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DETERMINATION OF VISCOSITY CURVE AND PVT PROPERTIES FOR WOOD-POLYMER COMPOSITE

Wiesław Frącz, Grzegorz Janowski
Rzeszow University of Technology
Faculty of Mechanical Engineering and Aeronautics
Rzeszów, Poland

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ABSTRACT

In the presented study the injection mold equipped with the test apparatus were used to determine the viscosity curve of wood – polymer – composite (WPC). During the test the polymer temperatures, flow rates and pressure data measured inside of mold cavity were recorded. In addition the pressure – volume – temperature (PVT) characteristic of this composite was determined using the capillary rheometer. The determined properties were used in numerical simulations of injection molding process of WPC. The results were verified on the basis of pressure profiles measured in the mold cavity during experiment. The simulation was performed using the Autodesk Moldflow Insight 2013 commercial code. The calculated and measured pressure profiles were compared.

KEYWORDS: Wood-polymer composites, numerical simulations, viscosity curve, PVT characteristic, injection molding.

INTRODUCTION

Wood - polymer composites (WPC) are the group of polymer composites which are now increasingly used. Their similar properties to the wood and the use of waste derived from processing of wood make the number of their applications continue to grow. One of the main reasons for increasing their use is also the possibility of using different methods of processing such as extrusion, compression and injection molding. In addition, it is worth paying attention to chosen properties of these materials such as: the ability to be recycled, good mechanical properties and first of all the environmental aspects of their use (Bouafif et al. 2009, Cui et al. 2008, Klyosov 2007, Żuchowska et al. 2007).

The numerical analysis results of injection molding process depends largely on the accuracy of the estimated plastic properties and methodology of their determining, which is generally known. Unfortunately, in the case of WPC, determination of many properties is very complicated

or not feasible using conventional methods. This is mainly due to the considerable heterogeneity of the composite structure or low heat resistance of wood filler (Birley and Haworth 1992, Kim and Pal 2011, Stokke et al. 2013). For example, attempts the attempts to determine the viscosity curve of WPC are not reliable. Too many pressure pulsations that prevent the stabilization of measuring signal may be due to inhomogeneities of WPC. Local, large agglomerates of flours or wood fibers interfere the stable level of signal required to obtain reliable results of pressure. This paper proposes the method to determine the viscosity curve of WPC using the injection mold. In addition, the PVT properties of this composite were determined. The examined WPC properties were used, among others, to perform the injection molding simulation of WPC using the Autodesk Moldflow Insight 2013 commercial code. The simulation results were compared with experiment.

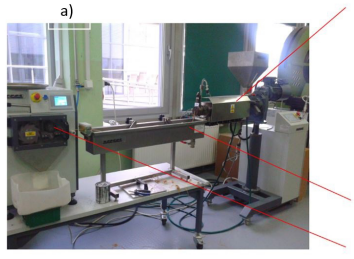
MATERIALS AND METHODS

The composite manufacturing

In order to assess the possibility of wood-polymer composites manufacturing, the samples with different wood fibers percentage were prepared. For the composite preparation the Moplen HP 648T as a polymer matrix (PP) from Orlen Basell Polyolefins company with a high melt flow rate (MFR) was used. As the filler the wood fiber (WF) from coniferous trees of Arbocell C350SR trade name from JRS - J. Rettenmaier & Söhne Gmbh + CO.KG with particle size of 70 to 150 μm was used. The percentage of WF were respectively: 10%, 20%, 30%, 40%. Moreover, 4% of coupling agent Fusabond P613 from DuPont company was added. The Fusabond is an anhydrite modified polypropylene (PP), which allows to receive homogeneous mixtures of incompatible plastics or composite with natural fillers. MFR index for this additive was 49 g.10 min^{-1} (test conditions: 190°C, 2.16 kg according to ISO 1133). In addition, the bulk density of composite components (for Arbocell C350SR - 167 $kg \cdot m^{-3}$, for PP - 565 $kg \cdot m^{-3}$) were determined.

