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Evaluation and effect of rice bran and microwave heating on the physicomechanical properties of low-density polyethylene film and packaged milk quality

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Abstract: Low-density polyethylene (LDPE) film was developed with a mixture of reference LDPE and rice bran (RB). Three mixtures of 5, 10, and 15% RB were used for the LDPE film preparation. The films were analysed for the physicomechanical properties before microwave heating (MH) and after MH and packaged milk quality was analysed regarding MH and pasteurisation. After MH, the physicomechanical properties of the material change significantly. LDPE film containing 15% RB was created to beat changes in tensile strength, elongation at break, bursting strength, and water vapour transmission rate (WVTR). The total migration of tested packaging materials was within the agreeable limit and after MH it also was within the permissible limit. Four different simulators were used to create food categories. These food simulators included distilled water, 3% acetic acid, 50% alcohol (ethanol), and *n*-heptane. The obtained results display that the total migration of food packages is dependent on the MH period, package material, and the simulator.

Keywords: microstructure; pasturisation; sensory quality; food packaging; elongation

Packaging represents an interface between the product and the environment (Kim and Pometto 1994). The principal roles of food packaging are to protect a food product from outside influences and damage, to contain food and to provide ingredient and nutritional information. The goal of food packaging is to contain food in a cost-effective convenient way that satisfies industry requirements and consumer desires, maintaining food safety and minimising any environmental impact (Marsh and Bugusu 2007). Thermoplastics are widely used in packaging and fabrication of bottles and films. The major type of thermoplastic material includes polyethylene, polypropylene, polyvinyl chloride, polystyrene and other resins (Andrady and Neal 2009). Low-density polyethylene (LDPE) was studied to enhance its biodegradability by incorporating different additives like starch. The rice bran (RB), the outer layer of a rice grain, is a low-cost (Kim and Pometto 1994) underutilised coproduct of rice milling, containing 65–70% of saccharides along with other constituents like protein and fat. The polyethylene type can be identified easily by the relationship between physical properties and density (Kumar et al. 2006). Generally, polyethylene clarity improves with decreasing density. LDPE is used in film applications because of superior tensile strength, elongation at break characteristics, and puncture resistance of films (Rennert et al. 2013). The starch granules are often used as a filler in plastics.

The level of starch addition is generally limited to about 10%. Another process was developed to make extrusion blown films from mixtures of starch, poly(ethylene-co-acrylic acid) and LDPE (Evangelista et al. 1991). For in-

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stance, an experiment was conducted by Evangelista et al. (1991) to examine the properties of starch blends containing 40% polyolefin and 60% plasticised starch or 50% polyolefin and 50% plasticised starch. These materials are not used for the packing of food being reheated in the microwave oven. Brans have been considered as a promising material in the field of certain thermoplastic applications involving food packaging, because of its low cost, availability, biodegradability, food grade, and high purity (Khan et al. 2017). Polyethylene plastics have the generally advantageous property of toughness, high tensile strength and good barrier properties to moisture (Risch 2009). LDPE films tend to be soft and relatively clear, whereas films made from high-density polyethylene (HDPE) have a crisp feel and are opaquer (Kumar et al. 2006). The growing uses of microwaves at home and in the industry have created an expanding market for packaging materials enabling the microwave oven heating of foods (Jagannath et al. 2006). Product homogeneity is very important in microwave heating (MH). Most of the conventional packaging materials are transparent to microwaves (Issue 2000).

MATERIAL AND METHODS

Material

LDPE was purchased from IPCL, Baroda, India. The density and the melt flow index of the reference LDPE were 0.922 g cm^{-3} and 4 g $(10\ min)^{-1}$, respectively. RB (Kalyani-II) was procured from Indian rice mills and pre-dried at 60 °C for 24 h in a hot air dryer (FD 115; Binder GmbH, Germany) before compounding. The average particle size of used RB was < 30 µm. Fresh milk was purchased from the local supermarket from the milk vendor machine. The fat content of the milk used in the packaging was 3.5%.

