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Note

Performance of an Accelerated Method for the Determination of Equilibrium Moisture Content

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The performance of a method that could accelerate the sorption process of biological materials was evaluated with respect to rapidness (expedition of sorption), comparability of equilibrium moisture content (EMC) values with the control (conventional static method) and effect of specific airflow rates on the rate of sorption, EMC, and sorption rate constant. For this purpose, a simple, compact, and inexpensive experimental setup was fabricated with a facility to agitate the conditioned air around the sample. Different forms of raw and parboiled rice kernels (rough rice, brown rice, and milled rice) of an Indica variety were used as sample material. Both adsorption and desorption processes were investigated under conditions of high humidity (80.27%) and tropical temperature (30℃). Sorption rate constants based on a first-order kinetic model showed a faster rate of sorption by the accelerated method compared to the control for determining the equilibrium moisture content (EMC). The accelerated method required 40–60% less time compared to the control method (static) to reach sorption equilibrium. Specific airflow rates of 79.14 m^3/min-m^3 and lower showed the most similar EMC values to those of the control. The results showed that this method can be applied in order to expedite the sorption process.

Keywords: equilibrium moisture content, accelerated, performance

Introduction

Due to their hygroscopic nature, biological materials can gain or lose moisture depending on the material-air interaction that take place during storage. An understanding of adsorption and desorption of moisture in farm products and the corresponding methods of calculation is very important for storage and drying technology.

The process of adsorption and desorption of moisture by farm products is very slow. The experimental determination of sorption isotherms is time-consuming; samples must be left in a particular environment for a prolonged period (several weeks) to reach equilibrium in a normal static process depending on the initial moisture content of the sample. A dynamic method is faster (Bala, 1997).

Cereals and grains are alive, expending glucose and generating CO_2, water vapor and heat through respiration. Temperature and moisture content are two important physical variables contributing to the deterioration of these products. Conditions of high temperature and high moisture content are ideal for promotion of mold growth; mold that propagates on kernels generates even more moisture and heat. Mold grows easily within a couple of days on moist grain if the ambient temperature expedites the process of mold growth. The moisture content obtained with a relative humidity of over 80% is not the true moisture content (Bala, 1997; Seo, 1995). It is necessary to maintain humidity below 75% to prevent mold growth (Yamashita, 1993). Respiration of grain tends to increase with higher moisture content, and temperature increases due to respiration can not be disregarded. It has been reported that at a moisture content of 17% and a temperature of 30℃, a loss of glucose of 1% occurs within 22 days, while moisture content and temperature increase by 0.46%/month and 63℃/month, respectively, due to respiration by rough rice, based on an assumption of complete heat insulation and no ventilation (Seo, 1995). Dillahunty *et al.* (2000) also reported that moisture content and temperature greatly affected the respiration of rice. All these factors contribute to serious error in the detection of the actual equilibrium moisture content (EMC) of the sample. It can be seen that grain is vulnerable at ambient conditions of high humidity and high temperature as well as over prolonged periods of exposure.

Sufficient water becomes available to sustain microorganism growth at a grain-water equilibrium relative humidity of 70–80% for molds and 90% for bacteria in the intergranular air (Semeniuk 1954). Even chemically treated grain experiencing a large moisture increase can deteriorate in quality due to microorganism growth (Stewart, 1975). Most equilibrium moisture data of hygroscopic materials have been obtained by placing a small sample of the material in a sealed container with a sulfuric acid solution of specific concentration or saturat-

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ed salt solution that produces a certain specific water vapor pressure or relative humidity. Henderson (1952) recognized difficulties associated with this procedure, as long periods may be needed to reach equilibrium if the air inside is not stirred. Consequently, samples may be removed before reaching an equilibrium condition. Moreover, due to the long test period sometimes required, samples exposed at approximately 85% relative humidity or higher may mold before the endpoint is reached. The moisture of respiration of the mold will give a falsely high equilibrium moisture content. Henderson (1952) also reviewed some methods adopted in observing equilibrium moisture relationships, such as absorption tower (Wilson and Fuwa, 1922), direct measurement of vapor pressure (Legault *et al.,* 1948), electric hygrometer (Brockington *at el.,* 1949), and wet- and dry-bulb readings (Hukill, 1950). He remarked that each of these procedures had certain advantages, but none had proved satisfactory in every respect.

