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RESEARCH ARTICLE

Drying Characteristics of the Furfural Residues of Biomass Hydrolyzation

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Drying behavior of furfural residues was studied in a laboratory type dryer. The effect of drying temperature, initial material load, particles size and heating rate on the drying characteristics of furfural residue was investigated. The results show that the increase in drying temperature resulted in a decrease in the drying time, the drying rate increases with the decrease of moisture content. The optimum drying temperature may be 130 °C. Higher initial material load induced longer drying time, and the moisture content decreases gradually. Constant drying rate period was not found in all runs. The drying rate of larger particle size is lower than that of small size, and the maximum drying rate occurred with furfural residues of 20 mesh. The drying rate of rapid heating rate is greater than the slow one as the moisture content below 48.2%. The experimental drying results of furfural residues obtained were fitted to several models. The accuracy of the models was estimated with the root mean square error analysis (*RMSE*), sum of squared errors (*SSE*), correlation coefficients (R^2) and reduced chi-square (χ^2). The results of Page model were better than other models. The parameters of page model were estimated with multiple regression analysis.

Keywords: Furfural Residues, Drying, Modeling, Biomass.

1. INTRODUCTION

Furfural is an important compound produced from agricultural waste biomass and residues contained pentosans, which are hydrolyzed to furfural and other byproducts. Pentosans are present in the cellulose of many woody plant tissues such as corn cobs, sugar cane bagasses, paper-pulp residues, grain hulls, wheat and rice straws, rice and oat hulls, etc.[1] China is the biggest producer and exporter of furfural production in the world. As the furfural residues are the main byproduct and plenty of waste residues are produced in the process of furfural production. Furfural residues are often utilized as fuel of boiler for energy recovery which can reduce production costs to a great extent. Also sometimes, furfural residues may be used as raw material to produce activated carbon by pyrolysis and as an additive or fertilizers for amelioration of alkaline soil.

In China, furfural residues actually in production were employed as the raw material of boiler for combustion to recovery energy. As fresh furfural residues came from hydrolysis reactor, the moisture content is rather high reaching about 60%. It is disadvantage to combust directly without any drying treatment. The wet residues may cause the efficiency of combustion decrease, the damage for boiler chamber, huge heating loss and increase the cost of tail gas treatment. So, it is necessary to dry furfural resides before burning. The other thermo-chemical process for high value application of furfural residues might be gasification and pyrolysis, however these technologies require the moisture content of furfural residues at low level. Studies on biomass gasification and pyrolysis showed that biomass moisture was in the range of 5.58–12.29%.[2–9] So drying treatment for wet furfural residues is a vital route prior to thermo-chemical process.

Drying is a complicated process involving simultaneous heat and mass transfer. Many researchers had carried out studies on biomass drying. Moreno[10] used vibrating-fluidized bed, mechanically agitated fluidized bed and fluidized bed with inert solids to study the drying characteristics of pinus radiate. Ståhl[11] researched the influence of industrial scale dryers on the quality of wood pellets. The drying medium, temperature and residence time were discussed as drying factors. Tore Filbakk[12] investigated the storage and drying methods for wood used as a raw material for pellet production influenced

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pellet durability, bulk density and energy consumption. As drying fruits and vegetables have gained commercial importance, most researchers had investigated the drying characteristics of fruits and vegetables. Karim[13] developed a mathematical model for drying of food products undergoing shrinkage. And the drying experiments were carried out on a tunnel type fruits dryer. As an alternative potential fuels, it is a promising method that fruit process residues can be used as fuel to produce the energy necessary during fruit drying. Nagle[14] investigated the drying process of fruit processing residues, which was used as an alternative fuel for energy utilizing.

Although many researchers focus on the drying of several biomass materials, such as wood, forest residues, bark, straw, energy crops, peat, and agricultural residues, according to our best knowledge, the drying of furfural residues has not any reports in literature. In this paper, the drying experiments of furfural residues were studied, and several effect factors (drying temperature, initial material load, particles size and heating rate) on furfural residues drying process were investigated. This study might be benefit for the energy utilization of furfural residues as fuel.

