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Interparticle Interaction Effects in Polymer Suspensions

Nico Laufer, Harald Hansmann, Christian Boss and
Stefan Ofe

Additional information is available at the end of the chapter

Abstract

Viscosity functions of particle-filled polymer melts are shifted to higher values in comparison with those of the unfilled polymer melts. This increase is affected by a number of factors such as the volume content, size, size distribution and the aspect ratio of the suspended particles. If interparticle interactions occur, which include the formation and breaking of agglomerates as well as the migration and rotation of particles during flow, the extent of the viscosity increase of suspensions also depends on the applied shear stress. Filler with a high aspect ratio, such as fibrous and plate-like particles, exhibits a strong tendency for interparticle interactions even at low volumetric concentrations. However, depending on the polymer matrix, spherical particles can exhibit a range of negligible interparticle interactions at low filler volume concentrations. Non-negligible interactions occur at higher filler concentrations. On the basis of the *generalized interaction function*, which considers the transition from negligibly interparticle interactions to the domain of non-negligibly interactions, the flow behavior of particle-filled polymer melts can be estimated. The subject of this chapter is the application of the *generalized interaction function* for the characterization of the flow behavior of particle-filled polyolefin melts.

Keywords: interparticle interaction, generalized interaction function, interaction exponent, rheology, flow function, suspension

1. Introduction

A suspension is a heterogeneous system in which solid particles are dispersed in a liquid. An example of a suspension is a particle-filled thermoplastic polymer melt in which

the dispersed phase is characterized by the fillers and the continuous or suspending phase (matrix) by the polymer melt. The rheological properties of suspensions that are relevant to processing are influenced by a variety of factors and significantly differ from those of the matrix fluid. When the suspending medium is a Newtonian fluid, then the suspension exhibits non-Newtonian behavior with increasing filler concentration. When the suspending medium is non-Newtonian in character, the presence of solid particles magnifies the complexities of its rheological behavior [1].

Rheological investigations on particle-filled polymer melts show that the viscosity functions exhibit higher values with increasing volumetric filler concentration (**Figure 1**). The offset depends not only on the volume fraction but also on the shape, size and size distribution of the suspended particles. In the case of interaction effects, the offset also depends on the applied shear stress and shear rate.

Several theoretical and empirical relationships have been proposed to describe the viscosity of suspensions [1]. Most of these relationships describe the influence of the volumetric filler concentration on the relative viscosity, which is defined as the ratio of the viscosity of the suspension to the viscosity of the suspending liquid.

$$\eta_r = \frac{\eta_s}{\eta_0} \tag{1}$$

where

η_r = relative viscosity [−]

η_s = viscosity of the suspension [Pa s]

η_0 = viscosity of the suspending liquid [Pa s]

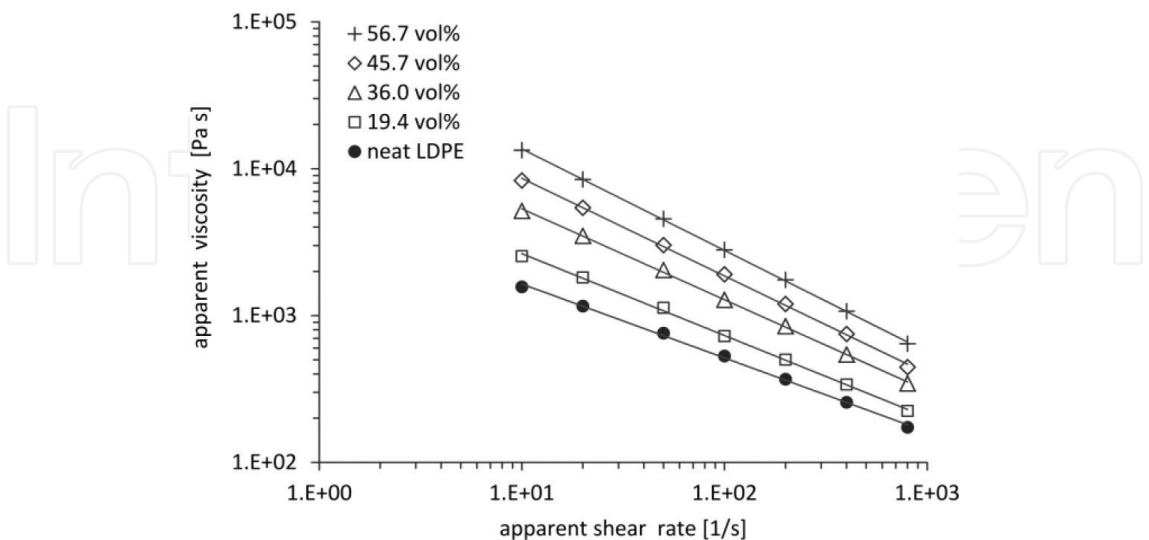**Figure 1.** Viscosity functions of LDPE filled with natural graphite (d50 = 266.6 µm; median particle size of a particle analysis) with varying volume content (T = 190°C; typical processing and recommended MFI temperature – ISO 1133).

The first theoretical relationship of the relative viscosity of a Newtonian fluid in which rigid spheres are suspended was proposed in 1906 by Albert Einstein (corrected in 1911) [2, 3]. He derived an analytical solution that is valid for dilute suspensions in which no particle-particle interactions occur:

$$\eta_r = 1 + 2.5\phi \tag{2}$$

Rutgers experimentally proved that (Eq. (2)) is valid for volume fractions up to $\phi < 0.1$ [1]. For more densely filled suspensions, Einstein's equation was extended by higher order terms to mathematically describe the nonlinear relationship between relative viscosity η_r and filler volume fraction ϕ:

$$\eta_r = 1 + B\phi + B_1 \phi^2 + \ldots \tag{3}$$

with B = 2.5 according to Einstein's constant and with $7.35 \leq B_1 \leq 14.1$ found by different authors [1, 4–6]. The main drawback of the polynomial function (Eq. (2)) is the poor consistency of calculated and measured values for particle volume fractions $\phi > 0.25$ [5].

Krieger & Dougherty developed an empirical equation describing the relative viscosity as a function of the filler content, taking into account the maximum packing of the filler ϕ_m (equivalent formulas or modifications by Maron-Pierce, Kitano, Queimada or Mooney) [1, 5–9]:

$$\eta_r = \left(1 - \frac{\phi}{\phi_m}\right)^{-B\phi_m} \tag{4}$$

This equation is one of the most widely used functions for fitting to experimental data, since B and ϕ_m can be used as fitting parameters for the approximation [5].

In addition to the abovementioned equations, there are a variety of other relationships for describing the flow behavior of suspensions based on the relative viscosity [1, 5–12]. Within a large number of publications, the relative viscosity as a function of the filler concentration is determined either at a constant shear rate or at a constant shear stress. However, studies on particle-filled LDPE and PP indicate that the relative viscosity (determined at both constant shear stress and constant shear rate) depends not only on the filler concentration but also on the applied shear rate (**Figure 2**).

Figure 2 (right) shows the impact of the apparent shear rate on the relative viscosity, which can be expressed by a power function. On the basis of this function, it can be shown that an increase of the shear rate by a decade leads to a reduction of the relative viscosity, determined at constant shear stress, by approximately 60%. The relative viscosity, determined at constant shear rate, is reduced by approximately 28%. This shows that the shear rate has to be taken into account for an exact prediction of the real flow behavior of particle-filled polymer melts.

