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# 1. Abstract

In this study, the drying kinetics of granular nylon-6 has been investigated. Single particle drying experiments were carried out in a magnetic suspension balance (MSB) using dry air as drying agent. The comparison of measured and simulated data using Vrentas-Duda diffusion model shows a satisfactory prediction of mean particle moisture content and drying rates. The Flory-Huggins model predicts well the measured sorption equilibrium. The simulation on the influence of air inlet humidity predicts well the residual moisture content and drying time.

Keywords: drying, sorption, diffusion, nylon, free volume theory.

# 2. Introduction

The moisture content, X, in nylon 6 is an extremely important variable affecting both processing and end use properties due to nylon 6 is a very hygroscopic polymer. For example, in 50% RH and 23°C (= normal condition), nylon can absorb moisture about 3%.<sup>[1-2]</sup> Therefore, drying process is needed to decrease the moisture content below 0.1% for product quality. On the other hand, the big problems in drying of nylon are low temperature limits (70-80°C) and the oxidative deterioration and discoloration at low moisture content. Usually nylon is dried in vacuum condition or in recirculating dehumidified air (dew point lower than -18 °C). For these conditions, the drying time ranges from 10 to 24 h.<sup>[1]</sup>

Many investigations on determination the diffusion and sorption equilibrium of water on nylon 6 have been done. The water vapour in nylon 6 is immobile at the lower water content, and considerably mobile at higher water contents.<sup>[3-4]</sup> The mechanism of water sorption in nylon is firmly bounded water, loosely bound water, and sites for capillary condensed water.<sup>[5]</sup> Indeed, this sorption phenomenon is important aspect in the prediction of residual moisture content, especially in terminal drying kinetics. Meanwhile, the previous experimental results show that the sorption and drying process of nylon is a diffusion controlled process. The diffusion of water vapour in nylon is dependent on temperature and moisture content.<sup>[3,5-7]</sup> The activation energy of water diffusion is function of water content and temperature.<sup>[5]</sup> However, those investigations still not discuss tail drying phenomenon in terminal drying process.<sup>[8]</sup>

Therefore, this paper present a detailed investigation both experiment and simulation drying kinetics of nylon 6.

#### 3. Experimental

A magnetic suspension balance (MSB) produced by Rubotherm (Bochum, Germany) has been used for determination of drying kinetics and sorption equilibrium. Dry nitrogen from the flask has been used to determine the dry sample mass with temperature 80°C. Single particle measurements are performed in drying process, while six particles measurements are conducted in sorption process. In addition, the stationary concept in time series analysis has been used to determine the constant mass of dry mass and equilibrium mass.<sup>[8]</sup> The material properties are 2.9 mm of particle diameter and 1150 kg/m<sup>3</sup> of solid density.

#### 4. Results and Discussion

#### *4.1. Sorption equilibrium*

Fig. 1 shows a good agreement between the experimental results of sorption equilibrium and prediction uses Flory-Huggins model<sup>[9-10]</sup>

$$\ln(\varphi) = \ln \varphi + (1 - \varphi) + \chi (1 - \varphi)^2, \qquad (1)$$

where  $\varphi$  is the water activity,  $\varphi$  is the volume fraction of dissolved water and  $\chi$  is the F-H interaction parameter. The constant  $\chi = 1.75$  is fitted value to experimental data. The influence of temperature is low, which is typical for polymeric materials.<sup>[10]</sup>



Fig. 1. Sorption equilibrium of water on Nylon 6

#### 4.2. Drying kinetics

Fig. 2. shows the comparison between experimental and simulation data for different gas temperatures. Both the temporal change of moisture content (Fig. 2a.) and the drying rates (Fig. 2b.) are predicted well. The model has developed on the assumption that the drying process is mainly controlled by diffusion of moisture within the particle and mass transfer kinetics at the phase boundary. The diffusion coefficient is predicted by means of Vrentas-Duda diffusion model.<sup>[11]</sup> The free volume parameters in Vrentas and Duda diffusion model are determined from thermodynamics,<sup>[13-14]</sup> except D<sub>0</sub> (constant pre exponential factor) and  $\xi$  (ratio of solvent and polymer jumping unit), which are estimated from experimental data by fitting. The fitting procedure is performed by solving the diffusion equation and fitting value of parameters of D<sub>0</sub> and  $\xi$  simultaneously which giving minimum value of the sum





Fig. 2. Comparison between measurement and simulation of single particle drying process; (a) mean particle moisture content, and (b) drying rates

It should be pointed out that the initial drying rates were an order of magnitude smaller than the theoretical first period drying rate for the given process conditions. For example at 60°C, the measured drying rate is  $0.1 \text{ kg/m}^2\text{h}$  where the calculated first period drying rate using Sh=2 is 1.31 kg/m<sup>2</sup>h. Consequently, the entire drying process of nylon 6 is mainly controlled by the internal diffusion of moisture. This conclusion is in agreement with previous investigation.<sup>[3-4,6-7,15]</sup>

In addition, after residual moisture content achieves 0.01, the drying curve shows tail drying or plateau diffusion phenomenon. It is caused by the diffusion coefficient decreases tremendously close to zero (see Fig. 3). Comparison value of diffusion coefficient with literature data<sup>[3,6,15]</sup> does not give a good agreement.

# 4.3. Simulation: the influence of air inlet humidity

Fig. 4 shows that the product quality (X<0.1%) can be achieved only when dew point of air inlet is lower than  $-3^{\circ}$ C. In industrial experience, drying of nylon 6 is conducted at 80°C, DP <  $-18^{\circ}$ C, and



Fig. 3. Dependency of diffusion coefficient on particle moisture content and temperature



Fig. 4. The prediction of residual moisture content on the influence of air inlet humidity

drying time is around 24 hours.<sup>[1]</sup> This condition agrees with the predicted results for

DP -18°C. The simulation is working well due to consideration mass transfer kinetics at the phase boundary.

### 5. Conclusion

The Flory-Huggins model predicts successfully the sorption equilibrium data. Drying process is mainly controlled by internal diffusion of moisture inside the particle. The Vrentas-Duda diffusion model gives satisfactory prediction of mean particle moisture content and drying rates. At the end of drying shows tail of drying or plateau diffusion phenomena, due to the diffusion coefficient decrease tremendously when moisture content is close to zero. The simulation also works well to predict the drying time and the residual moisture content on the influence of air inlet humidity.

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