To determine the sorption isotherms of chilli by a dynamic method, Hossain *et al.* (1998) used an arrangement with a facility for stirring the air inside a plastic container. The setup had some problems with durability and the mode of airflow, and it was not compact or rigid. A rapid method developed by Kimura and Masuda (1978) took 0.5–2 days to reach an equilibrium moisture content for rice. The principle of the method was to measure equilibrium relative humidity (ERH) between the air inside and a grain sample kept in a hermetic vessel using a hygrometer. The method needed large sample amounts (120 g) and the equipment was a little bigger, requiring a large setup to maintain a constant temperature. The accuracy of ERH measurement of the air inside will depend on the size of sample in relation to the space inside the chamber. Chen and Morey (1989) used a step-by-step ERH-determining method that needed 6–18 hours to reach ERH. The setup was complicated and equipped with temperature and RH sensors that required calibration by a complex method.

In light of the above circumstances, it is worthwhile to investigate the suitability of a more simple and compact and less costly method for determining the sorption behavior of agricultural products, which can shorten the time required in the sorption process. Since little information is available on the effect of agitation of air around the sample, current research is devoted to studying the effectiveness of an accelerated method during sorption, using rice kernels. Almost all the equilibrium moisture content (EMC) values on paddy in the previous literature are related to raw paddy of *Indica* or *Japonica* variety; there is no information, or very little, about the parboiled kernels. The objective of this study was to investigate the performance of the experimental setup with respect to rapidness (expedition of sorption), comparability of EMC values with those of the control (conventional static method) and the effect of specific airflow rates on the rate of sorption, EMC, and sorption rate constant for rice kernels (raw and parboiled rough, brown, and milled rice).

Materials and Methods

Materials Rice kernels of the indica variety (*Belle patna*), harvested in October 2004 at the farm of the Japan International Cooperation Agency (JICA), Tsukuba, Japan, and stored at 5℃ for five months in a refrigerated warehouse, were used in this study. The initial moisture content of the rough rice was about 16% (d.b.). Both raw (non-parboiled) and parboiled kernels, including rough rice, brown rice and milled rice, were used to observe the effectiveness of the method on the sorption process.

A saturated solution of potassium bromide (KBr) was used to create an environment of 80.27% (±0.21%) relative humidity at a temperature of 30℃ (Troller and Christian, 1978). The salt was of research grade (Wako Pure Chemical Industries Ltd., Japan).

Preparation of the sample To obtain parboiled paddy, raw paddy was washed with normal water to remove dust and immature kernels and soaked in a water bath at 65℃ for 3 hours followed by steaming in an autoclave at 120℃ for 5 minutes (Sudeepa, 2004; Kimura, 1995; Uddin *et al.,* 1987). The samples were then dried to a 12% moisture content under the shade at room temperature (24℃, 30% RH) in order to avoid any unwanted stress on the kernels as might occur in mechanical drying. After drying, samples were stored at room temperature in sealed polyethylene bags for moisture equilibration and stabilization.

Brown rice was obtained by dehusking the raw and parboiled rough rice in an impeller-type husker (FCS type, Otake Co., Oharu, Japan) in a single pass. Brown rice was then milled in a vertical friction-type laboratory milling machine (VP31T, Yamamoto Co., Tendu, Japan) to obtain milled rice. The degree of milling was kept at about 8% for both the non-parboiled and the parboiled sample.