Hinkelmann noted in his investigations on glass-filled SAN that a simple analytical description of the filler concentration on the relative viscosity is problematic due to the strong
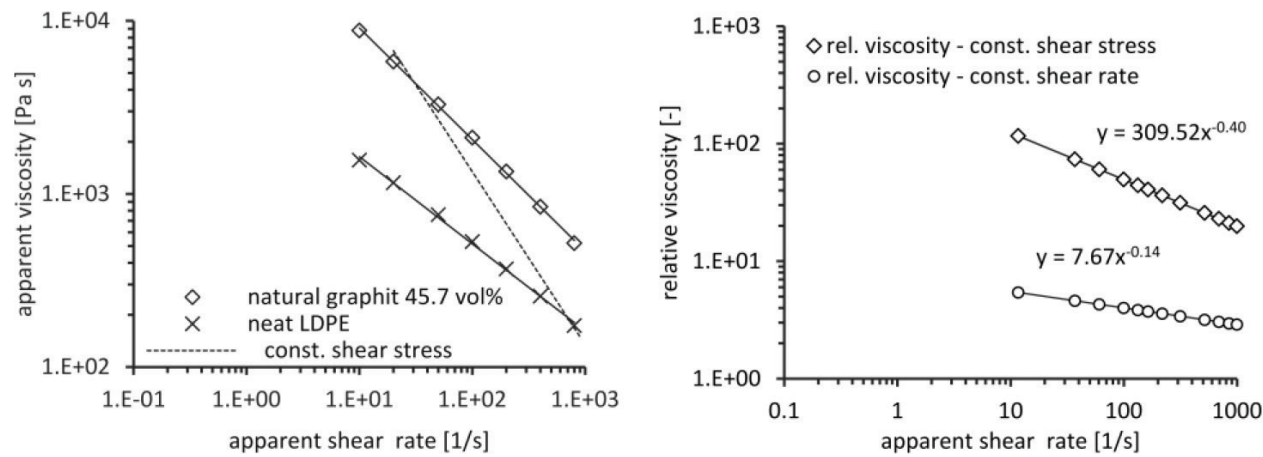
Figure 2. (left) Viscosity functions of LDPE and LDPE filled with 45.7 vol% natural graphite; (right) Relative viscosity determined at the same shear rate and shear stress, respectively.

impact of the shear rate on this value [12]. Gleißle *et al.* also pointed out in his investigations on charcoal-filled silicone oil that equations describing the relative viscosity as a function of the volume concentration cannot universally describe the increase in the viscosity of liquids by the addition of particles [13]. Alternatively, Gleißle and Baloch proposed a very vivid description to illustrate the flow behavior of suspensions based on the concept of shear-stress-equivalent shear rate.

Within this concept, the hydraulic diameter is reduced by the volume content of the particles. This leads to an increase in the shear rate of the continuous phase between these particles, the so-called inner shear rate (**Figure 3**).

The relative increase of this inner shear rate to the shear rate of the suspension (considered as a homogeneous liquid) is expressed by the shift factor B.
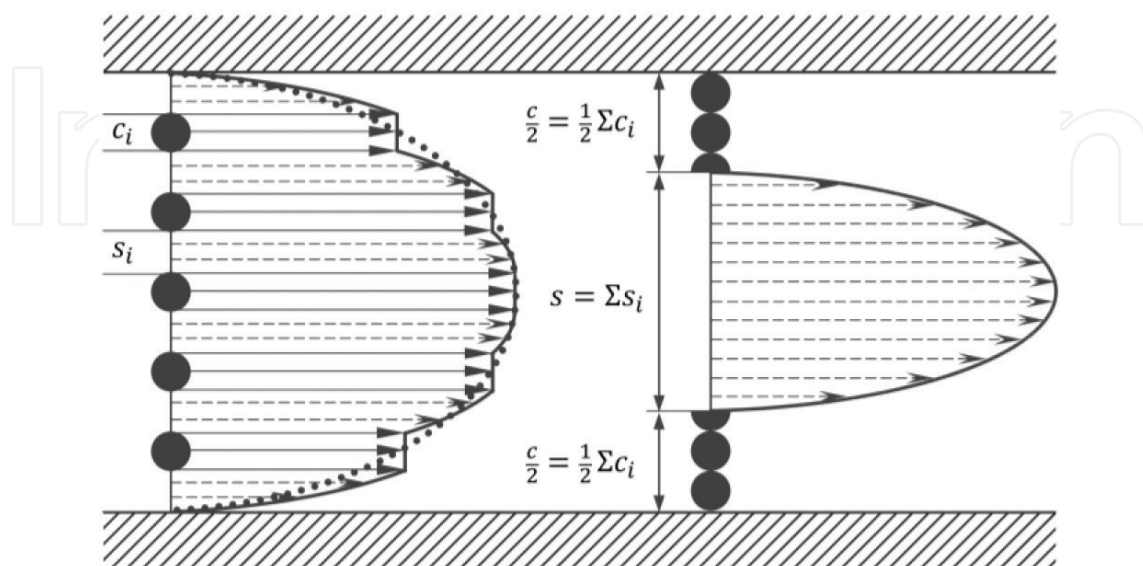

Figure 3. Idealization of Poiseuille flow (pressure-induced channel flow) processes in suspensions according to the concept of the shear-stress-equivalent shear rate.

$$B \; = \; \frac{\dot{\gamma}_c}{\dot{\gamma}_0} \tag{5}$$

where,

$\dot{\gamma}_c$ = inner shear rate [1/s]

$\dot{\gamma}_0$ = shear rate of the suspension [1/s]

In general, the flow behavior of a shear thinning fluid, such as particle-filled polymer melt, can be described by the power law (defined by consistency index K and flow behavior index n) of Ostwald/de Waele. On the basis of the power law, interaction effects can be taken into account in the calculation of the shift factor as follows [14]:

$$B \, (\tau)^{n_0} \; = \; \frac{K_c}{K_0^{\,\chi}} \cdot \tau^{(\chi-1)} \tag{6}$$

where,

K_c = consistency index (suspension) [Pa s^n]

K_0 = consistency index (polymer matrix) [Pa s^n]

τ = applied shear stress [Pa]

χ = interaction exponent $\left(\chi = \frac{n_c}{n_0}\right)$ [−]

n_c = flow behavior index (suspension) [−]

n_0 = flow behavior index (polymer matrix) [−]

Shift factor B is affected by a number of factors: interparticle interactions, the formation and breaking of particle aggregates, the solids content of the suspension, the particle size and morphology of the solid phase, the effects of surfactants, which in turn affect the viscosity of the suspension [15]. In case of negligible interparticle interactions, the flow behavior indices are the same for the flow functions of the suspension (n_c) and the matrix polymer (n_0); thus, the interaction exponent is equal to one. In this case, shift factor B is independent of the applied shear stress, and the filler content only affects the consistency index [13].

The quantitative impact of the volumetric filler concentration on the consistency index of the suspension (**Figure 4**) is expressed by the following empirical Equation [16, 17]:

$$K_c \; = \; K_0 \cdot \left(1 + A \cdot \phi_v^{\,\alpha} \cdot e^{\beta \cdot \phi_v}\right) \tag{7}$$

where,

ϕ_v = volumetric filler concentration [−]

The adjustable parameters A, α, β can be found by regression analysis to fit a given set of experimental data.

