

# Modification of Linear Low-Density Polyethylene Film Using Oxygen Scavengers for Its Application in Storage of Bun and Bread

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**ABSTRACT:** Linear low-density polyethylene (LLDPE) film was modified by blending with zinc, iron, or ascorbic acid as oxygen scavenger. The modified films were characterized for their physicochemical properties and their application for oxygen scavenging. There was no significant change in the physicochemical properties. The oxygen scavenging property of the films was studied by shelf-life studies of bun and bread. The shelf life studies were conducted using the modified films and LLDPE film as a control. The sensory analysis showed good taste and texture of bun and bread till the fifth day. The control

sample was not acceptable even on second day. This was supported by instrumental texture analysis. There was no significant change in force required to compress the sample in case of packed scavenging film while the control sample showed significant change. The moisture analysis also showed similar results. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 122: 55–63, 2011

**Key words:** extrusion; gas permeation; FTIR; polyethylene; barrier; water vapor transmission rate; shelf life

## INTRODUCTION

Packaging is one of the most important processes to maintain the quality of food products during storage and transportation as well as for end-use.<sup>1</sup> The four basic functions of packaging are protection, containment, information, and convenience. The quality of the most food products deteriorate because of mass transfer phenomena such as moisture absorption, oxygen invasion, flavor loss, undesirable odor absorption, and migration of packaging components into the food.<sup>2</sup> These phenomena can occur between the food product and the surrounding, between the food and the packaging material, or among the heterogeneous ingredients of the food product.<sup>3</sup>

The oxygen may be present in the package at the time of sealing or it may enter the pack by permeation or leakage during the storage period. The impact of oxygen on food quality, and ultimately shelf life, is dependent not only upon the quantity of oxygen available for chemical oxidation or support of growth of organisms but also upon the rate of the reactions with which oxygen is consumed. This, in turn, will be influenced by the solubility of the gas

in the medium provided by the food or beverage. The oxidation of fat in potato crisps is highly dependent upon water activity, with a minimum rate at  $a_w$  0.3–0.4, and the increase in reaction rate above this value is interpreted in terms of the formation of an aqueous multilayer on the food with consequent dissolution of oxygen and enhancement of the oxidation of food constituents.<sup>4</sup> Much of the trade literature on oxygen scavenging packaging presents a focus on the quantity of oxygen in a package without consideration of the widely different rates at which food quality can be degraded.<sup>5</sup> The quantities of oxygen lie in a range from a few ppm to a few hundred ppm based on the weight of the food. Removal of oxygen by conventional means is not generally achievable if the food or beverage has components that react rapidly. Beer is one of the most studied beverages, and it has been found that an uptake of around 1 ppm (or a little more) results in the beer reaching its shelf life. A conventional bottle closed with a crown seal allows an uptake or around 750–2000 ppb of oxygen over a period of 3–8 months.<sup>6</sup>

Lot of research work on sachet-based oxygen scavenger were done by Brody et al. (2001),<sup>7</sup> Tallgren (1938),<sup>8</sup> Buchner (1968),<sup>9</sup> the Mitsubishi Gas Chemical (the range of Ageless<sup>TM</sup>) sachets, Multisorb Technologies (FreshMax<sup>TM</sup>/FreshCard<sup>TM</sup>), Yoshikawa et al. (1977).<sup>10</sup> Although the performance of oxygen-absorbing sachets was quite satisfactory for a wide

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range of food storage conditions, a number of limitations to their use in practice were observed. The esthetics of inserts, coupled with a concern about possible ingestion or rupture as well as their unsuitability for use with beverages drove researchers to seek package-based solutions. The approach of using the packaging material as a medium for the oxygen-scavenging chemistry was developed independently in several laboratories and countries. Not surprisingly, the reactions were initially the same as used in the sachet technologies, but eventually it was recognized that the restrictions applying to package inserts need not apply to the package. This has allowed a multiplicity of oxygen access problems, arising from quite disparate packaging factors, to be addressed, thus permitting targeting of problems at their source rather than waiting for the oxygen to enter the package to be absorbed by an insert such as a sachet.

