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The universal temperature parameter of rigid PVC gelation in Brabender kneader

RAPID COMMUNICATION

Summary — Course of gelation of rigid PVC Polanvil S of *K*-value 52, 58, 61, 67 or 70, in the chamber of Brabender kneader at temperature range 160—185 °C and rotation speed of the rotors 10—50 min^{-1}, has been investigated. On the basis of torque and temperature changes as functions of process time the curves of torque changes versus real temperature of gelating blend were received. It has been observed that maximal torque value always occurred in the narrow temperature range, independently on chamber wall temperature or shear rate, and was dependent on PVC *K*-value. It was found that temperature related to torque maximum (T_{EG}) could be universal parameter characterizing PVC gelation. The proposed new way of analysis facilitates more precise comparison of torque curves obtained at various set temperature and rotational speeds of rotors.

Key words: poly(vinyl chloride), *K*-value, gelation, torque curve, torque maximum temperature, universal parameter.

The rigid PVC compositions are mainly processed by extrusion. The properties of the products strongly depend on PVC *K*-value, the type of additives used, and on the processing conditions [1—6]. For the estimation of the processing ability of PVC composition, and for the analysis of gelation effect the rotary rheometers with measuring mixers are frequently used. In this measurement technique the apparatus starts to record the torque and the temperature changes, as functions of time, just after loading a definite quantity of PVC composition into the hot chamber of the kneader.

A run of the typical PVC torque curve, as observed by means of Brabender and/or Haake rheometer, was often discussed in the literature [7—10]. This kind of analysis of the torque is specially useful when laboratory tests of new PVC compositions are realized, though usually the temperature of the processed composition is not taken into consideration [11, 12]. It has to be stressed that during the processing the temperature of PVC composition generally differs from those settled as invariable temperature of the chamber walls [13]. On the contrary, from the analysis of the gelation of the rigid PVC composition it follows that its final structure, after processing, mainly depends on the real temperature of the polymer [14].

The main task of our research was to study the processing induced temperature development of PVC of various *K*-value, and to relate the changes of the torque to the temperature and shearing. In this work a new procedure of the torque data analyses was used.

EXPERIMENTAL

Materials

The compositions of 100 parts of PVC Polanvil S (Anwil SA, Włocławek, Poland) of different *K*-values: 52, 58, 61, 67 or 70 [bulk densities (in g/cm^3) 0.611, 0.582, 0.573, 0.529 and 0.509, respectively], 4 parts of the tinorganic stabilizer MOK Mark 17 (Akcros) and 1 part of the paraffin — wax Loxiol G22 (Henkel) were used.

In the further text the numbers in the symbols of samples investigated refer to *K*-values of PVC used, *e.g.* PVC S-52, PVC S-67, *etc.*

Methods

The composition was processed in the chamber of the Brabender kneader (Plasti-Corder Pl 2200-3) at temperature range between 160 and 185 °C, and rotation speed of the rotors between 10 and 50 min^{-1}. The temperature sensor, isolated thermally from the chamber wall, was located in the bottom plate, in the center position between both rotors. The mean value of the shear rate was estimated for the highest and the lowest shearing, according to Schramm [15], taking into account, in both cases, only the flow of the polymer in the radial direction. In this study we neglected the effects related with chamber loading.

RESULTS AND DISCUSSION

Typical rheograms for sample PVC S-67 taken as an example are shown in Fig. 1 and 2.

An immediate increase in temperature of the processed PVC composition, by heating from the chamber wall, is observed (Fig. 1, curve 2). Subsequently, a slow temperature rise, corresponding to the transitory equilibrium state between sliding of broken grains and increasing degree of PVC gelation (point B) [10] is followed by a faster temperature increase.

