Drying Kinetics and Inversion Temperature in a Low-Pressure Superheated Steam-Drying System

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The present study aimed at investigating the drying rates and inversion temperatures of model porous particles undergoing low-pressure superheated steam and vacuum drying. Molecular sieve beads, which were used as the model material in this work, were dried as a single layer in a low-pressure superheated steam dryer. The effects of steam temperature and pressure on the drying rates of these particles were determined. The same dryer was also operated in a vacuum mode to determine the effects of the above-mentioned operating parameters as well as the variation of the inversion temperature of the processes with the operating pressure. The differences between the values of the inversion temperature calculated only from the rates of drying in the constant rate period and those calculated from the whole drying period are pointed out and discussed. Page’s equations and a single-term exponential equation were found to satisfactorily describe the kinetics of low-pressure superheated steam- and vacuum-drying systems, respectively.

Introduction

Despite the many advantages of near-atmospheric pressure superheated steam drying, there still exist some limitations, especially when applying it to drying of heat-sensitive materials, e.g., foods and bioproducts.1 Because most foods or other heat-sensitive products melt, undergo glass transition, or are damaged at the temperature of superheated steam corresponding to the atmospheric or higher pressures, one possible way to alleviate the above-mentioned problems is to operate the dryer at reduced pressure.1–3 In addition to being able to preserve the quality of heat-sensitive products, lowering the operating pressure may enhance the drying rates as well.1,4–8

Some investigators have recently applied the concept of low-pressure (or subatmospheric pressure) superheated steam drying (LPSSD) to various heat-sensitive materials. Elustondo et al.3 studied subatmospheric-pressure superheated steam drying of wood slabs and shrimp, banana, apple, pear, potato, and cassava slices both experimentally and theoretically. A semiempirical model was developed assuming that water removal was accomplished by evaporation in a moving boundary, allowing the vapor to flow through the dry layer built up as drying proceeds. The model proposed was found to predict the drying kinetics reasonably well. More recently, Devahastin et al.9 studied experimentally drying of carrot cubes in both low-pressure superheated steam and vacuum dryers. They observed that the differences between the two sets of drying rate data (low-pressure superheated steam and vacuum drying) were smaller at higher drying temperatures. This suggested that raising the drying temperature further would eventually lead to equal rates of drying at the so-called inversion temperature1,10 because of the increased temperature difference between the steam and product as well as a reduction in the initial steam condensation11 that occurs as the wet material enters the drying chamber at a temperature lower than the saturation temperature of steam at prevailing pressure. Such information on the inversion temperature for the LPSSD system and the effect of vacuum pressure on this temperature is still missing, however. Shibata et al. (respectively in refs 4 and 12) have studied the steam-drying mechanisms of sintered spheres of glass beads under atmospheric pressure and vacuum. They reported that the drying mechanisms of the two processes were different and that superheated steam drying under vacuum gave lower critical moisture contents as well as higher drying rates in the falling rate period (FRP) than those in air drying under vacuum. However, they have not reported any information about the inversion temperature of the systems.

The present work, therefore, aimed at investigating the effect of vacuum pressure on the value of the inversion temperature by comparing the single-layer drying rates obtained in LPSSD and in vacuum drying of model porous particles, viz., molecular sieve beads. In addition, the values of the inversion temperature calculated only from the rates of drying in the constant rate period (CRP) were compared with those calculated from the whole drying period (CRPs and FRPs) in order to point out the fundamental differences between the two sets of temperatures, obtained from two different sets of drying rate information, beyond which drying rates in LPSSD are higher than those in vacuum drying. Three simple mathematical models that enable prediction of the drying behavior of molecular sieves undergoing LPSSD and vacuum drying were also proposed and compared.
Experimental Setup, Material, and Methods