Samples with higher moisture contents were prepared by wetting the samples (Chen, 2000). This was done by fine spraying of distilled water on a single-kernel layer of samples. The amount of water was predetermined on the basis of initial weight, initial moisture content and the desired moisture content of the samples. Spraying was done intermittently and after a single spray, the sample was stirred and mixed thoroughly and left for couple of minutes in a heap to be homogenized. All the samples were then sealed in plastic packets and kept in a refrigerated warehouse at 5℃ for 2 weeks to homogenize the moisture content in the kernels. Before starting the experiment, samples were kept overnight at room temperature in the sealed packets for thawing. The sample size for each treatment was about 3 g, with two replications. Samples were set into small polyethylene packets (film thickness 0.04 mm, width 75 mm, and folding height 50 mm) which were opened wide by folding the upper half before being placed inside desiccators containing saturated salt solution (Anonymous, 2004). The thickness of the sample inside the sample holder was about two kernels.

Moisture content determination The ISO R712 standard method (ISO, 1979) was followed (Chen, 2003) to determine the moisture content of the kernels. Ground sam-

ples of 5 g were dried at 130℃ for 2 hours in an air oven. Samples were ground using a Kett TQ-100 grain grinder (Kett Electric Laboratory, Japan) and immediately weighed to avoid moisture loss. An air oven (ESPEC, convection oven LC-122: Tabai Espec Corp., Japan), was used in this study. Moisture content is expressed on a dry basis.

Apparatus/Experimental setup A schematic of the experimental setup for the accelerated method is shown in Fig. 1. A glass desiccator was used as a closed chamber maintaining a constant environment. The inside of the desiccator was equipped with a small but durable compact flat-type fan (Power Logic, model: PL80S12M-1) to agitate the air inside. This was a DC brushless, sleeve-bearing fan with an impeller diameter of 80 mm. It was suspended under the desiccator plate to direct the air stream towards the salt solution instead of the sample directly. The desiccator plate was kept elevated from its normal position in order to facilitate easy flow of the conditioned air between the upper and lower parts. The wires, connected to the fan and temperature monitoring system, were inserted through a small opening on the lead of the desiccator, which was then sealed. The setup evaluated in this study is more durable, rigid, and compact than that used by Hossain *et al.* (1998), and is improved with respect to the container, type of fan, position of fan relative to the sample, and direction of airflow. Moreover, the arrangement of a PID (proportional plus integral plus derivative action) temperature controller (TF3-10, Keyence Corp., Japan) with a relay switch was incorporated into the setup used in the current study in order to monitor and control the internal temperature of the desiccator. The effectiveness of the accelerated method (specific airflow rate 79.14 m^3/min-m^3) was first examined with respect to the control (static), and then the effect of different specific airflow rates on sorption was studied.

Temperature profile Thermocouples were used for monitoring temperature profiles inside the two desiccators (with and without a fan) and the incubator. Temperature profiles were monitored continuously and simultaneously by a data acquisition system (NR-250, Keyence Corp., Japan) interfaced with the software WAVE THERMO (NR-3H3W) for Windows. Both of the desiccators were of same size and placed inside an incubator (Eyela, SLI-600N, Tokyo Rikakikai Co. Ltd., Japan) with a temperature setting of 29.7℃.

Kinetic analysis A first-order kinetic model, as shown in Eq. (1), was used to perform least squares nonlinear regression analyses using SigmaPlot (Jandel Corp., San Rafael, CA). The kinetic parameters were estimated for a better understanding of the rate and index of change in moisture content of the samples during the sorption process. The equation is as follows:

$$M = M_e + (M_o - M_e)exp(-kt) \quad (1)$$

where M is the moisture content (%) at time t, M_e is the final moisture content (%), M_o is the initial moisture content (%), and k is the kinetic sorption rate constant (h^{-1}).

Specific airflow rate An arrangement consisting of a circular tunnel (80 mm diameter and 120 mm long) with the fan fixed at one end was used to determine the average airflow rate and air velocity, which was measured by a digital anemometer (Climomaster-6542, Kanomax Japan Inc.) following the JIS standard method B8330. The readability of the device was 0.01 m/s and its precision was ± (3% of reading + 0.1) m/s. The average airflow rate and air velocity obtained from the fan was 66 m^3/h (1.1 m^3/min) and 3.66 m/s, respectively. The specific airflow rate was calculated using the following relation:

$$Q_s = \frac{Q_{av}}{V} \quad (2)$$

where Q_s is the specific airflow rate (m^3/min-m^3); Q_{av} is the average airflow rate (m^3/min); and V is the volume of the desiccator (m^3). Three specific airflow rates (42.31, 79.14, and 203.70 m^3/min-m^3 volume) were applied to observe their effect on the sorption process at a constant temperature. For this purpose, desiccators of three different dimensions (0.026, 0.0139, and 0.0054 m^3) were used with the same type of fan inside.