2. MATERIALS AND METHODS

2.1. Experimental Setting and Material

Furfural residues were obtained from a local furfural manufactory located in Tongliao in Inner Mongolia, China. The moisture content of fresh furfural residues was above 60% at the initial ejection from hydrolyzation reactor with the pressure about 10–12 MPa. After storage and transforming, the average moisture content had decreased to 58%. In this study, furfural residues had bulk density of 303 kg/m^3, and after long transforming, the furfural moisture content is of order of 53%. The furfural residue was crushed and the range of particle sizes is 0.5 mm–10 mm.

The laboratory scale furfural residues drying experiments were carried out on a halogen moisture detector (Mettler Toledo, HR83P). The experiments were performed to measure the temperature and the moisture of the samples.

2.2. Characterization

In order to investigate the characteristics of furfural drying process and to evaluate the drying efficiency, two parameters, drying rate and average drying rate are proposed.

The drying rate of furfural is defined as the rate of mass loss due to moisture evaporation per unit time.

$$R = \frac{W_t - W_{t+1}}{\Delta t} \tag{1}$$

And average drying rate is defined as following equation,

$$R_p = \frac{\sum_{i=1}^{t} R_i}{t} \tag{2}$$

2.3. Model Formulation

During furfural residues drying, heat and moisture transferred based on diffusion mechanism. Part of heat is transferred to the particle surface of furfural residues to evaporate surface moisture while other heat is consumed to diffuse to particles interior. Under the action of the diffusion, moisture in the particles interior migrate to exterior with the moisture of surface evaporating. Usually the transport of water of biomass can be described by the Fick's second law of diffusion.[15,16] A diffusion based model was simplified.

The solution of Fick' equation for a slab, for constant diffusivity (D), in terms of an infinite series is given by Crank (1975):[15]

$$MR = \frac{M - M_f}{M_0 - M_f} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-\frac{n^2 \pi^2 D t}{r^2}\right) \tag{3}$$

For longer drying periods, Eq. (3) can be considered the first term of the above series. It is possible to describe the logarithmic reduction of the first term of Eq. (3) as a linear function of time.

$$\ln MR = \ln\left(\frac{M - M_f}{M_0 - M_f}\right) = \ln\left(\frac{6}{\pi^2}\right) - \frac{n^2 \pi^2 D t}{r^2} \tag{4}$$

Equation (4) can be written as

$$MR = k \exp(-ct) \tag{5}$$

This model is the famous Henderson and Pabis model, many other semi-empirical models can be deduced from Eq. (3), i.e., Lewis or Newton, Page model, Logarithmic, Wang and Singh, weibull. Many researchers had studied these drying models with different materials and dryers.[17,18] In this study, three models, Henderson and Pabis, Page and Logarithmic model (Eqs. (5)–(7)), were employed to describe the drying kinetics and predict the moisture ratio.

$$MR = \exp(-kt^n) \tag{6}$$

$$MR = a \exp(-kt) + c \tag{7}$$

Several statistical test methods can be used to evaluate statistically the performance of the drying models. The coefficient of correlation (R^2) is one of the primary criteria to estimate the simulation for the drying curves of furfural residues. The reduced chi-square (χ^2), sum of squared errors (SSE) and root mean square error analysis ($RMSE$) also were used to determine the best fit. These statistical criterions can be calculated as follows:

$$R^2 = \frac{\sum_{i=1}^{N}(MR_i - MR_{\text{pre},i}) \cdot \sum_{i=1}^{N}(MR_i - MR_{\exp,i})}{\sqrt{[\sum_{i=1}^{N}(MR_i - MR_{\text{pre},i})^2]} \cdot \sqrt{[\sum_{i=1}^{N}(MR_i - MR_{\exp,i})^2]}} \tag{8}$$

$$SSE = \sum_{i=1}^{N}(MR_i - MR_{\text{pre},i})^2 \tag{9}$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{\exp,i} - MR_{\text{pre},i}\right)^2\right]^{1/2} \quad (10)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{\exp,i} - MR_{\text{pre},i})^2}{N-z} \quad (11)$$

