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REGULAR PAPER

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Evaluation of solid state of rice flours produced by different milling processes using ATR-FTIR spectroscopy

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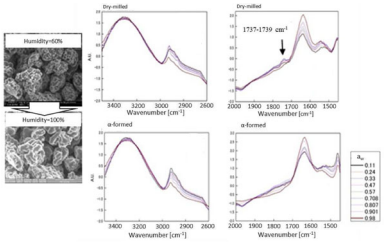
ABSTRACT

This study evaluated the influence of the milling process on solid state of rice flours according to water activity using ATR-FTIR. A band at 1740 cm^{-1} attributed to the C=O stretching of lipids was detected for crystalline samples, and it disappeared at a high a_w range. The CH band at 2930 cm^{-1} of crystalline samples gradually shifted to a higher wavenumber with a_w. This band of the α-formed and wet-milled samples shifted to higher wavenumbers above $0.8a_w$. A band due to OH stretching mode in the 3500-3000 cm^{-1} region did not shift with a_w. The result obtained from IR spectra suggests that the parameter *K* calculated by Guggenheim–Anderson–de Boar model reflected not only the interaction between water molecules but also the changes of state in solids. Consequently, the results from this study provide insights about the adsorption properties of nonideal solids such as rice flour.

Graphical Abstract



Changes in surface structure of rice flour and ATR-FTIR spectra in the 3400-1500 cm^{-1} during adsorption process.

Keywords: infrared spectra, milling, rice flour, starch, water activity

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Rice flour is a complex biopolymer consisting mainly of starch, protein, and lipids. Products made from rice flour have a unique texture and many advantages in terms of preventing celiac disease or other gluten-associated allergies (Okunishi 2015; Naquash *et al.* 2017; Matsuda 2019; Yano 2019). However, the complex and ambiguous physical properties of rice flour constrain the development of new products.

Milling is the most common food processing method for producing materials with new properties and different states due to the induced mechanical shock. Thus, changes in physicochemical properties, such as crystallinity, crystal granules, and spatial structure, of the starch in rice flour induced by the milling process have been investigated using various methods (Arisaka, Nakamura and Yoshii 1992; Tran *et al.* 2011; Asmeda, Noorlaila and Norziah 2015; Patindol, Siebenmorgen and Wang 2015; Leewatchararongharoen and Anutagool 2016). Especially, in recent years, research has demonstrated that the molecular structure of starch and functional properties of rice flour are affected by the processing procedure (Kadan, Bryant and Miller 2008).

Our research group has also studied the powder properties of rice flour subjected to different milling procedures toward optimizing the production process (Ishikawa *et al.* 2017; Fujii and Shoji 2012; Shoji *et al.* 2012). Fujii and Shoji (2012) reported that the surface and interior of rice flour subjected to the milling process formed a core–shell structure. Shoji *et al.* (2012) also found that the hydration behavior was closely related to the internal structure of rice flour. Moreover, Ishikawa *et al.* (2017) recently reported that the moisture isotherm for rice flours used agreed with the Guggenheim–Anderson–de Boar (GAB) sorption model, and the parameter *K* calculated from the GAB equation of α-formed rice flour was higher than that of the other rice flours (note that samples are same in the present study). Although the study demonstrated that the adsorption properties might be influenced by an ordered/disordered structure of rice flour, the interaction between solid and water during the adsorption process was not investigated.

It is well known that vibrational spectroscopy, such as infrared (IR), near-IR (NIR), and Raman spectroscopy, provide not only qualitative and quantitative information on components, but also insights regarding factors such as intra- and intermolecular interactions. Basic IR spectra of starch were investigated, for example, by Kizil, Irudayraj and Seetharaman (2002), and several studies have reported structural information on flours, such as their crystallinity and conformation, employing IR spectra, including the 1200-900 cm^{-1} region due to the skeletal vibration mode (van Soest *et al.* 1995; Solano and Rojas-de Gante 2014; Warren, Gidley and Flanagan 2016; Ashwar *et al.* 2018; Chen *et al.* 2018; Fetouhi *et al.* 2019). Recently, the effect of food additives on the ordering structure of potato starch was reported by Dankar *et al.* (2018). Lu *et al.* (2005) investigated the spectral changes in the IR region during natural fermentation of rice flour and concluded that some degree of modification of rice starch occurred because of fermentation.