Methods

Formulation of LDPE films with different percentages of RB. The concentration of 5, 10, and 15% of RB was incorporated into LDPE using a twin-screw extruder (ZK35; Dr. Collins GmbH, Germany) and blown into films of uniform thickness using film blowing equipment (ZK35; Dr. Collins GmbH, Germany) attached to the twin-screw extruder. For that, the reference LDPE was physically blended with RB at different concentrations of 5, 10, and 15% for a period of 20–22 min (Model CTW 100 Haake; ThermoFisher, Germany). Twin-screw speed was adjusted to 38 rpm while the torque was maintained in the range of 5–11.6 Nm. The diameter and length of the screw were 25 mm and 550 mm, respectively. The compounded polymer strands were cooled in a cool air stream and pelletised. The pellets were then blown into films using a single-screw extruder (ZK25; Dr. Collins GmbH, Germany) with lift to drag (L/D) ratio of 22 : 1 with the film blowing unit (ZK25; Dr. Collins GmbH, Germany) at the temperature range of 130–165 °C. The film blown ratio was fixed at 4 : 1 and the thickness was maintained uniformly at 68 ± 1 µm.

Microwave heating (MH). Reference LDPE and RB incorporated LDPE films were subjected to MH. For this purpose, a domestic microwave oven (MS2044V; LG, Czech Republic) having a frequency of 2 450 MHz was used. The experimental condition set was medium high for 5 min. The tested materials were cut into prescribed sizes (2 × 10 cm) in line with the requirement of the test conditions and placed in a beaker and allowed to expose to microwaves for 5 min.

Milk packaging method. An amount of 200 mL of fresh milk was packed in pouches of 15 × 20 cm in size (No. 24 lab, Vertrod, US). One batch of the sample was pasteurised at 90 °C in the steam kettle (SWK 1031SS; Sencor, Czech Republic) for 10 min. Another batch of the sample was heated in the microwave oven by following the same conditions applied to the packaging materials.

Evaluation of physicomechanical properties. Physicomechanical properties were tested by different test methods and standards [American Society for Testing and Materials (ASTM) and international standards], such as thickness (ASTM D-374), tensile strength (ASTM-882), tear strength (ASTM-1922), elongation at break point (ASTM-882), bursting strength (ASTM D-774), water vapour transmission rate (WVTR) (ASTM E-96), and total migration (IS 9845, IS 1986).

Surface morphology. The surface morphology of RB filled LDPE films was examined by scanning electron microscopy (SEM) and the photographs are shown below. The surface morphology of these LDPE films was evaluated using SEM (VEGA II LMU VG3720771CZ; TESCAN Essence™ EDS, Czech Republic) operating at 20 kV. The film samples were mounted onto copper stubs using double-sided sticky tapes. Mounted stubs were gold-coated (20 nm thickness). Micrographs of the samples were taken at higher magnifications (500× and 800×) to identify the changes on sample surfaces due to the incorporation of RB.

Sensory evaluation. Sensory evaluation of milk was carried out on a 9-point hedonic scale by 10 semi-trained panellists. The scope pattern was 1 for dislike extremely and 9 for like extremely. The points of the panellists were cumulated, and overall acceptability was calculated.

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Statistical analysis. The results were statistically analysed using analysis of variance (ANOVA). Statistical analysis was accomplished using Statistica CZ 9.1 software (Stat Soft Ltd., Czech Republic). The results are expressed as mean values ± standard deviation (SD) ($P < 0.05$).

RESULTS AND DISCUSSION

Thickness. The thickness of the food packaging materials is very critical as it ultimately determines the mechanical as well as permeability properties. The thickness of reference LDPE film was decreased by incorporated RB as shown in Table 1.

Tensile strength. Tensile strength is the measure of the force required to stretch the material at a constant rate to the breaking point/yield point. While the tensile strength of the tested materials (5, 10, and 15% RB) before MH was comparable with the reference LDPE film in the machine direction (MD), the results show increased tensile strength for 5% and 15% of RB and it was decreased for 10%. In cross direction (CD), the tensile strength of the test materials was increased in 5% of RB, however, in 10% and 15% of RB incorporated LDPE films, it was decreased while compared with the reference film. However, in the film with 10% of RB, the tensile strength was found to decrease in the MD. After, MH, the tensile strength of the test packaging materials in the MD was found to decrease marginally as shown in Table 1.

