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HEAT PUMP ATMOSPHERIC SUBLIMATION AND EVAPORATIVE DRYING OF BOVINE INTESTINES

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Abstract: Bovine intestine samples were heat pump dried at atmospheric pressure and at temperatures below and above the material freeze points. Studies were done on effects of drying conditions on product quality and properties. The GAB model well predicted the relationship between water activity and equilibrium moisture content. A modified solution of Fick´s second law described the moisture removal by combined sublimation and evaporation. This equation appears to account for the moisture removal irrespective if predominating mechanism is diffusion, capillary flow or compressive force by structural collapse. The bovine intestine samples can be dried with improved quality and properties.

Keywords: color, intermittent drying, kinetics, rehydration

INTRODUCTION

In preservation and energetic view points it is desirable to storage foodstuffs at ambient conditions. This can be done by drying where the material moisture content and water activity are reduced and spoilage avoided during long time storage at nearly ambient conditions. However, conventional industrial drying is a high demanding energy process. New developments in heat pump technology offer alternatives to overcome drawbacks in preservation and energy use.

A well designed heat pump efficiently supplies both heating and cooling that are required in a drying process. The heat pump evaporator can recover latent heat freely available in the water vapor coming out of the wet drying material and recycle it through the condenser. It is also possible to condensate and to recover valuable volatiles or to remove otherwise noxious condensable gases by controlling the surface temperature of the evaporator.

Compared to a conventional dryer, the additional advantages of a heat pump dryer is the possibility to handle sticky and sensitive materials while producing a better quality product (Alves-Filho, 2002). For a properly selected heat pump fluid the technology is also environmentally friendly. Aside from complying with ozone depletion and global warming regulations it operated in closed drying circuit there is no gas, fumes or fines discharge to atmosphere.

The drawback of the heat pump dryers is the low moisture removal rates for atmospheric pressure freeze drying that greatly increases the residence time for stationary bed mode. However, this problem can be avoided by agitation, fluidization and intermittent drying (Mujumdar and Alves-Filho, 2003).

The objectives of the present work on fluidized bed heat pump drying of bovine intestine are to study the effect of operating conditions on quality and property parameters and to describe the kinetics and drying rates. Fluidized bed heat pump drying of bovine intestine samples were done in atmospheric pressure at below and above the material freeze temperature. The particles were 4 mm cubes and the sample batches were composed of white and dark fractions that were kept at -25°C before drying. Measurements were made to study the effect of drying conditions on product quality and properties with focus on kinetics, color, rehydration and water activity.

MATERIALS AND METHODS

The bovine intestine is composed of a large fraction of white tissue and a smaller portion of dark tissues. This raw material was kept frozen of -25°C to maintain its original characteristics and heated close to melting point prior to size reduction to 2, 4 and 8 mm cubes. Batches of samples composed of white and dark fractions that were separated and kept at -25°C until drying commenced.

An experimental heat pump dryer was used for the trials. The dryer has a drying loop and the heat pump circuit. The drying loop has an air dehumidifier and heater and a blower. The conditioned air enters the chamber, contacts and fluidizes the bed of wet solids. The removed water vapor condenses on the evaporator surface and is drained out of the loop. The dehumidified air flows through the condenser and is heated and re-enter the drying chamber at the desired trial temperature. In this way the latent heat of condensation of the water vapor removed by drying is used to boil the fluid inside the evaporator. Next the energy is recovered and transferred to the airflow as the fluid liquefies inside condenser. The external parts of the drying loop and heat pump circuit are thermally insulated to minimize energy losses to surroundings (Alves-Filho et al., 2002).

The drying chamber is cylindrical with a diameter of 0.25m and the bed of particles had different heights. Sensors were properly placed along the loop and circuit to monitor and to record the inlet-outlet conditions for each component including the air velocity, relative humidity and temperatures.

This work describes the results of drying tests for seven runs each using a bed of 2×10^{-3} m^3 of 4 mm cubes with. The drying temperatures were -10, -5, 5, 15, 25°C and combinations of -10/25°C and -5/25°C. The experimental conditions are given in Table 1 and all runs were conducted under stable fluidization attained with air velocity between 1.5 and 2.5 m/s.

