Effect of processing and formulation on water migration through lipids

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Abstract

Moisture migration through multi-compartment foods negatively affects product quality and safety. The effect of processing condition, crystallization temperature, emulsifier, solid particle, and storage conditions on vapor migration through lipid moisture barrier were studied. Palm oil and palm oil blends were structured under laminar shear applications and static conditions. Gravimetric experiment was conducted by sealing uniform-sized lipid disks over plastic cups containing known relative humidity (RH), then put into desiccators containing a different known RH. For pure palm oil, lipid barriers formed under laminar shear gave lower water vapor permeability compared to barriers formed under static condition. The addition of emulsifier into the fat system agreed with this result, showing similar trend with sheared samples having lower moisture permeability. Interestingly, when solid particle (cocoa powder) was introduced into palm oil, the opposite trend was observed. These results suggest that media formulation in combination with processing conditions affect moisture barrier properties differently. It is suggested that solid fat content, crystal polymorphism, and crystal size and agglomeration be analyzed to identify potential cause(s) of the difference. Using more accurate and specific measurement methods such as magnetic resonance imaging can be helpful. It also would be of interest to look further into the role of formulation in functionality in future studies.

Key word: lipid structure, moisture migration, laminar shear, water vapor permeability.

1. Introduction

1.1 Moisture migration in food

Moisture migration happens in a food system when a water activity gradient exists between a food compartment and its surrounding environment, such as adjacent food components or exposed atmosphere, leading to transfer of vapor or liquid water from the region with higher water activity to that with lower water activity until equilibrium is reached (Guillard and others 2003). While progresses in packaging technologies are significantly improving the shelf-life of products in regard to moisture exchange with atmosphere, moisture migration remains problematic in multi-compartment foods, such as chocolate-covered confectioneries and pastries with soft centers. It promotes microbiological growth as well as quality deteriorations – cracking and collapsing of the outer shell, staling of pastry or sugar bloom of chocolate, drying and shrinking of soft center, and other flavor and texture changes (Barron 2007; Ergun and others 2010; Smith and others 2004; Talbot 2009). Since it is impractical to always match the water activities of different food compartments, edible barriers are commonly used to reduce moisture migration problem. The diffusion of moisture in a material is dependent on two groups of factor: the thermodynamic relationship between moisture form and the material, and the penetrated material's composition and structure (Fennema and others 1994). Based on these factors, moisture barriers are developed to introduce tortuosity to the migration path of water in product, and ideally would deter migration for sufficient shelf-life length while interfere minimally with product's sensory attributes (Talbot 2009). The barrier can be made in form of a film, which can be identified as a stand-alone sheet, or a coating which is directly formed on the product (Bourlieu and others 2009).

1.2 Evaluation of edible moisture barriers

Gravimetric method is a common method for evaluating moisture barrier. It is based on weight change due to average bulk moisture transfer and indicates the water vapor permeability (WVP) property of the sample (Bourlieu and others 2009). Modeling studies are often based on using saturated salt solutions and/or desiccants to maintain a controlled relative humidity gradient separated only by the barrier, as detailed in standard method ASTM E96 (1995). The WVP value would then be determined from the vapor pressure gradient, rate of steady change in sample weight, sample thickness, and area in contact with humidity of sample (Martini and others 2006). A great drawback of this method is the lack of information on mechanism of migration, diffusivity, and any interaction between the lipid system and moisture over time. This is especially important in cases where non-lipid materials need to be added for other sensory or functionality purposes.