It is found that at the lower concentration (5%) the dispersion and distribution of RB seem to be uniform but when RB reaches higher concentrations, the RB particles are grouped together leading to the formation of clumps, possibly due to the hygroscopic nature of RB. It is evident from the changes in the tensile strength of the test materials that the increased RB content decreases the homogeneity of the films, which in turn contributes to the decreased mechanical properties. At the lower concentration (5%) the dispersion and distribution of RB seem to be uniform but when RB reaches higher concentrations, the granules are grouped together leading to the formation of clumps, possibly due to the hygroscopic nature of RB as shown in Figure 1. After MH, the tensile strength of the tested films was decreased in LDPE films filled with 5, 10, and 15% of RB while compared with the reference film in the MD. In CD, the tensile strength of the films was also decreased while compared with the reference LDPE film.

Tear strength. Tear strength is the measure of the energy absorbed by the test sample in propagating a tear

Table 1. Changes in mechanical properties of tested packaging materials before and after MH (mean ± SD)

Mechanical properties		Before MH				After MH			
LDPE reference		LDPE reference	5% RB	10% RB	15% RB	LDPE reference	5% RB	10% RB	15% RB
Thickness (μ)		68.00 ± 2.00	63.00 ± 3.00	60.00 ± 2.00	58.00 ± 2.00	67.00 ± 3.00	63.00 ± 3.00	60.00 ± 4.00	57.00 ± 3.00
Tensile strength (MPa)	MD	15.40 ± 0.1	19.18 ± 0.10	7.49 ± 0.09	19.84 ± 0.06	15.73 ± 0.04	10.46 ± 0.05	15.42 ± 0.04	12.12 ± 0.03
	CD	18.30 ± 0.04	20.95 ± 0.06	8.12 ± 0.05	12.21 ± 0.04	20.41 ± 0.0	9.97 ± 0.08	16.29 ± 0.07	7.88 ± 0.06
Elongation at break (%)	MD	655.81 ± 1.00	165.50 ± 1.00	185.32 ± 0.80	254.61 ± 0.60	758.11 ± 1.00	146.73 ± 1.00	212.52 ± 1.00	240.41 ± 3.00
	CD	957.12 ± 3.01	107.82 ± 1.10	66.74 ± 2.21	68.62 ± 2.11	1054.04 ± 2.13	73.73 ± 1.12	109.26 ± 1.01	65.42 ± 2.04
Tear strength (g)	MD	168.03 ± 0.81	65.82 ± 2.01	64.05 ± 2.02	51.25 ± 2.05	179.22 ± 1.01	73.81 ± 1.02	65.06 ± 1.03	58.11 ± 1.04
	CD	363.20 ± 2.00	182.00 ± 1.00	104.60 ± 0.6	169.90 ± 2.00	492.60 ± 2.00	191.70 ± 1.00	108.70 ± 1.00	164.20 ± 2.00
Bursting strength (MPa)		0.060 ± 0.001	0.056 ± 0.003	0.038 ± 0.003	0.031 ± 0.004	0.057 ± 0.003	0.050 ± 0.001	0.045 ± 0.001	0.040 ± 0.006

SD – standard deviation; MH – microwave heating; LDPE – low density polyethylene; RB – rice bran; MD – machine direction; CD – cross direction