There was the first multilayer plastic structure in which antioxidants were claimed to function as an oxygen "getters." According to Brody et al. (2001),<sup>7</sup> the process involved dispersing very minor portions of conventional antioxidants in or between layers in a multilayer. The first plastic to incorporate dissolved reagents with known oxidation chemistry involved the light-energized excitation of oxygen diffusing into the plastic.<sup>11</sup> The substrate for oxidation did not react with ground-state oxygen, so the oxygen to be scavenged had to be excited to the singlet state. This was achieved by including a photosensitizing dye and exposing the scavenger film to visible light. The process occurred only when the scavenger film was exposed to the light. It was shown that ascorbic acid could perform as a singlet oxygen acceptor and that the photochemistry imposed limits on the scavenging rate. The use of one polymer as both the reagent and the reaction medium was investigated using natural rubber.<sup>12</sup> This work was extended to other rubbers, which displayed different inherent reactivities with singlet oxygen while having similar values of oxygen permeability. The process was applied in a poly(furyloxirane) designed to have even higher reactivity towards singlet oxygen.<sup>13</sup> An approach to create a polymeric film with total barrier to oxygen permeation based on the transition-metal-catalyzed oxidation of aromatic nylons was developed by the Carnaud Metal Box Company under the trade name Oxbar<sup>TM</sup>.<sup>14</sup> The key advantage initially seen for such a process was the ability to blend the polymer plus catalyst with PET in the manufacture of bottles for wine and beer.

Based on some oxidation chemical reactions of zinc and iron with ascorbic acid, incorporation of these reactive chemicals in linear low-density polyethylene (LLDPE) film was aimed. Some of the bakery products like bun and bread having short shelf-life and sensitivity to oxygen and moisture

were selected for the study, using oxygen scavenging films.

## MATERIALS AND METHODS

### Development of oxygen scavenging films

#### Materials

LLDPE resins from Reliance Industries, (Mumbai, India). Density = 0.924 g/cc; MFI = 6.5 g/10 min.

- i. Iron powder: molecular weight 55.85, minimum assay 95.0%, mesh size (300 mesh) min. 95.0%. manufacturer S.D. fine chemicals limited, Mumbai.
- ii. Zinc powder: molecular weight 65.38, minimum assay 95.0%, and mesh size 90% (minimum) passes through 300 mesh. Qualigens fine Chemicals, Mumbai.
- iii. Ascorbic acid: AR grade, manufacturer Merck Chemicals Pvt., Mumbai.

#### Instruments

Extruder: single screw extruder Lx-30, Model HM 300 (Klockner Windsor India, Chennai) with operational conditions L/D ratio: 26 : 1, Dig diameter: 25 mm, screw diameter: 30 mm, and plasticizing capacity: 8 kg/h.

#### Method

On the basis of literature survey and initial experiments, following chemical combinations are selected: ascorbic acid + Iron powder and ascorbic acid + zinc powder.

#### Preparation of master batch

The master batches of these chemicals were made for easier extrusion and mixing. The concentration of these master batches was taken in the range of 10–20%. The final batch predecided concentration was prepared by using their respective master batches.

#### Extrusion and palletizing

The extruded strands from the die of 2 mm diameter with zone temperatures from 150°C to 160°C, die zone 170°C, and screw speed of 30 rpm, were subjected to water at room temperature. The wet strands were dried in a hot blower and passed through the pelletizer. The speed of the pelletizer was synchronized with the speed of the extruder. The final product was in the form of uniform pellets.

**TABLE I**  
**Physicomechanical Properties of Packaging films C-LLDPE Film**

S. No	Test	Specification	C	2A	3A
1	Thickness ( $\mu\text{m}$ )		$99.5 \pm 0.2$	$101.7 \pm 0.8$	$101.5 \pm 0.2$
2	Tensile strength ( $\text{N}/\text{mm}^2$ )	ASTM D882(08.01)			
	Machine direction		$21.9 \pm 0.6$	$20.8 \pm 0.5$	$20.3 \pm 0.2$
	Cross direction		$19.3 \pm 0.2$	$18.6 \pm 0.4$	$18.4 \pm 0.1$
3	Heat seal strength ( $\text{N}/\text{mm}^2$ )	ASTM D882(08.01)			
	Machine direction		$19.3 \pm 0.19$	$17.8 \pm 0.46$	$19.14 \pm 0.09$
	Cross direction		$18.0 \pm 0.02$	$17.2 \pm 0.65$	$16.89 \pm 0.80$
4	Impact strength (k Pa)	ASTM D3998(08.03)	$15.0 \pm 0.7$	$15.3 \pm 0.4$	$15.1 \pm 0.3$
5	Tear strength ( $\text{N}/\text{cm}$ )	ASTM D1922(08.02)	$7.2 \pm 0.2$	$5.4 \pm 0.3$	$5.9 \pm 0.6$
6	WVTR ( $\text{g}/\text{m}^2$ 100 guage/day)	ASTM E 96	$20.0 \pm 0.8$	$17.2 \pm 0.4$	$17.4 \pm 0.5$
7	OTR ( $\text{cc}/\text{m}^2\text{day}/\text{atm}/65\%\text{RH},27^\circ\text{C}$ )	ASTM D 3985	$18,252 \pm 0.1$	$15,107 \pm 0.1$	$16,465 \pm 0.1$

2A, ascorbic acid and iron incorporated LLDPE film; 3A, ascorbic acid and zinc incorporated LLDPE film.

