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Change in oxygen permeability of plastic vacuum bags containing germinated brown and parboiled germinated rice

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Abstract

The study was conducted to investigate changes in oxygen permeability of Nylon/Polyethylene (Nylon/PE) and Low Density Polyethylene (LDPE) films, utilised for vacuumed packages of germinated brown rice and parboiled germinated rice, during 120-day storage at an ambient condition (35ºC). Weights of both Nylon/PE and LDPE films, immediately in contact with both types of rice, continuously increased and approached saturation. These were well described by the Langmuir-based model. Film weights of Nylon/PE were relatively higher than those of LDPE films. Absorption of fat from rice to the films was considered the key process attributing to the increase in film weight. Absorbed fat, effectively acting as a plasticiser in polymeric structure, caused an increase in the oxygen transmission rate (OTR) values of the films. Whilst OTR values of Nylon/PE films noticeably increased throughout the storage, those of LDPE films showed inconsistent trends. The present work demonstrates that estimation of oxygen flux across the Nylon/PE film into the package, by assuming constant film permeability to oxygen, would underestimate experimental results during storage, due to the increased film permeability as a result of rice-film interaction.

Introduction

Germinated brown rice (GBR) and parboiled germinated rice (PGR) have a high fat content in the rice bran layer (Sharp and Timme, 1986). Interactions of fat with oxygen accumulated in package headspaces, with the presence of moisture, can lead to hexanal vapour and hydroperoxide releases, thus causing the rice off-flavour. Accumulation of oxygen also causes other undesirable effects such as insect growth, especially Sitophilus zeamais and Tribolium castaneum, which are major pests in stored rice (Noomhorm *et al*., 2013). GBR and PGR are kept commercially in vacuum packages, in order to extend their shelf life (Tananuwong and Lertsiri, 2010; Baradi and Matinez, 2015). The vacuum package is typically a laminated Nylon/Polyethylene (PE) film bag, which has a high barrier property to oxygen, moisture, other gases and volatiles (Robertson, 2013a). An increase of oxygen concentration in the vacuum package during storage is considered a major problem that can limit the shelf life of rice grain (Khaosaeng *et al*., 2012). This may be attributed to the possibility of leaks at the film surface, which could be the result of rice grain punctures on film tightly placed on rice grain after vacuuming (Osborn and Jenkins, 1992). These leaks could be a passage for air permeation.

Even if there are no leaks on the film surface, oxygen permeation to plastic film could cause accumulation of oxygen in the package (Khaosaeng *et al*., 2012). When films are exposed to fat, film permeability to gases and volatiles is likely to increase. Johansson and Leufvén (1994b) reported that film permeability to oxygen (FPO) values of High Density Polyethylene (HDPE) and Polypropylene (PP) increased when the films contacted with and absorbed rapeseed oil. Absorption of oil was considered a key process causing rearrangements of polymeric structures that subsequently facilitated oxygen permeation through the film (Johansson and

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Leufvén, 1994b).

The contacts of GBR and PGR with Nylon/PE vacuum package can be considered physically similar to those of HDPE and PP films with rapeseed oil. Therefore, it can be hypothesised that the fat content of GBR and PGR will be absorbed by the PE film layer of Nylon/PE film into which fat content can be soluble and the absorptions will cause increases in FPO of the film. Absorption of fat content of rice by plastic film has also been anecdotally recognised in the printing industry. It can cause a fading away of ink printed on outer film surfaces (Nutthi, 2011). The effects of fat absorption on FPO values of the film packages used for GBR and PGR, or other types of rice, have not been investigated. Considering the importance of vacuum packages on storage quality of rice, this work purposely was conducted to evaluate changes of FPO of Nylon/PE film immediately in contact with GBR and PGR during storage.