As already stated, in case of negligible interparticle interactions, only the consistency index of the suspension changes as a function of the volumetric filler concentration, while the
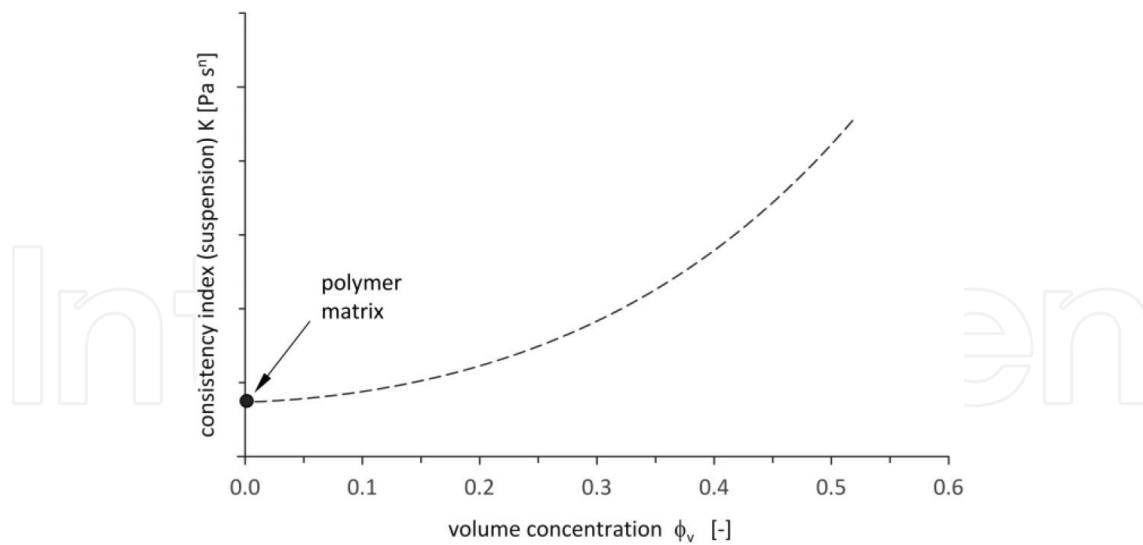
Figure 4. Typical function of the correlation between consistency index of a suspension and volumetric filler concentration.

interaction exponent remains constant at a value of one. The interaction exponent decreases only if interparticle interactions occur (**Figure 5**).

A general mathematical description of the interaction exponent over the entire range of negligible interparticle interactions with the transition to non-negligible interparticle interactions provides the following equation, which is called the *generalized interaction function* [18]:

$$\chi = \frac{1}{\left(1 + \left(\frac{K_c}{K^*}\right)^a\right)^{\frac{b}{a}}} \tag{8}$$

where,

K^* = transitional consistency index [Pa s^n]
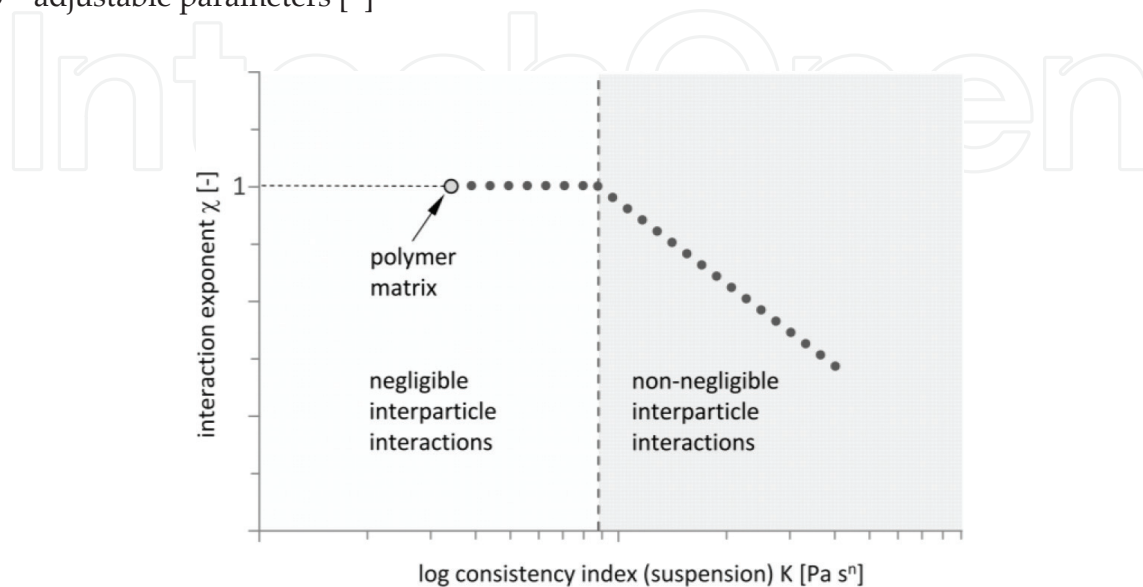
a, b = adjustable parameters [−]

Figure 5. Interaction exponent of a suspension as a function of the consistency index.

The model parameter (K*) describes the transition from negligible interactions to non-negligible interactions, whereas (a) expresses the width of the transition. The intensity of interaction effects is considered by parameter (b). The model parameters of this empirical equation can be found by regression analysis to fit a given set of experimental data with regard to minimize the sum of the relative error squares. To quantify the quality of the approximation, the mean of the absolute relative error values is used. This procedure was applied to all regression analyses in this study.

2. Comparison of interaction effects in long-chain branched and linear polymeric matrices

Interparticle interactions take place when the filler concentration increases. This includes an increase of particles per unit volume, which come into contact during flow. Furthermore, the rotation and migration of particles during flow, as well as the formation and breaking of aggregates, produces additional dissipative effects, which lead to an increase in the viscosity [1]. The mathematical consideration of interaction effects, which are dependent on the size, size distribution and morphology of the filler particles, as well as the filler volume concentration and the applied shear rate or shear stress, which is affected by volume output and the flow channel geometry in one general approach that describes the flow behavior of suspensions, is subject of this chapter. In addition to the particle properties and process conditions, the molecular structure of the matrix polymer has an influence on the formation of interparticle interaction effects and thus also on the flow behavior of the suspension. Linear and long-chain branched polymers differ in both shear thinning flow behavior and nonlinear, steady-state viscoelastic melt properties. The elastic melt properties, defined as the ratio of normal stress difference and shear stress, influence the particle migration during flow. The resulting differences in flow behavior are illustrated below using a long-chain branched LDPE and a linear PP as polymer matrices. In order to characterize the rheological properties of the particle-filled polymer melts, the fillers must be uniformly dispersed into the polymer matrix by means of suitable compounding techniques. The fillers mentioned in this chapter (**Figure 6**) can be classified based on their geometrical shape into the following categories:

- Spherical → glass beads, wood flour (small fraction)
- Plate-like → natural graphite
- Fibrous → wood flour (big fraction), wood fibers

The particle size distribution was determined using the Mastersizer 3000 (Malvern Instruments GmbH) via laser diffraction. The preparations of all formulations as shown in the following section were carried out by compounding on a corotating twin screw extruder (Brabender DSE 20) with a mass temperature of 190°C and mass output of 5 kg/h. Temperature profile: feed zone 170°C; plastification zone 180°C; conveying zone 185°C; mixing zone 185°C; extrusion zone 190°C.

