**ORIGINAL PAPER**



# **The efect of trans‑polyisoprene/LDPE based active flms on oxidative stability in roasted peanuts**

**Kirtiraj K. Gaikwad<sup>1</sup> · Suman Singh2 · Yuvraj Singh Negi3 · Youn Suk Lee4**

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#### **Abstract**

A natural *trans*-polyisoprene (PSN)-based rubber that functions as a light-activated oxygen-scavenging flm were developed and applied this material to the packaging of roasted peanuts to prevent loss of quality due to oxidative degradation. Results of the oxygen scavenging analysis indicated that flms exhibited reasonable oxygen scavenging rates. Roasted peanuts were packaged with or without LDPE/PSN flms, and they were stored for 90 days at 25 °C for oxidative analysis. The roasted peanuts packaged with LDPE/PSN films exhibited significantly lower (*P*<0.05) headspace oxygen content within the packages than that of controls containing pure LDPE flms. The Hunter *L*, *a*, and *b* values for peanuts packaged with LAE/PSN flms were significantly higher (*P*<0.05) than those of the control sample. After 90 days of storage, total peroxide, TBA values increased. Total peroxide values increased from 0 to 28.11 meqQ<sub>2</sub>/Kg in controls and  $17.51\pm0.11$  and  $15.28\pm0.01$  meqO<sub>2</sub> /Kg in peanuts packaged in 10 and 20% PSN LDPE flms, respectively, and similar results were observed for TBA values. These results demonstrated that experimentally developed LDPE/PSN oxygen scavenging flms inhibited oxygen-mediated deterioration of roasted peanuts. The LDPE/PSN 10 and 20% flms may be useful as active food‐packaging material.

**Keywords** Oxygen scavenger · Polyisoprene · Peanuts · Low-density polyethene · Film · Active packaging · Lipid oxidation

## **Introduction**

As the plastic processing industry continues to grow, consumers of food processing industry products currently demand innovative packaging solutions that are specifcally tailored to productable needs and market desires. Active packaging technologies that incorporate the use of novel plastics are designed to promote food preservation to extend

 $\boxtimes$  Kirtiraj K. Gaikwad gaikwad.msu@gmail.com

 $\boxtimes$  Youn Suk Lee leeyouns@yonsei.ac.kr

- <sup>1</sup> Department of Pulp and Paper Technology, Indian Institute of Technology Roorkee, Roorkee, India
- <sup>2</sup> Department of Food Engineering, Institute of Food Science & Technology, VCSG Uttarakhand University of Horticulture and Forestry, Majri Grant, Dehradun 248140, Uttarakhand, India
- <sup>3</sup> Department of Polymer and Process Engineering, Indian Institute of Technology Roorkee, Roorkee, India
- <sup>4</sup> Department of Packaging, Yonsei University, Wonju, Gangwon-do 26493, South Korea

the shelf life of the packaged products by actively controlling the atmosphere within the package [\[2,](#page-7-0) [12\]](#page-7-1). These conditions include techniques that remove oxygen from the package. Active packaging that incorporates the use of oxygen scavengers is the most widely used technique in oxygen removal from food packages [\[13](#page-7-2), [19](#page-7-3)].

Oxygen is one of the key causes of food deterioration. Oxygen present within packages is responsible for oxidative rancidity of unsaturated fats in food, loss of vitamins (especially C), browning of raw meat, oxidation in oil and pigments, and the development of the growth of aerobic microorganisms that are responsible for food spoilage [[11,](#page-7-4) [20](#page-7-5)]. To overcome such issues, oxygen scavengers/absorbers have been utilized to eliminate oxygen or to avoid the permeation of oxygen into the packaging atmosphere. Specifcally, oxygen scavenging agents can combine with the packaging materials to reduce high oxygen content that either penetrates through the packaging wall or that exists within the package headspace [[2,](#page-7-0) [6](#page-7-6)]. Iron-based oxygen scavengers are commonly used in food processing industries, as these scavengers prolong the food shelf life from 3 to 4 days to 14 or more days [\[7\]](#page-7-7). Oxygen scavengers that are primarily ironbased, however, require moisture to activate the scavenging reaction, and these scavengers are therefore not useful for dry food products, including peanuts, almonds, and other dry fruits. Activation by UV-light could provide one possible solution to allow for the control of the oxidation of these scavengers until the fnal use in the package.