The higher the value of R^2 and lower the values for *SSE*, *RMSE* and χ^2, the better the goodness of fit. In this study, the effects of dying temperature, initial material loads, particle size and heating rate on the coefficients and constants of the drying expression which describe the drying characteristics of furfural residues were also investigated. The constants and coefficients of the models involving the mentioned drying variables were determined by investigating different type of equations such as simple linear, logarithmic, exponential, power, Arrhenius and rational.

3. RESULTS AND DISCUSSION

3.1. Effect of Drying Temperature

Experiment results of effect on drying temperature are studied. The initial loading is 2 g, the particle size of furfural residues is 10 mesh, and the initial moisture content is about 53%. Drying process was carried out at four drying temperatures, 80, 105, 130 and 150 °C, respectively (show in Fig. 1(a)). An inverse relationship between drying temperature and drying time was observed, that is, an increase in drying temperature resulted in a decrease in the drying time. The shortened drying time is the result of higher drying temperature which led to higher heat transfer. The moisture content from the initial moisture of about 53% to zero cost time were found to be between 13 and 30 min. 10% moisture content of furfural residues is a key criterion because of favoring for thermo-chemical transform, for example, combustion, pyrolysis and gasification using furfural residues as fuel.[19] As the moisture contents reached 10% under the various drying temperature, the time cost were 15.5, 10, 5.5 and 5.5 min respectively. Drying rates of furfural residues were calculated using Eq. (1). The changes in the drying rates versus moisture content are shown in Figure 1(b). It can be seen that the drying rate decreases with moisture decreases under all drying temperatures. With the drying temperature increase, the drying rate increases with the moisture content declining. This is due to, as the drying temperature rises, the moisture migrates to the biomass surface and the evaporate rate from the surface to air increases.[20] It also can be seen, under the drying temperature of 130 and 150 °C, the change trend of drying rate is similar. This may be because the evaporation of water in furfural residues is heated instantaneously to a high level. The water inside and surface of materials is overheated and evaporates fiercely at higher drying temperature. The average drying rates of furfural residues in different drying temperature are 3.596, 5.177, 8.143, 6.835% d.b. min^{-1}, respectively.