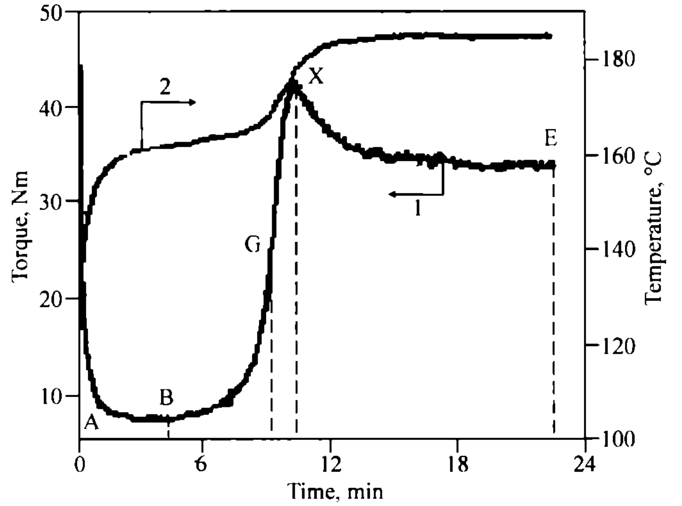

Fig. 1. Torque curve of Polanvil PVC S-67 composition processed at temperature 160 °C and shear rate 13 s^{-1}: 1 — torque, 2 — temperature; A — point of loading, B — minimum point, G — point of inflexion, X — maximum point, E — end of the test

In the range between the inflexion point (Fig. 1, point G) and the maximum at the torque curve (point X) a rapid shear induced growth of PVC composition temperature, up to 20 °C above the temperature of the chamber walls, may be noted. In spite of the decrease in the torque value, the temperature of PVC composition does not change any more. Other torque curves (for PVC of various *K*-values) show similar courses, although the

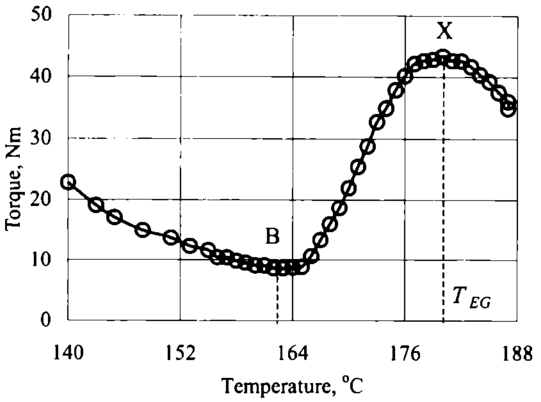

Fig. 2. Torque of Polanvil PVC S-67 composition processed at temperature 160 °C and shear rate 13 s^{-1} as a function of the real composition temperature; T_{EG} — temperature of maximum torque value; for another symbols see Fig. 1