These studies have demonstrated the potential of IR spectroscopy as an effective tool for analyzing the physical properties of starch subjected to mechanical processing. However, the spectral changes observed below 1200 cm^{-1} are more complicated, and to the best of our knowledge, the spectral behavior of rice flour subjected to different milling processes in the 4000-1200 cm^{-1} region, depending on water activity, has not yet been fully discussed. Therefore, in this study, rice flour samples subjected to different milling processes were prepared, and their spectral behavior in the 4000-1200 cm^{-1} region was investigated to provide insights about the solid state of rice flour during water sorption.

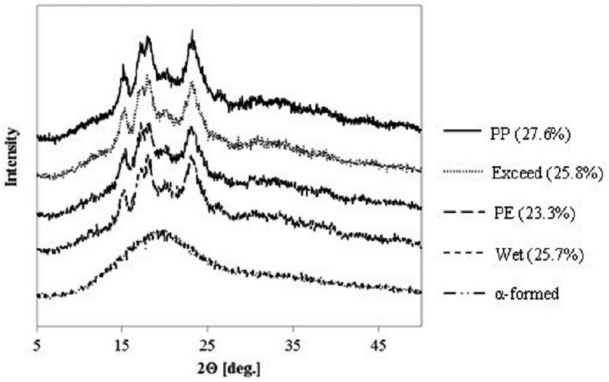

Figure 1. X-ray diffraction patterns and crystallinity for rice flours referred from Ishikawa *et al.* (2017). The percentage in blank of each sample indicates the crystallinity.

Materials and methods

Sample preparation

White rice harvested in 2015 from Miyagi Prefecture, Japan, was used as the material for rice flour production in this study. The flour sample, "PP," was produced by successive milling at a screen diameter of 3.0 and 0.3 mm at 3600 rpm (1/min); the sample "Exceed" was produced by pulverized milling at 11 000 rpm (1/min). For the "PE" and "Wet" samples, the white rice was milled at 3600 rpm and then pulverized at 10 000 rpm (1/min) with a screen diameter of 3.0 mm. For the Wet sample, 5 wt% of water were added prior to the milling process. For the "α-formed" sample, the white rice was subjected to α-type milling at 130°C.

In our previous study, an X-ray diffraction pattern of each rice flour was measured by a diffractometer (PHILIPS, X'pert MPD) within the 2θ range of 5-50° and CuKα1 radiation ($\lambda = 1.5405$), as shown in Figure 1. The crystallinity of PP, PE, Exceed, and Wet was calculated using the ratio of the peak area of the α-formed sample to that of each rice flour in the 14°-25° range. The calculated crystallinity of PP, Exceed, PE, and Wet was 27.6%, 25.8%, 23.3%, and 25.7%, respectively (Ishikawa *et al.* 2017).

Water activity and moisture contents

Eight desiccators that contained LiCl, CH_3COOK, $MgCl_2$, $LiNO_3$, NaBr, $SrCl_2$, KI, $BaCl_2$, or $K_2Cr_2O_7$ were prepared to control the relative humidity. Using these desiccators, the water activity of the rice flours was controlled in the 0.11-0.98a_w range. The rice flour was kept in each desiccator at 25°C until water sorption equilibrium was reached, and then dried at 105°C for 72 h.

Spectral measurement

Attenuated total reflectance (ATR)-IR spectra of the flour samples in the 4000-1200 cm^{-1} region were obtained using a FT/IR-6300 spectrometer equipped with a DLATGS detector (JASCO Co., Tokyo, Japan). Each sample was subjected to the ATR-IR measurement immediately after removing from the desiccator to avoid any possible changes in the sample conditions. The ATR unit (ATR-ProOne, JASCO, Tokyo, Japan) was equipped with a diamond prism and operated in a single reflection mode. ATR-IR spectra in the 4000-1200 cm^{-1} region were acquired with 256

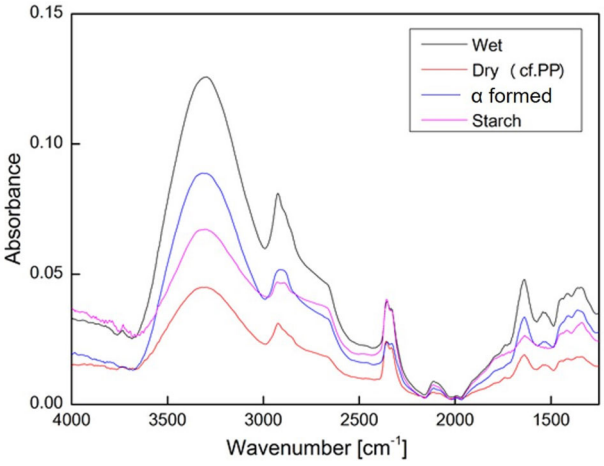

Figure 2. ATR-IR spectra in the 4000-1200 cm^{-1} region of rice flours and starch. Note that the bands at around 2400 and 2100 cm^{-1} originated from air were excluded from the analysis.