Table 1. Experimental runs and drying conditions with x_0=54.5 %wb for all runs

Run	T, °C	t, hr	x_f, %wb	Sampling, min
1	-10	21:00	12.9	30
2	-10/25	5:00/2:00	11.6	30/10
3	-5	20:00	9.0	30
4	-5/25	5:00/2:00	9.6	30/10
5	5	5:30	13.4	15
6	15	7:45	6.8	15
7	25	2:20	9.2	10

Quality and properties

Sampling and measurement were done during each drying test to characterize quality and properties.

The water activity was measured using an Aqualab meter operating at ambient conditions (25±2°C). The color was measured with an X-Rite 948 spectrocolorimeter. This device records and display the color component mean values from four sets of consecutive measurements.

The rehydration was evaluated in duplicate samples by 10 minutes immersion in water bath at temperatures of 20°C and 37.5°C. The procedure consisted of weighting the samples before and after wetting. The rehydration is expressed as the mass ratio of the wetted to the dried sample, as follows

$$r_a = \frac{m_w}{m_d} \qquad (1)$$

Desorption isotherm

The water activity and equilibrium moisture content was described by Guggenheim-Anderson-De Boer (GAB) model (Keey, 1992)

$$x = \frac{M_m \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 + (C-1) \cdot K \cdot a_w)} \qquad (2)$$

where C, K and M_m are the GAB equation parameters determined from experimental data for all drying runs. Equation (2) can be rearranged as follows

$$x = \frac{a_w}{\alpha \cdot a_w^2 + \beta \cdot a_w + \gamma} \qquad (3)$$

Equations for drying kinetics and rates

Kinetics is often described by Fick's second law irrespective if the mechanisms are diffusion, capillary flow or compression force by shrinkage. Then, the law will be slightly modified to describe moisture diffusion by sublimation and evaporation as occurring in atmospheric freeze-medium temperature heat pump drying. For an unsteady state process and for a spherical material with uniform initial and equilibrium moisture contents, the general equation is

$$\frac{\partial x}{\partial t} = D_e \cdot \nabla^2 x \qquad (4)$$

And the solution is

$$x = \frac{6 \cdot [(x_o - k) - x_e]}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{n^2} \exp(-n^2 \cdot O_d \cdot t) + x_e \qquad (5)$$

The specific drying rate is expressed by

$$N = \frac{6 \cdot [(x_o - k) - x_e]}{\pi^2} \sum_{i=1}^{\infty} -O_d \cdot \exp(-n^2 \cdot O_d \cdot t) \quad (6)$$

where k is a modified drying rate curvature factor. The inverse time constant O_d in the above equations is defined by

$$O_d = \frac{D_e}{r_e^{\prime 2}} \quad (7)$$

The cumulative water removal in the time interval from 0 to t_f is the integral

$$M_w(0 \le t \le t_f) = \int_0^{t_f} \left[\frac{6[(x_o + k) - x_e]}{\pi^2} \times \right.$$
$$\left. \times \sum_{i=1}^{\infty} -O_d \cdot \exp(-n^2 O_d t) \right] dt \cdot \ddot{M}_d \quad (8)$$

where $\ddot{M}_d$ is the ratio of dry matter mass in the bed to the particle surface area.

RESULTS AND DISCUSION

Water activity and moisture content

The measured desorption isotherms points for all drying runs are plotted in Fig. 1. The plot shows that the bovine intestine samples water activity varies from 0.45 to 1 in the moisture content range from 5 to 55 %wb.

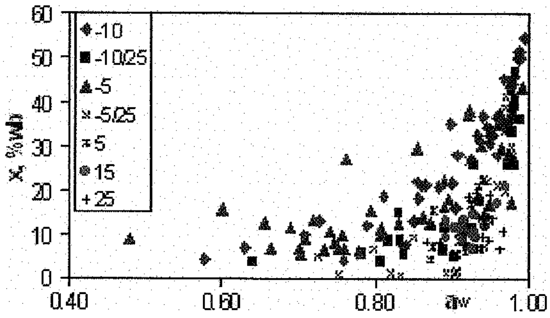

Fig. 1. Desorption isotherms measured data for all bovine intestine samples

The Equations (2) and (3) constants C, K, M_m, α, β and γ for all tests were determined. The parameters α, β, γ and the determination coefficients for selected drying runs are presented in Table 2.