Experimental Setup. A schematic diagram of the low-pressure superheated steam dryer and its accessories is shown in Figure 1. The dryer consists of a stainless steel drying chamber, insulated carefully with rock wool, with inner dimensions of $45 \times 45 \times 45$ cm$^3$; a steam reservoir, which received steam from the boiler and maintained its pressure at around 200 kPa (gauge); and a liquid ring vacuum pump (model ET32030, Nash, Trumbull, CT), which was used to maintain a vacuum in the drying chamber. A steam trap was installed to reduce excess steam condensation in the reservoir. An electric heater, rated at 1.5 kW, controlled by a proportional–integral–derivative controller (model E5CN, Omron, Tokyo, Japan), was installed in the drying chamber to control the steam temperature and to minimize condensation of steam in the drying chamber during start-up; with the use of a heater, the initial steam condensation during the start-up period was reduced considerably. A variable-speed electric fan was used to disperse steam throughout the drying chamber. The steam inlet was made into a conical shape and was covered with a screen to help in the distribution of the steam in the chamber. The sample holder was made of a stainless steel screen with dimensions of $12 \times 12$ cm$^2$. The change of the mass of the sample was detected continuously (at 1 min intervals) using a load cell (model Ucg-3kg, Minebea, Nagano, Japan), which was installed in a smaller chamber connected to the drying chamber by a flexible hose (in order to maintain the same vacuum pressure as that in the drying chamber) and also to an indicator and recorder (model AD 4329, A&D Co., Tokyo, Japan). The temperature of the steam was also measured continuously using type K thermocouples, which were connected to an expansion board (model no. EXP-32, Omega Engineering, Stamford, CT). Thermocouple signals were then multiplexed to a data acquisition card (model no. CIO-DAS16Jr., Omega Engineering, Stamford, CT) installed in a PC. LABTECH NOTEBOOK software (version 12.1, Laboratory Technologies Corp.) was used to read and record the temperature data.

Material and Methods

Molecular sieve beads (no. 69837, Fluka, Buchs, Switzerland) that have a pore size of 0.4 nm and an average diameter of 3.02 mm with a standard deviation of 0.34 mm and a particle density of 750 kg/m$^3$ were used as the test material in this work. Prior to the start of each experiment, distilled water (6.7 g) was slowly sprayed onto the beads (22 g) to make the initial moisture content of the beads around 0.3 kg/kg (db), which was roughly the maximum moisture-holding capacity of the beads. The particles were then left in a tightly closed box at room temperature for about 6 h to allow them to equilibrate. The drying experiment was performed by placing roughly 28.7 g of water-saturated particles (about 1000 beads) on the sample holder as a single layer. The drying chamber was then sealed tightly, and valve 2 was opened to allow the steam from the boiler to flow into the reservoir; the steam pressure was maintained at about 200 kPa (gauge) in the reservoir. A vacuum pump was then switched on to evacuate the drying chamber to the desired operating pressure, and the steam regulator was opened to slowly flash the steam into the drying chamber. Because of the low-pressure environment in the chamber, the steam became superheated. An electric heater was used to maintain the steam temperature at the desired drying temperature. Although the ratio of the steam pressure in the steam reservoir to that in the drying chamber was rather high, the effect of the adiabatic expansion of steam introduced into the drying chamber on the steam temperature was rather small. This is because

![Figure 1. Schematic diagram of the low-pressure, superheated steam dryer and associated units: 1, boiler; 2, steam valve; 3, steam reservoir; 4, pressure gauge; 5, steam trap; 6, steam regulator; 7, drying chamber; 8, steam inlet and distributor; 9, electric fan; 10, sample holder; 11, electric heater; 12, online temperature sensor and logger; 13, vacuum breakup valve; 14, insulator; 15, online weight indicator and logger; 16, vacuum pump; 17, PC with installed data acquisition card.](image1)

![Figure 2. Drying curves of molecular sieve particles.](image2)
an electric heater was installed in the drying chamber to help in the stabilization of the steam temperature. In addition, the temperature measurement was done carefully to ensure that the variation of the steam temperature within the drying chamber was negligible. At the end of drying, the breakup valve was opened to allow air into the drying chamber before opening up the chamber door and loading off the sample.

The experiments were performed under the following conditions: steam absolute pressures of 7, 10, and 13 kPa; steam temperatures of 80, 90, and 100 °C. The flow rate of steam into the drying chamber was maintained at about 26 kg/h, and the speed of the fan was fixed at around 2100 rpm. All experiments were performed in duplicate.

For vacuum-drying experiments, the same experimental setup was used but without the application of steam to the drying chamber. The same operating conditions as those used for LPSSD were therefore achievable.