The pellets of composites were prepared in the extrusion process by means of the single screw extruder of EHP25 (the screw diameter of 25 mm) model made by Zamak company, equipped with a cooling bath and granulator (Fig. 1). During the extrusion process the adjustable parameters: heating zones temperature – 140°C, 170°C, 175°C, 180°C and screw rotation of 10 min^{-1} were used. Wood fillers were dried before extrusion at 100 °C for approx. 4 hours.

a)



b)

Screw	
screw diameter [mm]	25
screw length [mm]	670
screw rotation [min^{-1}]	200
Cylinder	
Maximum pressure in cylinder [MPa]	20
Heating power [W]	6x250
Range of temperature control [°C]	30 – 400
The number of heating /cooling zones	3
Cooling medium	air

Fig. 1: a) The test stand for extrusion of WPC pellets: 1- the extruder: Zamak EPH-25, 2- the cooling bath, 3 – the granulator; b) the technical data of extruder.

To prepare the samples the Dr Boy 55E injection molding machine was used. There were assumed five kinds of WPC samples made of a polypropylene matrix and wood fiber with different volume content, respectively: 10 %, 20 %, 30 %, 40 %, 50 %. The cross-section of

the mold cavity was constant, so it was assumed that the flow rate during the cavity filling was constant. During the tests the following flow rates: 2, 5, 10, 15, 25, 35, 40, 45, 50, 60 $cm^{3} \cdot s^{-1}$ were used.

Test stand

In the preliminary studies, to determinate the viscosity characteristics of the composite the capillary rheometer Smart Rheo 2000 of Ceast producer was used. These characteristics were determined at temperature: 180, 190 and 195°C. These results did not allow to determine the proper viscosity curve. During the measurement there was a big problem with the stabilization of pressure signal in the nozzle (Fig. 2), which may be the result of non-homogeneous properties of the composite. The viscosity characteristics of composite were obtained later in the research on the basis of the material flow in real mold cavity.

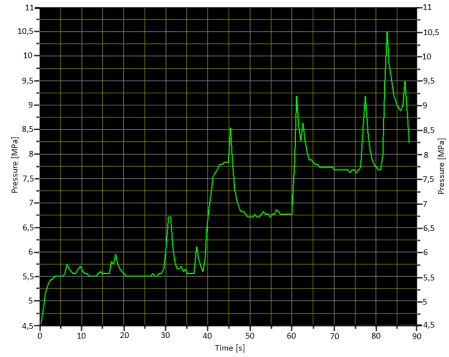


Fig. 2: Instability of pressure signal during viscosity measurement using a capillary rheometer.

In order to carry out the experimental studies (to determine the viscosity curve) the single-cavity injection mold with cavity thickness of 2 mm was used (Fig. 3). The total length of the cavity was 195 mm. The test stand consisted of: the injection molding machine, the injection mold, pressure and temperature sensors mounted in the cavity and the measurement accessories. The cold runner system consisted of: the tapered sprue of 80 mm length), the runner of 8.5 mm diameter and tapered gate with angle of 1.5°. The injection mold has two circuits of cooling channels of 8 mm diameter. Inside the cavity three pressure sensors were placed with a distance of 80 mm from each other. The first sensor was placed at a distance of 17.5 mm from the gate.

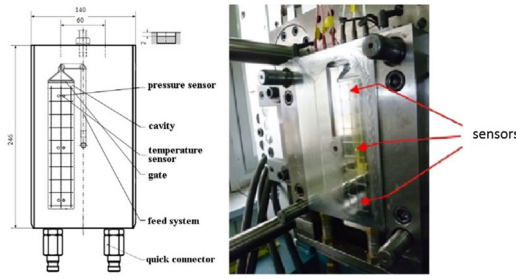


Fig. 3: The single-cavity mold with pressure sensors: a) scheme of the injection mold, b) the mold mounted in the injection molding machine.

To measure the pressure the piezoelectric sensors of Priamus^{TM} named 6002B were applied. The technical characteristics of used sensors are shown below:

- measurement range: 0-2000 bar,
- maximum temperature of the material: no limits,
- maximum mold temperature: 200°C,
- deviation from the linear characteristic: <±1,
- vibration frequency: > 80 kHz,
- measuring sensor diameter: 4 mm.