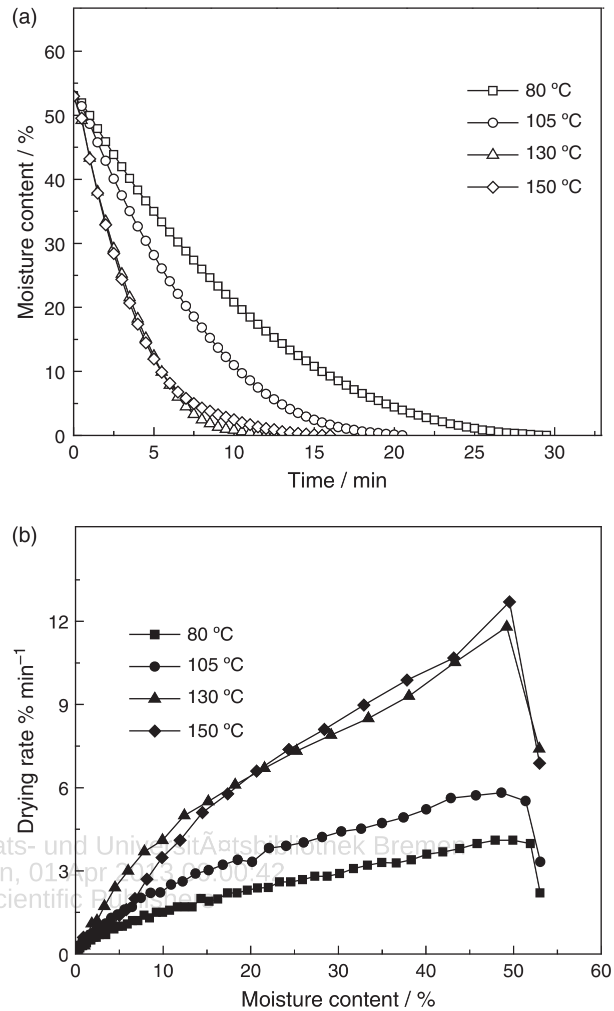

Fig. 1. Effect of drying temperature on the moisture content (a) and drying rate (b) of furfural residues.

3.2. Effect of Initial Material Loads

The effect of initial material loads on the drying process of furfural residues was illustrated in Figure 2(a). The drying temperature was kept at 105 °C, and the particle size of furfural residues is 10 mesh. Drying process was carried out at three initial material loads, 1 g, 4 g and 10 g with initial moisture contents (wet basis) of 54.93%, 53.65% and 53.14%, respectively. It was visually observed that the higher initial material loaded the longer drying time taken. As the drying time increases, the moisture decreases gradually. Total time of 14, 20 and 25 min were taken after drying moisture content reached zero for the runs. The time of moisture content of furfural residues, as the moisture content reach 10%, spent between 7, 10 and 13.5 min under different initial loads, and they accounted for 50%, 48% and 54% of total time.

Figure 2(b) shows the changes in the drying rates versus moisture content. From Figure 2(b), since the nonexistence of a constant drying rate period, it can be seen that the rate of furfural residues drying decrease gradually

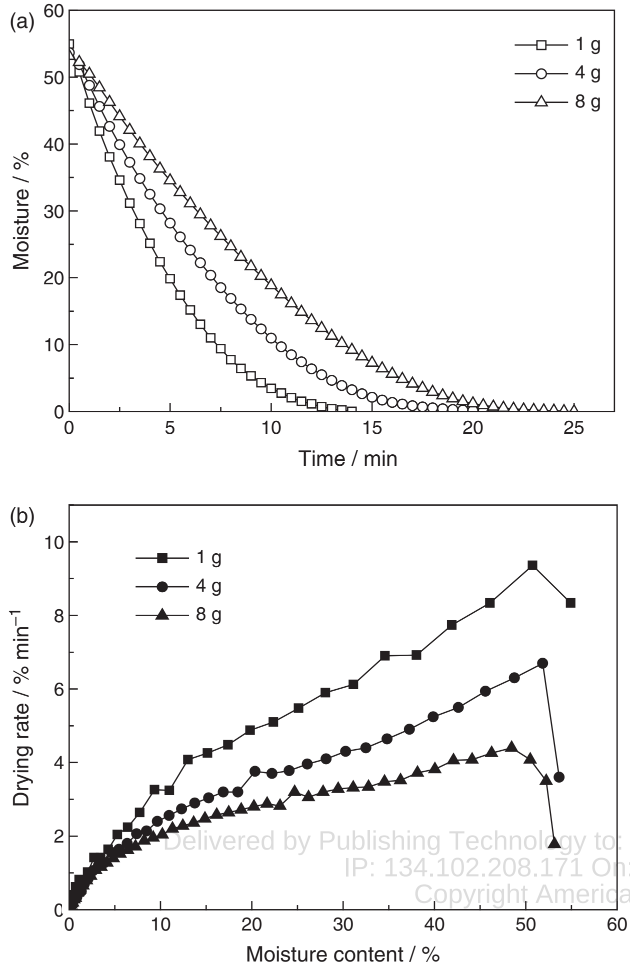

Fig. 2. Effect of initial material load on the moisture content (a) and drying rate (b) of furfural residues.