Data recording Weight loss or gain by the samples was measured using an electronic balance (LIBROR AEG-220G, Shimadzu Corp., Japan), the readability of which

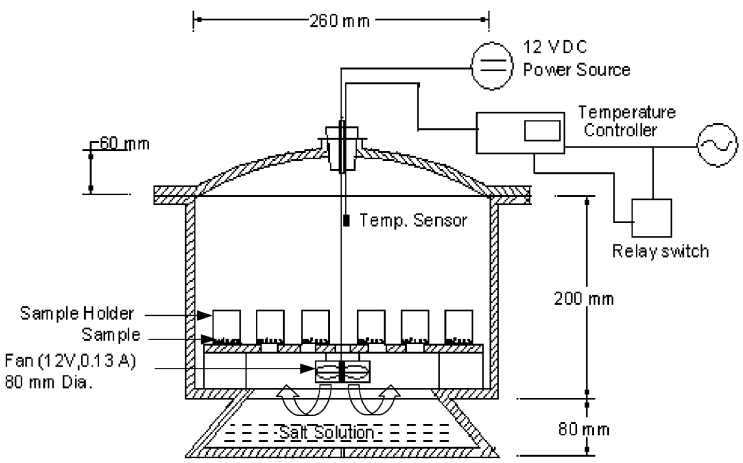

Fig. 1. Schematic showing the experimental setup of the accelerated method.

was 0.0001 g. The sample inside the sample holder was weighed quickly and immediately replaced in the desiccator. Weighing of each sample was performed in the closed chamber of the balance within a few seconds to minimize moisture loss or gain from atmosphere. Equilibrium conditions were considered to have been reached when the change in moisture content of the sample was $\leq$ 0.004% per hour, or three consecutive readings were the same.

Results and Discussion

The performance of the method used to determine the EMC in this study is described in terms of potential to expedite the sorption process, reliability of the results and the effect of specific airflow rates on the sorption process.

Temperature Profile Temperature profiles inside the desiccator fitted with a fan used in the accelerated method, the control (without fan), and the incubator are shown in Fig. 2. Among these, temperature profiles did not show any remarkable change. The temperature inside the control was found 0.3℃ higher than inside the incubator. From an initial temperature of 29.7℃, the temperature scale inside the accelerated system ran parallel with that of the control, with a rise of only 0.3–0.4℃, and that of the incubator, with a rise of 0.6–0.7℃. This micro-level increase in temperature may also be attributed to the specific heat capacity of the thick glass desiccator wall. This minute increase in temperature can easily be eliminated by use of a PID (proportional plus integral plus derivative action) temperature controller with a relay switch.

Rate of Sorption Figure 3 depicts the rate of moisture adsorption and desorption by the kernels. For both parboiled (Figs. 3 a, b, and c) and raw (Figs. 3 d, e, and f) kernels, the accelerated method resulted in a faster rate of adsorption and desorption during the sorption process. The rate of sorption was very fast during the early stage and most of the sorption took place within 24 hours from initiation. In case of the accelerated method, equilibrium was reached for both adsorption and desorption within 48–72 hours for all the samples. In contrast, samples in the control method with the same initial moisture contents took at least 120 hours for desorption and even more for adsorption (264 h, not shown) to reach equilibrium, showing a very slow rate. In both methods (accelerated and control), a normal hysteresis effect was found for all the samples.