Materials and Methods

Rice and plastic film materials

Both GBR and PGR were purchased from the T.M. Inter-Rice Company, Don Mod-Dang, Ubon Ratchathani, Thailand in 2013. The rice harvesting season was November 2012-January 2013. Foreign materials, such as stones, were removed from the rice. The cleaned rice was kept in a PP woven sack with a plastic film liner until the research commenced. The approximate moisture contents of both GBR and PGR were 12% (dry basis). Nylon/PE (80 µm thick: 15 µm for Nylon and 65 µm for PE) and LDPE (30 µm thick) film bags were purchased from Jittra Packaging Co. Ltd. Thailand and Thepwichit Industry Co. Ltd., respectively. Oxygen transmission rate (OTR) values of plain LDPE and Nylon/PE film were approximately 3480.99 and 39.60 $ml \cdot m^{-2} \cdot day^{-1}$, respectively. The research was undertaken at the Postharvest Technology, Faculty of Agriculture, Ubon Ratchathani University, Thailand.

Preparation for change of film weight and FPO value measurement

Fifty grams of either GBR or PGR was packaged into Nylon/PE and LDPE bags (size 130 × 130 mm) containing a rectangular Teflon frame (100 × 100 × 30 mm width × length × thickness) inside the bag. The frame was used to arrange rice packaged as a single layer and to structurally keep the bag in a flat-shape form, after it was vacuumed, i.e. the vacuum level was 1.9 kPa (an absolute pressure), and heat-sealed using a vacuum packaging machine (Multivac, Japan). Following the sealing and applying vacuum process, plastic films became tightly exposed to rice surfaces. The rice-film contact allowed fat (defined as the general term representing both fat and lipid contents) from the rice to be absorbed and transported into the films. Individual packages were kept at 30ºC for 120 days. At an interval of 30 days, samples were randomly chosen and packaged films were measured in order to determine changes of film weight and FPO values.

It should be noted that, although an LDPE film bag is technically considered improper to be used as a vacuum packaging material, due to its low oxygen barrier, the utilization of an LDPE bag in the present work was purposely used in order to compare the extent of fat absorption between LDPE and Nylon/PE films. Since the latter contains a PE film layer, it could be hypothesized that fat absorption of Nylon/PE could occur through this layer.

Change of film weight and FPO value measurement

Plastic film weight (g) was determined using an electronic balance with an error range of 0.001 g. Weight changes of individual films after packaging were expressed as differences of film weights after packaging, in comparison with the initial film weights. The values of FPO were measured and reported in terms of oxygen transmission rate (OTR). OTR values of Nylon/PE films were measured at 23ºC and 0% RH (ASTM D3985) using an Oxygen Permeation Analyser (Model 8000, Illinois Instrument). For the LDPE films, OTR values were measured at 23ºC and 0% RH (ASTM D3985) using an Oxygen Permeation Analyser (MOCON Ox-tran Model 2/21). In order to measure the OTR of individual films, the sample film was greased on the rim and placed on the sample chamber with an exposed testing area of 100 cm^2. Nitrogen gas was blown onto one side, whilst pure oxygen gas was blown onto the other side at the exposed testing area. The OTR value ($ml \cdot m^{-2} \cdot day^{-1}$ as an OTR unit) was subsequently recorded. The OTR measurements of Nylon/PE films were conducted at the Package Testing Laboratory, Thai Packaging Centre: and those of the LDPE films were undertaken at the Plastic Laboratory, National Metal and Materials Technology Centre (MTEC), Thailand, of which its MOCON Ox-tran Model 2/21 had been technically set for measuring only high OTR films including LDPE.

The Langmuir-based model (Robertson, 2013a) was utilised in the present work, due to its simplicity and ability to describe experimental results showing saturation over a certain timeframe.

$$Wt_{\theta} = Wt_{max} \cdot b_{Lgm} \cdot \theta / 1 + (b_{Lgm} \cdot \theta) \quad (1)$$

where wt_θ= change in film weight at time θ (g), wt_{max}= maximum change in weight (g), b_{Lgm}= Langmuir-based model constant (dimensionless), and θ = storage time (day). Fitting (1) to empirical data was conducted using a nonlinear regression approach. Root mean square error (RMSE) (2) were calculated following Yang and Chinnan (1988) to evaluate the goodness of fit between the model's predictions and empirical data.