Efforts have been made to quantify the diffusion of liquid water within multi-component foods. One approach using weighing techniques utilizes simulations consisted of a ‘dry compartment’ (industrial sponge cake, bulk gelatin, or dry biscuit) placed in contact with a ‘wet compartment’ (agar gel, process cheese, or concentrated jam) inside a tight container, with optional air gap or moisture barrier in between (Bourlieu and others 2008, Gulilard and others 2003, Ramos-Cabrer and others 2006, Rougier and others 2007). For each time point in the experiment, samples are cut into thin slices and measured for moisture content using the drying oven method. Moisture content is then plotted against distance from contacting surface for each food compartment. A mathematical model providing good fits and predictions have been developed in some studies; however the model is based on the assumptions that water migration is the only migration process happening between the compartments to cause weight changes, that diffusion is uniform, and that the model takes into account physical changes in samples over time (such as shrinking of the wet compartment as it loses moisture). Further studies would be needed to evaluate how this method may be applied to other food systems and experiment conditions. Magnetic resonance imaging (MRI) is a potent and informative non-invasive, non-destructive technique that can provide detailed internal moisture distribution map and insights on relaxation and diffusion behavior of material (Ziegler and others 2003). Lodi and others (2007) used MRI to investigate the effect of adding soy ingredients on water redistribution in bread during storage. For ham products, which is affected in both quality and safety by moisture, Fantazzini and others (2009) used this technique to monitor moisture distribution during curing process, and Antequera and others (2007) proposed that using MRI to measure moisture content during ripening process is superior to the gravimetric method used in industry. Troutman and others (2001) monitored freshly made jelly bean for both migration of water from the shell to the center and redistribution of moisture in the center. Ramos-Cabrer and others (2006) noted that the effectiveness of a MRI protocol depends greatly on balancing the trade-off relationship between sensitivity, spatial resolution, temporal resolution, and the range of molecular mobility to be studied. For multi-component foods, monitoring moisture migration can be challenging when great difference in water contents means very different targeted molecular mobilities. They proposed two protocols for this type of product, using sandwich bread containing various ingredients with unmatched water activities (cheese, tomato, lettuce, ham, etc.) as sample. One protocol gave detailed result but long experiment time; while the other protocol focused on faster moisture transportation processes, thus reduced scanning time but compromised other imaging requirements. The same drawback was observed by Weglarz and others (2008) when crunchy cereal-based snacks with lipid moisture barriers were tested by placing in contact with hot water or moist. This showed that the development of MRI protocol for quantitating moisture transport in multi-compartment foods need to be balanced specifically for each sample and targeted migration process.

1.3 Lipid barriers functionality

Due to their hydrophobic nature, lipids are very effective for protection against moisture. The efficacy of edible lipid barrier is greatly influenced by their composition, crystal habit, physical state, and performing environment (Bourlieu 2009; Ghosh and others 2002; Martini and others 2006; Morillon and others 2002). The crystal habits of lipid ingredients are affected by physical factors during crystallization process such as transfer of heat, mass, and momentum. Bourlieu and others (201) observed that slower cooling rates significantly decreased the WVP of a blend of white beeswax and acetic acid ester of mono and diglycerides, but not the WVPs of other materials under investigation such as white beeswax, a palmitic-stearic acids blend, and acetylated monopalmitin. Tempering procedures and aging time result in changes in polymorphism, crystal size and crystal distribution of lipids, in turn affecting water permeability properties (Morillon and others 2002). Cocoa butter films were observed to have WVPs lowered by approximately 15 folds when tempering at room temperature is applied instead of quick cooling at 3^{o}C (Landman and others 1960). The same study found that the WVP of quick-cooled film decreased over time, while the WVP of tempered film increased. The authors suggested that the former phenomenon happened as polymorphic forms with higher melting points were formed, while the later phenomenon was due to interaction between solid and liquid lipids, which could happen following tempering process at room temperature but was unlikely at 3^{o}C. Kester and Fennema (1989) observed that when lipid polymorphism was kept unchanged, resistance to water vapor transport in tempered stearyl alcohol increased by 50% after 35 days. Application of laminar shear on cocoa butter was observed to affects crystal habit with both temperature ladder and breaking forces (Maleky and Marangoni 2011, Padar and others 2009). Laminar shear can arrange lipid crystallites to be oriented in a direction parallel to the external shear field, reduce the size of crystal clusters, and lower the density of the lipid (Maleky and others 2011; Maleky and others 2012). These changes reduce oil permeability of lipid. For example, sheared cocoa butter was shown to have lower rate of oil diffusion compared to cocoa butter that was statically crystallized (Maleky and others 2012). It could be expected that shearing might also reduce moisture permeability of lipid because water vapor and oil move through the same route through the system. Optimization of a lipid-based barrier for functionality or sensory purpose might call for blending of different lipids as well as incorporation of ingredients containing proteins, polysaccharides, and/or plasticizers (Baldwin and others 2012). Gosh and others (2005) observed that cocoa powder and lecithin increased the WVP of a coconut oil-based film, while sugars decreased the WVP. Replacing lecithin with Citrem and replacing sucrose with lactose or dextrose also increased WVP. Pfeifer and others (2008) demonstrated that different dairy powders, sugars, emulsifiers, and cocoa powder treatments affect WVP lipid-based films differently. Interaction between water and non-lipid ingredients can be especially significant. At water activities <0.85, lipid-sucrose-based films were found to have similar WVPs when in contact with liquid water as when in contact with water vapor; while at water activities >0.85, these films had had much higher WVPs when in contact with liquid water than vapor (Morillon and others 1998). The phenomenon was not observed in lipid-based film. The authors explained this as due to partial solubilization of sucrose and swelling of the matrix at the same time. Therefore, **the objective** of this study was to evaluate if there was a difference in moisture migration through lipid barriers that were structurally modified (sheared) and moisture migration through lipid barriers that were not structurally modified (static) in a controlled temperature and humidity environment, with consideration to commonly used non-fat ingredients.