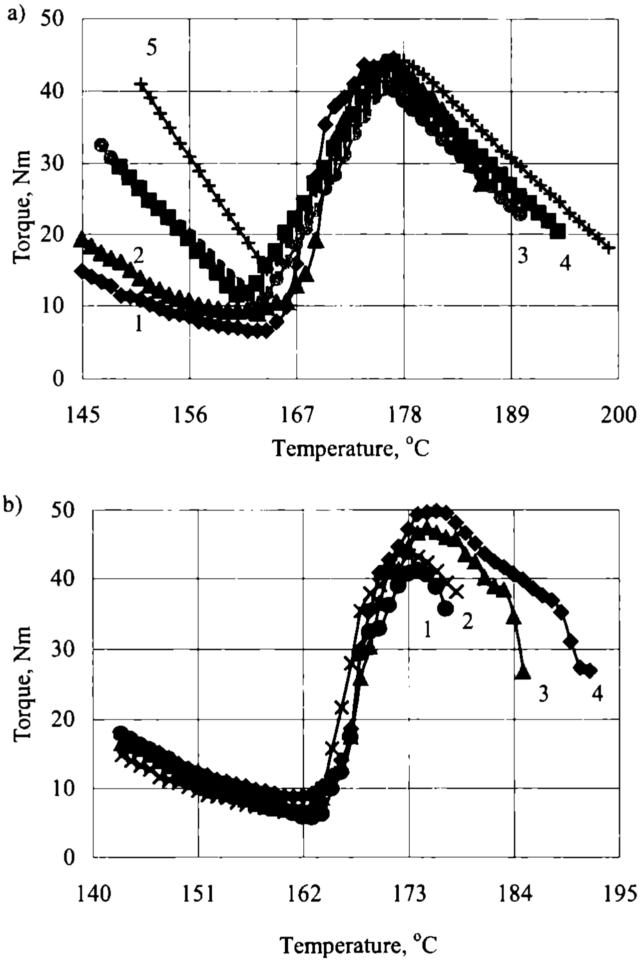

Fig. 3. Torques of Polanvil PVC S-61 as the functions of real composition temperature a) constant shear rate 13 s^{-1} and different chamber temperature: 1 — 160 °C, 2 — 170 °C, 3 — 175 °C, 4 — 180 °C, 5 — 185 °C; b) constant chamber temperature 160 °C and different shear rates: 1 — 9 s^{-1}, 2 — 13 s^{-1}, 3 — 17 s^{-1}, 4 — 26 s^{-1}

lower is *K*-value the shorter is the time of the process (position of the point X on the torque curve — Fig. 1).

The maximum of PVC S-61 torque, as a function of the real compound temperature, for both experimental conditions, *i.e.* various chamber temperatures and constant shear rate (Fig. 3a), and for various shear rates and constant temperature of chamber wall (Fig. 3b), appears always in the temperature range between 175 and 178 °C. The torque curves as functions of the real compositions' temperature, for PVC of various *K*-values, during processing at 160 °C and shear rate 13 s^{-1} are presented in Fig. 4. So, the most evident gelation effect, *i.e.* the real composition temperature corresponding to the maximum at the torque run, depends on PVC *K*-value. Namely we have observed: 158—160 °C for *K* 52; 167—169 °C for *K* 58; 174—177 °C for *K* 61; 179—182 °C for *K* 67 and 184—187 °C for *K* 70.

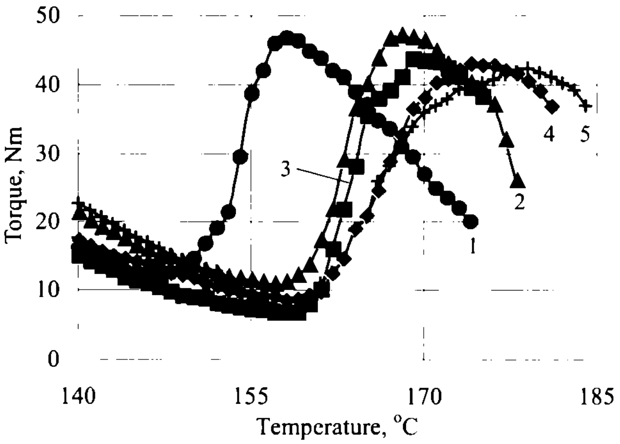

Fig. 4. Torques of Polanvil PVC S compositions of various K-values, processed at 160 °C and shear rate 13 s^{-1}, as the functions of the real composition temperature; K-value: 1 — 52, 2 — 58, 3 — 61, 4 — 67, 5 — 70

From the analysis of the relationship of the torque and the real composition temperature it follows that for PVC with defined *K*-value the maximum of torque appears in the same temperature, independent on the temperature of the chamber wall and on the rotation speed of the rotors (variable shearing in the mixing chamber). This effect is probably related to the mechanism of PVC gelation in the kneader [9]. Up to the processing time when PVC virgin grains start to be progressively disintegrated, its interfriction and simultaneous gelation leads to an increase in the temperature and in the torque to its certain maximum. After that the temperature still increases, and the torque decreases to the equilibrium states attained by these values.

CONCLUSION

We have found that the maximum of torque, related to the most evident PVC gelation effect, appears in the narrow temperature range, strongly dependent on PVC *K*-value. The range of temperature of the maximal torque value T_{EG} is practically independent on the temperature of the chamber wall and on the rotation speed of the rotors. Based on these results it may be supposed that this temperature presents an universal parameter allowing to estimate PVC gelation appearance. Probably, this is also the best temperature for the PVC processing by extrusion. This new methodology of torque analysis facilitates the comparison of the torque data obtained in various conditions of the temperature and shear rate.

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