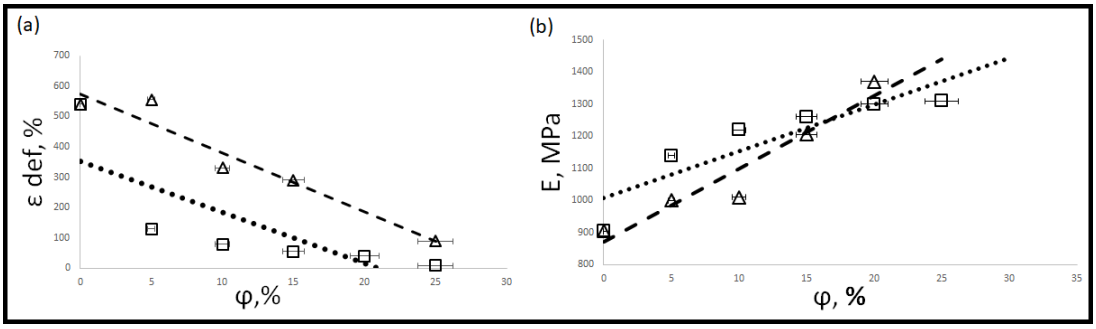


**Figure 5.** Graphics of (a) elongation at break ($\epsilon$, %) and (b) Youngs modulus (E, MPa) versus concentration of the filler $CaCO_3$ [wt.%] for □ $d_1$=4.1$\mu$m and △ $d_2$=3.3$\mu$m with linear trend lines.

## 4. Conclusion

From the rheological tests it seems that viscosity depends on the concentration of the filler for $d_1$=4.1$\mu$m and can be explained by Einstein-Guth-Gold model.

Mechanical tests open a new interesting correlation between the size of the filler and elongation properties. With an increase in the concentration of powder filler increases the value of Young's modulus (E, MPa) and also there is a significant decrease in the elongation at break ($\varepsilon$, %), and the strength also decreases, but rather weakly. The cause may be the occurrence of agglomerations, or insufficient adhesion between PP and $CaCO_3$.

These results give an opportunity to understand better the reasons of unusual rheological properties, using SEM analysis and the other techniques.

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