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\left[(Wt_{exp})^i - (Wt_{pred})^i\right]^2} \quad (2)$$

where wt_{exp} and wt_{pred} are experimental and model predicted data on change in film weight, respectively, is n number of data points. Fick's first law model (3) (Roberston, 2013a) was used to describe a steady-state flux of O_2 permeation across Nylon/PE films.

$$J_{fl}^{O_2} = P_{fl}^{O_2} \cdot \Delta p / L_{fl} \quad (3)$$

where $J_{fl}^{O_2}$ is steady-state flux of O_2 permeation ($mol \cdot s^{-1} \cdot m^{-2}$), $P_{fl}^{O_2}$ is film permeability to oxygen ($mol \cdot m \cdot s^{-1} \cdot m^{-2} \cdot Pa^{-1}$), L_{fl} is film thickness (m), and Δρ= pressure gradient (Pa). Fluxes ($J_{fl}^{O_2}$) were estimated using (i) film permeabilities ($P_{fl}^{O_2}$), which were calculated from OTR values of Nylon/PE of both films contacting with rice and a plain film, (ii) 80 μm film thickness (L_{fl}), and (iii) 21 kPa as pressure gradient (Δρ) between inner (0 kPa) and outer (21 kPa) bag. It should be noted that the partial pressure of O_2 inside the bag was assumed to be 0 kPa for simplifying the flux calculations.

Fat content determination of rice grain

The analysis method utilised for quantifying the fat content of rice grain packaged in both LDPE and Nylon/PE bags was carried out using the Association of Official Analytical Chemists (AOAC) method, of which the Soxhlet extraction with petroleum ether (AOAC Method 945.16) was employed with some modifications. Five grams of ground rice grain was placed in a porous thimble and later placed in an extraction chamber. The thimble was suspended above a flask containing 60 ml of petroleum ether, below a condenser. The flask was heated and the solvent evaporated and moved up into the condenser. The solvent subsequently turned to liquid and accumulated in the extraction chamber. After 6 h, the flask containing the solvent and lipid was removed. The solvent in the flask was then evaporated and the mass of remaining lipid was measured. Fat content was reported in % dry basis.

Table 1. Changes in film weight (g) of LDPE (initial weight 1.38 g) and Nylon/PE (initial weight 4.21 g) films in contact with GBR and PGR during 120 days

Time (day)	Changes in film weight (mg)			
	LDPE		Nylon/PE	
	GBR	PGR	GBR	PGR
30	4.30±1.16^{Db}	4.10±1.60^{Cb}	20.20±5.96^{Ca}	17.40±2.59^{Da}
60	6.00±0.67^{Cc}	5.90±0.57^{BCc}	22.90±1.37^{BCa}	21.30±1.42^{Cb}
90	7.30±0.82^{Bc}	6.90±0.74^{Bc}	26.20±2.20^{ABa}	24.60±1.58^{Bb}
120	8.20±1.03^{Ab}	9.90±3.60^{Ab}	29.60±3.84^{Aa}	27.80±2.94^{Aa}

Data represent mean ± standard deviation (n = 10). Means followed by different upper-case letters in the same column, and lower-case letters in the same line differ by the DMRT at 5% probability ($p<0.05$). LDPE = Low Density Polyethylene, GBR = Germinated Brown Rice, and PGR = Parboiled Germinated Rice.

Experimental design and statistical analysis

The experimental design and statistical analysis were conducted in accordance with a completely random 2 × 2 × 4 factorial design. The factors were two types of plastic films (Nylon/PE and LDPE), two types of rice (GBR and PGR) and four storage time (30, 60, 90 and 120 days). Ten samples of plastic films and five samples of rice from each treatment were utilised for analysing changes of film weight and, fat content respectively. After measuring film weight, two films of individual treatments were randomly chosen for analysing the OTR values.