2.1. Low-density polyethylene

The polymer matrix of all following compounds in this section is a low-density polyethylene (LDPE), which is highly branched and exhibits shear thinning flow behavior.
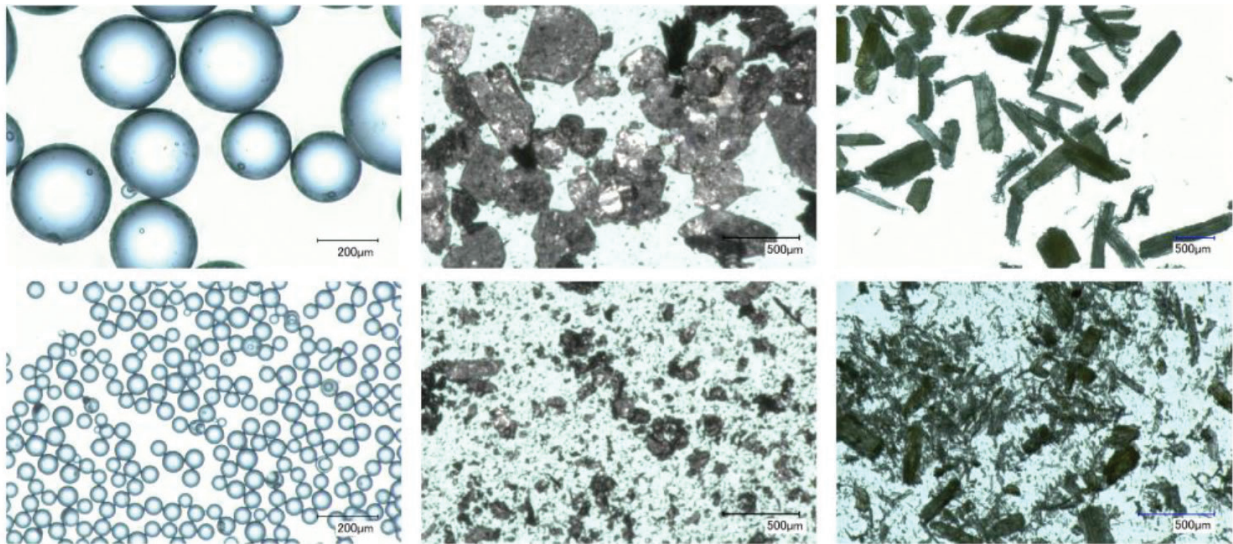
Figure 6. Light transmitted microscopy of spherical, plate-like and fibrous particles.

Figure 7 shows the flow functions (shear stress as a function of shear rate) of unfilled LDPE and LDPE filled with different volumetric concentrations of wood fibers. The volume concentrations correspond to typical mass fractions and have been discussed in previous studies [14].

All flow functions exhibit shear thinning flow behavior, which can be mathematically described by the power law of Ostwald/de Waele. With increasing volumetric content of the wood fibers, the level of the flow functions is shifted to higher values. Accordingly, the consistency indices (K) increase with the increasing volume fraction of the filler particles. The extent of the consistency increase depends on the size and geometrical shape of the filler particles. **Figure 8** shows flow functions of various filled LDPE systems at a fixed loading of 46 vol% to illustrate the impact of the filler morphology on the flow behavior. At low shear rates, the relative increase
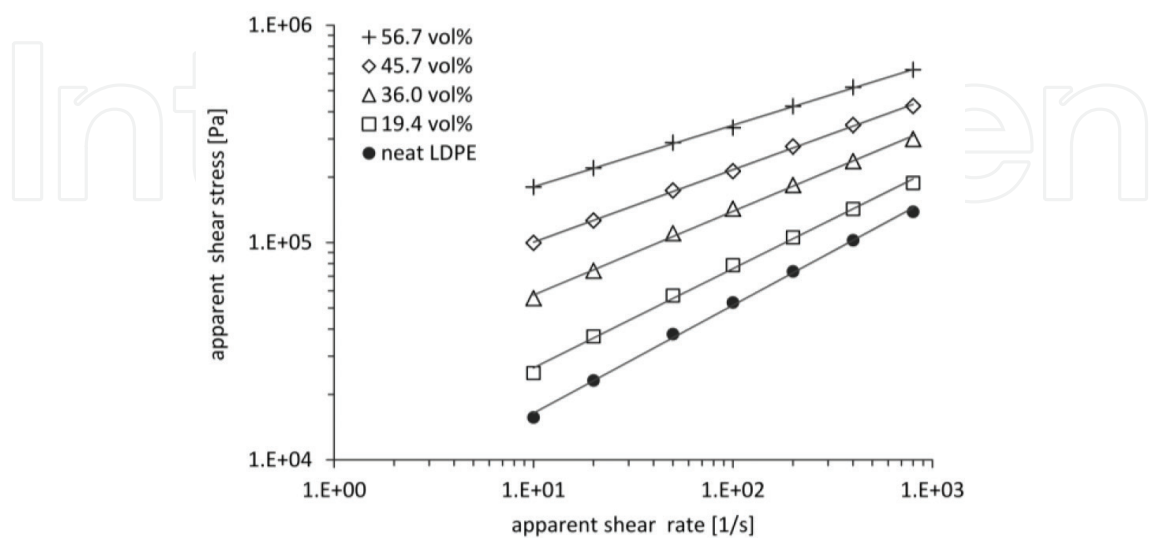

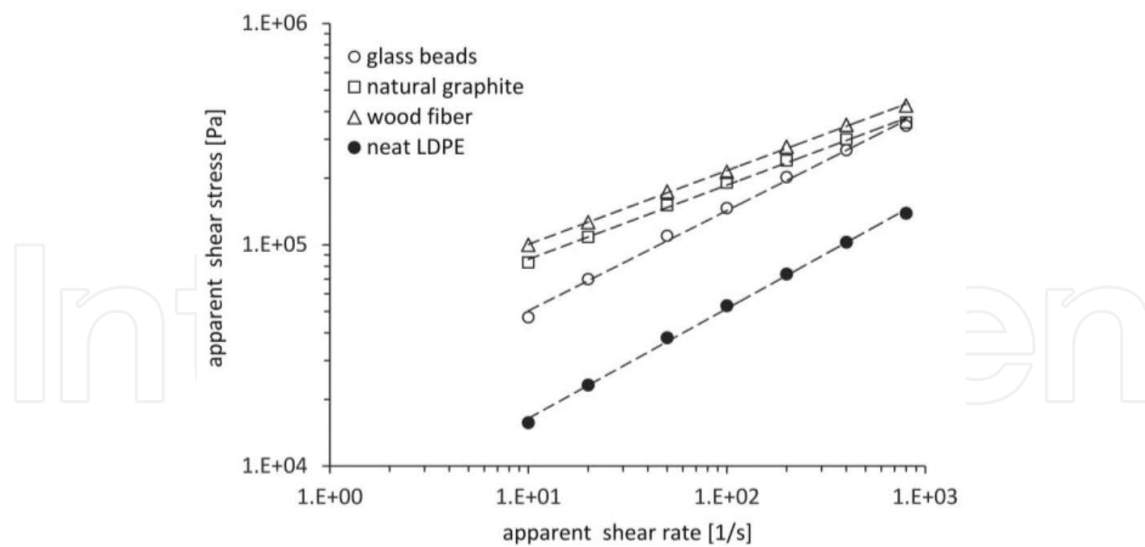**Figure 7.** Flow functions of LDPE filled with varying volume fractions of wood fibers (d50 = 527 μm).

Figure 8. Flow functions of LDPE filled with 46 vol% of different fillers; glass beads (d50 = 346 μm), natural graphite (d50 = 267 μm), wood fibers (d50 = 527 μm) in comparison with the unfilled LDPE (T = 190°C).

in shear stress of the suspension to the shear stress of the polymer matrix is the highest for the fibrous particles (*wood fibers*), slightly lower for the plate-like particles (*natural graphite*), and the lowest for the spherical particles (*glass beads*). With increasing shear rates, the impact of the filler morphology on the flow behavior decreases.

In order to describe the impact of the volumetric filler concentration on the consistency index of particle-filled LDPE for various fillers in different size fractions, Eq. (7) has been used. **Figure 9** shows that experimental data (*symbols*) can be excellently fitted (*lines*) on the basis of this equation.