Table 2. The GAB model parameters for selected drying runs

Run	r^2	α	β	γ
1	0.990	31.4145	-76.3514	31.9860
3	0.903	8.6169	-44.2543	36.3544
5	0.921	-112.6012	187.0871	-72.7826
6	0.972	159.0051	-360.2817	203.6438
7	0.974	-886.0037	1554.9560	-672.8944

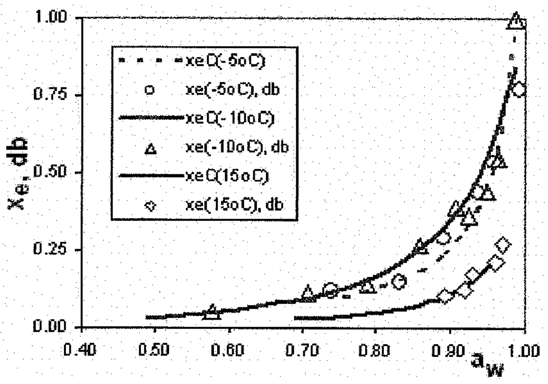

Fig. 2. Experimental isotherms for runs 1, 3 and 6 and description by Equation (2)

The relationship between equilibrium moisture content versus water activity is obtained by Equations (2) and (3) with parameters. This is shown in Fig. 2 for runs 1, 3 and 6, which were dried from -5 to 15°C. The results indicate that the bovine intestine runs have similar trend in water activity and equilibrium moisture content. The values obtained by GAB equation were in good agreement with measured data as shown in Fig. 2 and the r^2 values in Table 2.

Drying curves and rates

Atmospheric freeze drying combined with medium temperature drying involves removal of moisture from the solid both by sublimation and evaporation. In such combined process the moisture is removed sequentially by ice sublimation and liquid evaporation by avoiding structural collapse (Alves-Filho and Roos, 2006). An important aspect is that as drying progresses the drying curve moves asymptotically to equilibrium and the drying rate drops quickly to 0. Therefore, it is relevant to study the behavior of the graphs for the kinetics and drying rates and to select the most feasible way to remove the residual moisture content.

The measured data were fitted using Equations (5) to (7) and the constants determined by minimizing the sum of squares of deviations between observed and calculated values. The kinetics and rates for runs 5, 6 and 7 are plotted in Figures 3 to 8 and the equation constants k, O_d and D_e are given in Table 3. As expected, the values of O_d and D_e decrease as the drying air temperature drops and the curvature k factor varies according to the initial moisture and the dispersion or spread of the measured data. Consistently, the effective diffusivity reduced from 1.0053×10^{-9} to 0.2132×10^{-9} m^2/hr as the drying temperature decreased from 25 to 5°C.