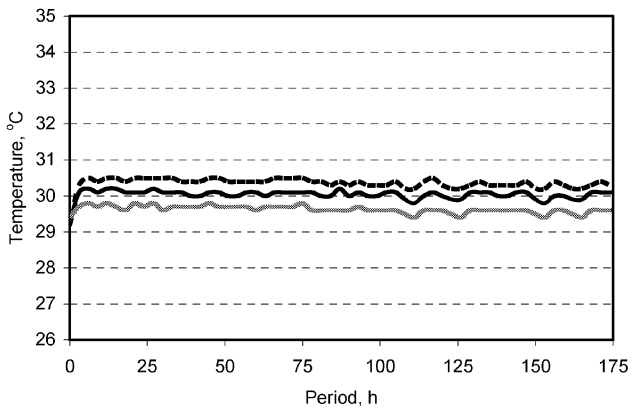

Fig. 2. Temperature profiles inside the arrangements: ------ accelerated method; —— control; —— incubator.

Equilibrium Moisture Content Table 1 shows the equilibrium moisture content (EMC) of both parboiled and raw rice kernels obtained by the accelerated method (specific airflow rate 79.14 m^3/min-m^3) and the control method at 30℃ and 80.27% RH. Parboiled rice kernels showed slightly lower EMC values than raw rice kernels. EMC values of parboiled kernels were lower by approximately 1.5 percentage points and less than 0.5 percentage points at desorption and adsorption, respectively, according to both methods. This phenomenon might be attributed to the change of state of the parboiled kernels during parboiling and subsequent drying (gelatinization and retrogradation) affecting its physico-chemical properties (Bhattacharya and Subba Rao, 1966; Raghavendra Rao and Juliano, 1970; Bhattacharya, 2004; Kimura, 1995; Sudeepa, 2004). The milled rice kernel showed the highest EMC values, followed by brown rice and rough rice. This might be due to the presence of extra coating, (bran in brown rice and both bran and husk in rough rice), which have different moisture diffusivity values compared to the endosperm. The equilibrium moisture content values obtained by the accelerated method (sp. airflow rate 79.14 m^3/min-m^3) and control method for all types of kernels showed high consistency. This indicates that the accelerated method can be used to determine the EMC of the materials accurately.

Sorption Rate Constants For all types of rice kernels, rate constants based on the first-order kinetic model were found to be considerably higher for the accelerated method than for the control (Table 2). This trend existed for both adsorption and desorption processes, indicating faster sorption by the accelerated method. Rate constants for raw kernels in the accelerated method (with a specific airflow rate of 42.31 m^3/min-m^3) were within a range of 0.79–0.83 h^{-1} for adsorption and 0.73–0.81 h^{-1} for desorption, while those of the control were 0.50–0.73 h^{-1} and 0.52–0.67 h^{-1}, respectively. Similar trends were found for the parboiled kernels. It is evident from Table 2 that the sorption rate constants during the adsorption process (unlike the desorption process) did not increase, but rather slightly decreased with increasing specific airflow rate. The change was not very significant. The probable reason is explained with respect to the effect of specific airflow rate in a later section.

A steeper slope for sorption rate yields a higher-value sorption rate constant. Therefore, the accelerated method, which showed higher rate constants compared to the control method, indicated faster sorption. This section supports the findings of the previous section on sorption rate; the accelerated method expedited the sorption.

Effect of Specific Airflow Rate The specific airflow rates applied in this study in order to observe the effect on rapidness of sorption process did not result in marked-