At low filler concentrations up to approx. 20 vol%, the particle properties, for example, particle size and morphology, have no significant impact on the extent of the consistency index
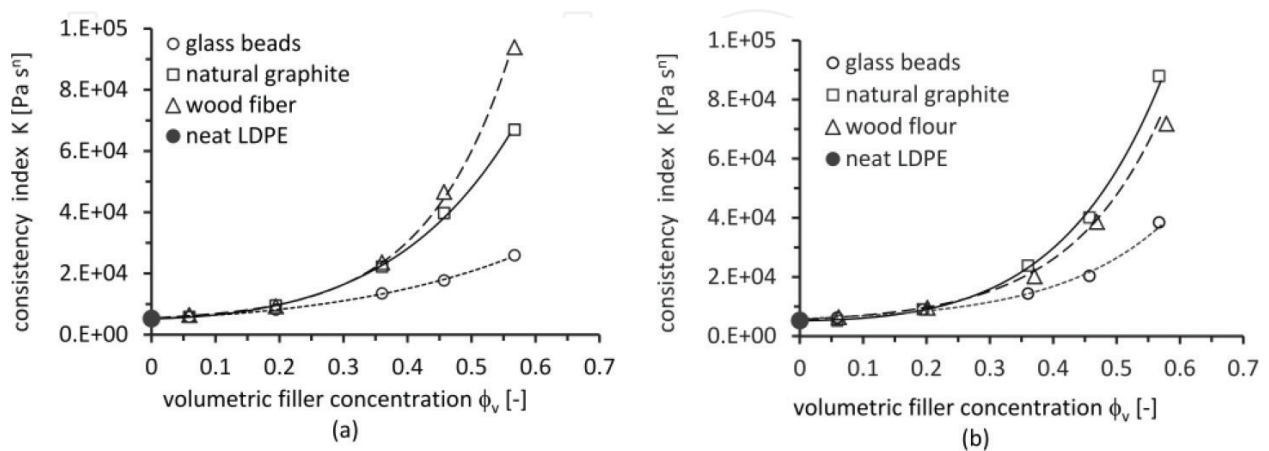

Figure 9. Consistency index as a function of volumetric filler concentration for LDPE filled with various fillers with different particle sizes, (T = 190°C); (a) glass beads (d50 = 346 μm), natural graphite (d50 = 267 μm), wood fibers (d50 = 527 μm); (b) glass beads (d50 = 60 μm), natural graphite (d50 = 77 μm), wood flour (d50 = 110 μm).

increase. At higher filler concentrations, the differences between the filler types are substantial. High aspect ratio particles such as wood fibers and natural graphite, regardless of their size, have a significantly greater impact on the consistency index than the spherical glass beads. Generally, the viscosity of a suspension and thus the consistency index exhibit higher values with decreasing particle size at a constant filler volume fraction. An exception can be observed here with the wood fillers. The small particle fraction contains a considerable amount of wood dust, which has a much lower aspect ratio as compared to the larger wood fibers. Since the impact of the aspect ratio on the consistency index increase is stronger in comparison with the particle size, the larger fibrous wood fillers cause a higher increase in consistency than the smaller particles of wood flour.

The *generalized interaction function* (Eq. (8)) is used in order to describe the interaction exponent as a function of the consistency index, taking into account the transition from negligible interparticle interactions to the domain of non-negligible interactions. **Figure 10** shows that experimental data (symbols) can be fitted (lines) on the basis of this equation with high accuracy.

The interaction function illustrates that glass beads in LDPE exhibit less interparticle interactions in comparison with particles with larger aspect ratio. In particular, large glass beads even show a range of negligible interparticle interactions with a transition to non-negligible interactions. Regardless of type and size, high aspect ratio particles have a characteristic relationship between consistency index and interaction exponent, which is distinctive of the polymer matrix LDPE.

Based on the *generalized interaction function* (Eq. (8)) and the relationship between consistency and volumetric filler concentration (Eq. (7)), the shift factor B can be derived as a function of the volume fraction for variable shear stresses (Eq. (6)). **Figure 11** comparatively illustrates the influence of the filler volume concentration and the applied shear stress for various fillers in
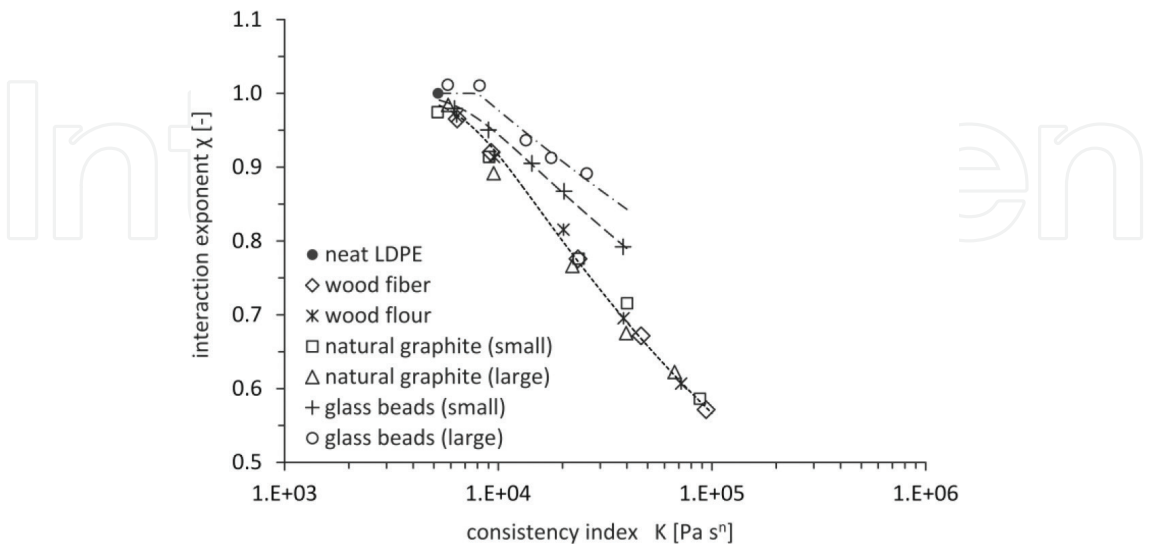

Figure 10. Interaction exponent as a function of consistency index of LDPE filled with various fillers (T = 190°C).
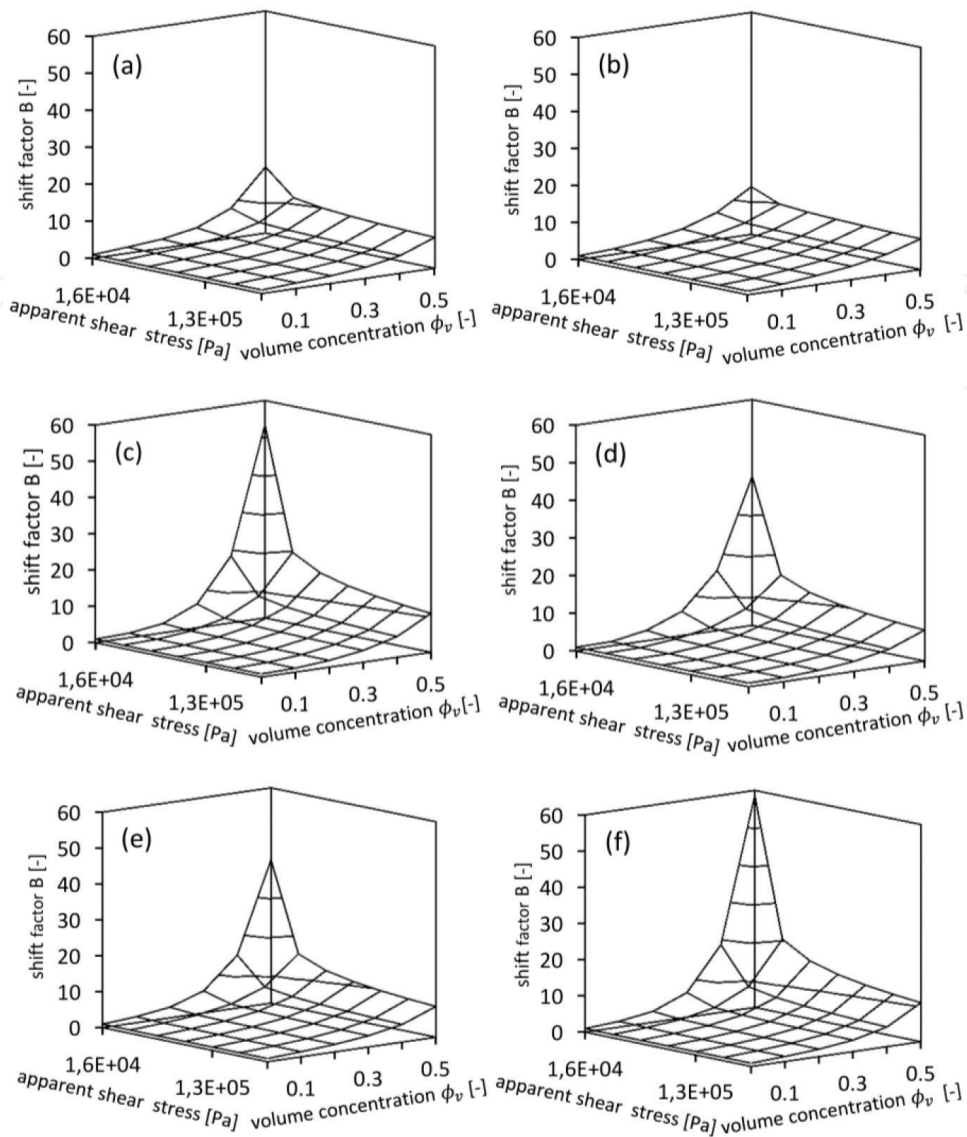
Figure 11. Shift factor B as a three-dimensional function of filler content and applied shear stress for LDPE filled with various fillers; (a) glass beads (d50 = 60 μm), (b) glass beads (d50 = 346 μm), (c) natural graphite (d50 = 77 μm), (d) natural graphite (d50 = 267 μm), (e) wood flour (d50 = 110 μm), (f) wood fibers (d50 = 527 μm).