Results and Discussion

Drying Rates of Porous Particles. Molecular sieve beads, which were saturated with water [the initial moisture content was about 0.3 kg/kg (db) or 23% (wb)], were dried to their equilibrium moisture content at each
operating condition in the dryer using both low-pressure superheated steam and vacuum conditions. During each experiment, the drying rates were calculated from the recorded weight changes, during both the CRPs and FRPs. Because, strictly speaking, the inversion temperature is defined only for surface moisture evaporation and not for internal moisture removal, the drying rates in the CRP are reported first. The overall average drying rates, based on the rates in both the CRPs and FRPs, are then calculated and compared with the rates in the CRP to point out the fundamental differences between the values of the inversion temperatures calculated using only the rates in the CRP and those obtained using the overall average drying rates in both drying periods.

First, the drying curves for the single layer of particles undergoing LPSSD and vacuum drying at some selected conditions are shown in Figure 2. While the drying curves for LPSSD at different conditions are quite different and the effect of the temperature on the drying curves is greater than the effect of the pressure, the drying curves for vacuum drying at different operating conditions are rather similar. It is seen that the drying times of LPSSD at an operating temperature of 80 °C were longer than those of vacuum drying for all operating pressures tested. However, the drying times for both processes operated at 100 °C were quite similar. This is due to the fact that an increased drying temperature led to higher drying rates, resulting from sharply increased temperature differences or gradients between the samples and the ambient steam in the case of superheated steam drying. However, the differences between the air temperature and the particle surface temperature in vacuum drying increased only slightly as the drying temperature increased. In addition, it can be observed that the equilibrium moisture contents of the beads undergoing LPSSD were much higher than those undergoing vacuum drying. For example, the equilibrium moisture contents of particles dried at 80, 90, and 100 °C using LPSSD at an operating pressure of 7 kPa were 1.5, 0.9, and 0.2% (db), respectively, while the equilibrium moisture contents of particles were 0.09, 0.05, and 0.02% (db), respectively, in the case of vacuum drying at the same pressure. This is due to a higher humidity condition in the drying chamber of LPSSD and, hence, a reduced vapor pressure gradient, which is the driving force of the drying process. Therefore, the drying times of most LPSSD were longer than those of vacuum drying.

Figure 3 shows a comparison of the observed drying rate curves in superheated steam drying with those in vacuum drying at different operating conditions. For all conditions, the drying rates fluctuated marginally but remained nearly constant as the moisture content decreased until the critical moisture content for each condition was reached. The drying rates then decreased continuously in the FRP. It can be seen from this figure that the critical moisture content was different for different conditions in the case of superheated steam drying [the critical moisture contents of particles dried, for example, at 80, 90, and 100 °C were 20, 17, and 15% (db), respectively, at an operating pressure of 7 kPa] but were quite similar in the case of vacuum drying [17% (db) over the temperature range of 80–100 °C at an operating pressure of 7 kPa]. It was also observed that the lower-pressure and higher-temperature su-
difference is the difference between the superheated steam temperature and the saturation temperature at the corresponding operating pressure, while for vacuum drying, the difference is between the air temperature and a temperature between the wet-bulb temperature and the saturation temperature (not saturation temperature because, in this case, the level of vacuum was not so high that the effect of convection by the fan could be negligible). While the temperature differences (or driving forces for heat transfer) in vacuum drying were higher at lower operating temperatures than those observed in LPSSD, the values of the heat-transfer coefficient of the medium in the case of vacuum drying were lower because of the inferior thermal properties of air. Raising the drying temperature, however, led to higher temperature differences and, hence, higher CRP drying rates. The counteracting effects of the heat-transfer coefficient and the temperature difference led to the inversion phenomenon, as is evident in Figure 4, where the CRP drying rates of vacuum drying and LPSSD are seen to be equal. Beyond the inversion temperature, the CRP drying rates of steam drying were higher than those of vacuum drying because of both the increased temperature difference and the higher heat-transfer coefficient.

When the operating pressure was increased (at the same operating temperature), it can be seen that the drying rate was lower. This was due to the fact that the boiling temperature of water at higher pressure is higher; this leads to a decreased temperature difference and, hence, a lower drying rate.