scans at a spectral resolution of 4 cm^{-1}. The measurement room was maintained at 25°C. The obtained spectra were transferred to an analytical software and subjected to ATR collection.

Pretreatment of spectra

IR spectra typically exhibit baseline drift due to changes in measurement conditions. In this study, the obtained spectra were subjected to the standard normal variate (SNV) procedure to correct the baseline drift prior to the analysis of the spectral data (Dhanoa *et al.* 1994; Fearn *et al.* 2009). In this study, the SNV method was used to perform a normalization of the spectra so that spectral characteristics of rice flours could be compared under different conditions of production. The SNV procedure, an effective method for the standardization of spectra, is described by the following equation:

$$x_j^{SNV} = \frac{x_j - \bar{x}}{s} \quad (1)$$

where x_j is the spectrum of the dataset, and $\bar{x}$ and s are the mean and standard deviation, respectively, for each spectrum in an arbitrary range. In this study, the mean and standard deviation of the 4000-2000 and 2000-1200 cm^{-1} regions were calculated separately. The band in the 2400-2100 cm^{-1} region was excluded from the calculation, and the Savitzky–Golay smoothing method (using a second-order polynomial and 21 data points) was applied to reduce the influence of noise on spectral data.

Results and discussion

Assignment of ATR- IR spectra of rice flour and starch

ATR-IR spectra in the 4000-1200 cm^{-1} region of the rice flours and starch are shown in Figure 2. A broad feature in the 3600-3100 cm^{-1} region is assigned mainly to the OH stretching vibration mode of water (Leewatchararongharoen and Anutagool 2016; Sarekha *et al.* 2017). The group of bands centered around 2930 cm^{-1} is due to the CH stretching modes, mainly that of starch, lipids, and protein; however, the contribution of protein is usually weak. Hence, the lipid-derived band is assigned to the steep band at 2930 cm^{-1} (Diop *et al.* 2011; Colussi *et al.* 2014; Sarekha *et al.* 2017). In the 1600-1400 cm^{-1} region of the rice flour, 2 bands appeared at 1540 and 1640 cm^{-1}, whereas the starch spectrum contained only a broad band at 1640 cm^{-1} in this region. The 1640 cm^{-1} band consisted of 2 bands: one due to the HOH bending vibration mode of water and another one due the amide I mode of protein (Wilson *et al.* 1991; van Velzen *et al.* 2003; Fetouhi *et al.* 2019).

Water activity-dependent spectral changes in the 2000-1200 cm^{-1} region of ATR-IR spectra of rice flour

The SNV spectra in the 2000-1400 cm^{-1} region of starch and each rice flour are shown in Figure 3. As described above, a broad feature in the 1700-1600 cm^{-1} region of the rice flour arises from the HOH bending mode of water, and the protein amide I band also contributes to it. The weak bands in the 1570-1520 cm^{-1} region are due to the amide II modes of proteins of the rice flour. On the other hand, in the case of starch, a broad band was observed in the 1700-1600 cm^{-1} region; this band is assigned to the OH bending mode of water. Kizil, Irudayraj and Seetharaman (2002) investigated IR spectra of several types of starches and suggested that this band corresponds to water molecules absorbed in amorphous regions. The gradual intensity change of the band in the 1700-1600 cm^{-1} region of starch indicates that water molecules were absorbed homogeneously.