Peanuts are a signifcant crop worldwide in both developing and developed commercial markets. Lipid oxidation due to oxygen is a major issue in the peanut processing industry due to the high amount of polyunsaturated fat present in peanuts [\[16](#page-7-8)]. Roasted peanuts are extremely susceptible to lipid oxidation, as they contain high amounts of polyunsaturated fatty acids. Lipid oxidation in roasted peanuts results in spoilage due to rancidity formation, which is a major concern for the shelf life stability of many confectionary products that contain roasted peanuts [[15\]](#page-7-9). Aside from this, unpleasant favour alterations and a reduction in pleasant attributes also occur in roasted peanuts due to oxidation.[\[4](#page-7-10)]. Therefore, it is important to develop a safe, efficient, and cost-efective packaging system to prevent the slow oxidation process in roasted peanuts. The use of oxygen scavenging packaging is recommended to maintain the quality of roasted peanuts during storage [[14\]](#page-7-11).

*Trans*-polyisoprene (PSN) is a natural rubbery biopolymer that oxidizes upon exposure to atmospheric oxygen at ambient temperature. PSN is utilized in many packaging materials, including flms and tapes [\[21\]](#page-7-12). Generally, a small amount of a catalyst in the form of a transition metal is added into polymer-based scavengers to accelerate the oxidation reaction. Numerous studies examining thermal oxidation, photo-oxidation, and ozonolysis of PSN have been performed during the last 20 years, and several oxidation methods have been suggested [\[1\]](#page-7-13). None of these studies, however, have evaluated the oxidation rate to determine the capacity of PSN as an oxygen scavenging polymer for food packaging applications.

The objectives of the present study were to develop UVactivated low-density polyethylene (LDPE) based oxygen scavenging flms containing diferent amounts of PSN and to investigate their oxygen scavenging properties under various storage conditions. The oxidation levels of the roasted peanuts that were packed into the prepared oxygen scavenging flms under non-commercial storage conditions were investigated using modern measuring equipment. Alterations in

the physicochemical qualities of the roasted peanut samples were also assessed.

## **Materials and methods**

#### **Materials**

*Trans*-polyisoprene (PSN) as a active agent in the form of resin and benzophenone (white crystals) were purchased from Sigma-Aldrich (CA, USA). Anhydrous cobalt chloride was purchased from Daejung Co., Ltd. (Kyungki, South Korea). Low-density polyethylene (LDPE) (Lutene LB7500) was purchased from LG Chem. Ltd. (Seoul, South Korea). All chemicals used in experiments were of reagent grade and used without further purifcation.

## **Preparation of trans‑polyisoprene/LDPE based active flm**

LDPE/PSN composite flms were developed using a laboratory scale, twin screw extruder (BA19, BauTek Co., Seoul, South Korea) possessing a length/diameter (L/D) ratio of 40:19. Prior to melting the compounds and extruding them, all materials were dried at 100 °C for 24 h to remove moisture. LDPE/PSN composite flms with PS loadings of 0, 5, 10, and 20% (w/w) were processed in the presence of diferent amounts of cobalt chloride (metal catalyst) as presented in Table [1](#page-1-0). Benzophenone was used as an initiator during the preparation of flms. The weighed materials and the LDPE resin were placed in a plastic pouch and thoroughly mixed by shaking prior to the melt extrusion process. In the twin screw extruder, the header zone was set to 120 °C, zones 1–6 were set to 140 °C, and the feed zone was set to 110 °C. The barrel pressure for melting the compounds was set to 5.2 kgf/  $\text{cm}^2$ , and the extrusion process was performed at 4.65 kgf/ cm<sup>2</sup>. The developed oxygen scavenging film samples were labelled LDPE/PSN 5%, 10%, and LDPE/PSN 20% for 5%, 10, and 20 wt% PSN loadings, respectively. LDPE flm without PSN was considered as a control LDPE sample. The LDPE/PSN flms were maintained at a thickness of 50 μm to allow for estimation of their physical properties.