different size fractions on the shift factor B. On the basis of the shift factor B, the flow behavior of polymer suspensions can be estimated for arbitrary volume concentrations and shear stresses or shear rates on the basis of the following equation:

$$\tau_c = B^{n_0} \cdot \tau_0 = B^{n_0} \cdot K_0 \cdot \dot{\gamma}^{n_0} \tag{9}$$

2.2. Polypropylene

The polymer matrix of all following compounds in this section is a polypropylene (PP), which exhibit a linear molecular structure and shear thinning flow behavior.

Figure 12 shows the flow functions (shear stress as a function of shear rate) of PP filled with different volumetric concentrations of wood flour, as well as of the unfilled polymer matrix.

As with particle-filled LDPE, all PP-based flow functions exhibit shear thinning flow behavior. In the observed shear rate range, the flow behavior can also be described very well by the power law. Compared to the LDPE-based compounds, the flow behavior index (n) of the PP-based formulations has slightly smaller values and thus a higher pseudoplasticity.

The influence of the filler morphology on the flow behavior of PP filled with different filler types at a fixed loading of 46 vol% is shown in **Figure 13**. Compared to long-chain branched LDPE, the impact of filler type on the flow function of linear PP is lower. Furthermore, it is obvious that the flow behavior index is only slightly affected by the different aspect ratio of the filler types.

On the basis of (Eq. (7)), the impact of the volumetric filler concentration on the consistency index of particle-filled PP has been described. **Figure 14** shows that experimental data (*symbols*) can be excellently fitted (*lines*).

Over the entire range of volume fraction, the differences between the filler types are substantial regarding the impact on the consistency index increase. Basically, a strong influence can be observed of the aspect ratio of the filler particles on the consistency increase. High aspect ratio particles such as wood fibers and natural graphite, regardless of their size, have a significantly greater impact on the consistency index than the spherical glass beads, especially at high filler contents.

Figure 15 presents the correlation between interaction exponent and consistency index, which have been mathematically described by the *generalized interaction function* (Eq. (8)). This contains all fillers; wood fibers/flour, natural graphite and glass beads, in the entire concentration range from 6 to 56 vol%, in each case in two particle size fractions.

It is a remarkable fact that there is a characteristic relationship between interaction exponent and consistency index for all PP-based compounds, regardless of filler type, size and volume
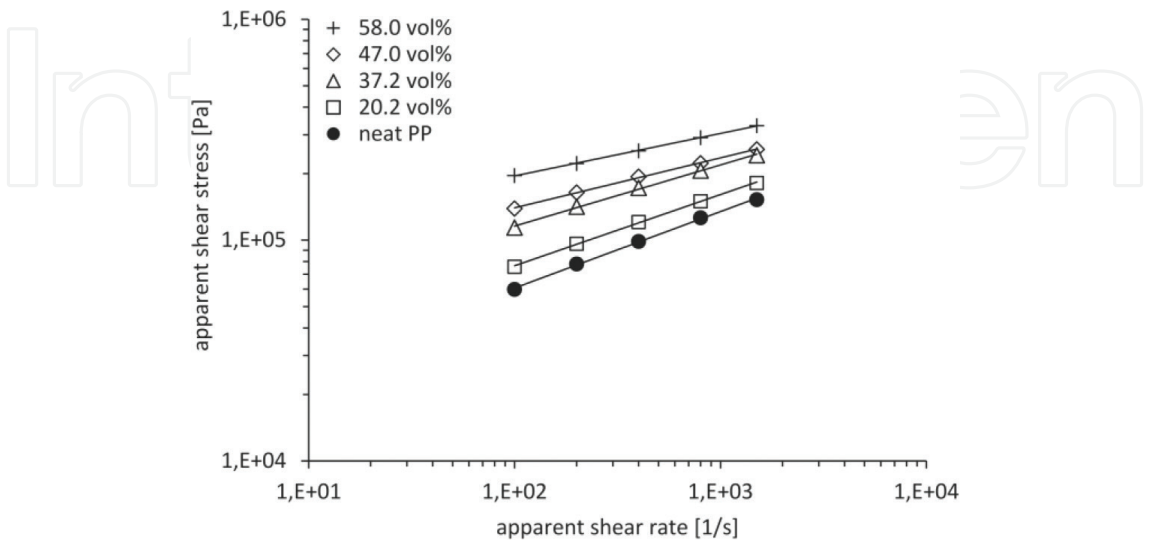

Figure 12. Flow functions of PP filled with varying volume fractions of wood flour (d50 = 110 μm), (T = 200°C).