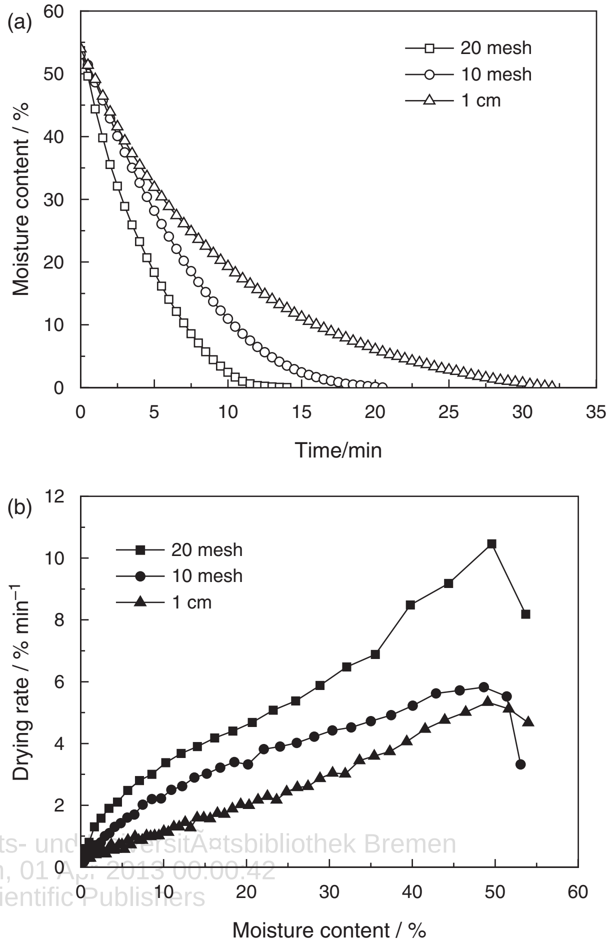

Fig. 3. Effect of particle size on the moisture content (a) and drying rate (b) of furfural residues.

at all runs. As initial material loads increase, the drying rates showed decrease trends. In each curve, the drying rate dropped as the moisture content decrease. Higher drying rates were obtained at lower initial material loads due to the decrease in the amount of moisture mass that should be removed from the dried material.[21] These results are in agreement with previous literature studies on drying of biomass.[21–23]

3.3. Effect of Furfural Residues Particle Size

Furfural residue samples were crumbed and sieved to produce three different sizes: 1 cm, 10 mesh and 20 mesh. The experiments were conducted for an initial load of 4 g and the drying temperature of 105 °C. The initial moisture contents for the three particle sizes used in the experiments were 53.69, 53.06 and 54%. Figure 3(a) shows the effect of particle size on the drying process of furfural residues. The final moisture contents reaching zero require 14, 20.5 and 32 min for the particle size of 1 cm, 10 mesh and 20 mesh, respectively. To reach the moisture content of 10% mentioned above, drying time of about 7.1, 10.2 and 16 min is needed, which account for 50.71, 49.76 and 48.48% of total time. That is, half of total drying time may be spent removing the last one-tenth of the moisture content. It also can be seen that the increasing particle sizes caused an increase in the drying time and the decrease in the moisture ratio.

The influence of the particle size on the drying rate of furfural residues was shown in Figure 3(b). It is clear that except the start of drying process (the first 1 min), the drying rate descend with the moisture content decrease. The drying rate of larger particle size is lower than that of small size. In this study, the maximum drying rate occurred at furfural residues of 20 mesh, and the particle size of 1 cm had a minimum value. The larger furfural residues particle sizes dried slower due to the increased distance. The moisture travels and the reduced surface area exposes for a given volume of samples.[21] Compared to the larger particle size samples, the small one presents a rather large surface area, and it enhances the evaporation of moisture from the surface to air. The diffusion resistance is also smaller than that of the larger particle size, which resulted in the moisture inside particle migration to the surface. The results were in good agreement with the drying studies on other research.[21,23]

