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The influence of the chamber temperature in the Brabender measuring mixer on the state of equilibrium of the torque of rigid poly(vinyl chloride)

RAPID COMMUNICATION

Summary — The analysis of the plastograms obtained during kneading of poly(vinyl chloride) (PVC) compound in the Brabender mixing chamber (Plasti-Corder Pl 2200-3) in temperature range 160—200 °C, and rotation speed of the rotors from 5 to 40 min^{-1} has been presented in this paper. From the analysis of characteristic values of the torque at equilibrium state (M_E), the time necessary to reach equilibrium (t_E) and the real temperature of the compound at this state (T_E) it results that the mechanical charge decreases with the rise of chamber temperature (T_{ch}). It has been found that the shear rate ($\dot{\gamma}$) and T_{ch} values, within the tested range, influence the time of the occurrence of the final stage of gelation namely since time of reaching of maximum torque and the real temperature of PVC compound.

Key words: poly(vinyl chloride), gelation, kneading, torque curve.

WPŁYW TEMPERATURY KOMORY PLASTOGRAFOMETRU BRABENDERA NA STAN RÓWNOWAGI MOMENTU OBROTOWEGO NIEPLASTYFIKOWANEGO POLI(CHLORKU WINYLU)

Streszczenie — Przedstawiono wyniki badań ugniatania nieplastyfikowanej mieszaniny poli(chlorku winylu) (PVC) w czasie potrzebnym do osiągnięcia równowagi momentu obrotowego (punkt E na plastogramie). Ugniatanie prowadzono w komorze plastografometru Brabendera Plasti-Corder Pl 2200-3 w zakresie temperatury 160—200 °C z prędkością obrotową rotorów 5—40 min^{-1}. Z analizy zmian wartości momentu obrotowego w stanie równowagi (M_E), czasu potrzebnego do osiągnięcia równowagi (t_E) i rzeczywistej temperatury mieszanki w tym stanie (T_E) (rys. 1) wynika, że obciążenia mechaniczne maleją wraz ze wzrostem temperatury komory (T_{ch}) (rys. 2) o czym świadczy gwałtowny spadek momentu obrotowego. Stwierdzono, że szybkość ścinania ($\dot{\gamma}$) i wartość T_{ch} wpływają jednocześnie na czas trwania ostatniego etapu żelowania mieszaniny PVC tzn. od chwili osiągnięcia maksymalnego momentu obrotowego (rys. 3) oraz na rzeczywistą temperaturę żelującej mieszaniny (rys. 4). Wpływ $\dot{\gamma}$ na wartość T_E mieszaniny PVC w stanie równowagi momentu obrotowego jest szczególnie istotny przy najniższej nastawionej temperaturze komory i najwyższej szybkości ścinania w zakresie stosowanych w pracy.

Słowa kluczowe: poli(chlorek winylu), żelowanie, ugniatanie, plastogram.

The rheometric investigations, realized commonly by means of the Brabender and Haake devices, are usually carried out until the torque reaches an equilibrium state or/and until the polymer will undergo a certain destruction. For the clarification of poly(vinyl chloride) (PVC) gelation effects, the compound may be discharged from the kneading chamber and applied for further investigation, in order to reveal the state of gelated PVC compound.

According to the papers [1—4], the state of equilibrium of torque, measured during kneading by means of Brabender or Haake rheometer, corresponds to the time when the melt becomes homogeneous, and as a consequence the equilibrium between the heat of the friction and the temperature of the chamber is achieved [3]. Shah [4] stated that the further kneading of PVC compound, even after the time of the maximum torque, leads only to a certain heating of the compound, and consequently to an increase in temperature of the polymer, followed by a decrease in viscosity of the melt. The lowering of the viscosity of the melt may be reached by the stabilization of torque which achieves the state of equilibrium. Hawkins [2] claims that at equilibrium period, visible at the processing curve, the melt is homogeneous and it is the state required for fabrication. In this circumstances the torque

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run should be smooth, its level and the melt temperature are stable. Portingell [5] noted also that the torque level, after the maximum point on the torque—time curve, is influenced by the flow properties of the melt.

In our previous papers [6—8] we have discussed another characteristic points of the Brabender plastograms, *i.e.* the minimum of torque (point B), the inflection point G and the maximum of torque, called point X. In the last paper [8] we have stated that the real temperature of the compound, considerably different from the temperature of the kneader chamber walls, significantly influences the PVC structure and the degree of gelation of the compound processed to the point X. The intensive self heating of the compound takes place especially by a low chamber temperature, and by high shear rate.