To register the pressure during the injection cycle the four-amplifier of Priased^{TM} 5080 type with computer software from Priamus company was used. The measuring system consisted of: pressure sensors mounted inside the cavity, the electrical wires connecting the sensors to the amplifier, and the amplifier from which the signal was transmitted by means the A / D converter via BUS industrial interface to the PC computer. The amplifier used in the system enabled an adjustment of sensors sensitivity, which allowed to detect automatically the plastic front presence, as well as the lack of connection between the amplifier and the sensors due to damage or failure in connection of above chain elements. Using the Fill Control software the data from the pressure sensors were recorded. This software also enabled to do the simplified graphical presentation and statistical results analysis. The complete system were recorded measurement data with a frequency of 2 kHz. During the measurements the signals values of active channels and injection cycles were saved automatically. When the front of flowing material reached the sensors location, it started recording the pressure. With the plastic flow, the pressure rose along its flow path, reaching its maximum value when the cavity was completely filled. In the investigated case the pressure change was recorded between the two initial pressure sensors

The third sensor (at the end of flow) was used to control the switching point pressure from filling to packing phase. The measuring system can also record a temperature change in places related to pressure sensors. The plastic temperature was measured in the mold cavity at the first pressure sensor by a thermocouple of N type. The temperature measurement was made at half the thickness of the cavity.

The methodology of viscosity curve determination

The polymer viscosity depends, among others, on the temperature, pressure and shear rate. The viscosity value is calculated from the equation in which these variables must be known. Taking into account the rheological properties, the polymer may behave as Newtonian or non-Newtonian fluid. Newtonian behavior takes place, if the polymer does not lose energy during collision of macromolecules, so the shear stresses associated with the flow are a linear function of shear rate and the viscosity is constant. This phenomenon can be expressed by the relationship:

$$\eta = \frac{\tau}{\gamma} \tag{1}$$

where: τ– shear stress,
η – viscosity,
γ_p – shear rate.

Non-Newtonian behavior occurs if a collision energy of macromolecules is dissipated, so the shear stresses are proportional to the shear rate. The relationship between the stress and the shear rate is often referred to using a Power law, where the viscosity is expressed by the relationship:

$$\eta = K \cdot \gamma^{n-1} \tag{2}$$

where: K – the consistency coefficient,
n – the flow exponent.

The polymers behave like non-Newtonian fluids if n <1. They are known as pseudoplastic fluids. During the injection molding process, however, the shear rate reaches very high values and therefore the interaction between the macromolecular is reduced. This phenomenon occurs when the specific flow rates of the polymer chains are oriented along the direction of flow. In some ranges of shear rates, polymers behave as Newtonian fluids. Then, the viscosity of the polymer can be calculated from Eq. 1, where the shear stresses and shear rates are associated with other process parameters such as pressure, flow rates and the geometry of the mold.

To determine the viscosity of the WPC the method in which the polymer flows through cavity of rectangular cross-section was proposed (Fig. 3). When one knows the geometry of the cavity and the pressure values in two areas of the mold cavity, shear rate and shear stress can be calculated from the relationship:

$$\gamma = \frac{3 \cdot Q}{4 \cdot a^2 \cdot b} \tag{3}$$

where: Q – the volumetric flow rate,
a – the cavity height,
b - the mold cavity width.

$$\tau = \frac{\Delta p \cdot a}{L} \tag{4}$$

where: Δp – pressure drop between the two sensors in the cavity,
L – the distance between the pressure sensors.

On the basis of Eqs. 3 and 4 the Eq. 1 can be written as:

$$\eta = \frac{4 \cdot \Delta p \cdot a^3 \cdot b}{3 \cdot Q \cdot L} \tag{5}$$

The viscosity is normally determined using a capillary rheometer when the diameter, the length of the capillary and the pressure drop between the beginning and the end of the capillary (Bouafif et al. 2009) are known. This is called the apparent viscosity. To calculate the actual value of the viscosity the Bagley and Rabinowitsch corrections were used (Bagley 1957, Bociąga 2007, Friesenbichler et al., Rabinowitsch 1929). The Bagley correction improves the incorrect assumption that the cross-section of a capillary, through which the polymer flows are fixed. In fact, the pressures are measured before the inlet and at the end of capillary outlet. The cross-section changes affect the measured viscosity. By skipping the inlet losses, the experimental shear stress is overestimated in relation to their actual value. Taking into account the above-mentioned correction, the actual values of shear stress τ is calculated using the relationship:

$$\tau = \frac{p}{4\left[\left(\frac{L}{D}\right) + \left(\frac{L}{D}\right)_c\right]} \tag{6}$$

where: p - pressure,
$(L/D)_c$ - the ratio of the dimensions of the capillary,
where p = 0

For the studies the mold pressure drop was measured inside the mold cavity. There were not pressure changes resulting from cross-section changes of the cavity. The Bagley correction was not needed in this case.