<span id="page-1-0"></span>**Table 1** Recipe for the LDPE/ PSN flms with diferent polyisoprene (PSN) percentages



## **Oxygen scavenging capacity and rate of flms**

The oxygen absorption cell used for the oxygen scavenging experiment was comprised of a 125 mL transparent glass vial containing two pieces ( $8 \text{ cm} \times 6 \text{ cm}$ ) of  $4 \text{ g}$  (w/w) LDPE/ PSN film under ambient oxygen (20.1%  $O_2$ ). The oxygen absorption cell was exposed to UV light for 10 s to activate the scavenging reaction in the flms before sealing the vial with a metal cap that possessed a polytetrafuoroethylene (PTFE) septa to collect the oxygen during the experiment. The vial was stored at constant ambient temperature  $(25 \degree C)$ for 8 days using a digital temperature meter (HTC, Seoul, South Korea). The oxygen scavenging abilities of the LDPE/ PSN flms were assessed according to the method reported by Gaikwad et al. [\[8](#page-7-14)]. The amount of oxygen in the vial was assessed each day using an oxygen analyzer (MOCON, CA, USA). The probe of the analyzer was injected into the vial via the PTFE septa and the oxygen content (%) within the headspace was measured.

## **Sample preparation for storage test of roasted peanuts**

Roasted peanuts were procured from a local market of Wonju, South Korea. The 10% LDPE/PSN and 20% LDPE/ PSN flm pouches (8 cm×8 cm) were prepared and exposed to black UV light for 10 s on a conveyer belt at the Department of Packaging (Yonsei University, Wonju, South Korea) to activate the oxygen scavenging reaction in the LDPE/ PSN flm. The roasted peanuts (50 g) were added into the activated LDPE/PSN pouches, and these were then sealed. LDPE/PSN pouches containing peanuts were then packed into coextruded, multilayered, high barrier transparent pouches (C5045, nylon/PE/nylon/PE/nylon/LLDPE, Boss-Pack, Seoul, South Korea) as indicated in Fig. [1.](#page-2-0) Roasted peanuts packed without the LDPE/PSN pouch were used as a control. All packages were then stored at controlled room temperature ( $25 \pm 1$  °C) for 90 days, and samples of the peanuts were taken at regular intervals every 10 d throughout the storage period to evaluate quality.

#### **Evaluation of oxygen content in packages**

The volume of oxygen in the headspace of the roasted peanut packages during 90 days storage was monitored using a digital headspace analyzer (CheckMate3, Topac Inc., Cohasset, MA, USA). Prior to opening the packages, the syringe of the headspace analyzer was inserted into the package through a polytetrafuoroethylene (PTFE) septum attached to the surface of the package, and then the oxygen data were recorded. The results were expressed as  $\%$  O<sub>2</sub>. Calibration was performed using ambient air [[10](#page-7-15)].

#### **Color of roasted peanuts**

The Hunter color values, L<sup>\*</sup> (brightness or darkness),  $a^*$  (redness or greenness), and  $b^*$  (yellowness or blueness), of roasted peanuts were evaluated using a digital spectrophotometer (NS800, Shenzhen Threenh Technology Co., Ltd., Shenzhen, China) that was calibrated using a white reference plate. Measurements were taken at the surface of the roasted peanuts using an 8 mm aperture diameter. Each minced sample was then stirred into 10 mL of deionized water for measurement of pH (Orion Research, Inc. pH model 501, Boston, Mass., U.S.A.). The diferences in the total color values (*Δ*E) of the roasted peanut samples were determined using Eq. ([1](#page-2-1)).

<span id="page-2-1"></span>
$$
\Delta E = (\Delta L^2 + \Delta a^2 + \Delta b^2)^{0.5}
$$
 (1)

Here,  $\Delta L$  (lightness variance) =  $L^*$  –  $L$ ,  $\Delta a$  (green chromaticity variance) =  $a^*$  − *a*, and  $\Delta b$  (yellow chromaticity variance) =  $b^*$ −*b*. Additional,  $L^*$ ,  $a^*$ , and  $b^*$  are the color

<span id="page-2-0"></span>**Fig. 1** Storage test of oxygen scavenging packaging containing LDPE/PSN flms (10 and 20%) and roasted peanut samples



values of the standard plate, and *L*, *a*, and *b* are the color parameter values of the roasted peanuts samples.

## **Peroxide value**

Peroxide values (PV) of the roasted peanuts were evaluated using the Association of Analytical Communities (AOAC) 965.33 method. Briefly, roasted peanuts were coarsely ground and compressed using a hydraulic press **(**TMA103, TENAQUIP Limited Calgary, AB, Canada) to obtain the oil. Then,  $5 \pm 0.05$  g of oil were weighed, poured into 250 mL fasks, and dissolved using 30 mL of an acetic acid and chloroform solvent mixture (ratio- 3:2 v/v). Next, 0.5 mL saturated KI was poured into the fask and shaken for 1 min. After shaking, 30 mL water was added, and then 0.5 mL starch solution (1 g/100 mL water) was added as an indicator. The obtained solution was titrated by 0.01 N sodium thiosulfate until the blue color disappeared. The PVs of oil were calculated by the below equation:

$$
PV = S \times N \times 1000/W
$$
 (2)

Here, S is the volume of sodium thiosulfate used for titration, N is the normality of sodium thiosulfate, and W is the mass of peanuts oil in g.