Therefore, in this paper the main interest will be given to the equilibrium state of the torque, called point E. The effects occurring in the PVC compound, processed in the Brabender kneader until the time of reaching the equilibrium state of the torque, will be analyzed.

EXPERIMENTAL

Material

The material used in this study was the compound containing PVC S-61 Polanvil (M_n = 47 500, M_w/M_n = 2.25), produced by Anwil Włocławek (Poland) (100 weight parts) mixed with tinorganic stabilizer MOK Mark 17M, produced by Acros (4 weight parts), and paraffin wax Loxiol G22, produced by Henkel (1 weight part).

Processing

The compound was homogenized using a two-stage hot-cold mixer Kontinomix 40/80, produced by Mixaco, operating at mixing speed 1500 min^{-1}. The dry blend compound (weight of 54 gram) was processed in the Brabender mixing chamber (Plasti-Corder Pl 2200-3) at the temperatures (T_{ch}) of 160, 170, 175, 180, 185 and 200 °C, respectively, and the torque values were registered as a function of time. The processing was performed always up to the time when the equilibrium state of torque was achieved, *e.g.* point E on the torque-time curve (for the description of the characteristic points at the torque curve see [6—9]). The rotation speeds of the blades were: 5, 10, 20, 30 or 40 min^{-1} and the applied friction was 1:1.5.

The shear rates ($\dot{\gamma}$ = 2.18, 4.34, 8.69, 13.03, 17.38 s^{-1}) were evaluated as the average values from the highest and the lowest shearing, according to Schramm [10], taking into account only the radial melt flow direction. The real temperature of the compound was determined by means of resistance temperature detector situated between the rotors, directly in the kneaded PVC compound, isolated from walls of the chamber.

RESULTS AND DISCUSSION

In the case of PVC processing where the gelation takes place, the run of plastograms is generally similar and corresponds principally to literature data [6, 11].

Typical plastogram for rigid PVC is presented in Fig. 1.

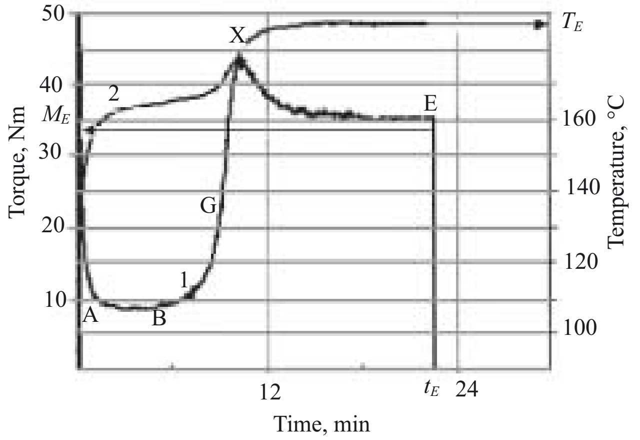

Fig. 1. Typical torque rheometer process curve of rigid PVC: 1 — torque, 2 — temperature; M_E — torque at the equilibrium state, T_E — real temperature of the compound at point E, t_E — the time necessary to reach the point E

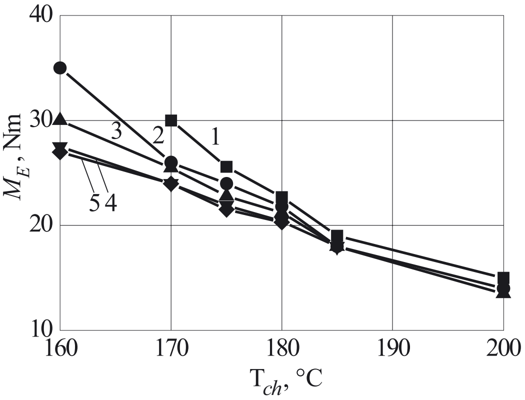

Fig. 2. Torque at the equilibrium state (M_E) as a function of the chamber temperature (T_{ch}) at various shear rates: 1 — 2.18 s^{-1}, 2 — 4.34 s^{-1}, 3 — 8.69 s^{-1}, 4 — 13.03 s^{-1}, 5 — 17.38 s^{-1}

On the basis of such figures we have analyzed the values of torque at the equilibrium state (M_E), the time necessary to reach the point E (t_E) and the real temperature of the compound at this point (T_E).