As described above, the 1700-1600 cm^{-1} region in the rice flour spectra contained 2 overlapping bands; one from the water HOH bending mode and another one from the amide I of proteins. Since the water band was wider than the amide I protein band in the Dry and Wet samples (see Figure 3a and b), the band width of the overlapping bands broadened at higher water activities. Furthermore, the band intensity at 1640 cm^{-1} in the PP and Wet spectra gradually changed with increasing water activity (the data of PE is not shown). In contrast, no broadening occurred in the band of α-formed rice flour, and the change in the band intensity in the 1700-1600 cm^{-1} region was not constant in the water activity range. These results suggest that the protein of α-formed rice flour may be denatured during the preparation process because it was subjected to high temperature and shear stress. Moreover, as described above, the intensity increase of α-formed rice flour was not constant, which indicates that the amorphous state of the starch was inhomogeneous. In other words, the amorphous regions in the Dry and Wet samples contained crystalline regions and maintained a semi-ordered structure. Although the structure of the α-formed rice flour was collapsed by the milling process, the evolution of amorphous regions was not uniform.

A clear band appeared at approximately 1740 cm^{-1} in the Dry and Wet flour spectra. This band is well known as a key band of lipids and oils in food samples. The dry milling samples, such as PP, showed a small intensity change at 1740 cm^{-1} in the 0.11-0.8 range of a_w, and the band disappeared at high water activities. The band of rice flour subjected to wet milling decreased clearly compared with that of dry milling in the 0.11-0.80 range of a_w and disappeared at high water activities. In contrast, this band was unclear or very week in the α-formed spectrum.

A previous study by Chen *et al.* (2018) reported that this a_w-dependent band behavior at approximately 1740 cm^{-1} of a fried starchy sample was affected by changes in the interactions occurring between the matrix of starch and oils on the starch surface. In other words, in this study, it is likely that the band behavior of approximately 1740 cm^{-1} corresponded to the solid state of rice flour.

Chen *et al.* (2018) also reported a change in the crystalline structure of starch induced by the moisture content, where a

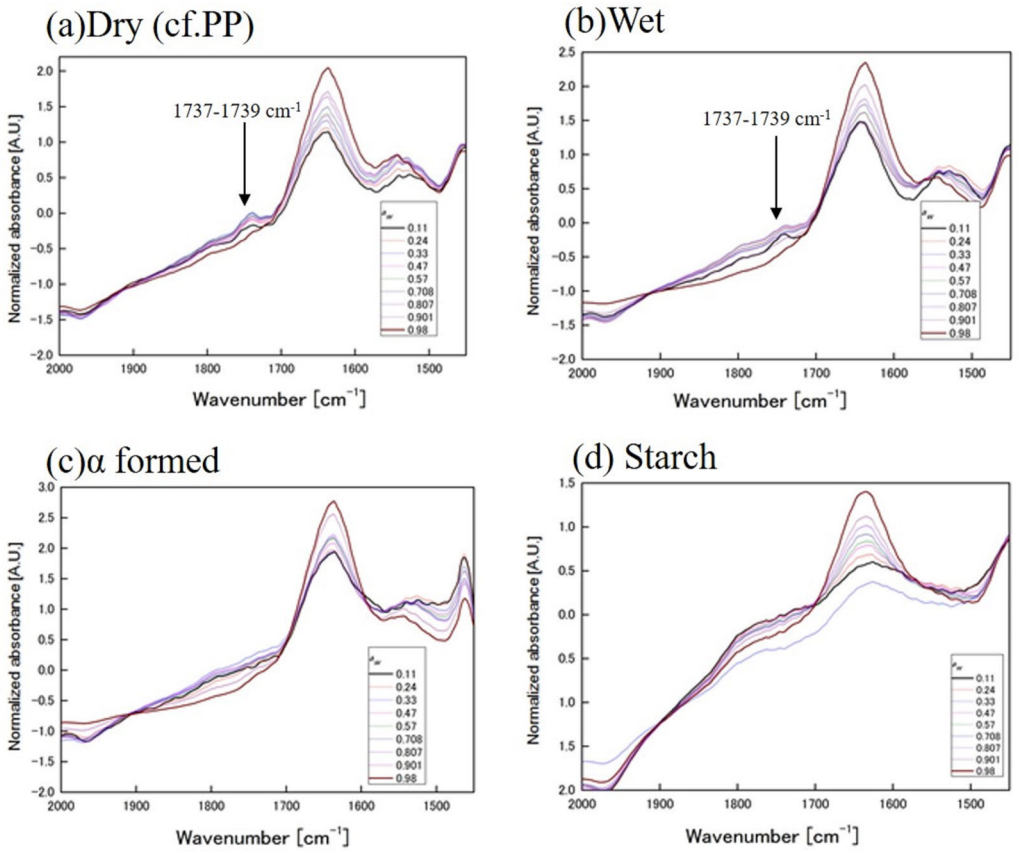

Figure 3. Changes in the normalized absorbance by standard normal variate procedure in the 2000-1450 cm^{-1} region of (a) Dry (cf. PP), (b) Wet, (c) α-formed, and (d) starch over a water activity range of 0.11-0.98.