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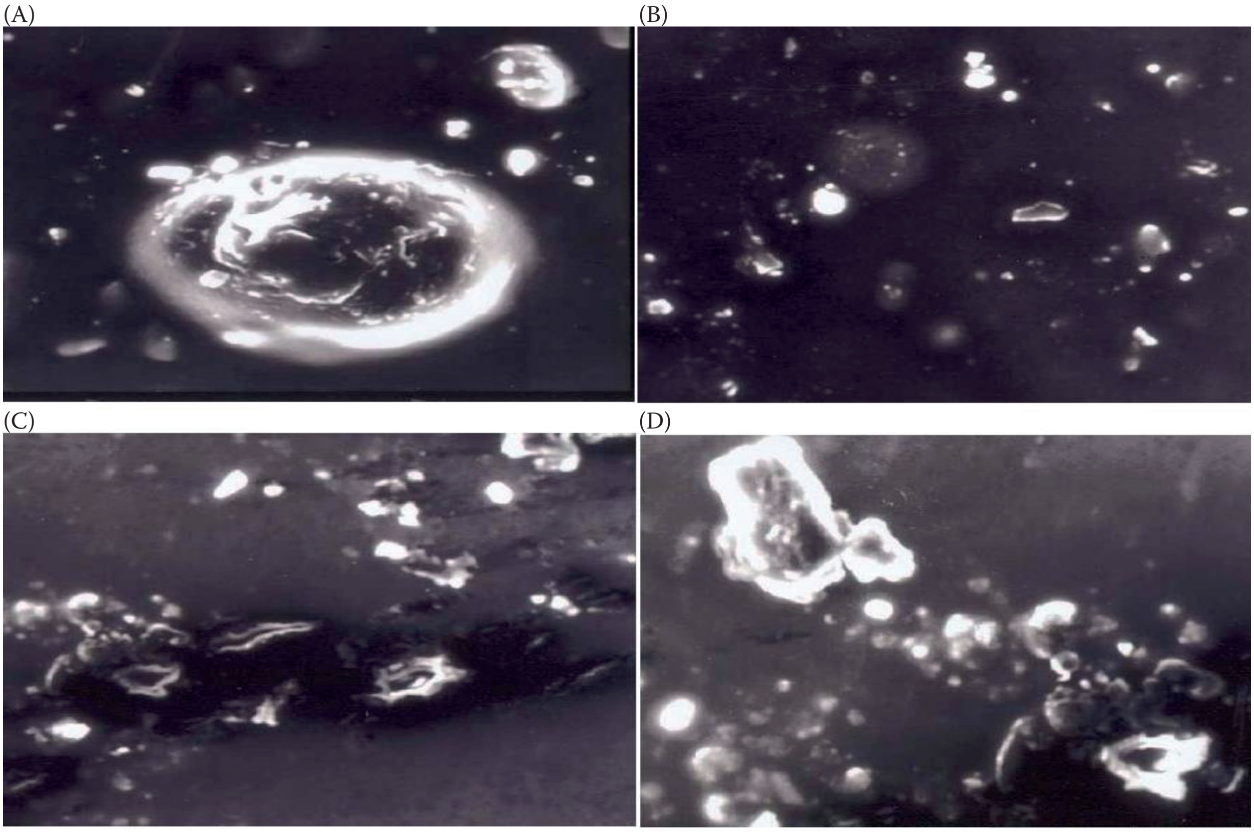

Figure 1. SEM micrographs of LDPE/RB blends: (A) RB, 800×, (B) 5% RB filled LDPE, 500×, (C) 10% RB filled LDPE films, 500×, (D) 15% RB filled LDPE films, 500×

SEM – scanning electron microscopy; LDPE – low density polyethylene; RB – rice bran

that has already been initiated by cutting a small notch in the test sample with a knife. Tear strength of the test materials before MH was decreasing for 5, 10, and 15% RB filled LDPE films while compared with the reference LDPE film in MD. In CD, tear strength of the test materials was decreased for 5, 10, and 15% RB filled LDPE films while compared with the reference LDPE film as well, as shown in Table 1. After MH, tear strength of the test materials was also decreasing for 5, 10, and 15% RB incorporated LDPE films while compared with the reference LDPE film in MD. In CD, tear strength of the test materials was decreased also in RB incorporated LDPE films while compared with the reference LDPE film. The changes in the tear strength of the test materials due to MH were found statistically significant as shown in Table 1.

Elongation at break. The elongation at break of tested packaging materials in MD as well as CD was decreased by the incorporation of RBs. The elongation at break of the tested materials before MH was decreased while compared with the reference LDPE film in MD. In CD, the percentage of elongation of the test materials was also decreased while compared with the reference LDPE film. After MH, the elongation at break of the tested materials was decreased while compared with the reference LDPE film in MD. In CD, the elongation at break of the tested LDPE films containing 5, 10, and 15% of RB significantly changed while compared with the reference LDPE film as shown in Table 1.

Bursting strength. The bursting strength of LDPE films before MH was found decreased if RB was filled while compared with the bursting strength of the reference LDPE film. After MH, the bursting strength was found to be decreased for 10% and 15% of filled RB while compared with the bursting strength of the reference LDPE film. The changes in the bursting strength of the test materials due to MH were found statistically insignificant and the same is shown in Table 1.