2. Materials & Method

2.1 Materials

Silica gel grade 60 (230-400 mesh), magnesium chloride hexahydrate ($MgCl_2.6H_2O$), and sodium chloride were purchased from Fisher Scientific (Pittsburg, PA), hydroxypropyl methyl cellulose (HPMC E15) was purchased from The Dow Chemical Co. (Midland, MI). Palm oil was provided by Loders Croklaan (Channahon, IL). Lecithin Alcolec® S was obtained from American Lecithin Company (Oxford, CT). Calcium chloride was purchased from Sigma-Aldrich Co. LLC (St. Louis, MO). Commercial cocoa powder was used for palm oil and cocoa powder blend.

Three different lipid materials were studied, as indicated in table 1. The materials were completely melted at appropriate temperatures to destroy crystal memory before formation of test samples. For palm oil blends, cocoa powder or lecithin was added after pure palm oil had been melted at 70^{o}C and held at 50^{o}C for 10 min.

Samples were formed into flat round disks of 1mm thickness using custom-made molds, each comprised of a solid plastic base, a removable aluminum sheet with holes of 42mm in diameter, and a solid plastic top. A layer of aluminum foil was placed between the base and the aluminum sheet. Two crystallization processes were tested. For static samples, melted lipid materials were pipetted into molds at room temperature (22°C), covered by a layer of parafilm, and then covered by the plastic top. For sheared sample, lipid materials were sheared using a turbine-type impeller with 3 blades in a 400 mL glass beaker, submerged in a water bath with temperature ranging between 22 to 25^{o}C. After partial crystallization, the materials were transferred to the molds. Specific shear rate, time, and starting temperature of the water bath are shown in Table 1. Sheared samples were crystalized for 3 hours and static samples were crystalized overnight; crystallization was done in an incubator at 20^{o}C.