### Film blowing

The extruder was fitted with a cross-head annular die for film blowing operations using extruder conditions similar to pellet extrusion. The homogenous film was extruded in the form of tube and sealed at one end and pulled between the nip rollers 1 : 2.5 m/min which were placed above the collapsing frame. As the film was being pulled, air was pumped into the tube. The air was maintained at the same level as there was no escape of the air from the bubble once it reached the nip rollers. The thickness of the film was adjusted by tightening/loosening the spider leg screws. The thickness of the film was measured at frequent intervals and the necessary correction was made. The lay flat width was noted down and the film was wound up on a winding station.

### Physicomechanical properties

Thickness, tensile strength, heat seal strength, impact strength, tear strength, water vapor transmission rate (WVTR), and oxygen transmission rate (OTR) of the film were determined as per the standard procedures shown in the Table I.

#### Thickness

Thicknesses of oxygen scavenging film as measured in this method was defined as the perpendicular distance between the two principle surfaces of film under specific condition. The thickness of film was measured at various points using micrometer MI20, Messmer Instruments (Gravesend, Kent, UK). The tip of the micro meter was cleaned before measuring the thickness of the samples to avoid errors. Five measurements were made and the average value was reported.

#### Tensile strength

Tensile strength was carried out using Instron (LR 5K) LLOYD, UK with load cell 100 kgf/1 kN as per

ASTM 882(08.01). The tests were carried out with initial grip separation of 50 mm and crosshead speed of 200 mm/min. Tensile strength was calculated by dividing the maximum load for breaking film by cross-sectional area, and the values were measured both in machine direction and cross-direction, at an ambient temperature of  $28 \pm 2^\circ\text{C}$  to observe whether any difference in the orientation of polymer chain occurred. Five measurements were made and the average value was reported.

#### Heat seal strength

Heat seal strength is the measure of the force required to pull apart the pieces of film that have been sealed together. The heat seal strength was conducted by using T peel specimen on an Instron (LR 5K) with load cell, 100 kgf/1KN. Tests were performed on both machine and cross direction as per ASTM D882(08.02).<sup>15</sup> The two strips of same plastic films ( $6.25 \text{ cm} \times 2.5 \text{ cm}$ ) were sealed together using a HP impulse sealer (Sunray Industries, Mysore, India) at  $8.4 \text{ kgf}/\text{cm}^2$  pressure for 14 s to get a standard seal width of 10 mm. The free ends of the sample were mounted on two grips of the tensile testing machine and the movable jaw driven at a constant rate of 200 mm/min, breaking the sealed area apart. The load required to break the seal of the sample was noted. The seal strength was calculated by dividing the load at break by the seal width.

#### Impact strength

Impact strength was performed using Elmendorf tearing and impact tester (Thwing Albert Instrument Co. Philadelphia) as per ASTM D3998 (08.03) at  $27^\circ\text{C}$  and a relative humidity of 65%. The sample was fixed between the two grips. When the pendulum was released, the ball stroked the specimen and broke the specimen. The energy required was measured on the scale attached to the pendulum by

means of a pointer. The reading obtained directly gave the impact strength in terms of grams per mil of thickness and converted to kPa.

#### Tear strength

Tear strength is defined as the measure of energy absorbed by the specimen in propagating a tear that has already been initiated by cutting a small nick in the sample. Tear strength was carried out according to ASTM D1922(08.02), using Instron with load cell 100 kgf/1 KN. The sample was fixed in the grips and a crack initiated by the machine. When the pendulum was released, it swung down and tore the specimen. The energy required to complete this tear was measured on the scale attached to the pendulum by means of a pointer. The reading was noted down from the calibrated scale. Tear strength of packaging films was expressed in gram-force, which was converted to N/cm.