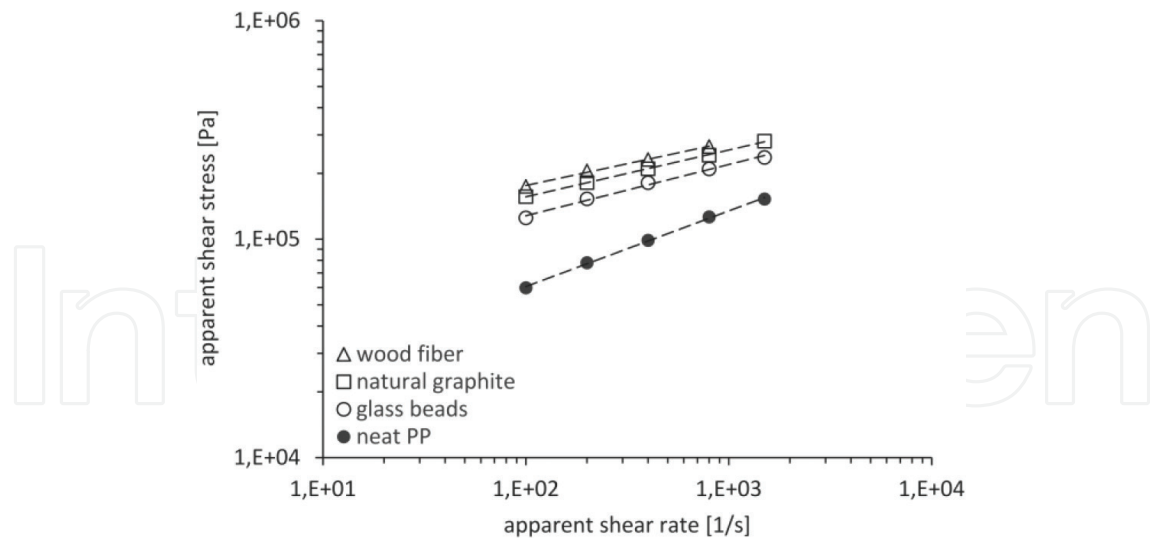**Figure 13.** Flow functions of PP filled with 46 vol% of different fillers; glass beads (d50 = 346 μm), natural graphite (d50 = 267 μm), wood fibers (d50 = 527 μm) in comparison with the unfilled LDPE (T = 200°C).
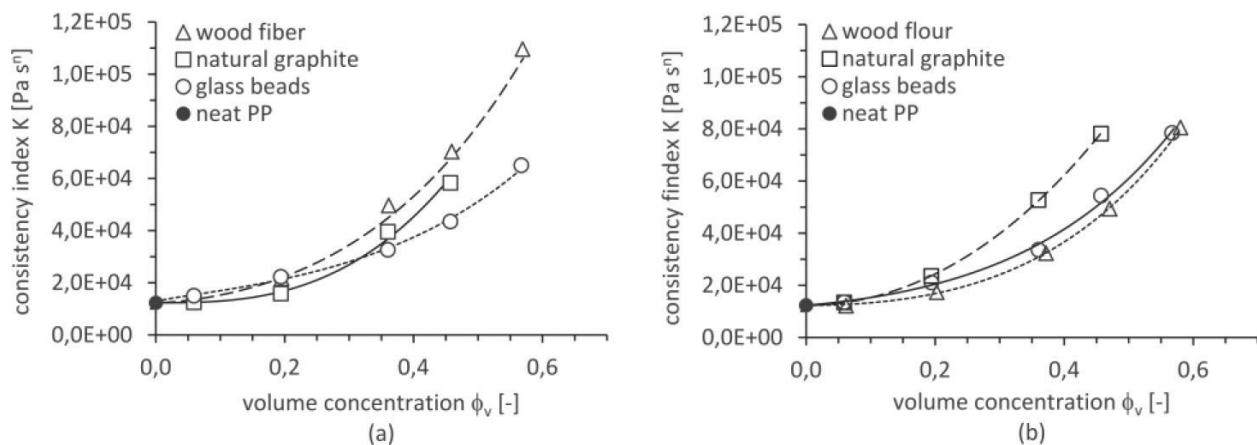

Figure 14. Consistency index as a function of volumetric filler concentration for PP filled with various fillers with different particle sizes, (T = 200°C); (a) glass beads (d50 = 346 μm), natural graphite (d50 = 267 μm), wood fibers (d50 = 527 μm); (b) glass beads (d50 = 60 μm), natural graphite (d50 = 77 μm), wood flour (d50 = 110 μm).

fraction. On the basis of this characteristic correlation, interparticle interactions in PP-based suspensions can be universally described with only one set of parameters of the generalized interaction function. The parameters that are generally valid for particle-filled PP to describe the interaction exponent as a function of consistency (Eq. (8)) index are:
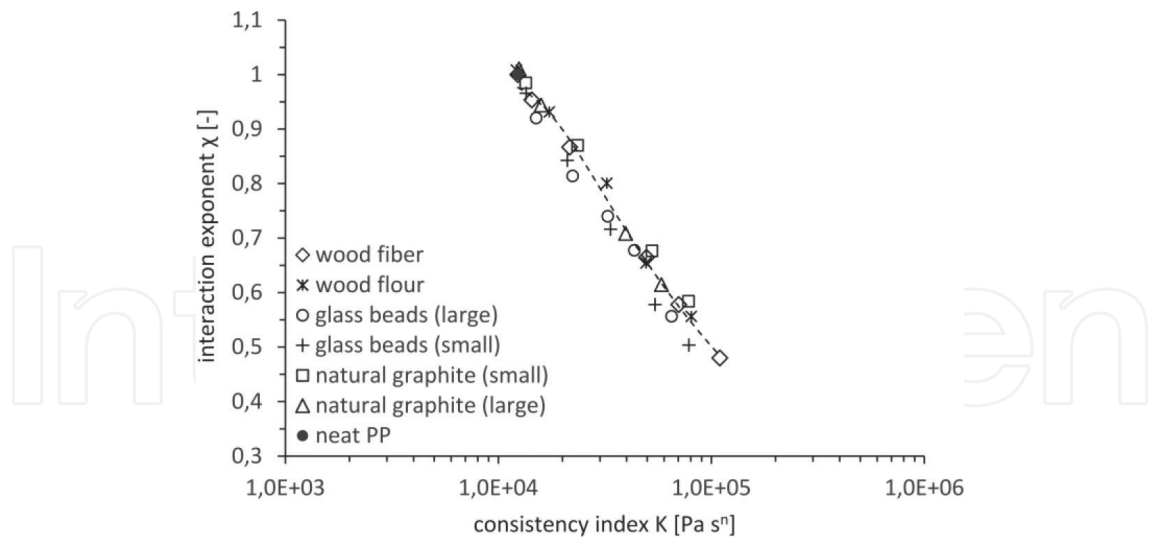
$$K^* = 16706 \ Pa \ s^n$$

$$a = 4.076$$

$$b = 0.385$$

Figure 15. Interaction exponent as a function of consistency index of PP filled with various fillers (T = 200°C).

On the basis of these parameters, the flow curves of particle-filled PP can be estimated, regardless of the filler type, particle size, and volume fraction. **Figure 16** exemplarily illustrates this on different formulations. The experimental data (symbols) have been excellently fitted (lines), using that generally valid parameter set of the interaction function.

Figure 17 comparatively illustrates the influence of the filler volume concentration and the applied shear stress for various fillers in different size fractions on the shift factor B, which has been derived on the basis of (Eq. (6)). On the basis of the shift factor B, the flow behavior of polymer suspensions can be estimated for arbitrary volume concentrations and shear stresses or shear rates, respectively.
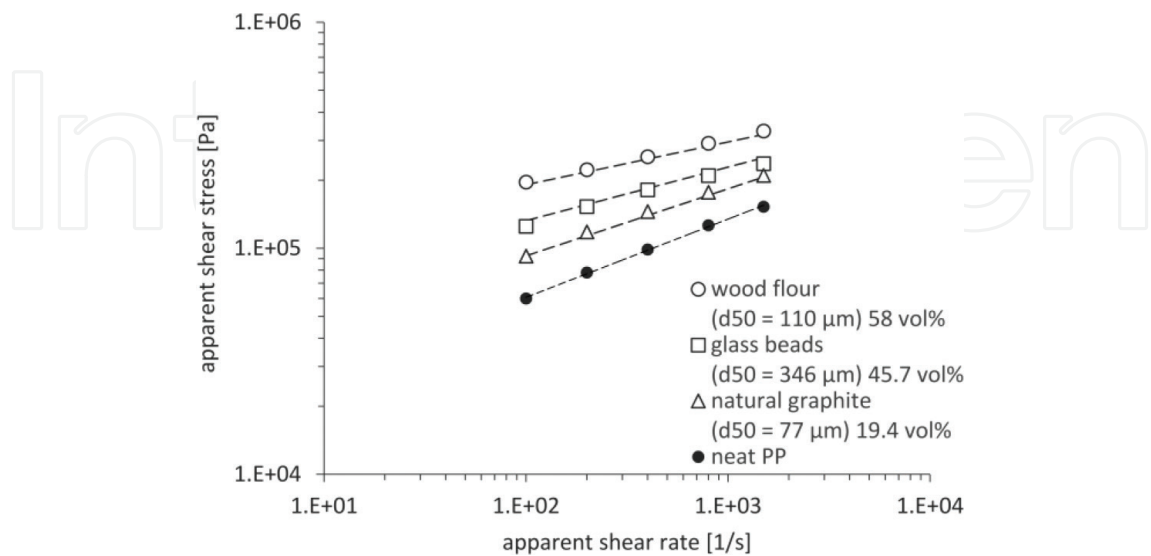