The Rabinowitsch correction is based on the idea that when the polymer flows through the capillary there is no slip at the walls. In fact the slip, although sometimes small, takes place.

It is suggested to adopt a more flat profile of the polymer flow rate, which forces the use of this correction (Kloziński 2007a, Kloziński 2007b). Correction should, therefore, be made even in the form proposed in this research. The actual value of the shear rate was determined from the relationship:

$$\gamma_r = \frac{\gamma}{4} \cdot \left(3 + \frac{d \log \gamma}{d \log \tau} \right) \tag{7}$$

where: γ - shear rate,
τ - shear stresses.

The methodology of determining the viscosity curve in an actual process conditions consisted of the following steps:

a) setting of injection molding process parameters (temperature range, flow rate, pressure),
b) plastic injection into the cavity at set technological parameters and variable flow rate,
c) analysis of the pressure, recorded by the sensors,
d) calculation of the polymer viscosity based on the recorded pressure,
e) calculation of the viscosity curve parameters on the base of Cross-WLF model, using DataFit 9 commercial code,
f) determination of composite viscosity curve.

To determine the viscosity curve the 7-parameter of Cross-WLF mathematical model was used, which allows for rather high accuracy of experimental data approximation. In Autodesk Moldflow Insight commercial code, the 7-parameter model was used as a basic model describing the viscosity properties of polymers for many years. This allows to calculate the viscosity at any temperature. In this model the viscosity is a function of temperature, pressure and shear rate. Unfortunately, the basic problem of its use is the difficulty of defining the dependent parameters. In this model the viscosity of the polymer is determined by Cross equation (Dealy and Larson 2006, Kwiatkowski et al. 2011, Pötsch and Michaeli 2008):

$$\eta(\gamma, T, p) = \frac{\eta_0(T, p)}{1 + \left(\frac{\eta_0 \cdot \gamma}{\tau^*} \right)^{1-n}} \tag{8}$$

The zero viscosity η_0 was calculated on the base of William-Landel-Ferry (WLF) equation (Williams et al. 1955):

$$\eta_0(T, p) = D_1 \cdot \exp\left[- \frac{A_1 \cdot (T - T_g)}{A_2 + (T - T_g)} \right] \tag{9}$$

The parameters $\widetilde{A}_2$ =51.6 K, T_g=263.15 K, D_3=0 K/Pa of Cross-WLF model were determined using a differential scanning calorimeter DSC Q2000, TA Instruments and polymer data base, integrated with Autodesk Moldflow MPI 2013. To calculate the dependent parameters of Cross-WLF model, the DataFit 9 software of Oakland Engineering was used. The calculated coefficients of this equation allowed to determine the viscosity properties of composite over a wide range of shear rates. This makes it possible to carry out the numerical simulation of the injection molding to determine the effect of viscosity curve determining method on the plastic parameters in mold cavity. When one knows the parameters of the cross-WLF equation the viscosity of plastic in non-standard shear rates and temperature can be calculated. By applying this equation for different values of temperature and shear rate, the viscosity values were determined in non-standard conditions of the injection process.

RESULTS

Determination of viscosity curve

Based on the geometry of cavity and pressure and temperature data measured by sensors the shear stress and shear rate were calculated. The injection mold used in the study allowed to receive the data directly from the real process. The WPC specimens with 15% of wood fiber (WF) were injected changing the injection speed. The following flow rate were used: 10, 20, 40, 60, 80, 100 $cm^3 \cdot s^{-1}$. The experiments were carried out at four temperatures: 170, 180, 190 and 200°C. On the basis of the measured data (Tab. 1) the viscosity characteristics were plotted (Fig. 4).

Tab. 1: The viscosity test results obtained using the experimental injection mold.