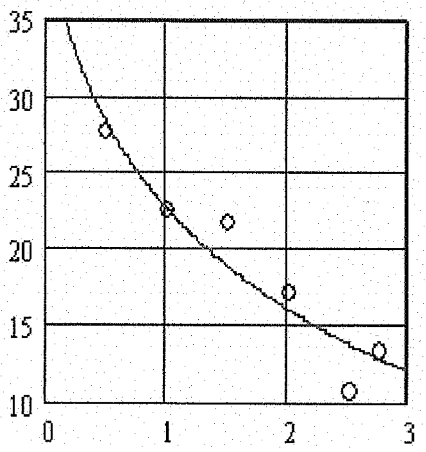

Fig. 3. Moisture content (%wb) versus time (hr) for run 5 at 5°C

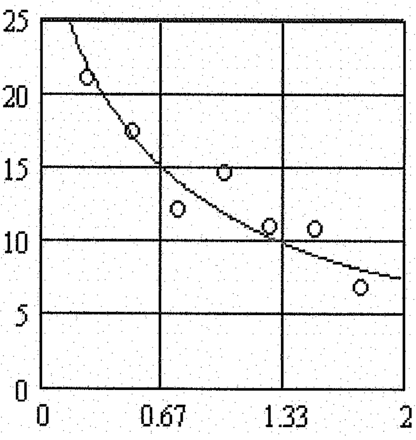

Fig. 4. Moisture content versus time for run 6 at 15°C

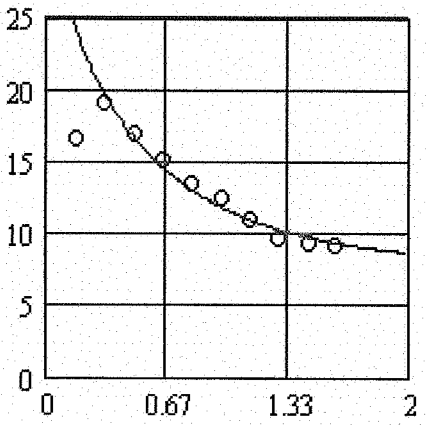

Fig. 5. Moisture content versus time for run 7 at 25°C

The drying rates were determined using Equations (6) and (7) and the parameters in Table 3. The results are plotted in Figures 6 to 8 and indicate that the drying rate curves have the same pattern for all runs. It is initially high and decreases asymptotically to zero as drying time increases or as the moisture content approaches the equilibrium. Moreover, the plot shows that the higher the drying air temperature the higher the initial drying rate.

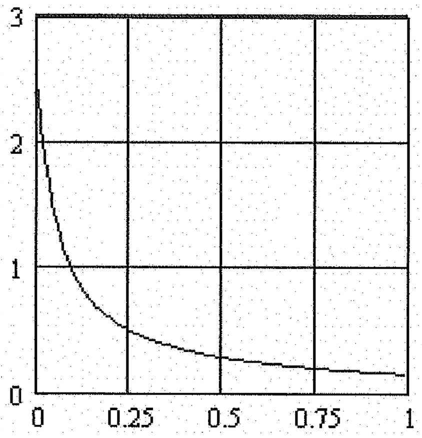

Fig. 6. Specific drying rate (kg water per kg dried matter per hr) versus drying time for run 5 at 5°C

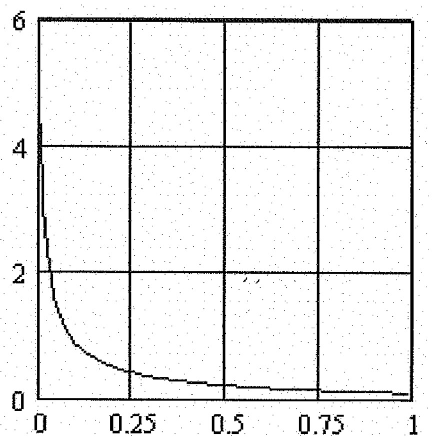

Fig. 7. Specific drying rate versus drying time for run 6 at 15°C

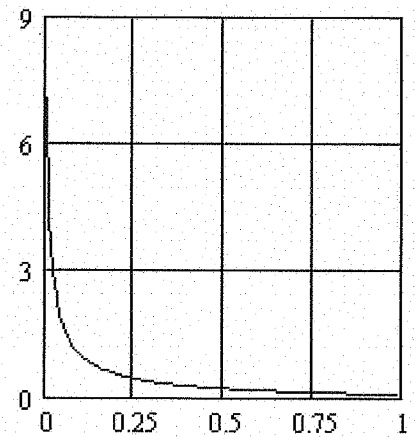

Fig. 8. Specific drying rate versus drying time for run 7 at 25°C

Table 3. Kinetics parameters for the drying runs of bovine intestine

Run	T	k	x_e	O_d	$D_e \times 10^9$
5	5	9.5	3	0.35	0.2132
6	15	17.5	5	1.05	0.6397
7	25	15.5	8	1.65	1.0053

Also, the plots indicate that bovine intestine drying rates are similarly to gels since it has no clear constant drying period and no apparent critical moisture content. Thus, it behaves as a nonporous hygroscopic material where the drying rate drops sharply from a high initial value and in an ever decreasing rate that moves asymptotically to zero as drying time progress.