3.4. Effect of Heating Rate

The heating rate is also an important factor in furfural residues drying. The used halogen moisture detector had two modes, slow drying rate and rapid drying rate. Drying programs controlled heating rate of sample drying. In this study, the two different heating rates were set to investigate the characteristics of furfural residues in the laboratory scale drying experiments. Because the limited condition of the halogen moisture detector, the slow drying mode was defined that the sample was heated to the preset drying temperature (105 °C) and was held constant at this temperature. However, under the mode of the rapid drying rate, following the start, the selected temperature (105 °C) was exceeded by 40% (about 147 °C) for 3 minutes to compensate the cooling due to vaporization and accelerate the drying process. The drying temperature is then lowered to the set value (105 °C) and maintained.

The experiments were conducted for an initial load of 4 g, the sample particle size is 10 mesh, and the initial moisture contents is 54.21 and 53.06%. Figure 4 displayed the variations of moisture content and drying rate against the drying time and moisture content at the abovementioned two heating rates for furfural residues.

Figure 4(a) shows the effect of heating rate on the drying process of furfural residues. The final moisture contents reach zero and require 20.5 and 18.5 min for the slow heating rate and rapid heating rate, respectively. It spent about 10 and 10.5 min to decrease the moisture content from 54.21 and 53.06% to reach the moisture content of 10%, about 50% of total drying time spent to evaporate the 90% moisture content. At the first about 10 min, the moisture content of rapid heating rate is higher than that of slow one. After 10 min, the drying trend inverses. The moisture content of furfural residues drying process in the two heating rates decreased with drying time increase. At about 9.5 min, the moisture content under the two heating rate reached equal. Figure 4(b) shows the effect of heating rate on the drying rate of furfural residues. From Figure 4(b), it can be seen that the drying rates under rapid and slow heating rate decline with moisture content decrease, whereas the drying rate of rapid heating rate is greater than the slow one as the moisture content below 48.2%. This is because that the sample reaches the preset drying temperature quickly under rapid heating rate, and the drying temperature of rapid heating rate is higher than that of slow one in the forepart time. The sample has longer time exposed in high temperature, and it induces the evaporation of water removed faster than the one under the mode of low heating rate. A rapid increase of drying rate is found in the start 2 min of drying process. This is because the samples are heated from room temperature to the preset temperature in short time, and the drying rate increase from low to high level in a short time influenced by the instable heating condition.

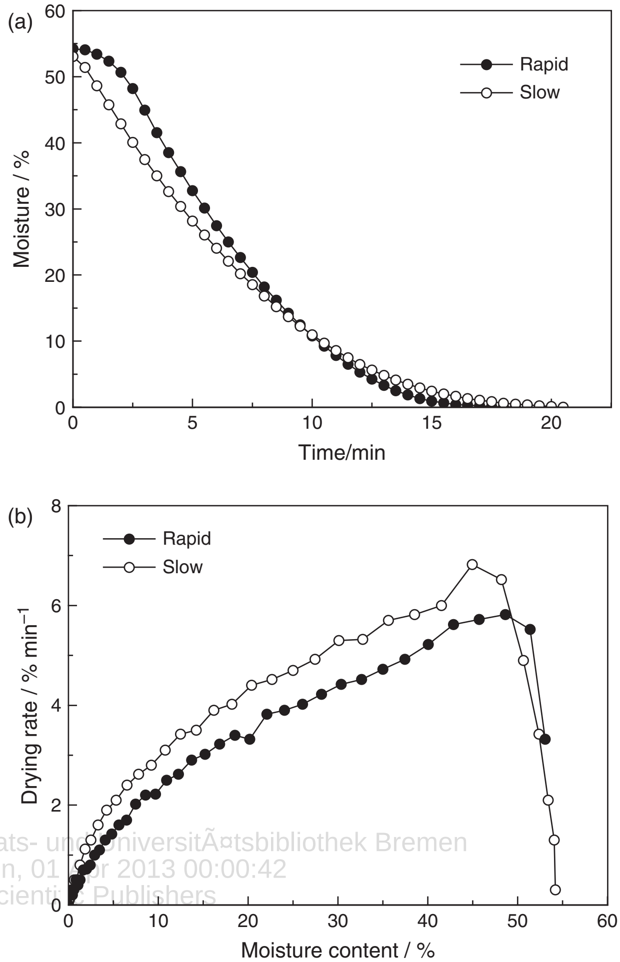

Fig. 4. Effect of heating rate on the moisture content (a) and drying rate (b) of furfural residues.