Melt temperature (°C)	Flow rate ($cm^3 \cdot s^{-1}$)	Share rate (s^{-1})	Viscosity (Pa·s)	Standard deviation
T1 = 200	10	260	231.2	21.3
	20	600	132.1	13.5
	40	1081.5	84.5	9.2
	60	1595.9	63.9	5.8
	80	2128.1	49.8	6.5
	100	3845.9	30.5	3.4
T2 = 190	20	273.5	265.5	19.7
	40	756.8	126.8	10.5
	60	1995.3	60.5	6.3
	100	4178.3	32.3	4.8
	10	4285.8	30.5	3.7
T3 = 180	20	633.9	165.2	16.3
	40	1366	90.8	9.2
	80	2703.9	52.6	5.7
	100	4425.9	35.6	3.5
T4 = 170	10	312	331.9	25.3
	20	824	158.5	12.1
	40	1774.2	84.5	8.6
	60	3380.6	50.5	5.3
	100	4425.9	40.4	4.9

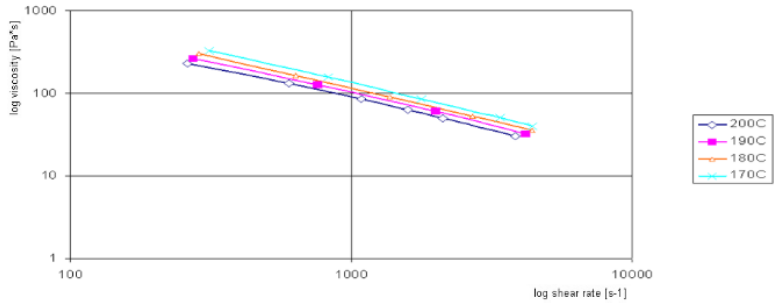


Fig. 4: The viscosity characteristics obtained using the experimental injection mold.

The viscosity curves used in the simulation are determined for a broader range of shear rates. The shear rates used in an injection molding process can reach $10^5 \cdot s^{-1}$. The shear rates lower than $10^2 \cdot s^{-1}$ are in this process rarely used, but they require the definition using a mathematical models. For the numerical calculations the 7-parameter mathematical model - Cross-WLF is often used. The parameters of this equation were calculated using the DataFit 9.0 software. The calculated model parameters are shown in Tab. 2. The calculated viscosity curves of WPC 15% WF composite are shown in Fig. 5.

Tab. 2: Parameters of Cross-WLF model for WPC with 15% WF.

Parameter	Unit	Value
n		0.2744
τ^*	Pa	31511.2
D1	Pas	1.3524e+10
D2	K	263.15
D3	K/Pa	0
A1		19.457
A2~	K	51.6
Determination coefficient -R^2	-	0.998

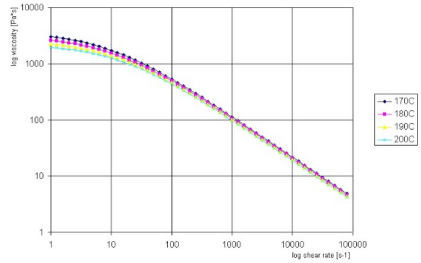


Fig. 5: The viscosity curves of the WPC with 15% of WF calculated using the Cross-WLF model at temperatures of 170°C, 180°C, 190°C and 200°C.

Determination of PVT properties

The pressure – volume - temperature properties of plastics in injection molding process are mainly used to control the packing phase. The variables in this phase are a packing pressure and plastic temperature. Thanks to these properties the specific volume of the polymer in the mold cavity is possible to calculate. The data on changes of specific volume allow to calculate, among others, the shrinkage of molded piece.

The packing time depends on the maximum rate at which the heat flux can be discharged from the polymer while maintaining a uniform cooling of the molded piece. For the composite containing 15% wood fiber the PVT properties were determined too. The capillary rheometer Smart Rheo 2000 of Ceast was used during the experiment. Theses characteristics were determined at temperature: 120, 140, 150, 155, 160, 170, 180, 190 and 200°C. The working pressures: 10, 20, 40, 80 and 100 MPa were applied. The resulting PVT properties of the composite containing 15% of wood fibers are shown in Fig. 6. The parameters of the mathematical model were then determined based on the Tait model (Kowalska and Sikora 2003, Pötsch and Michaeli 2008). These parameters are summarized in Tab. 3. The PVT properties used in the numerical simulation of the injection molding process are shown in Fig. 7. On the basis of the sample used to determine the PVT characteristics the density of the polymer was calculated in the solid state (0.953 g·cm^{-3}) and in melt state (0.796 g·cm^{-3}).

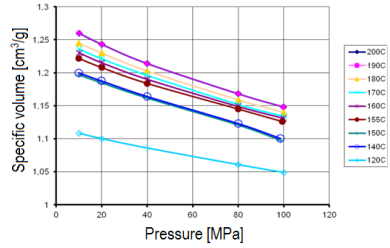


Fig. 6: The PVT properties of WPC with 15% WF based on experimental.