Table 1. Lipid materials and corresponding melting and shearing procedure

Lipid material	**Shearing procedure**
Palm oil	180.06 s^{-1}, 26 min, 24°C starting water
Palm oil + 20% cocoa powder (w/w)	260.09 s^{-1}, 5 min, 22°C starting water
Palm oil + 0.5% lecithin (w/w)	260.09 s^{-1}, 8 min 30 sec, 24°C starting water

2.2 Methods

Pure palm oil samples were tested in a 95%/33% relative humidity (RH) gradient. A gel of 95% RH was made (37.5% w/w of silica gel, 3% w/w of HPMC, 13.2% w/w of saturated solution of $MgCl_2.6H_2O$, and 46.3% w/w of deionized water) and filled into plastic AQUALAB cups (Decagon Devices, Inc., WA) until about half full. Sample disks were put on top of these cups and sealed on the edge with additional palm oil. Sample cups were placed in a desiccator that had 33% RH (using a saturated $MgCl_2.6H_2O$ solution) in an incubator set at 5°C.

Palm oil blend samples were tested in a 3.5%/75% RH gradient. $CaCl_2$ was filled into plastic AQUALAB cups until half full. Lipid disks were put on top of these cups and completely sealed with each of the respective materials. Sample cups were placed in a glass desiccator that had 75% RH (using a saturated NaCl solution) in an incubator set at 20°C

At least 4 replications were made for each sample set. Samples were weighed at day 0, 4, 7, then intervals of at least 7 days afterward, until at least 4 straight points had been obtained. Weighing was done with a Mettler Toledo XS205 Dual Range (Columbus, Ohio, USA) analytical scale at maximum amount 81g and d=0.01

The water vapor transmission rate (WVTR) values were calculated using the following equation:

$$\text{WVTR} = \text{slope}/A$$

where slope is the slope of a straight line portion of the plot of weight change vs. time (mg/days) and A is the area of the film in contact with humidity. As the disks were bigger than an AQUALAB cup’s 3.9 cm diameter, A was determined from a 3.9 cm diameter circle to be 11.946 cm^2. Therefore, the unit for WVTRs is $mg.days^{-1}.cm^{-2}$. Weight loss was a linear function of time for all samples studied ($0.9 < r2 < 0.95$). The WVP was calculated as:

$$\text{WVP} = \text{WVTR} * \Delta x/\Delta p$$

where Δx is the thickness of the film (mm) and Δp (mmHg) is the vapor pressure difference between both sides of the film. The unit for WVPs is $mg.mm.days^{-1}.cm^{-2}.mmHg^{-1}$. The thickness of the films was 1 mm, as determined by the mold's thickness. Pressure gradients (Δp) were to be 3.86 mmHg for pure palm oil sample (95%/33%RH gradient) and 12.5125 mmHg for all other samples (3.5%/75% RH gradient).

Weight change in replicates were averaged in each day to create one single slope for calculation.

3. Results and discussion

Palm oil and palm oil blends

Table 1. Water vapor permeability (WVP) of palm oil and palm oil blend samples

Lipid material	Testing condition	Average WVP ($g.mm.days^{-1}.cm^{-2}.mmHg^{-1}$)	
		Static samples	Sheared samples
Pure palm oil	95%/33% RH gradient, 5°C	5.4×10^{-2}	2.1×10^{-2}
Palm oil + 20% cocoa powder	3.5%/75% RH gradient, 20°C	10.3×10^{-2}	5.4×10^{-2}
Palm oil + 0.5% lecithin	3.5%/75% RH gradient, 20°C	3.4×10^{-2}	5.2×10^{-2}

The testing condition and results of the experiment were summarized in Table.1 and figure 1-3. In general, sheared samples gave lower WVP on average in case of pure palm oil and palm oil with 0.5% lecithin, but gave higher average WVP in case of palm oil with 20% cocoa powder. A replicate experiment confirmed the trend observed with the palm oil and cocoa powder blend.