Therefore, the drying rate tends asymptotically to 0 during combined moisture removal by sublimation and evaporation or similar processes. Assuming that $\ddot{M}_d$ equal to 1 kg/m^2, Equation (8) provides the actual cumulative (M_w) and relative cumulative (M_w/M_{wo}) moistures, which were calculated for different time intervals and are presented in Table 4. It indicates that, for run 5 and time interval from 0 to 0.5 hour, the M_w and M_w/M_{wo} are 0.339 and 0.48, and for time interval from 0 to 2 hours M_w and M_w/M_{wo} are 0.542 and 0.77 while at maximum time M_w/M_{wo} is 1. For run 6 in and time intervals from 0 to 0.5 hour and from 0 to 2 hours the values for M_w/M_{wo} are 0.68 and 0.94. For run 7 in and time intervals from 0 to 0.5 hour and from 0 to 2 hours the values for M_w/M_{wo} are 0.78 and 0.98.

Thus, after 2 hour of drying all these runs have lost most of their original moisture. The percentage of relative cumulative moisture removal for runs 5, 6 and 7 is 77%, 94% and 98%. Therefore, based on the cumulative rates, a feasible drying process would remove moisture up to 2 hours only. Certainly, it would be more practical and economical to remove the residual moisture fraction by simply resting the product or by using an intermittent drying process.

Table 4. Cumulative water removal for different time intervals

Run	t_f	0.5	1.0	2.0	Max
5	**M_w**	**0.339**	**0.440**	**0.542**	**0.702**
	M_w/M_w	**0.48**	**0.63**	**0.77**	**1.00**
6	**M_w**	**0.331**	**0.403**	**0.458**	**0.485**
	M_w/M_w	**0.68**	**0.83**	**0.94**	**1.00**
7	**M_w**	**0.391**	**0.456**	**0.493**	**0.501**
	M_w/M_w	**0.78**	**0.91**	**0.98**	**1.00**

Drying temperature effect on product quality and properties

The impact on color and rehydration ability was studied during and after the drying process.

The product surface color depends on drying conditions and is an important factor on quality. The product surface may change due to close thermal contact with the drying air.

Spectrophotometric methods are used to measure differences in color during the drying time. The *L*, *a* and *b* coordinates in CIELAB space were measured. The *L* component is associated with the sample's brightness and it ranges from full black at 0 to full white at 100 units. The coordinate *a* is associated with red-green spectrum, coordinate *b* with yellow-blue and both range from -60 to +60 units. The color change in component *L* for run 1 (-10°C) and run 6 (15°C) are plotted in Fig. 9.

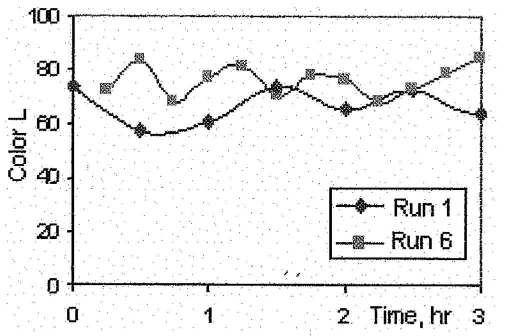

Fig. 9. The color component *L* versus drying time for runs 1 and 6

It shows that samples dried at positive temperatures had higher brightness than the run freeze dried.

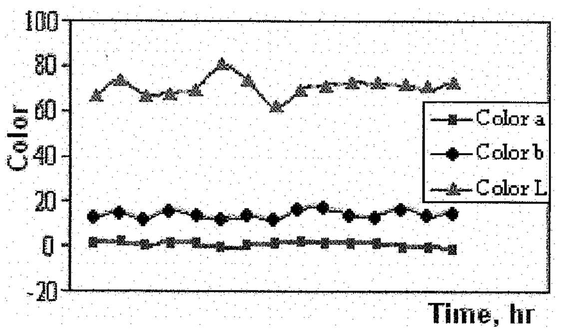

Fig. 10. Color components versus time for samples dried at 25°C

The color components for run 7 were continuously measured during drying and the results are plotted in Fig. 10. It is observed that the *L* and *b* values fluctuate only in the intermediate position while the initial and final colors remain unchanged. The *a*, *b* and *L* values of 0, 18 and 75 suggest that this sample is pale-yellow and bright, which is the preferred color by the end-users. Therefore, the temperature of 25°C maintained the colors for run 7 during drying. This temperature was the highest used and the atmospheric freeze dried samples kept the original color constituents.