3.5. Modeling of Drying Curves

The models of Page's, Henderson and Pabis and logarithmic were employed to fit the drying data of furfural residues under different effect factors. Parameters, root mean square error analysis (*RMSE*), sum of squared errors (*SSE*), correlation coefficients (R^2) and reduced chi-square (χ^2) were calculated (see in Table I). Generally, the highest R^2, and the lowest *RMSE*, *SSE* and χ^2 are the best fitting effect. From Table I, it can be seen that, the values of R^2 were varied between 0.9617 and 0.9997. The highest value of R^2 and the lowest value of χ^2 were obtained by using Logarithmic model. But under most circumstances, the results of Page model were better than Henderson and Pabis model. The values of R^2 are all lower than the other two models, and the values of *RMSE, SSE*, and χ^2 are larger than Page model and Logarithmic model.

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Table I. Results of regression analysis for the empirical constants of the page and the Henderson and Pabis model.

	Drying temperature/°C				Initial load			Particle size			Heating rate	
Model and parameters	80	105	130	150	1 g	4 g	8 g	20 mesh	10 mesh	1 cm	Rapid	Slow
Page MR $= \exp(-kt^n)$												
k	0.0497	0.0738	0.1764	0.2058	0.1421	0.0778	0.0405	0.1599	0.0738	0.0931	0.0261	0.0738
n	1.2927	1.3427	1.3303	1.2144	1.2583	1.3252	1.4363	1.2251	1.3427	1.0564	1.8071	1.3427
RMSE	0.0149	0.0133	0.0103	0.0068	0.016	0.0163	0.0193	0.0203	0.0133	0.0121	0.0109	0.0133
SSE	0.0133	0.0074	0.003	0.0015	0.0074	0.0114	0.0191	0.0119	0.0075	0.0096	0.0045	0.0074
R^2	0.9978	0.9983	0.9989	0.9994	0.9975	0.997	0.9967	0.996	0.9983	0.9982	0.9992	0.9984
χ^2	$2.334E-04$	$1.915E-04$	$1.225E-04$	$5.202E-05$	$2.866E-04$	$2.889E-04$	$3.992E-04$	$4.574E-04$	$1.937E-04$	$1.535E-04$	$1.374E-04$	$1.915E-04$
Henderson and Pabis MR $= a^* \exp^*(-kt)$												
a	1.0892	1.0991	1.0869	1.0714	1.0656	1.0924	1.1204	1.0583	1.0991	1.0153	1.2024	1.0991
k	0.1109	0.1644	0.3081	0.3002	0.2344	0.1659	0.1256	0.2457	0.1644	0.1085	0.1653	0.1644
RMSE	0.0358	0.0394	0.0359	0.0204	0.0348	0.0394	0.0505	0.0341	0.0395	0.0144	0.0743	0.0395
SSE	0.0768	0.0654	0.0359	0.0138	0.0351	0.0668	0.13	0.0338	0.0654	0.0134	0.2096	0.0654
R^2	0.9888	0.9869	0.9893	0.9956	0.9895	0.9869	0.9791	0.99	0.9869	0.9978	0.9617	0.9869
χ^2	$1.333E-03$	$1.691E-03$	$1.404E-03$	$4.442E-04$	$1.327E-03$	$1.634E-03$	$2.695E-03$	$1.322E-03$	$1.639E-03$	$2.146E-04$	$5.836E-03$	$1.623E-03$
Logarithmic MR $= a^* \exp(-kt) + c$												
a	1.1728	1.1818	1.1244	1.081	1.1463	1.1675	1.2676	1.1293	1.1818	1.0372	1.4292	1.1818
k	0.0814	0.1217	0.2589	0.2814	0.1757	0.1242	0.0818	0.1866	0.1217	0.0953	0.0985	0.1217
c	−0.1336	−0.1303	−0.0657	−0.0214	−0.1227	−0.1217	−0.2108	−0.1122	−0.1302	−0.045	−0.2989	−0.1302
RMSE	0.012	0.0176	0.0238	0.0174	0.0125	0.018	0.0207	0.0118	0.0175	0.0041	0.0431	0.0176
SSE	0.0086	0.0129	0.0158	0.0099	0.0045	0.014	0.0219	0.0041	0.0129	0.0011	0.0705	0.0129
R^2	0.9984	0.9967	0.994	0.9963	0.9982	0.9965	0.9957	0.9984	0.9967	0.9997	0.9849	0.9967
χ^2	$1.523E-04$	$3.322E-04$	$6.345E-04$	$3.327E-04$	$1.763E-04$	$3.510E-04$	$4.579E-04$	$1.575E-04$	$3.322E-04$	$1.792E-05$	$2.000E-03$	$3.322E-04$