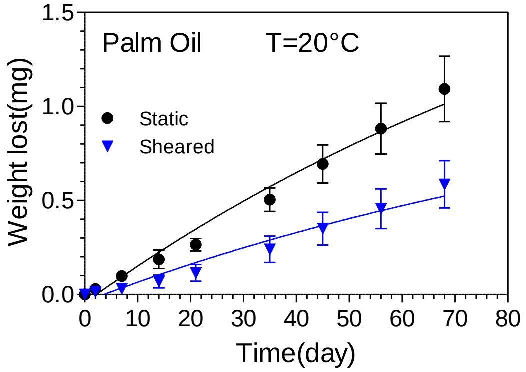
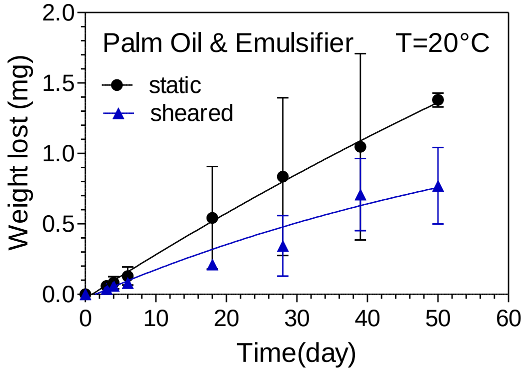


Figure 1. Average weight change over time of pure palm oil samples formed under static condition vs. sheared condition



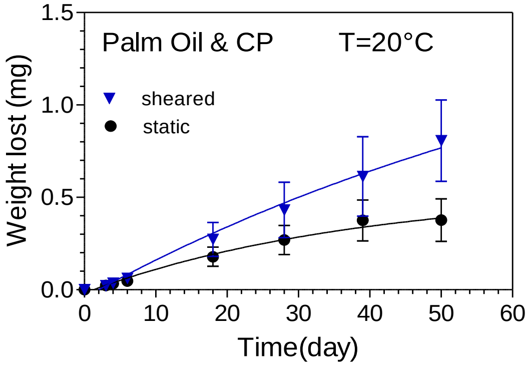


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Figure 2. Average weight change over time of palm oil + 0.5% lecithin samples formed under static condition vs. sheared condition.

Figure 3. Average weight change over time of palm oil + 20% cocoa powder formed under static condition vs. sheared condition.

Martini and others (2006) observed that under the same storage condition, crystallization at 20^{o}C with or without shear did not make a significant difference in WVP values of films made from partially hydrogenated palm kernel oil (PKO) blended + 28% canola oil (w/w), PKO + 27% canola oil + 1% polyglycerol monostearate (w/w), distilled monoglyceride + 27% canola oil (w/w), or Benefat. This suggested that the lipid composition of the barrier could affect the effectiveness of shear application in increasing moisture migration inhibition through the network.

Gosh and others (2005) showed that WVP of coconut oil-based films increased with increasing proportion of cocoa powder or lecithin, and that lowered temperature reduced WVP as well as increased solid fat content for blend containing both cocoa powder and lecithin. It could be of interest to study the effect of laminar shear on the WVP of lipid-based barriers under different temperature or with other proportion and type of non-lipid materials.

In order to identify causes of difference between WVPs of static and sheared sample, it is suggested that the solid fat content, crystal polymorphism, and crystal cluster size and agglomeration be investigated. These information can be obtained using nuclear magnetic resonance, X-ray diffraction, and polarized light microscopy, respectively. For future studies, it would also be beneficial to use more robust methods to observe water diffusion in the materials, such as magnetic resonance imaging.

4. Conclusion

The structure of edible lipid barrier can be modified to improve inhibition against moisture migration in foods. Application of laminar shear during crystallization induced significant decrease in water vapor permeability (WVP) of palm oil barriers at 5°C. For palm oil blended with cocoa powder or lecithin, no significant difference in WVP was observed between samples crystalized sheared and static conditions. In general trend, sheared samples had higher average WVP in case of blend containing 20% cocoa powder, and had lower average WVP in case of blend containing 0.5% lecithin. This suggested that the pathway of moisture distribution could be uniquely affected by the type of non-lipid component used in the blend. In future studies on shear application for lipid barriers, it might be of interest to use more potent methods to investigate the diffusion of water in the network, as well as how the technique specifically affect the functionality of different lipids and blends.

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