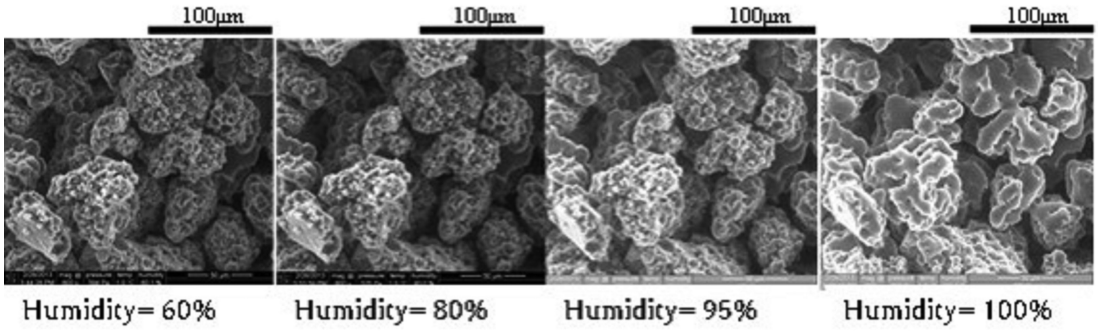

Figure 4. Changes in surface structure observed by SEM images of rice flour in the humidity of 60%-100% referred from Ishikawa *et al.* (2017).

V-type structure was formed at high moisture contents in the case of a fried maize starchy sample. In our study, the band at approximately 1740 cm^{-1} was not observed for the whole a_w range of the α-formed sample, and for other samples, it disappeared at high a_w. As shown in Figure 1, the structure of the α-formed sample was apparently amorphous (Ishikawa *et al.* 2017). Hence, the results suggest that disordered structural changes of starch arise not only from the milling process but also from moisture conditions.

We previously reported variations in the surface structure of starch granules observed by scanning electron microscopy (SEM) due to water sorption (Ishikawa *et al.* 2017). SEM images of rice starch granules under 60%-100% humidity are depicted in Figure 4. Structural changes occurred on the starch glandules, particularly at high humidity. Therefore, it is suggested that rapid evolution of the disordered structure of rice flour was induced by a high a_w. Moreover, Chen, Lu and Lii (1999) reported that dry-milled flour retained components such as proteins, lipids, and ash at higher levels than wet-milled flour. Thus, the band behavior at approximately 1740 cm^{-1} of the samples processed by dry milling did not change markedly compared with those prepared by wet milling. Moreover, the peak position in the 1600-1500 cm^{-1} range due to the amide II mode of protein in the rice flour changed with a_w. The band was weak for the α-formed rice flour. Notably, in Figure 3a-c, the amide II band region varied with a_w, indicating that the secondary structure of protein on the surface of rice flour is affected by adsorption of water. The band due to the amide II mode did not overlap with the water band and,