Experiments were conducted during January-November 2013. Data collected on changes of film weight and fat content were subjected to an analysis of variance (ANOVA) and the means were compared by the Duncan Multiple Range Test (DMRT) at a level of 5% probability ($P<0.05$). The statistical analyses were performed using SAS. Meanwhile changes of OTR values of plastic films sampled were graphically presented in relation to change in film weight during storage.

Results and Discussion

Changes in film weight and fat content

The analysis of variance (ANOVA) of change in film weight shows that rice types, film types and storage time significantly affected changes of the film weight ($P<0.05$). Significant interactions of these factors on the film weight were found between film

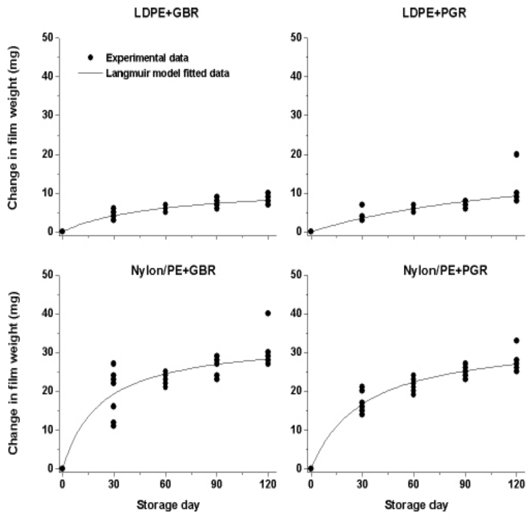

Figure 1. Experimental and predicted data of changes in film weight for both Low Density Polyethylene (LDPE) and Nylon/PE films contacting with germinated brown rice (GBR) and parboiled germinated rice (PGR). The solid lines are the Langmuir-based model predictions

types and (i) rice types and (ii) storage time (data not shown). Weights of both LDPE and Nylon/PE films in contact with either GBR or PGR continuously increased. Weights of Nylon/PE films increase approximately 2.8-4.70-fold higher than those of LDPE films given the same rice type and storage time (Table 1). It also can be noticeable from the data of Nylon/PE film that both the films contacting with GBR were likely to gain higher weight compared to those contacting with PGR, although such differences regarding the types of rice were not significant. The similar trend noted was observed among LDPE films, except on Day 120 where an average weight of films contacting with GBR was slightly lower than that contacting with PGR (Table 1). By observing film surfaces of Nylon/PE, there were no delamination signs noticed between Nylon and PE film layers. Increases in film weights were attributed to absorption of fat from rice grains, which transfer from the aleurone layer through the tegmen (seed coat) and pericarp to the plastic films. The aleurone layer of rice grain contains high lipid and fat content (Marshall and Wadsworth, 1994). Nylon/PE films contacting with rice gained higher weight than LDPE were due to the thickness of the PE layer of Nylon/PE (65 μm), which is about 2-fold thicker than that of LDPE film (30 μm), thus allowing a higher extent of fat or liquid to be absorbed by Nylon/PE film.

By plotting data on changes in film weight for all films against the storage time, there is a non-linear trend (Figure 1). It can be observed that the rate of film weight accumulation was rapid during the first two months of the storage period. These were attributed to the high gradient of fat concentration between rice grain and films. The rate subsequently decreased and became stable at the end of the study. These observations may be results of films gradually becoming saturated with absorbed fat and the concentration gradient subsequently being reduced.