As mentioned earlier, the CRP drying rates depend on the rate of heat transfer and, hence, the difference between the surface temperature of the sample and the drying medium temperature. For this reason, the drying temperature had only a small effect on the rates of vacuum drying as compared with the case of LPSSD because the particle surface temperature in the case of vacuum drying (which relates somehow to the wet-bulb temperature) changes only slightly with an increased drying temperature compared with the change of the boiling temperature, especially at lower operating pressures.

Inversion Temperature. Figure 5 shows the effect of the operating pressure on the inversion temperature, which was calculated from the CRP rates (Figure 4). The inversion temperature at each operating pressure was obtained from the plots of the drying rates at the point where the rates of vacuum drying and LPSSD were equal.

Figure 6 shows the overall average drying rates calculated from combined CRP and FRP drying rates at various operating pressures. As mentioned earlier, the CRP drying rates depend only on the external heat-and mass-transfer conditions because free water is always available for evaporation at the surface of the sample. However, in the FRP, the rates depend not only on the rate of external heat transfer but more on
in the case of LPSSD. Hence, lead to smaller differences between the overall drying rates of superheated steam drying and, therefore, increase the values of the combined (or overall) drying rates. This is no diffusional resistance of water from the sample into the drying medium. These effects of the FRP drying rates were not the same as the CRP drying rates. It can be seen from Figures 4 and 6 that the differences between vacuum- and steam-drying CRP rates are somewhat material-dependent. Therefore, the average drying rates calculated from combined CRP and FRP rates were not the same as the CRP drying rates. It can be seen from Figures 4 and 6 that the differences between vacuum and steam drying. No inversion temperature was observed from the plots of the overall drying rates as well.

Mathematical Modeling. Simple mathematical models that enable prediction of the drying curves of molecular sieves undergoing LPSSD and vacuum drying were developed based on the well-known Page’s equation, single-term exponential equation, and two-term exponential equation (Arrhenius-type model). Exponential equations were listed, and their prediction abilities were compared with that of a simpler Page’s equation with no real physical basis but just to illustrate that different sets of equations are suitable for different drying techniques.

Page’s Equation. 

\[
\text{MR} = \frac{X_i - X_{eq}}{X_i - X_{eq}} = \exp(-kt^n) \quad (1)
\]

The parameters \( k \) and \( n \) in the equation were determined from the experimental data and were correlated as follows:

For LPSSD

\[
k = -2.39 \times 10^{-1} + 2.86 \times 10^{-3}T - 7.13 \times 10^{-5}P - 3.83 \times 10^{-6}TP + 5.42 \times 10^{-2} \ln P \quad R^2 = 0.95
\]

\[
n = (1.87 - 3.91) \times 10^{-3}T + 1.11 \times 10^{-1}P - 5.44 \times 10^{-4}TP - 4.35 \times 10^{-1} \ln P \quad R^2 = 0.86
\]

For vacuum drying

\[
k = -7.42 \times 10^{-2} - 7.07 \times 10^{-5}T - 1.01 \times 10^{-2}P + 1.22 \times 10^{-5}TP + 1.12 \times 10^{-1} \ln P \quad R^2 = 0.96
\]

\[
n = (1.12 - 2.40) \times 10^{-3}T - 7.88 \times 10^{-2}P + 4.92 \times 10^{-4}TP + 2.21 \times 10^{-1} \ln P \quad R^2 = 0.73
\]

Single-Term Exponential Equation.

\[
\text{MR} = \frac{X_i - X_{eq}}{X_i - X_{eq}} = a \exp(-bt) \quad (2)
\]

For LPSSD

\[
a = (1.12 - 1.41) \times 10^{-3}T + 4.53 \times 10^{-3}P - 6.87 \times 10^{-6}TP + 6.53 \times 10^{-3} \ln P \quad R^2 = 0.75
\]

\[
b = -6.15 \times 10^{-2} + 1.12 \times 10^{-3}T - 9.75 \times 10^{-3}P + 7.56 \times 10^{-5}TP + 3.77 \times 10^{-2} \ln P \quad R^2 = 0.91
\]