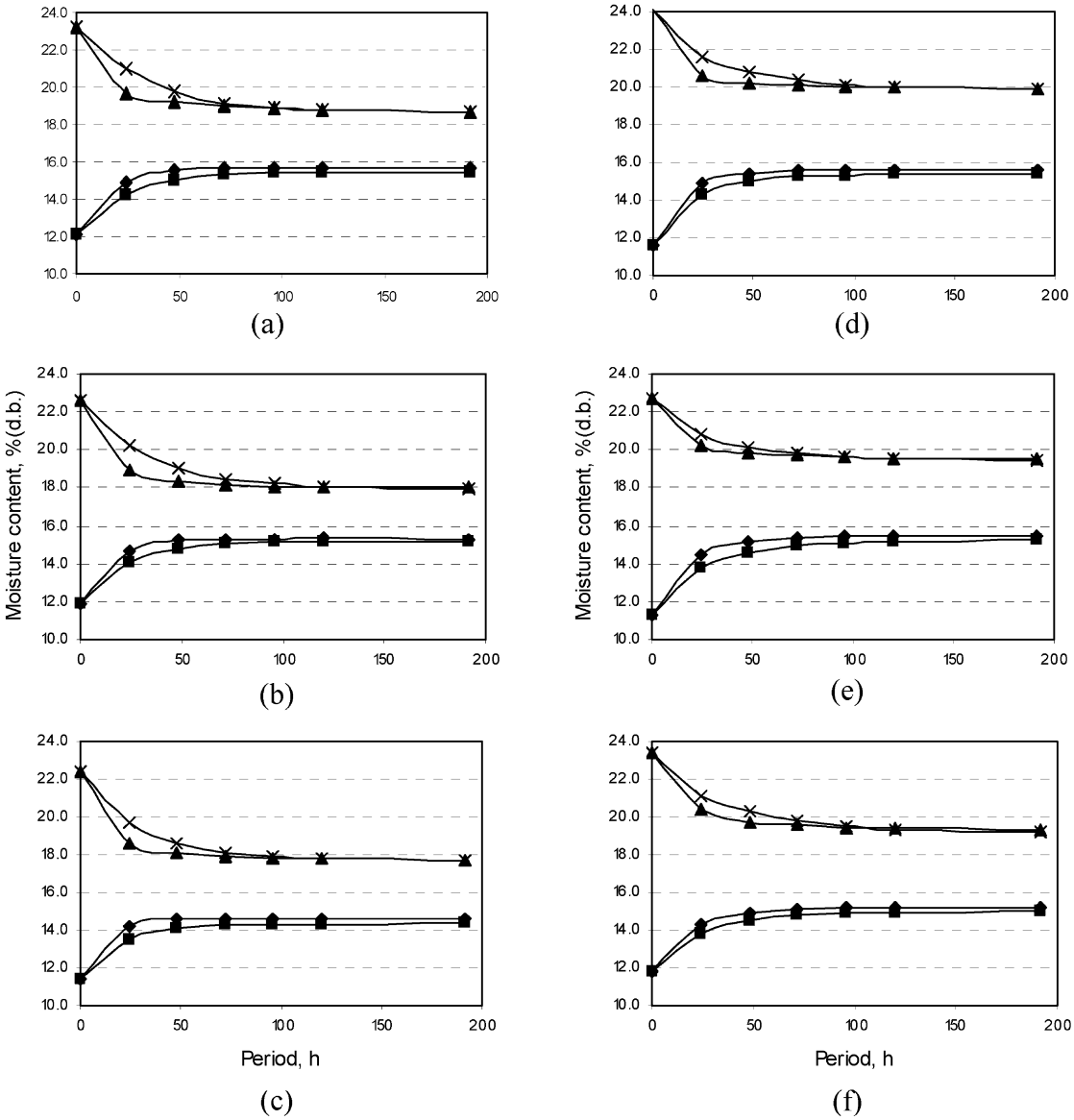

Fig. 3. Patterns of moisture content changes during sorption of rice kernels by accelerated and control methods: ◆ adsorption-accelerated; ■ adsorption-control; ▲ desorption-accelerated; × desorption-control
(a) milled, (b) brown, and (c) rough rice, parboiled kernels and (d) milled, (e) brown, and (f) rough rice, raw kernels.

Table 1. Equilibrium moisture content (%, d.b.) of rice kernels at 30℃ and 80.27% RH.