## **Thiobarbituric acid reactive substances (TBARS) assays**

The TBARS values of the roasted peanuts samples were measured by the method described by Yang et al. [\[22](#page-7-16)]. Samples (0.4 g) were weighed into 30 mL Pyrex screw-cap test tubes, and two drops of antioxidant solution (A: 0.3 g butylated hydroxyl anisole+5.4 g propylene glycol, B: 0.3 g butylated hydroxyl toluene  $+4.0$  g tween 20), 3 mL of thiobarbituric acid solution, and 17 mL of TA–HCl solution (25 g trichloroacetic acid, 0.6 N HCl 60 mL, QS to 1000 mL with doubly distilled water) were added. The obtained mixture was thoroughly vortexed and incubated in a 100 °C water bath for 30 min to induce a color change. The sample was then cooled in ice water for approximately 10 min. Next, 5 mL of supernatant was transferred into a 10 mL glass tube, 2 mL of chloroform was then added, and the sample was centrifuged for 15 min at 5000 rpm. The absorbance was measured at 532 nm. The TBARS values were calculated in triplicate for every sample using the following formula:

$$
TBARS = \{(absorbance sample - absorbance blank) * 4\} / \{sample weight(g) * 5\}
$$

#### **Statistical analysis**

All tests were performed in triplicate unless otherwise stated. Data evaluations were performed using the Statistical Analysis System (SAS) software version 9.4 (SAS Inst., Cary, N.C., U.S.A.). Diferences were identifed using analysis of variance. Pairwise diferences were identifed using Duncan's multiple range test and were considered signifcant at the 95% confidence level ( $P \le 0.05$ ).

# **Results and discussion**

#### **Oxygen scavenging mechanism of LDPE/PSN flms**

The oxygen scavenging reaction in the present study involves oxygen free radicals produced by transition metal (cobalt chloride). Free radicals of oxygen result from the non-enzymatic reaction of oxygen with transition metals [\[9\]](#page-7-17). The oxygen scavenging reaction for the prepared LDPE/PSN flms is provided below.

 $O_2$  + RH  $\rightarrow$  ROOH  $Co<sup>2</sup> + ROOH \rightarrow Co<sup>3</sup> + OH<sup>-</sup> + RO$  $Co<sup>3</sup> + ROOH \rightarrow Co<sup>2+</sup> + H<sup>+</sup> + ROO$ 

In the reaction, "RH" is a segment of the *trans*-polyisoprene molecule capable of facilitating the oxidation process. Typically, the allylic carbon and hydrogen bonds would be the bonds most susceptible to oxidative degradation due to their lower (85 kcal/mol) bond energy as compared to that of the tertiary carbon and hydrogen bonds energies in saturated hydrocarbons (90–103 kcal/mol)and the vinylic carbon (105 kcal/mol). There are also other possible sources of oxygen uptake such as oxidation of the double bonds to produce epoxides and the development of carboxylic acid (R–COOH) from aldehydes (–CHO). It is believed that increasing the amount of cobalt chloride will increase the rate of free radical generation. Bauman and Maron [\[3\]](#page-7-18) observed that an induction time followed by acceleration is typical of an auto-oxidation reaction, as reactions involving chain transfer typically follow photo initiation as part of an autocatalytic reaction.