The value of M_E is related to the temperature of the kneading chamber (T_{ch}). Dependences of M_E on T_{ch} for different $\dot{\gamma}$ are shown in Fig. 2. The highest values of M_E (from 27 to 35 Nm) are characteristic for the compound processed at T_{ch} = 160 °C. The rise of T_{ch} causes a considerable decrease in M_E, to the value between 12 and 14 Nm at 200 °C.

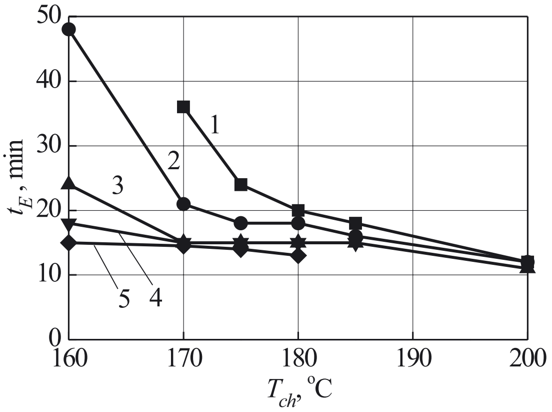

Fig. 3. Time of reaching the equilibrium state of torque (t_E) as a function of the chamber temperature (T_{ch}) at various shear rates: 1 — 2.18 s^{-1}, 2 — 4.34 s^{-1}, 3 — 8.69 s^{-1}, 4 — 13.03 s^{-1}, 5 — 17.38 s^{-1}

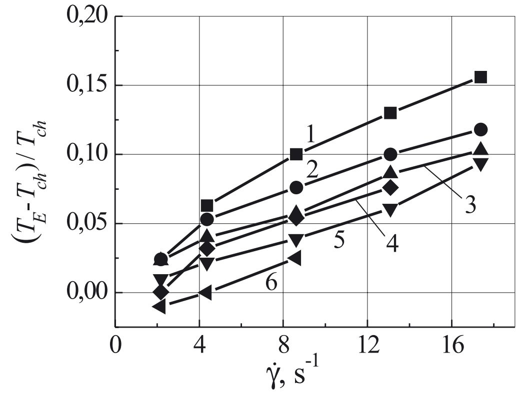

Fig. 4. The relative increment of the real temperature of PVC S61 compound at equilibrium state of torque as a function of the shear rate ($\dot{\gamma}$) at various chamber temperatures: 1 — 160 °C, 2 — 170 °C, 3 — 175 °C, 4 — 180 °C, 5 — 185 °C, 6 — 200 °C

The most significant influence of $\dot{\gamma}$ on M_E value may be observed in the case of kneading at the temp. 160—175 °C. For higher T_{ch} the M_E values practically do not depend on the shear rate. The similar dependence was noticed for the time t_E of reaching of the equilibrium state of torque presented in Fig. 3.

The higher T_{ch} value, the shorter the time of the attainment of the state of torque equilibrium. Longest t_E value, about 48 min, was observed for the compound processed at T_{ch} = 160 °C, and for $\dot{\gamma}$ = 4.34 s^{-1}. On the contrary, at T_{ch} = 160 °C and $\dot{\gamma}$ = 17.38 s^{-1} the time of the equilibrium state of torque (13—15 min) was similar to the t_E observed at the temperature between 170 °C and 185 °C for $\dot{\gamma}$ = 8.69 s^{-1}.

The relative growth of compound temperature at equilibrium state $(T_E - T_{ch})/T_{ch}$ as the function of $\dot{\gamma}$ for various T_{ch} shown in Fig. 4 is approximately linear. The most significant growth was observed for the compound processed at $\dot{\gamma}$ = 17.38 s^{-1} and T_{ch} = 160 °C.

CONCLUSIONS

The investigations of the PVC compound gelation, processed to the time of reaching the equilibrium state of the torque, allows to formulate the following conclusions:

— the shear rate and the chamber temperature influence both, the real compound temperature and the residence processing time;

— an increase in the chamber temperature and shear rate leads to shorten time of reaching of equilibrium state of torque, in the whole range of temperature and shear rates;

— the mechanical charge decreases with the rise of chamber temperature (the abrupt decrease in M_E values);

— the shear rate significantly influence the real compound temperature at the equilibrium state; an observation concerns especially the lower adjusted chamber temperature.

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