Polycarbonate melt shear viscosity in wide range shear rate

G Toth¹, A Bata¹ and K Belina¹

¹Department of Materials Technology, GAMF Faculty of Mechanical Engineering and IT, Pallasz Athéné University, Kecskemét, Hungary

E-mail: togoagc@gmail.com

Abstract. In our work we present an investigation of polycarbonate melt shear viscosity in wide range shear rate. In the course of our research we aggregated more testing method results and mathematically determined viscosity equation of the tested material.

1. Introduction
The majority of the processing thermoplastic polymers is in melt condition. For example, extrusion, or injection moulding which are the two biggest plastic processing technology. Nowadays there is a need to simulate these processes. The simulations are help for the plastic product and mould designers. In these simulations one of the most important internal parameter the shear viscosity what depend on the temperature, pressure, molecular weight or structure. So easy to see how a top priority precise knowledge of the shear viscosity of plastic melts [1].

The shear viscosity basically is a rheological property. Some references are describing the rheology than science which describe the deformation of materials. For a more detailed description the rheology establishes the relationship between a body deformation and the tensions and their changes over time. Also explore the dynamic (stress) and kinematics (deformation) variables linking the material characteristics of the status and structure of the material impact [2]. In this paper we narrow our experimental to research rheology area.

The polymer materials science one of the youngest sciences, but it serves with more and more results for over 40 years thanks to a variety of polymers, material structure and chemical structure. Day by day there is continual development of new equipment, the researchers get more accurate, faster, more easily interpretable results. In parallel with the rheological studies show evidence for these processes are constantly adding new results.

In our work we tested polycarbonate (DOW Calibre 303 EP) melt shear viscosity in wide range of shear rate (0.05 s⁻¹ – 240 000 s⁻¹), with variable temperature steps. It is a thermoplastic water clear polycondensation type plastic. In the course of our research we summarized the results of several test methods are sections covering each other in good agreement. The procedures used were the rotational viscometry and measurement techniques based on the principle of capillary viscometry. After evaluating the results of those tests, information is given about the tested polycarbonate shear viscosity dependence on the shear rate and temperature. From the crude results we are defined the Cross viscosity equations (with three variables), on the temperature steps. The results are aggregated and summarized in a diagram. It shows viscosity curves in wide range shear rate around the processing temperature.
2. Method
Our results we got from three shear viscosity investigation methods, which covers a wide range of shear rate and temperature steps near the processing temperature. These were in the low shear rate range of the rotational viscometry (0.05 – 30 s\(^{-1}\)), in the middle-high range of the capillary viscometry (100 – 10,000 s\(^{-1}\)) and in the high range we used a special technique (10,000 – 240,000 s\(^{-1}\)) which was based on the capillary viscometry method.

2.1. Rotational viscometry
With the rotational viscometry we could measure torsion flow in polymer melt between parallel plates. The measuring technique conceptual layout is shown in Figure 1. The CMT mean combined motor and transducer concept.

![Figure 1. Rotation viscometry CMT conceptual layout and marking system [3]](image)

From the marking system the stress, deformation and deformation rate components can be calculating with the equations described below (1, 2, 3).

\[
\sigma = \frac{2}{\pi r^3} \times M
\]  

(1)

\[
\gamma = \frac{r}{h} \times \Theta
\]  

(2)

\[
\dot{\gamma} = \frac{r}{h} \times \Omega
\]  

(3)

Where:
- \(r\) - circle plate radius;
- \(h\) - gap between the plates;
- \(\Theta\) - driving engine rotation (rad);
- \(\Omega\) - driving engine angular speed (rad/s);
- \(M\) - torque (\(\mu\)Nm)

With the equations (1, 2, 3) the stress and deformation rate can be calculated from these considering the shear viscosity definable. It is a frequent measuring technique to determine shear viscosity for small molecular liquids, or oils and thermoplasts at low shear rate. With this method we can measure on higher temperature (to 600°C) and perception degradation process.

For our testing, we used SMT (Separate motor and transducer) conceptual TA Ares G2 type rotation viscometer with 25mm parallel circle plates and 0.4mm gap. In this type of rotation viscometer, the drive engine and the transducer are separated.
2.2. Capillary rheometer
The capillary rheometer is a measuring device in which we are able to measure thermoplastic shear viscosity.

50-10 000 s⁻¹ shear rate ranges near to the processing temperature [4]. We used a Goettfert Rheograph 25 capillary rheometer. The measuring method is based on continuous melt flow in known geometry capillary(s) while the pressure drops measured between capillary inlet side and atmospheric pressure (Figure 2.).

![Figure 2. Capillary rheometer conceptual layout](image)

The variable parameters are the barrel temperature and piston speed (parallel with the deformation rate). In this case, the shear viscosity depend on temperature and deformation (shear) rate. In our test the shear rate changed between 100-10,000 s⁻¹ and the barrel temperatures changed in three steps around the material supplier recommended process temperature. The measuring method use two other geometry capillaries because of the entrance and exit effects (pressure drop). With this equipment in one step we can do the Bagley correction (correct the pressure drop) which gives the real shear viscosity.