Tab. 3: Parameters of the Tait model for the WPC with 15 % of wood fillers.

Parameter	Unit	Value
b_5	K	423.15
b_6	K·Pa^{-1}	1.101e^{-007}
b_{1m}	m^3·kg^{-1}	0.001207
b_{2m}	m^3·kgK	9.13e^{-007}
b_{3m}	Pa	8.67312e^{+007}
b_{4m}	1/K	0.004739
b_{1s}	m^3·kg^{-1}	0.001102
b_{2s}	m^3·kgK	5.129e^{-007}
b_{3s}	Pa	1.61599e^{+008}
b_{4s}	1/K	0.004835
b_7	m^3·kg^{-1}	9.48e^{-005}
b_8	1/K	0.1489
b_9	1/Pa	2.112^{-008}

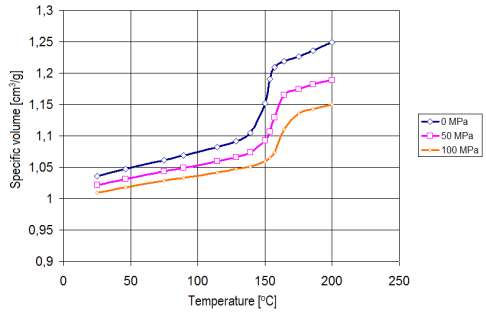


Fig. 7: The PVT properties of the WPC with 15% WF.

The composite properties verification

In order to verify the obtained viscosity curve and the PVT properties the numerical analysis of the injection molding process was performed and the received results were compared with the experiment. The simulation studies included the following stages: the 3D model designing, the model discretization by means of 3D tetra type finite elements (FE), the setting of initial and boundary conditions, carrying out the numerical calculations and results interpretations. The geometrical model was designed using NX 8 CAD software.

Based on geometrical model, the FE mesh was done using the Autodesk Moldflow Insight 2013 commercial code. The injection mold numerical model took into account both the runner and cooling systems.

The analyses were performed for model of 2 mm thickness which was shown in the Fig. 8.

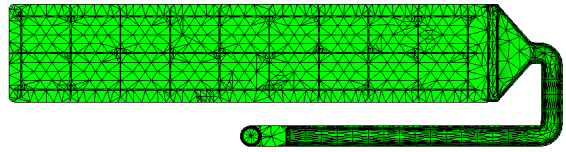


Fig. 8: The numerical model of the cavity (146 693 of finite tetra type elements).

The key moment during simulation was the introduction to software code the data describing the properties of processed composite. These data included both thermal, rheological and mechanical properties of processed composite. Then the injection molding process parameters were introduced.

Both in experimental and simulation studies, the injection molding process was analyzed under variable injection conditions, i.e. from impeded plastic flow (extremely low injection rate, mold temperature and plastic temperature and pressure) and vice versa, to flow easily due to low plastic viscosity.

One of the significant results of the numerical analysis, which enable verification of designated composite properties, is the plastic pressure profile inside mold cavity (Fig. 9). The pressure characteristics in the mold cavity were measured by means of sensors. The approved measuring points corresponding to the position of the sensors mounted in the cavity mold during the experimental study: point P1 - the point position closest to the gate, P2 - the point position in the middle of the cavity length, P3 - the point position at the end of the mold cavity.

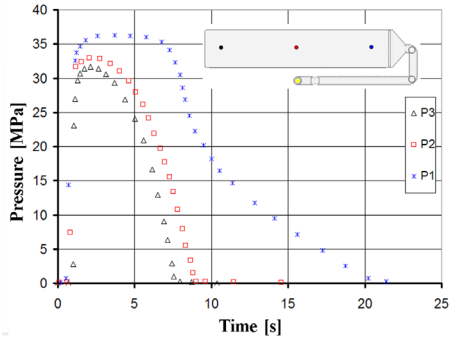


Fig. 9: The calculated pressure profiles at chosen nodes of model for the WPC with 15% WF at 170°C.