For further understanding of this result, the relationship between change in film weight and storage time was analysed using the Langmuir-based model (1). Weight changes of all films predicted by (1) are apparently in good agreement with empirical data (Figure 1), with root mean square error (RMSE) values of 0.0002-0.002. Model coefficients hence wt_{max} and b_{Lgm} were estimated using nonlinear regression. Values of wt_{max} for LDPE and Nylon/PE contacting with both types of rice are 11.84-20.70 mg and 33.56-33.89 mg, respectively, meanwhile the values of b_{Lgm} for those film-rice contacts are 0.007-0.018 and 0.032-0.045, respectively. Since values of RMSE estimated by (2) are lower than 2, which is considered an important criteria to justify model accuracy in accordance to Yang and Chinnan (1988), the Langmuir-based model (1) can be employed to predict film weight. This finding suggests that the films became nearly saturated with rice grain fat, and by leaving a longer contacting time between films and rice the film weights would reach their maximum values. It can be observed that there were slower increases in weights of Nylon/PE films contacting with PGR (Figure 1). Because the outer bran of parboiled rice tends to have high fat contents due to disruptions oil globules in the rice kernel during the parboiling process causing the oil to move toward the bran (Bhattacharya, 1985; Luh, 1991), it accordingly may be presumed that there would be a higher extent of fat transfer from PGR to the plastic films immediately contacted. However the slower changes in film weight would be attributed to alterations occurred to parts of the kernel including pericarp and aleurone layers which are parts of the bran. The parboiling processes could make these become softer and tightly attached to the kernel (Luh, 1991), potentially slowing down the fat transfer out from the bran layer.

It can be observed that there were losses of vacuum condition in some LDPE bags during the storage period and the films subsequently became loose. The film on the top side of each bag subsequently became separated from the rice grains. However, the bottom side of each bag continuously stayed in contact with the rice, due to the rice weight pressing on the film. Microbial appearances were visually observed

Table 2. Fat contents of GBR and PGR kept in LDPE and Nylon/PE film vacuum bags during 120 day

Time (day)	Fat contents (% dry basis)			
	LDPE		Nylon/PE	
	GBR	PGR	GBR	PGR
30	2.29±0.21^{Aa}	2.31±0.07^{Aa}	2.12±0.09^{Ab}	2.40±0.08^{Aa}
60	2.04±0.13^{Ba}	2.11±0.10^{Ba}	2.04±0.15^{Aa}	2.17±0.10^{Ba}
90	N/A	1.96±0.15^{Ba}	1.88±0.12^{ABa}	1.99±0.15^{Ca}
120	N/A	1.64±0.19^{Ca}	1.61±0.15^{Ca}	1.65±0.17^{Da}

Data represent mean ± standard deviation (n = 5). Means followed by different upper-case letters in the same column, and lower-case letters in the same line differ by the DMRT at 5% probability (p<0.05). N/A represents no available data because of microbial incidence on rice. LDPE = Low Density Polyethylene, GBR = Germinated Brown Rice, and PGR = Parboiled Germinated Rice.

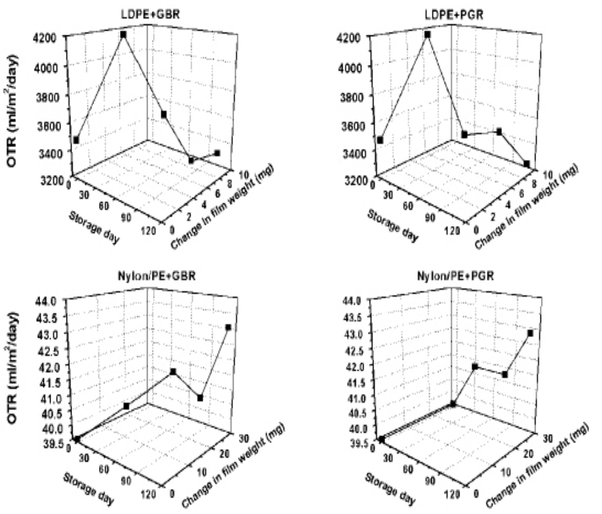

Figure 2. Changes of oxygen transmission rate (OTR) of Low Density Polyethylene (LDPE) and Nylon/PE films contacting with germinated brown rice (GBR) and parboiled germinated rice (PGR), in relation to change in film weight during storage