Water vapour transmission rate (WVTR). WVTR of the test packaging materials was significantly increased by the presence of RB, however, the differences between samples with incorporated RB were not significant as shown in Figure 2. After MH, the WVTR of the tested packaging materials was found increased

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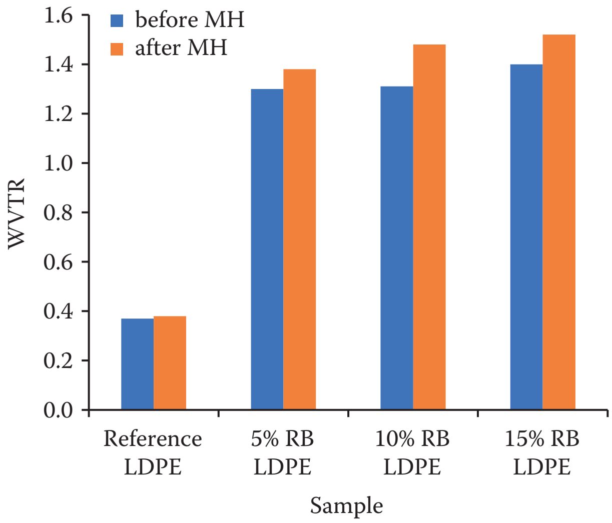

Figure 2. WVTR variation between before MH and after MH

WVTR – water vapor transmission rate; MH – microwave heating; LDPE – low density polyethylene; RB – rice bran

due to the onset of molecular reorganisation. Intermittent weighing determines the rate of the water vapour movement through the specimen into the desiccant. In this study, the systems have been found to equilibrate to the test conditions after 20 days of exposure. Further, due to MH, the values of WVTR are found to increase, which is statistically significant as shown in Figure 2.

Total migration. The tested packaging materials were evaluated for their compatibility through the total migration into food simulants such as distilled water, 3% acetic acid, 50% ethanol, and *n*-heptane under simulated conditions. Total migration values for all the RB filled LDPE films as shown in Table 2 revealed that there was an insignificant increase in the total migration value for the films containing RB. The total migration values of RB filled LDPE films were within the permissible limit and these films may be suitable food contact applications. Further, after exposure to MH, the migration values were found to remain within the permissible limit and thus these films may be suitable for MH of foods.

Surface morphology. Figure 1A shows the morphology of RB particles at higher magnification (800×) when the particles appear to be spherical in shape, having some irregularities on the surface. Figures 1B–1D depicts the images of 5, 10, and 15% RB domains dispersed in the LDPE matrix. At the lower concentration (5%) the dispersion and distribution of RB seem to be uniform but when they reach higher concentrations, the granules are grouped together leading to the formation of clumps, possibly due to the hygroscopic nature of RB. The SEM images reveal better compatibility between RB and LDPE fractions at lower concentrations (Figure 1).

Sensory evaluation. Deterioration in the sensory attributes, nutritional content and safety of food is caused principally by physical and chemical changes in the food during storage and by microbial spoilage (Meng et al. 2012). Tested biodegradable packaging materials, as conventional packaging, should minimise these deteriorative changes in food products (Osman et al. 2003). The milk was well accepted on a 9-point hedonic scale in reference LDPE, pasteurised milk in reference LDPE, pasteurised milk in 5% RB LDPE, and pasteurised milk in 10% RB LDPE as shown in Table 3.

Pasteurised milk in 15% RB LDPE and microwave heated milk in reference LDPE were liked slightly. Microwave heated milk in 10% and 15% RB LDPE were disliked. With microwave heated milk in 5% RB LDPE was neither like nor dislike.

In 5% and 10% RB filled LDPE films, the mean diameter of RB particles was increased while the specific surface of these particles was decreased (Madera-Santana

Table 2. Total migration variation before heating and after heating food simulants [mg (L m^{-1})]^2