Figure 16. Comparison of experimental data (symbols) to predicted flow curves (lines) for PP filled with various fillers by using just one parameter set of the interaction function (K* = 16,706 Pa s^n, a = 4.076, b = 0.385), (T = 200°C).
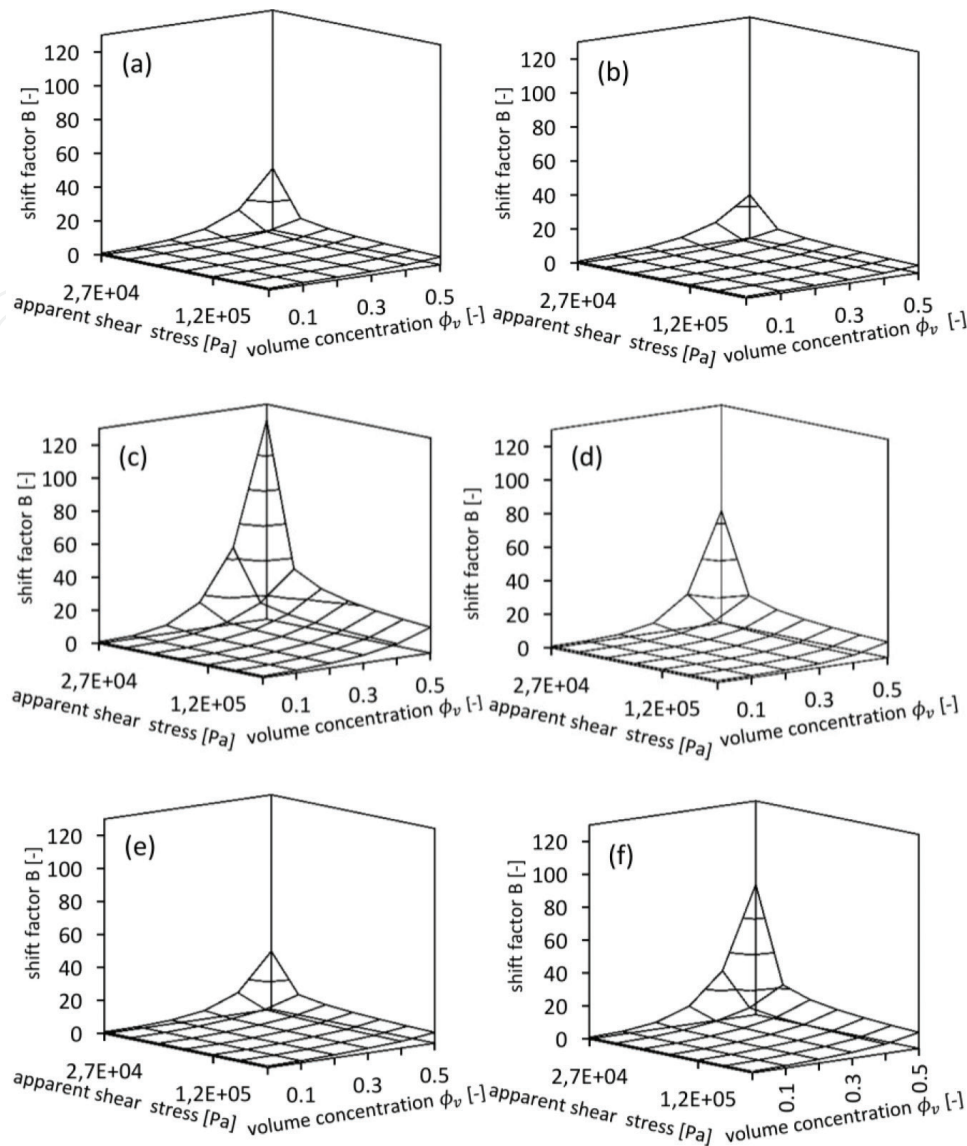
Figure 17. Shift factor B as a three-dimensional function of filler content and applied shear stress for PP filled with various fillers; (a) glass beads (d50 = 60 μm), (b) glass beads (d50 = 346 μm), (c) natural graphite (d50 = 77 μm), (d) natural graphite (d50 = 267 μm), (e) wood flour (d50 = 110 μm), (f) wood fibers (d50 = 527 μm).

3. Conclusion

In this chapter, new mathematical models describing interparticle interaction effects in long-chain branched and linear polymer matrices have been presented. In the context of studies with variable volumetric filler concentrations, the influence of filler type (morphology and aspect ratio) and particle size on interparticle interactions has been compared.

On the basis of the *generalized interaction function*, the correlation between interaction exponent and consistency index of particle-filled polymer melts can be mathematically described with high accuracy to experimental data. This correlation is characteristic and valid for each individual polymer matrix.
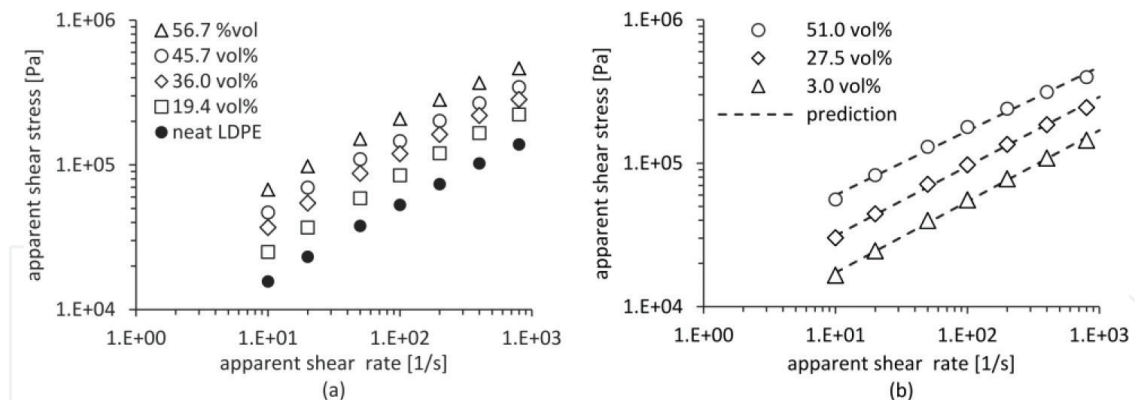
Figure 18. Flow functions of LDPE filled with varying volume fractions of glass beads (d50 = 60 μm), (T = 190°C); (a) measured values; (b) comparison of experimental data (symbols) to predicted flow curves (lines).

Based on the *generalized interaction function*, the shear thinning flow behavior of polymer suspensions can be estimated with high accuracy, regardless of filler type, particle size and volume fraction. The procedure for this is illustrated below using the example of LDPE filled with glass beads (d50 = 60 μm):

1. analysis of the flow functions for different filler concentrations (**Figure 18a**)

2. determination of the functional relationship between consistency index and volumetric filler concentration (Eq. (7))

3. determination of the functional parameters of the generalizes interaction function (Eq. (8))

4. derivation of the shift factor B (Eq. (6))

5. estimation of the flow functions for arbitrary volumetric filler concentrations (Eq. (9); **Figure 18b**)

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