#### **Oxygen scavenging capacity and rate**

(3)

Figure [2](#page-4-0) details the oxygen scavenging capacities of the developed LDPE/PSN flm with varying amounts of PSN. During the 8 days storage period, the amount of oxygen in the headspace of the glass vial holding the LDPE/PSN flms decreased in all samples. The LDPE/PSN 20% sample exhibited the highest oxygen scavenging activity compared to that of the other samples. Specifcally, the oxygen



<span id="page-4-0"></span>Fig. 2 Oxygen scavenging capacities of the LDPE/PSN films possessing varying amounts of PSN

<span id="page-4-1"></span>**Table 2** Oxygen scavenging capability of LDPE/PSN flms

Sample	Weight of film sample $(g)$	Efficiency (mL $O_2/gm^*$ day)	Capacity (mL O <sub>2</sub> /g)
Pure LDPE		0.00	0.00
LDPE/PSN 5%		0.84	6.72
LDPE/PSN 10%	4	1.70	13.6
LDPE/PSN 20%		2.09	16.72

content decreased from  $21.1\%$  (v/v) to  $15.53\%$  (v/v) after 8 days. With decreasing amounts of PSN in the LDPE flm, the oxygen scavenging activity also decreased. The oxygen scavenging rates and capacities of the LDPE/PSN flms are presented in Table [2](#page-4-1). The LDPE/PSN 20% flm exhibited the highest oxygen scavenging capacity (16.72 mL of  $O_2/g$ ) and oxygen scavenging rate  $(2.09 \text{ mL of } O_2/\text{g per day})$ . All samples showed acceptable oxygen scavenging capabilities and rates, which is necessary for their use as oxygen scavengers in food packaging applications [\[10](#page-7-15)]. Our results demonstrate that the prepared oxygen scavenging LDPE/PSN flms with 10 and 20% PSN can be used as efective oxygen scavenging materials for packaging dry and moist food products.

#### **Color of roasted peanuts**

Whiteness or lightness  $(L)$ , "*a*" and "*b*" values, were signifcantly diferent across all three samples during storage at  $25 \pm 1$  °C for 90 days ( $p > 0.05$ ) as shown in Table [3.](#page-4-2) The peanuts packed in LDPE/PSN 20% packages were darker than peanuts packaged in neat LDPE and LDPE/PSN 10%. Roasted peanuts packaged in LDPE/PSN 20% were gener ally more reddish than samples packaged in neat LDPE during storage (Fig. [3\)](#page-5-0). The reddish color of roasted peanuts



Values are mean  $\pm$  standard deviation Values are mean±standard deviation

<span id="page-4-2"></span>20%



<span id="page-5-0"></span>Fig. 3 The effects of the oxygen scavenging packaging on the whiteness (L value), a, and b values of the roasted peanuts assessed during storage

may be attributed to red pigment formed during the Maillard browning reaction among free radicals of polyisoprene, a reaction that occurs when polyisoprene reacts with oxygen in the packages and scavenges free radicals. Redness (*a*)



<span id="page-5-1"></span>**Fig. 4** Efect of low-density polyethylene/trans polyisoprene flms on the peroxide value (PV) value of roasted peanuts during storage at  $25 \pm 1$  °C

values of samples packaged in neat LDPE and LDPE/PSN 10% were signifcantly decreased (*p*>0.05) during storage, and this is may be associated with the lipid oxidation process in the peanuts due to the presence of sufficient oxygen within the package to allow for the oxidation reaction [\[17](#page-7-19)]. Overall, the peanuts packaged in LDPE/PSN 20% packages were slightly redder than the other types of peanuts during storage, but this did not detract from their appearance. Our results are in agreement with the results reported by Min and Krochta [[15](#page-7-9)], who evaluated roasted peanuts coated with ascorbic acid in whey protein flm coatings to control lipid oxidation.

## **Peroxide value**

The peroxide value of roasted peanuts has been evaluated as an indicator of the development of hydroperoxides, which are major products of primary lipid oxidation [\[10](#page-7-15)]. Figure [4](#page-5-1) illustrates the changes in peroxide values of the roasted peanuts stored with neat LDPE and LDPE/PSN flms at  $25 \pm 1$  °C. The peanuts packaged in neat LDPE exhibited an initial value of  $0.21 \pm 0.15$  meqO<sub>2</sub> /kg, and a steady increase was noted during storage. After the 30 days of storage, there was a significant increase to  $12.743 \pm 0.53$  and up to  $27.13 \pm 2.25$  meqO<sub>2</sub> /kg at 90 days of storage. In contrast, the peanuts packaged in LDPE/PSN 20% exhibited an initial value of  $0.21 \pm 0.15$  meqO<sub>2</sub> /kg on day one, and this value increased to  $8.21 \pm 0.14$  meqO<sub>2</sub> /kg by day 30. The peroxide value of oils after 90 days of storage was  $17.51 \pm 0.11$  and  $15.28 \pm 0.01$  meqO<sub>2</sub> /kg, respectively, for samples LDPE/ PSN 10 and 20% stored at  $25 \pm 1$  °C. As shown in Fig. [5](#page-6-0), the roasted peanuts packaged with LDPE/PSN with 20% *trans* polyisoprene displayed better stability compared to that of samples packaged in neat LDPE and LDPE/PSN 10%. This