Process	Method	Parboiled kernels			Raw kernels		
		Milled	Brown	Rough	Milled	Brown	Rough
Adsorption	Accelerated*	15.5	15.2	14.6	15.6	15.4	15.1
	Control	15.5	15.2	14.4	15.5	15.3	15.1
Desorption	Accelerated*	18.8	18.1	17.8	20.1	19.7	19.4
	Control	18.7	18.0	17.8	20.0	19.5	19.3

*Specific airflow rate 79.14 m^3/min-m^3.

ly different values. Figure 4 shows the effect of specific airflow rates on the sorption of parboiled (Figs. 4. a, b, and c) and raw (Figs. 4. d, e, and f) milled, brown, and rough rice kernels. A higher specific airflow rate (203.70 m^3/min-m^3) resulted in a minute rise in desorption associated with a slightly slower adsorption. Rate constants (Table 2) from the kinetic model obtained for three different specific airflow rates also showed agreement with this finding. A higher specific airflow rate (203.70 m^3/min-m^3) was associated with higher deviation of the EMC values from those of the control EMC.

In the accelerated method, turbulence in the fluid forced

Table 2. Effect of specific airflow rates on sorption rate constants (k-value, h^{-1}) of sorption process (at 30℃ and 80.27% RH) of rice kernels in accelerated method and control (without airflow).

Process	Sp. airflow rate, (m^3/min-m^3)	Parboiled			Raw		
		Milled	Brown	Rough	Milled	Brown	Rough
Adsorption	42.31	0.87	0.86	0.89	0.82	0.83	0.79
	79.14	0.81	0.84	0.88	0.75	0.66	0.76
	203.70	0.81	0.84	0.87	0.77	0.81	0.78
	Control	0.61	0.64	0.75	0.62	0.50	0.73
Desorption	42.31	0.78	0.74	0.86	0.77	0.81	0.73
	79.14	0.68	0.75	0.76	0.78	0.82	0.71
	203.70	0.85	0.84	0.86	0.81	0.88	0.76
	Control	0.28	0.27	0.35	0.52	0.67	0.57