among GBR kept in LDPE bags after 90 storage days (data not shown). The microbial evidence on GBR packaged in LDPE film bags supports the knowledge that brown rice tends to rapidly deteriorate under ambient conditions, as in the case of LDPE bags used in the study. High O_2 concentration, which is at or near 21% (v/v) in the LDPE package, could promote microbial proliferation (Kader *et al*., 1989; Villalobos *et al*. 2014; Oliveira *et al*. 2015).

Significant effects of films, rice, and storage time (P<0.05) on the increase of film weight aforementioned suggest that rice manufacturers should aware on importance of these factors upon storage qualities of vacuum packaged rice. Given a set of rice and packaging film, the time period for which vacuum packages would be marketed is an important factor, because the longer the exposure time, the greater the amount of fat that can be absorbed from rice to film packaging.

Moisture content of rice grain was stable during the storage period (data not shown). This result suggests that the moisture absorption by film was practically negligible and could be attributed to relatively low water absorption by Polyethylene film (Robertson, 2013b). Tananuwong and Lertsiri (2010) similarly reported that both moisture content of organic brown rice grain (12-14% dried basis) kept in OPP/Al/LLDPE and Nylon/LLDPE film bags did not change significantly during 12 months of storage. Since Nylon film has hydrophilic properties, absorption of water vapour present in the atmosphere surrounding the plastic bag would be likely. However, there was no change in the weights of empty Nylon/PE film bags which were kept in the same storage condition, together with other experimental samples. This may be attributed to, among other factors, saturation of Nylon/PE films with water vapour while being kept at the suppliers, prior to being used in the present work: or film surface treatment with proprietary coating material to minimise water vapour absorption of Nylon. Based upon the research findings noted above, the hypothesis on absorption of fat from rice, to which plastic films had immediate contact thus causing increases in film weight, has been substantiated.

The ANOVA of fat content using three factors was significant (P<0.05) indicating types of rice and films, as well as the storage time all affected changes of fat contents. There were also significant interactions between effects of film, rice and storage time on fat contents (data not shown). The total fat content of rice packaged in all plastic bags continuously decreased during storage (Table 2). The fat contents of rice measured on Day 120 for all treatments except GBR packaged in LDPE decreased approximately 1.85-fold from those measured prior being packed into the bags (3.04-3.24% dry basis). Absorption of fat by plastic film would contribute to a loss of fat from rice (Table 2). As discussed earlier, the extent of weight increases of Nylon/PE films are higher than those of LDPE. It could be implied that the fat content left on rice grain packaged in LDPE should be higher than those left on rice packaged in Nylon/PE. However, the fat content of both types of rice grain packaged in both types of plastic bags were comparable. By summing the fat mass left on

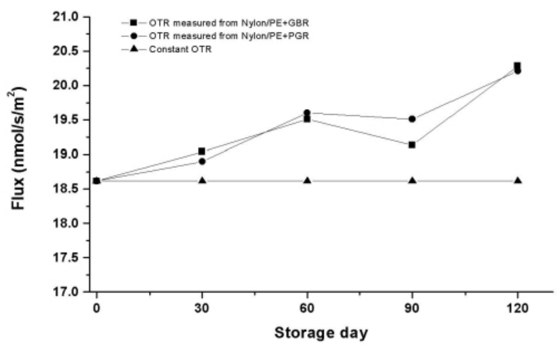

Figure 3. Demonstration of O_2 fluxes estimated across Nylon/PE films contacting with germinated brown rice (GBR) and parboiled germinated rice (PGR) using average oxygen transmission rate (OTR) values measured during storage, in comparison to those estimated using a constant OTR value

plastic film and that left on rice grain, the values were approximately half of the initial value of fat (Table 2). This suggests that there exists a loss of fat content in rice grain, which may be attributed to triglyceride degradation to free fatty acid and other components (St. Angelo and Ory, 1983).