<span id="page-6-0"></span>**Fig. 5** TBARS (thiobarbituric acid reactive substances) values (μg 1, 1, 3, 3-tetraethoxypropane/kg oil) of roasted peanuts stored in packaging with or without low-density polyethylene/trans polyisoprene films at  $25 \pm 1$  °C for 90 days

could be explained by the free oxygen passage in the neat LDPE package, as the presence of oxygen within the headspace of packages can increase lipid oxidation in roasted peanuts, ultimately afecting the fatty acid composition of the lipids [[5\]](#page-7-20). In the case of peanuts, oxygen molecules contribute to the production of oxidative degradation products from their precursor fatty acids [[18](#page-7-21)]. Our results are in agreement with Darko et al.  $[5]$  $[5]$ , who evaluated the effects of oxygen absorbing packaging and pre-storage treatments on the production of afatoxin and oxidation in roasted peanuts stored under controlled conditions.

#### **TBARS assays**

TBARS analyses are used to determine the amount of malondialdehyde, a key secondary by-product generates by aldehydes and ketones during the oxidation reaction, in a given substance  $[10]$ . The effect of the prepared oxygen scavenging LDPE/PSN flms on the TBA assays of roasted peanuts during a 90-day storage period at  $25 \pm 1$  °C was refected by the TBARS values, which provide an index of lipid peroxidation and are presented in Fig. [4.](#page-5-1) The TBARS values of the sample packed with LDPE/PSN 20% were signifcantly lower than those of samples packaged with neat LDPE flm during storage, and this indicates a signifcant reduction in lipid oxidation in roasted peanuts caused by the LDPE/PSN oxygen scavenging flm. These results can be attributed to the small amount of oxygen present in the LDPE/PSN 20% package due to the oxygen scavenging capability of the flm. The oxygen scavenging activities of *trans*-polyisoprene have been attributed to various mechanisms, including inhibition of radical chain initiation due to lack of oxygen, binding of transition metal ion catalysts, and free radical interaction to prevent lipid oxidation. Gaikwad et al. [\[9\]](#page-7-17) reported that active polyvinyl alcohol (PVA) flms incorporated with apple pomace exhibited a high free radical scavenging capacity. This activity increased with increasing amounts of apple pomace, and these flms exhibited oxidative stability even at high temperature. In general, lipids are extremely sensitive to oxygen. Several previous studies indicate that efective control of lipid oxidation by oxygen scavengers can pro-long the shelf-life of food products [[10](#page-7-15), [18\]](#page-7-21). These findings suggest that lipid oxidation in roasted peanuts could be decreased by using LDPE/PSN 20% flms due to the confrmed oxygen scavenging capacity of these flms.

### **Conclusion**

In present study, UV-initiated active oxygen scavenging LDPE flms containing *trans* polyisoprene were created using a cast extrusion process, and they were evaluated in terms of oxygen scavenging performance. LDPE with 10% and 20% *trans* polyisoprene flms exhibited the highest oxygen scavenging capacity upon UV initiation. Prepared LDPE/PSN flms were utilized as an active packaging material for roasted peanuts. The qualitative aspects of roasted peanuts, such as color, TBARS, peroxide value, and p Ansidien value, were within the acceptable range even after 90 days of storage in the LDPE/PSN packaging. The TBARS and peroxide values were signifcantly lower in roasted peanuts packed with LDPE/PSN 20% than those of peanuts packed in LDPE/PSN 10% or those of control samples up to 90 days of storage  $(P < 0.05)$ . Decreased TBARS and peroxide values in roasted peanuts appear to be related to the low oxygen concentration found in the headspace of the LDPE/PSN package. The samples packaged with LDPE/PSN flm maintained their red color during storage. The active LDPE/PSN oxygen-scavenging flms created and examined in this study exhibit the potential for application in active food packaging. The present results suggest that the developed LDPE/PSN flms should be exposed to UV light during packaging prior to sealing food packages. We recommend that the developed LDPE with *trans* polyisoprene flms be used for dry food or low water activity food packaging applications.

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## **Compliance with ethical standards**

**Conflicts of interest** All authors declare that they have no confict of interest.

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