For vacuum drying

\[
a = 9.68 \times 10^{-1} + 1.22 \times 10^{-3}T + 1.59 \times 10^{-4}P - 2.73 \times 10^{-5}TP - 1.12 \times 10^{-1} \ln P \quad R^2 = 0.94
\]

\[
b = 1.97 \times 10^{-2} - 3.74 \times 10^{-4}T + 2.13 \times 10^{-2}P + 1.08 \times 10^{-4}TP + 1.03 \times 10^{-1} \ln P \quad R^2 = 0.70
\]

Two-Term Exponential Equation.

\[
\text{MR} = \frac{X_i - X_{eq}}{X_i - X_{eq}} = a_1 \exp(-b_1t) + c_1 \exp(-d_1t) \quad (3)
\]
The equations were fitted with the experimental data; the fitted equations were evaluated based on their $R^2$ and standard error of estimation. When the three drying models are compared, the results show that Page’s equation fits the experimental data better than the single-term exponential equation and the two-term exponential equation in the case of LPSSD, while the single-term exponential equation fits the experimental data well in the case of vacuum drying at operating temperatures in the range of 80–100 °C and pressures of 7–13 kPa as exemplified in Figures 7 and 8. The minimum $R^2$ of Page’s equation was 0.997 and its maximum standard error of estimation was 0.0181 in the case of LPSSD, while the minimum $R^2$ of the single-term exponential equation was 0.998 and its maximum standard error was 0.0233 in the case of vacuum drying. Drying constants of Page’s equation ($k$ and $n$) and of the single-term exponential equation ($a$ and $b$) are found to depend on the operating temperature as well as pressure.

Concluding Remarks

The effects of operating parameters, i.e., drying temperature and pressure, on the rates of vacuum drying and LPSSD of model porous particles were investigated experimentally. In addition, the values of the inversion temperature calculated only from the rates of drying in the CRP were compared with those calculated from the whole drying period (CRP and FRP) in order to point out the fundamental differences between the two sets of temperatures beyond which the drying rates in LPSSD were higher than those in vacuum drying. It was found that the inversion temperatures calculated from combined CRP and FRP rates were not the same as the inversion temperatures calculated from only CRP rates; in the former case, no inversion temperature was indeed observed within the ranges of study. Empirical models that describe the experimental drying curves are also proposed. It is found that Page’s equation and the single-term exponential equation can fit well the experimental data for LPSSD and vacuum drying, respectively, over the ranges of operating temperature of 80–100 °C and pressure of 7–10 kPa.

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Nomenclature

$a = \text{constant of the single-term exponential model}$

$a_1 = \text{constant of the two-term exponential model}$

$b = \text{constant of the single-term exponential model}$

$b_1 = \text{constant of the two-term exponential model}$

For LPSSD

\[
\begin{align*}
a_1 &= (0.699P^{-0.051}) \exp(-51.312/T_{\text{abs}}) & R^2 &= 0.62 \\
b_1 &= (354.576P^{-0.0267}) \exp(-2958/T_{\text{abs}}) & R^2 &= 0.95 \\
c_1 &= (5.63 \times 10^{-3}P^{0.408}) \exp(-1269.76/T_{\text{abs}}) & R^2 &= 0.59 \\
d_1 &= (827.98P^{-0.132}) \exp(-3175.72/T_{\text{abs}}) & R^2 &= 0.93
\end{align*}
\]

For vacuum drying

\[
\begin{align*}
a_1 &= (3.709P^{0.554}) \exp(-1056.055/T_{\text{abs}}) & R^2 &= 0.53 \\
b_1 &= (0.164P^{0.059}) \exp(-185.677/T_{\text{abs}}) & R^2 &= 0.49 \\
c_1 &= (1.63 \times 10^{-3}P^{-1.082}) \exp(2773.12/T_{\text{abs}}) & R^2 &= 0.75 \\
d_1 &= (4.73 \times 10^{-2}P^{-0.903}) \exp(871.105/T_{\text{abs}}) & R^2 &= 0.95
\end{align*}
\]
constant of the two-term exponential model

constant of the two-term exponential model

constant of Page’s equation

moisture ratio

absolute pressure, kPa

drying time, min

temperature of the drying medium, °C

temperature of the drying medium, K

equilibrium moisture content, kg/kg (db)

initial moisture content, kg/kg (db)

moisture content at any time, kg/kg (db)

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