2.3. Special capillary technique
This special capillary technique equations are the same as the capillary viscometry, but we used an injection machine’s injection unit for the measurement, with changeable piston (screw) speed and barrel temperatures. An injection units piston can be faster than a measuring equipment and work on higher pressures. We used an ENGEL Victory 1050/300 injection-moulding machine and two special geometry capillary nozzles. In this case the injection machine was used as a capillary rheometer. With the injection-moulding machine repeatedly plasticating and injecting with varied injection settings through of the nozzles into the open air. Variables adjusted were for example the injection speed and
the temperatures of the barrel zones. With this method the effect of temperature and injection speed on the viscosity can be shown.

The capillaries used were very important in terms of measurement because this gave us the possibility to calculate the corrected viscosity parameters. The main difference between the two capillaries were the length and diameter ratio. The diameter is unified (2mm) and the length was changed, one is very short just 2mm and another 20mm (Figure 3).

![Figure 3. Geometry of the capillaries [5]](image)

This difference is necessary for applying the following Bagley correction. During capillary rheometer tests, pressure is measured above the die inlet. The true pressure drop along the capillary is therefore ‘hidden’ by an additional pressure drop at the entrance of the die, where the flowing material goes from a wide reservoir (the main cylinder or barrel) to a narrow capillary, possibly also creating turbulences. Assuming that the same additional pressure drop takes place with different capillary lengths (but keeping constant the barrel and capillary diameters, and inlet shapes), it’s possible to correct the pressure reading and estimate much more accurately the true pressure drop [7].

3. Results
Measurements were carried out at three different temperatures. Figure 4 shows the viscosity curves measured by different method. It can be clearly seen that the whole measured range covers almost eight scales.

The primer data were processed by the Cross equation [6]:

$$\eta = \frac{\eta_0}{1 + \left(\frac{\eta_0\dot{\gamma}}{\eta}\right)^{1-n}}$$

where

- $\eta$ - viscosity (Pa*s);
- $\dot{\gamma}$ - deformation rate (s$^{-1}$);
- $\eta_0$ - zero shear viscosity (Pa*s);
\[ \tau^* \]
- stress limit between the Newtonian and non-Newtonian region (Pa)
- slope of the transient region (-)

Figure 4. Shear viscosity curves separated by measuring methods

According to our results, the different methods can be merged, and the resulted viscosity curve describes the shear flow of a polymeric melt in very wide deformation rate range.

Figure 5. Shear viscosity curves calculated by Cross equation
The main advantage of this method is in the simulation. Using only one measuring method, the extrapolation gives around 15% deviation from the measured values. In our method the maximum deviation was only 5%.

4. Conclusion
Our measuring techniques can be used in the range 0.05 l/s – 240 000 l/s. Rotational viscosity covers the Newtonian region of flow [6-8], the capillary viscometry covers the transient region, the instrumented mould covers the non-Newtonian region. It was shown that the heat generated by the shear force has significant effect on the measuring temperature from around 50 000 l/s. Cross equation describes well the overall viscosity curve shows less than 5% deviation.

Acknowledgments
This project consumed huge amount of work, research and dedication. Still, implementation would not have been possible if we did not have a support of many individuals and organizations. Therefore, we would like to extend our sincere gratitude to all of them. We are also grateful to University of Miskolc, Department of Polymer Technology for provision of expertise, and technical support in the implementation. Without their superior knowledge and experience, the Project would lack in quality of outcomes, and thus their support has been essential. Nevertheless, we express our gratitude toward our families and colleagues for their kind co-operation and encouragement, which help us in completion of this project.

References
[1] T Czikovszkzy, P Nagy and János Gaal 2004 A polimertechnika alapjai, Műegyetemi Kiadó, Budapest http://www.tankonyvtar.hu/hu/tartalom/tkt/polimertechnika-alapjai/adatok.html
[2] L Halasz, I Molnar and I Mondva 1978 A polimerek feldolgozásának reológiai alapjai, Műszaki Könyvkiadó, Budapest (in Hungarian)
[3] TA instruments factory Ares G2 rotational viscometer equipment education material – Study of rheology theory and applications; http://www.tainstruments.com
[4] A Szűcs 2010 Műanyag ömledékek nagysebességű áramlásának tanulmányozása, PhD thesis, University of Miskolc (in Hungarian).
http://www.kerpely.uni-miskolc.hu/downloads/docs/cikkek/szucsandras-ertekezes.pdf
[5] G Toth and T Szabó 2015 Polymer melt viscosity measuring by an injection machine GARDUS; 2 (2) ISSN: 2064-8014; 112 http://gradus.kefo.hu/index.php/gradus/article/download/203/218
[6] F.N.Cogswell 1981 Polymer Melt Rheology, London
[7] G E Baumann and S Steingiser 1963 Journal of Plymer Sci. Part A 1 3395
[8] F Yang 1996 Polymer Eng. and Sci. 37 (1)