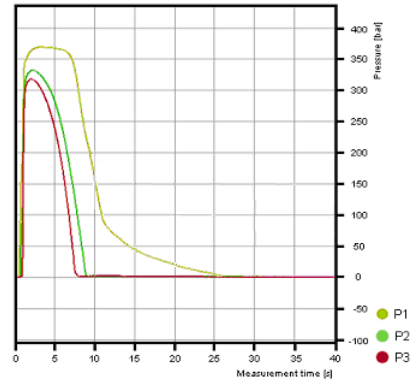


Fig. 10: The pressure profiles measured in the mold cavity for the WPC with 15% WF measured at the flow rate of 20 $cm^3 \cdot s^{-1}$ and melt temperature of 170°C.

For the analyzed cases the polymer flow and pressure profiles were registered by means of Priamus software. Fig. 10 shows the pressure profiles in the mold cavity for the WPC with 15% of WF at the chosen flow rate and 170°C of melt temperature.

DISCUSSION

The reliability of numerical simulations of the injection molding process depends mainly on the rheological characteristics of polymers or composites. The rheological characterization of WPC for injection molding is commonly carried out by a capillary rheometer. However, in this method the measurement is carried out on a sample of unprocessed polymer, neglecting, among others, the influence of the plastification phase. The use of capillary rheometers to determine the viscosity curve of WPC is also difficulty by the fact that the heterogeneity of the composite properties poses problems with the stabilization of the pressure profile recorded during its measurement, on the basis of which the stabilization area of the pressure signal is determined. This is confirmed by numerous works, (Klyosov 2007) among others. Pressure changes are dynamic and significant, what limits the possibility of determining the correct course of this rheological characteristic. This characteristic is simplified. The best properties provide in-line measurements (Kloziński 2016).

To improve the accuracy of rheological data by rejecting these simplifications, a laboratory test method has been developed for the physical simulation under industrial process conditions. The use for measuring the viscosity of the actual processing tool, i.e. the designed injection mold in this case, gives the possibility of in-line viscosity measurement. In practice, various technical solutions of this type are used (Bariani et al. 2007, Friesenbichler, and et al. 2011, Fernandez et al. 2014). These solutions confirm the possibility of determining the viscosity curve in real polymer processing conditions. Rheological measurements have been used to improve the accuracy of the results, in accordance with accepted principles (Kloziński 2007).

The proposed method is similar to an in-line slit-die rheometer which would be mounted as a mold on an injection molding machine. By means of this test it is possible too to relate a polymer viscosity not only to its temperature and shear rate but also to those parameters that characterize the plastication process: screw rotation speed, residence time, temperature and geometry of the system. The viscosity curves obtained in this way are consistent with the characteristics presented in the publications (Mazzanti et al.2015). The PVT properties were determined by a capillary rheometer. Their course is consistent with the data from literature (Golzar and Sadeghian 2007) for typical WPC.

The registered characteristics of measured pressure in selected injection mold cavity areas provide the data for verifying the viscosity curve and PVT properties.

The received pressure profiles correlate in a fairly good degree with numerically calculated profiles using Autodesk Moldflow MPI 2013. The significant difference is the shape of the profile in point P1. Furthermore, the actual pressure characteristics in points P2 and P3 are more lowered than characteristics in the point P1. The maximum pressure is lower for this case. Pressure values from the numerical calculations are a little bigger. The maximum pressure difference between experiments and calculations in (for point P1) for WPC with 15 % WD injected at 170 ˚C was up to 2 %.

CONCLUSIONS

Each proposal of a new product must be preceded by a comprehensive analysis, including assessment of manufacturability and analyzes in the field of manufacturing technology, where a huge role take place the numerical simulations of manufacturing process.

The correct simulation results are not possible without well defined material properties. The viscosity curves, as major polymers characteristics used in their processing simulation, are determined mostly by rheometer. In the case of heterogeneous materials, including WPC, the determination of these rheological properties is very difficult to obtain due to the inhomogeneity of the composite structure. The proposed methodology to determine the viscosity curves of WPC allowed to overcome the barriers with using standard methods.

The experimentally obtained pressure profiles in selected locations of mold cavity allow to verify the viscosity curve characteristic.

The well defined PVT properties, important in packing phase simulations of injection molding process, allow exactly to calculate the shrinkage of molded piece made of composite.

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Wiesław Frącz*, Grzegorz Janowski
Rzeszow University of Technology
Faculty of Mechanical Engineering and Aeronautics
12 Powstańców Warszawy Ave.
35-959 Rzeszów
Poland
* Corresponding author: wf@prz.edu.pl
Phone : +48 17 865 17 14