The rehydration ability was taken as the average value obtained from triplicate measurements. Figs 11 and 12 show the results for white and dark bovine intestine tissues samples.

The Figs 11 and 12 show that all freeze dried dark tissue samples had 2.5 to 4 times higher rehydration than freeze dried white tissue samples. When dried between 5 the dark and white tissue samples had similar rehydration but the white run had lower r_a at water bath at 20°C.

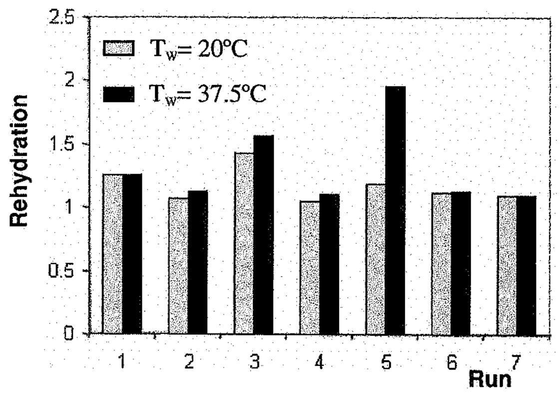

Fig. 11. Rehydration ability for white samples

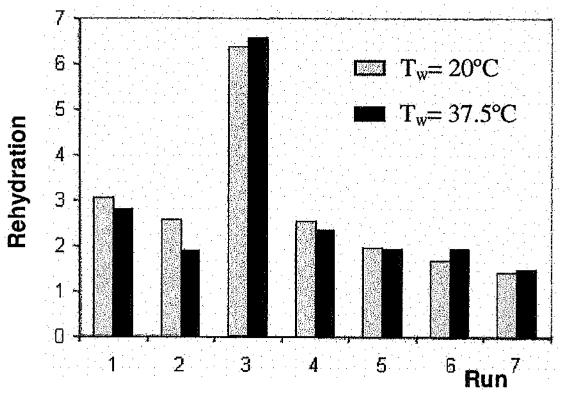

Fig. 12. Rehydration ability for dark samples

For the same tissue batch the freeze dried dark samples had slightly higher rehydration in water bath temperatures of 20°C. The highest value of rehydration was for run 3 dried at -5°C and lower values for runs 6 and 7 that were dried at 15 and 25°C, respectively.

For the white tissue samples the rehydration was slightly higher at water bath of 37.5°C. The higher values were for runs dried at 5 and -5°C and lower for runs 2, 4, 6 and 7, from which the last two dried at 15 and 25°C, respectively.

CONCLUSIONS

This work provides the basis for selection of the operating conditions to dry bovine intestine with improved quality and properties. Freeze dried samples had higher rehydration than medium temperatures dried runs in both water batch conditions. Also, in both water bath, the dark run 3 dried at 5°C had the highest rehydration among all runs (6.5 compared 1.5 when dried at 25°C). Runs dried at both at low and medium temperatures were bright. A typical sample was pale-yellow and bright, which is a preferred color by the end-users. The results suggest that atmospheric freeze drying conditions keeps the original bovine intestine colors.

The estimated water activity values by the GAB model with proper parameters were in good agreement with experimental data. The modified solution for Fick´s law described well the diffusion in combined sublimation and evaporation with consistent values for effective diffusivity. An important conclusion based on the determined drying rates is that, after 2 hours of drying, most runs had lost 77% to 98% of their initial moisture contents. Thus, an economical drying process would be stopped at or before 2 hours of operation. The residual moisture fraction would be removed simply by letting the product to rest, tempering or by an intermittent drying process.

NOMENCLATURE

a_w	water activity	
C	constant	
D_e	effective moisture diffusivity	m^2/hr
K	constant	
k	modified rate curvature factor	%wb
M_m	constant	
M	cumulative water removal	kg
m	mass	kg
N	specific drying rate	kg/kg·hr
n	coefficient 1, 2, ... ∞	
O_d	inverse drying time	hr^{-1}
r_a	rehydration	kg/kg
r_e	radius of a sphere with the same volume as a cube	m
T	temperature	°C
t	time	hr
x	moisture content	%wb, %db

Greek letters

α	constant
β	constant
γ	constant

Subscripts

e	equilibrium
d	dried
f	final
o	initial
w	wetted, water

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