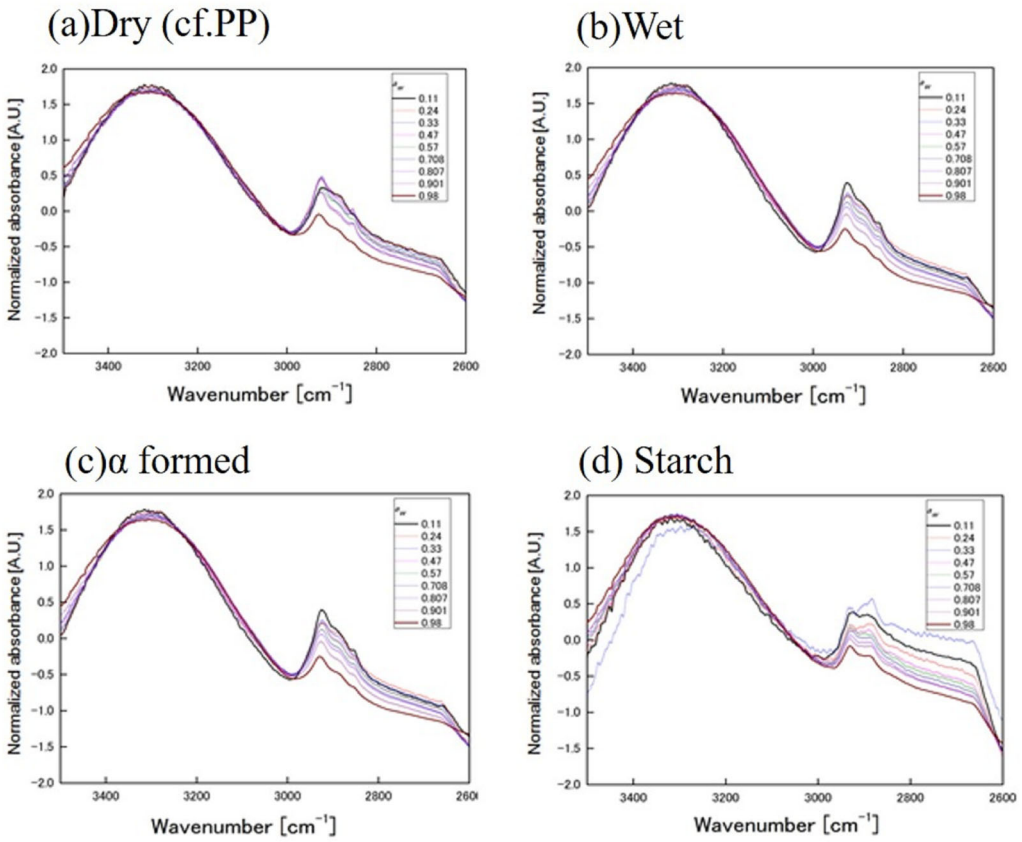

Figure 5. Changes in the normalized absorbance by standard normal variate procedure in the 3500-2600 cm^{-1} region of (a) Dry (cf. PP), (b) Wet, (c) α-formed, and (d) starch over a water activity range of 0.11-0.98.

thus, the amide II band is a good indicator for the state of proteins on the surface of rice flour.

Water activity-dependent spectral changes in the 4000-2000 cm^{-1} region of ATR-IR spectra of rice flour

The SNV spectra in the 4000-2000 cm^{-1} region of Dry, Wet, and α-formed rice flours and starch are shown in Figure 5a-d, respectively. A band intensity ratio between 2930 and 2850 cm^{-1}, originating from the CH stretching vibration mode of starch shown in Figure 5d, did not change obviously. Thus, the band at ~2930 cm^{-1} due to lipids on the surface of rice starch was dominant in the 3000-2800 cm^{-1} region of the rice flour spectra. The intensity of this band for all the rice flour samples decreased with a_w. The 2 peaks in the 3000-2800 cm^{-1} region of starch due to the CH_2 asymmetric and symmetric stretching modes appeared more clearly in the SNV spectra of starch. The peak positions of the CH stretching band at around 2930 cm^{-1} are plotted versus a_w in Figure 6. These bands of the Wet and α-formed samples remained almost constant in the 0.11-0.8 range of a_w, and then redshifted to 2930 cm^{-1} at a higher a_w. The band position in the PP spectrum gradually changed to a higher wavenumber in the 0.11-0.8a_w range and reached 2930 cm^{-1} at $a_w = 0.98$. On the other hand, the peak position of starch remained almost constant at 2930 cm^{-1} in the 0.11-0.98a_w range. This behavior is attributable to a stronger interaction at higher a_w values between the C=O groups of lipids and the OH groups of water with an increasing number of water molecules, leading to a loosely ordered structure of the alkyl chains of lipids. It is well known

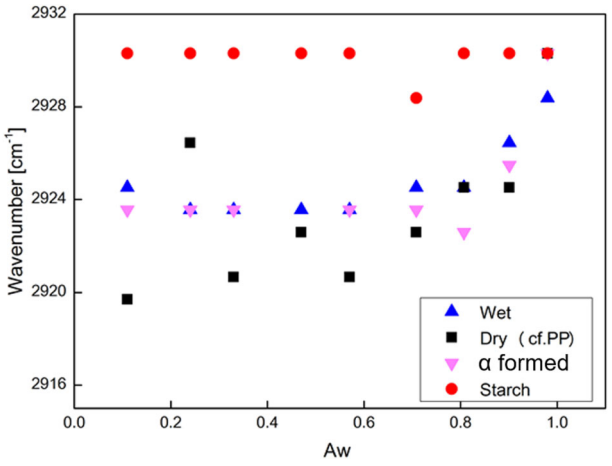

Figure 6. Changes in the peak positions of the CH stretching band at around 2930 cm^{-1} region for the rice flours and starch versus water activity.