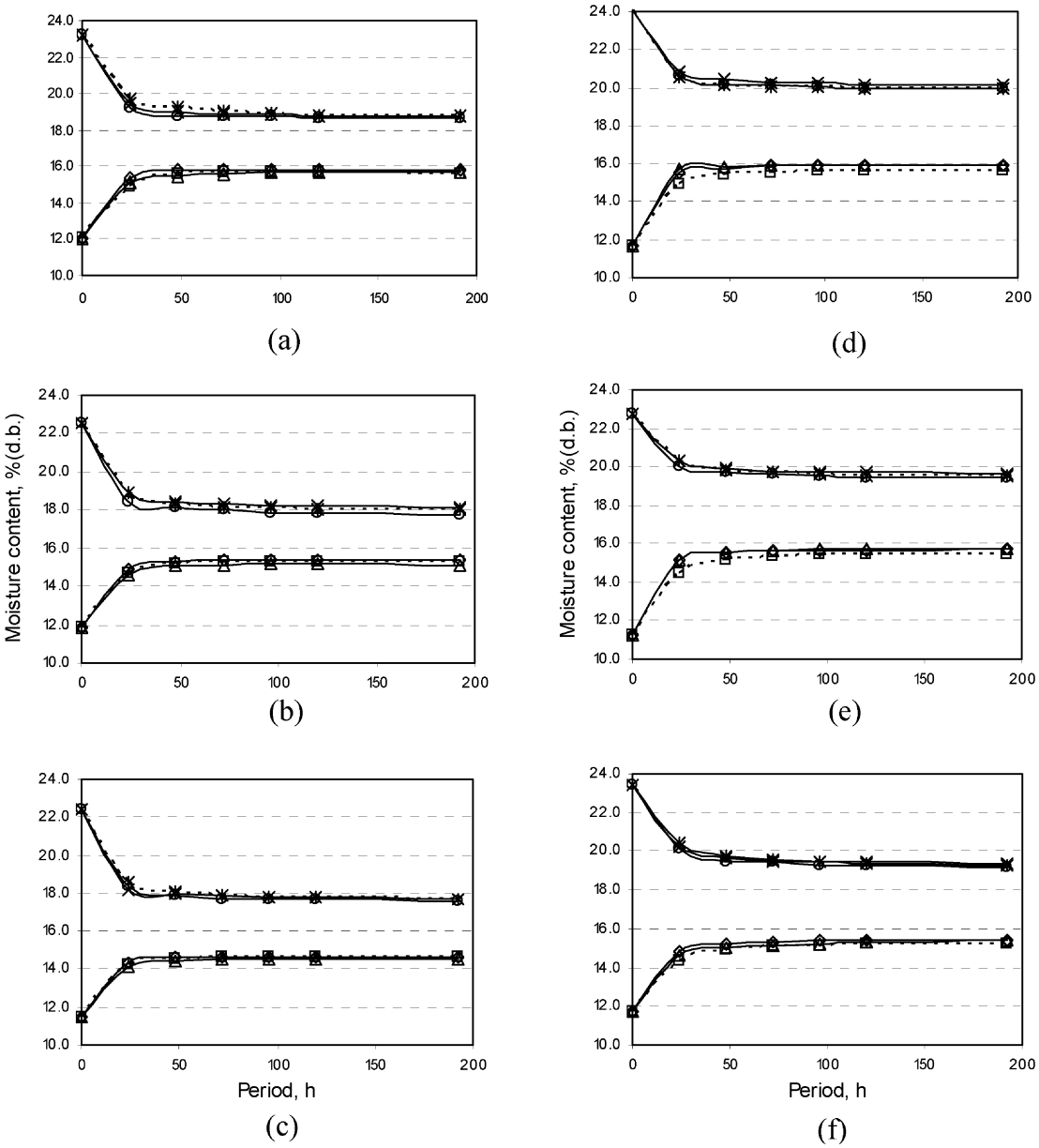

Fig. 4. Effect of specific airflow rates on rapidness of sorption of rice kernels by the accelerated method: ◇ adsorption-1; -⊟- adsorption-2; △ adsorption-3; × desorption-1; -✳- desorption-2; ○ desorption-3. Specific airflow rates: (1) 42.31; (2) 79.14; (3) 203.70 m^3/min-m^3. (a) milled, (b) brown, and (c) rough rice, parboiled kernels and (d) milled, (e) brown, and (f) rough rice, raw kernels.

the vapor molecules to become attached to the material surface containing a moisture gradient, which resulted in a faster adsorption rate compared to the control. However, the increased airflow rate may have dominated the convective mass transfer by carrying away the vapor molecules from the solid surface as well as from the fluid with a higher mass concentration at the boundary layer. With the increased airflow rate, the water vapor molecules may have been disturbed and allowed a shorter period to stay on the solid surface than those required for settling. These are all possible reasons that the increased airflow rate did not raise the adsorption rate, instead slightly decreasing the adsorption rate.

The EMC values obtained for specific airflow rates of 79.14 m^3/min-m^3 and lower showed the most similar EMC values to those of the control. Therefore, specific airflow rates within this range may be applied to obtain the most reliable results.

Conclusions

The performance of the method used in this study to determine EMC values is described in terms of potential to expedite the sorption process, reliability of the results and effect of specific airflow rate on rapidness of sorption process as well as accuracy of the EMC values.

The accelerated method resulted in a faster rate of sorption, particularly at the early stage of the sorption process. This method required 40–60% less time compared to the control method (static) to reach sorption equilibrium. Rate constants for both raw and parboiled kernels were higher in the accelerated method, confirming that this method can expedite the sorption process. The EMC values obtained by the accelerated method with specific airflow rates of 79.14 m^3/min-m^3 and lower showed the most similar EMC values to those of the control, indicating the accuracy of readings obtained by the accelerated method. The range of specific airflow rates (42.31–203.70 m^3/min-m^3 volume) applied in this study did not show much difference in terms of rapidness of sorption. A higher specific airflow rate (203.70 m^3/min-m^3) resulted in a minute rise in desorption but slower adsorption, and caused higher deviation of the EMC values from the control values. The setup of the accelerated method evaluated in this study is simple, compact, and less costly than previous methods and may be employed to determine the equilibrium moisture contents of biological materials under conditions of relatively high temperature and relative humidity.

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