Research of heat and mass transfer parameters at drying poly (ethylene terephthalate) granules

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Abstract. In this article, drying characteristics were studied for polyethylene terephthalate (PET) granules. For data analysis, a modified quasistationary method was used. The values of the reduced time and the hydrodynamic intensity index were obtained for the generalized drying curve of the entire drying process in the temperature range from 120 to 185°C. The duration of the first drying period of PET granules was determined for all experiments.

1. Introduction
For a stable process of molding products from poly (ethylene terephthalate) (PET), it is necessary that the moisture content in the polymer granulate does not exceed 0.005-0.01% (mass) [1]. Therefore, a drying process for these polymers is necessary.

It is known [2, 3] that the entire drying process can be divided into two periods: the period of constant drying speed and the period of decrease in drying speed. Sometimes they are preceded by the material warming. The drying process in the first period (constant rate) is characterized by an intensive decrease in the material moisture content and can be described by the linear dependence \( w(\tau) \). In the first drying period, it is mainly the surface moisture that is removed, and in the second it is the inherent moisture. Therefore, during the second drying period, the rate of change of moisture content of the material decreases.

It was previously shown [6] that drying PET in shaft dryers technologically makes sense. In this case, it is recommended to heat the material in the mold in front of the dryer or in a separate compartment to increase the crystallinity of the polymer. This recommendation is follows from the fact that at the stage of heating, the crystallization process occurs, which is necessary to increase the glass transition temperature of the polymer.

The glass transition temperature of isotropic amorphous PET is in the range \( t_c = 60-70 °\text{C} \), and the crystalline glass transition temperature is \( t_c = 230-240 °\text{C} \). Thus, the orderliness (crystallinity) of the structural elements of the granulate eliminates the danger of adhesion (agglomeration) of particles in the zone of loading it into the extruder [1].

The necessary indicators of the granulate at the beginning of the first drying period should be:
- the degree of crystallinity of the polymer, \( \alpha_k \), equal to 0.4;
- granulate temperature above 120 °C.

By the end of the first drying period, the moisture content should not exceed 0.03% (mass) [6].
The intensity of drying in each of the periods can be influenced by various factors. Therefore, the task of determining (clarifying) the duration of drying periods to search for optimal technological solutions is relevant.

2. Research methods
To calculate the productivity of the entire drying process, a modified quasi-stationary method (MK method) is used [4]. The parameters of the MK method are used to describe the mass transfer process: dimensionless complex of MR concentrations; characteristic time \( s \), hydrodynamic parameter \( m \), and drying rate \( N \).

The dimensionless concentration complex MR (moisture ratio) is necessary to find the kinetic parameters \( s \) and \( m \), since, on the one hand, it characterizes the physics of the process.

\[
MR = \frac{w - w_p}{w_0 - w_p}
\]  

(1)

Here \( w \) is the moisture at the considered time, \%;
\( w_p \) is the equilibrium moisture, \%;
\( w_0 \) is the initial moisture, \%.

On the other hand, this allows taking into account additional drying conditions:

\[
MR = \frac{1}{1 + \left( \frac{\tau}{s} \right)^m}
\]  

(2)

Here \( \tau \) is the time, min;
\( s \) is the characteristic time, depending on the set of properties of the dried material and the drying agent, min;
\( m \) is the hydrodynamic parameter, depending on the speed of the medium relative to the processed material.

In this work, using the MK method, the drying process is analysed for three types of granules at different temperatures. For each experiment, kinetic parameters \( s \) and \( m \) are obtained and kinetic curves are constructed.

Three pilot batches of granules are prepared by the method of underwater granulation. The granules have the shape of a cylinder with an elliptical cross section. To perform the calculations, diameters of a ball have equal size [6]. The obtained three types of samples of granules with equivalent diameters are: sample 1 – 4.16 mm, sample 2 – 3.3 mm, and sample 3 – 2.98 mm. The experiments are carried out at temperatures of 120 °C, 140 °C, 160 °C, 180 °C and 185 °C.

Based on the data obtained, a period of a constant drying rate is determined; it is shown with a straight line on the graph \( w (\tau) \) (period I in Fig. 1). The duration of this period is determined as the length of the projection of the straight section.
Figure 1. The kinetics of drying of sample 1 at a temperature of 120 °C.

For example, at a temperature of 120 °C, the rectilinear section will be located in the time interval from 60 to 93 minutes (Fig. 1). Therefore, in the figure, I is the first drying period, and II is the second drying period.

3. Results
As a result of data processing, it has been found that the kinetic parameters m in the mathematical description of the entire drying process depend on temperature and on the size of the granules. (fig. 2).

Figure 2. The dependence of the hydrodynamic index m on temperature t.
Figure 3. The dependence of the characteristic time $s$ on temperature $t$.

For the kinetic exponent $s$ (Fig. 3), no clear dependence on temperature and granule size has been found. However, the scatter of the values of the exponent $s$ from the average is small, which allows taking it as a constant equal to 94.6 min.

The durations of the first period (the period of constant drying rate) for each experiment have been determined (Fig. 4). An analysis of the results shows that the shortest time of the first period is characterized by 18 minutes of sample 3 at a temperature of 185 °C, and the largest (50 minutes) for sample 1 at a temperature of 120 °C.

Figure 4. Dependence of the duration of the first drying period (period of constant drying speed) of granules on temperature.
Thus, the duration of a constant drying rate depends on the size of the granules and temperature, i.e., decreases with increasing temperature and decreasing granule size.

Conclusions
1. The used quasistationary model is applicable for processing data on the drying of granular PET both for the entire process and for its various periods. MKM can be used with a minimum amount of experimental data.
2. The values of the kinetic indices m and s of the entire drying process have been obtained for the studied granulate samples in the temperature range from 120°C to 185°C.
3. The tendency to an increase in the index m with an increase in the drying temperature has been determined. An analysis of the obtained data uncertainty does not allow drawing a conclusion about the dependence of the s exponent on temperature and granule size. However, the scatter of values of the exponent s from the average is small, which prevents from accepting it as a constant with a small loss of calculation accuracy.
4. With known kinetic parameters m and s, it is possible to construct the curves of the drying kinetics and, therefore, to determine the duration of the first period according to formulas (1) and (2) for drying temperatures and granule sizes different from those studied.
5. The duration of the first period for the samples under study in the temperature range as well as the degree of the influence of temperature and sample size on the duration of the first drying period has been determined.

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