Due to the multiple regression analysis, the parameters of the below drying model, constants and coefficients were expressed in terms of the drying temperature and initial load.

$$MR = \exp(-kt^n)$$

For the furfural residue drying model of the effect on drying temperature,

$$k = 511.85\exp(-3281.4/T_{\text{abs}}), \quad R^2 = 0.9556,$$
$$n = -1.0023 \times \ln(T)^2 + 9.3118 \times \ln(T) - 20.269$$
$$R^2 = 0.9181$$

For the furfural residue drying model of the effect on particle size,

$$k = -0.0142m + 0.1485, \quad R^2 = 0.9460$$
$$n = 0.00256m + 1.2292, \quad R^2 = 0.9946$$

4. CONCLUSIONS

Furfural residue is a main waste in the process of furfural production. Commonly, high moisture content (above 50%) exists in the fresh furfural residues. In this study, an experimental investigation of drying process of furfural residues under various operating conditions was carried out.

It is found that the increase in drying temperature resulted in a decrease in the drying time. As the drying temperature increase, the drying rate increases with the decrease of moisture content. The optimum drying temperature may be 130 °C.

Higher initial material load induced longer drying time, and the moisture decreased gradually. Constant drying rate period was not found, and the rate of furfural residues drying decrease gradually at all runs. As initial material loads increase, the drying rates showed decrease trends.

The increased particle size caused an increase in the drying time and the decrease in the moisture ratio. The drying rate of larger particle size is lower than that of small size. The maximum drying rate occurred with furfural residues of 20 mesh.

The drying rates under rapid and slow heating rate decrease with moisture content, whereas the drying rate of rapid heating rate is greater than the slow one as the moisture content below 48.2%.

The results of Page model were better than other models. The parameters of page model were estimated with multiple regression analysis.

NOMENCLATURE

a, c, n, k, i, n, Empirical constants of drying models
D Is diffusion coefficient
M Mean moisture content at time t (g water/g dry matter)
M_f Is the moisture content at equilibrium (g water/g dry matter)
M_0 Initial moisture content (g water/g dry matter)
MR Fractional moisture ratio
$MR_{\text{exp}, i}$ Experimentally observed moisture ratio
$MR_{\text{pre}, i}$ Predicted dimensionless moisture ratio
m The particle size
N Number of observations
n Number of constants
r The radius of particle sphere (m)
R Drying rate
R_p The average drying rate
r^2 Coefficient of correlation
$RMSE$ Root mean square error
SSE Sum of squared errors
t Drying time (min)
T Drying air temperature (°C)
T_{abs} Absolute temperature (K)
W Water content of the sample
z Number of constants.

Greek Letter
χ^2 Reduced chi-square.

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