that loosely ordered alkyl chains have a CH stretching group at a higher wavenumber (Sapper, Cameron and Mantsh 1981). In the 0.11-0.80a_w range, the interaction sites of lipids were already occupied by water molecules since water was added during milling for the Wet and α-formed samples. Thus, the band was mostly constant in this range. On the other hand, the interaction between lipids on the starch surface and water molecules in the other samples, such as PP, increased gradually with a_w because the interaction sites of these samples may remain available for interaction with water molecules. Therefore, the results

suggested that the band behavior at 2930 cm^{-1} was affected by the milling process. The band at 1740 cm^{-1} due to lipids was too small to directly evaluate its interaction.

Interestingly, the band position at 3500-3000 cm^{-1} from the OH-stretching vibrations of water did not significantly change with a_w. In other words, it is possible that a phase transition occurred within the adsorption process, but the state of water did not show considerable changes.

It is well known that *K* calculated from the GAB equation reflects the multilayer interaction of water molecules on ideal surfaces, such as activated carbon. In other words, in the adsorption theory, the change in parameter *K* does not depend on the solid state. However, in a previous study, we suggested that the parameter K might change according to the ordered structure of solids (Ishikawa *et al.* 2017). A similar insight is also suggested by Purohit and Rao (2017); they concluded that the parameter K could be considered as an indicator for the crystalline status of rice flour. In this study, in the IR spectra, the shift of OH stretching in the IR spectra was not confirmed as shown in Figure 5. Thus, no significant change was observed in the interaction between water molecules on the surface. The result obtained from IR spectra suggests that the phenomenon determined by the parameter *K* includes not only the interaction between multilayer water molecules but also the change in the solid state. The results support the insight that *K* of the GAB model is a parameter that reflects the solid state of nonideal materials, such as rice flours.

Conclusions

This study aimed to understand the dependence of rice flour characteristics on the milling process. Rice flour samples were prepared by different milling processes, and the water activity of the rice flour was controlled by using saturated salt in the 0.11-0.98a_w range. ATR-FTIR spectra in the 4000-1200 cm^{-1} region were measured for each rice flour over the above-a_w range.

Two bands at 1640 cm^{-1}, one attributed to the amide I of protein and the other to the OH bending mode of water, were observed. Compared with the α-formed sample, the bandwidth at 1640 cm^{-1} of the crystalline samples was wider, and the intensity gradually changed with a_w. A band at 1740 cm^{-1} due to lipids or oils occurred in the spectra of the rice flour with a crystalline structure. This band disappeared at high a_w values for these rice flours, whereas it was not observed for the α-formed sample in the whole a_w range. Therefore, it is likely that starch disorder occurred not only due to milling, but also due to the adsorption process. The lipid band at 1740 cm^{-1} can be used for evaluating the solid state of rice flour. Moreover, a band at 1534 cm^{-1} attributed to amide II shifted to a higher wavenumber according to a_w. Thus, the secondary structure of the protein of rice flour might be affected not only by milling but also the water adoption process.

The band at 2930 cm^{-1} of the rice flour shifted to a longer wavenumber. This band may have changed as a result of the interaction of C=O of lipids and water when subjected to water sorption. The bands at 3500-3000 cm^{-1} due to OH stretching did not change notably with a_w across the rice flour samples. The results suggest that the state of water on the rice flour surface during the adsorption process did not change significantly. Thus, it is very likely that the adsorption parameters affect the change in the solid state of rice flour during the adsorption process. This study revealed that IR spectroscopy can facilitate the intrinsic understanding of sorption characteristics of objects with nonideal surfaces such as foods.

Authors contribution

D.I. and J.Y. performed the experiments and analyzed the spectra. They also drafted the manuscript. C.I. performed the experiments, especially the monitoring of water adsorption of rice flour. T.F. designed the study and suggested the experimental method used in the study. He also reviewed the manuscript. Finally, all authors have viewed the manuscript and agree to its final form.

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Disclosure statement

No potential conflict of interest was reported by the authors.

Data availability

The data underlying this article are available in [the Dryad Digital Repository], at https://doi.org/10.5061/dryad.80gb5mkqh.

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