Changes in FPO values

There were noticeable different trends in changes of OTR values of LDPE and Nylon/PE, in regard to changes of film weights (Figure 2). Whilst weights of LDPE film continuously increased (Figure 1), changes of OTR values were inconsistent. The OTR values increased and reached the highest value (approximately 4,100 ml·m^{-2}·day^{-1}) on Day 30, however, the OTR value thereafter sharply decreased to a value which was comparable to that of the plain film, i.e. no contact with rice. Similar trends of changes in OTR values of LDPE films were found in both GBR and PGR (Figure 2). These could be hypothesised as a result of loss in the vacuum condition under which the films became departed from the rice, subsequently limiting fat absorption from rice.

In contrast to LDPE, OTR values of Nylon/PE were consistently increased following changes of film weight throughout the storage period (Figure 2). The OTR values measured on Day 120 were about 1.10-fold higher than that reported for the plain film. It can also be observed that OTR values of Nylon/PE films measured on Day 90th were slightly lower than those measured on Day 60 (Figure 2). These may be attributed to the accumulation of fat absorbed to the film and interactions between absorbed molecules and polymeric structure, or among absorbed molecules. Accordingly, the interactions could slow down the permeation process, as reported by Johansson and Leufvén (1994a) for hexanal vapour permeation through LDPE and other films at different relative humidity levels. Johansson and Leufvén (1994a) postulated that clusters formed between hexanal and/or water molecules with polymeric structures potentially impeded diffusions of the molecules because more sorbed water molecules may compete with other permeants for available spaces. However, increases in OTR values were observed again on Day 120 (Figure 2). These could be a result of an accumulative amount of fat being absorbed and the exposure time that the films were in contact with the rice (Johansson and Leufvén, 1994b).

One key mechanism underlying the increased OTR of plastic film materials is attributed to the interactions between the film and fat absorbed, which potentially could cause the so-called swelling effect on the film that can alter the polymeric film structure (Johansson and Leufvén, 1994b). Whilst the swelling effect typically widens the film's thickness, as reported in studies on the immersion of plastic film into rapeseed oil (Johansson and Leufvén, 1994b), there were no apparent changes in thickness (measured using the micrometre) of film immediately connecting to GBR and PGR. Such experimental finding would hypothesise that the amount of fat absorbed into the film may not be sufficiently high for making film swelling to the degree at which the film thickness could be enlarged. Such absorption would also be a reason for no apparent delamination between Nylon and PE film layers. Fat absorbed into the film functionally has a role as a plasticizer, which can increase the void volume available in polymeric structure and subsequently facilitate permeation of absorbed molecules (Pascat, 1985). The fat absorbed from GBR and PGR by the films could, therefore, act as plasticizer inside the polymeric structure of LDPE and Nylon/PE films.

Since GBR and PGR, in general, are kept in plastic film packages for long periods (for example, three months or longer), the rate of oxygen permeation through plastic film can reasonably be considered a steady-state. In Figure 3, fluxes of O_2 permeation estimated by (3) through the Nylon/PE film in contact with both types of rice continuously increased during the storage period. The illustration also shows that an approach to estimate flux using constant OTR value, can underestimate the flux calculated from increased OTR values during the storage period. It could be implied that a higher rate of O_2 permeation into the package would stimulate a reduction of rice quality and potentially limit its shelf life from the time period expected.

Conclusion

The present work proves that oxygen permeability of Nylon/PE film in contact with GBR and PGR continuously increases during storage. Such change is a result of fat absorption from packaged rice grain. The absorptions also cause increases in the film weights of which have a non-linear relationship with storage time. Experimental findings importantly provide technical information for further development of Nylon/PE film for minimising fat absorption and, in turn, slowing the rate of O_2 permeation into the rice package.

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