Samples	Condition	Distilled water	3% acetic acid	50% alcohol	*n*-heptane
LDPE control	before MH	2.5 ± 0.1	4.5 ± 0.1	9.0 ± 0.4	8.5 ± 0.7
	after MH	4.0 ± 0.4	4.6 ± 0.1	23.5 ± 0.5	33.5 ± 0.6
5% RB	before MH	3.6 ± 0.1	4.9 ± 0.3	12.1 ± 0.2	10.3 ± 0.7
	after MH	4.5 ± 0.1	6.0 ± 0.2	26.2 ± 0.3	24.6 ± 0.4
10% RB	before MH	4.5 ± 0.3	4.9 ± 0.2	9.3 ± 0.5	11.5 ± 0.3
	after MH	4.5 ± 0.4	6.4 ± 0.1	26.9 ± 0.5	21.2 ± 0.4
15% RB	before MH	5.0 ± 0.3	5.5 ± 0.4	12.5 ± 0.4	9.2 ± 0.2
	after MH	6.0 ± 0.2	7.7 ± 0.4	28.6 ± 0.5	24.5 ± 0.4

LDPE – low density polyethylene; RB – rice bran; MH – microwave heating

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Table 3. Sensory analysis and OAA score of pasteurised and microwave heated milk

No.	Sample	OAA	Observation
1	reference milk	8.07 ± 0.33^{a}	like very much
2	pasteurised milk in reference LDPE	7.16 ± 0.24^{a}	like moderately
3	pasteurised milk in 5% RB LDPE	7.15 ± 0.13^{a}	like moderately
4	pasteurised milk in 10% RB LDPE	7.01 ± 0.07^{ab}	like moderately
5	pasteurised milk in 15% RB LDPE	6.90 ± 0.08^{b}	like slightly
6	microwave heated milk in reference LDPE	6.62 ± 0.18^{bc}	like slightly
7	microwave heated milk in 5% RB LDPE	4.25 ± 0.51^{c}	neither like nor dislike
8	microwave heated milk in 10% RB LDPE	4.20 ± 1.28^{de}	dislike
9	microwave heated milk in 15% RB LDPE	4.05 ± 1.28^{de}	dislike

$^{a–e}$Data with different superscript letters within a column are significantly different ($P < 0.05$); OAA – over-all acceptability; LDPE – low density polyethylene; RB – rice bran

et al. 2010). During food package interaction, polymer packaging materials tend to leach unprocessed monomers, processing aids and other undesirable substances present on the surface of the film (Arvanitoyannis et al. 1998; Madera-Santana et al. 2010). Due to the leaching process, the quality attributes of the food products may undergo changes, which make the product unacceptable (Meng et al. 2012). Therefore, it is necessary to evaluate the quantity of the migrating substances into the food. As food is a heterogeneous complex system, it is cumbersome to isolate and quantify the substances. Therefore, regulatory bodies like The United States Food and Drug Administration (FDA) and Bureau of Indian Standards (BIS) (IS 9845, IS 1986) have recommended food simulants of distilled water, 3% acetic acid, 15% ethanol and *n*-heptane as representatives of the food systems (Lahtinen and Kuusipalo 2007; Wang et al. 2017). Migration values for all the LDPE starch blend films revealed that there was no significant increase in migration value for the films containing starch. The transfer values of bran filled LDPE films were within the permissible limit and these films may be suitable for food contact applications (Kim and Pometto 1994). It is evident from the micrographs that the increased RB content decreases the homogeneity of the films, which in turn contributes to the decreased mechanical properties (Kumar et al. 2006). Further, after exposure to MH, the migration values were found to remain within the permissible limit and thus these films may be suitable for MH of foods.

CONCLUSION

The use of RBs also facilitated the integration of more fillers in LDPE. Most of the work has been attentive to the expansion of stiff injection moulded items having a higher concentration of bran. Mechanical properties of LDPE were found to decrease by the addition of RB. After MH of the tested materials, mechanical properties have significantly changed. In this study, the test packaging materials were evaluated for their compatibility through the overall migration into food simulants such as distilled water (for aqueous food products), 3% acetic acid (for acidic products), 50% ethanol (for alcoholic beverages) and *n*-heptane under simulated conditions (Kumar et al. 2018). Migration values for all prepared LDPE bran films revealed that there was an insignificant increase in the migration value for the films containing bran. A similar trend was observed in elongation at break. LDPE with 5% incorporated RB is more suitable for milk MH, in comparison with LDPE combined with 10% or 15% RB. As the RB incorporated amount increases in LDPE, the quality for MH of milk decreases as shown in Table 3. Total migration in RB incorporated LDPE increased as shown in Table 1. Due to total migration, the milk sensory quality decreased. The development of RB filled LDPE film is low-cost in view of the economics.

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