#### WVTR

WVTR was determined using aluminium dishes as per ASTM E-96. About 50 cm<sup>2</sup> diameter samples were sealed on a cup containing highly hygroscopic material like anhydrous CaCl<sub>2</sub>. The specimen was then placed on the cup and sealed all-round leaving 50-cm<sup>2</sup> surface area in a circular form for the exposure using hot wax. The cups with the wax were then rested for few minutes to reach room temperature. The prepared cups were weighed and placed in the humidity chamber maintained at 38°C and 90% RH gradient. Consequently, there was an increase in weight because of absorption of moisture by CaCl<sub>2</sub> permeated through the film. Weight gain was plotted against time and linear least-square method used to calculate WVTR as per the following equation.

$$\text{WVTR} = \text{Slope (wt gain/day)}/\text{Film area (m}^2\text{)}$$

#### OTR

OTR of the films was measured volumetrically using Permeability cell, Model CS-135:319, Custom Scientific Instruments, NJ with ASTM D 3985. The test makes use of permeability cell consisting of two stainless steel disks that form cylindrical cavity when disks are superimposed. The film to be tested was clamped between the two disks using six equally spaced bolts after placing filter on the upper disks (as support) and a rubber gasket to ensure a pressure tight fit. The cell consisted of a glass capillary in a vertical position to an opening in the center of the upper disk. Suitable gas inlet and vent lines were provided on both sides of the cells. Oxygen

was supplied from surge tank at a constant pressure to the bottom of the cell. A short plug of mercury, contained in a capillary, was displaced upward by the permeating gas, and this displacement gave the rate of permeation of the gas through the packing material. An electromechanical vibrator was used to avoid friction to the movement of the plug. The change in volume of permeate was measured as a function of time. The displacement of mercury versus time was plotted, and slope of the straight line was obtained. The gas transmission rate (GTR) was calculated using the following formula:

$$\text{GTR} = 34,029 \times \text{slope}/\text{Pressure} \\ (\text{cc/m}^2/\text{day atmosphere})$$

where 34,029 is the capillary constant.

#### Fourier transform infrared spectroscopy

Thin films with uniform thickness were used for obtaining the IR spectra using Fourier transform infrared spectroscopy (FTIR)-Raman-Nicolet 5700 to analyze the presence of oxygen scavenging chemicals in the extruded film. All measurements were carried out at 20°C in anhydrous conditions with air as the background. For each sample, 32 scans at a 2-cm<sup>-1</sup> resolution were collected in the range of 4000–400 cm<sup>-1</sup>. The spectra were then analyzed with a curve-resolving technique based on a linear least square analysis to fit a combination of Lorentzian and Gaussian curve shapes.

#### Shelf life studies of bun and bread

The pouches were prepared by using ascorbic acid + iron (2A), ascorbic acid + zinc (3A) LLDPE film combinations of oxygen scavenging films with a control of LLDPE film (C).

Freshly prepared bun and bread were procured from a local market.

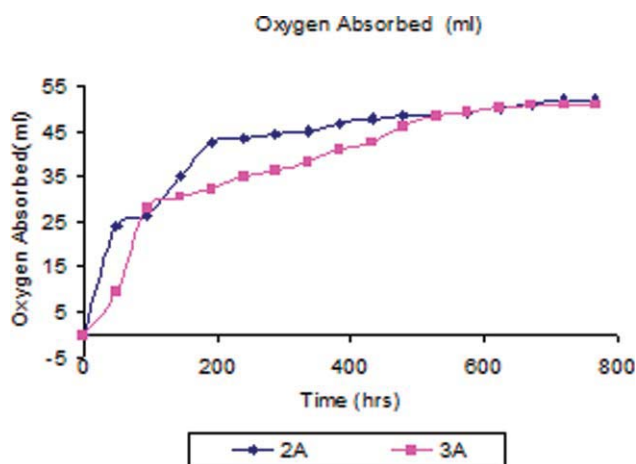
Bun: weight = 55.5 ± 0.5 g, volume = 200 ± 10 cc, height = 50 ± 10 mm, moisture content = 28 ± 3%.

Bread: weight = 400 ± 5 g, volume = 1400 ± 17.5 cc, moisture content = 28 ± 3%, slices used for the experiment, weight = 15.5 ± 0.5 g, height = 10.5 ± 0.5 mm.

Seventy pouches per combinations were made, and the bun and bread were packed. The four sides of each pouch were sealed by using heat sealing machine. The samples were stored at room temperature.

#### Sensory analysis

Sensory analysis was carried out with a score of 5–3 with acceptable conditions and 2–1 with poor acceptability for softness and taste of bread/bun.



**Figure 1** Oxygen absorbed versus time [ascorbic acid + iron (2A) and ascorbic acid + zinc (3A)]. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

### Texture analysis

The texture analysis of bun/bread was carried out by using a texture analyzer-TA-HDi (Stable Microsystem Surrey, UK). The sample was placed on the platform of stable micro system and was comprised up to 80% of its original height by using a probe of 100 mm diameter at a cross head speed of 100 mm/min. The force used to compress the sample was recorded. The samples were tested in quadruplicate, and the average height of the sample, average force required to compress the sample, and the standard deviation were calculated.

### Moisture analysis

Moisture analysis of bread/bun was carried out by using AACC method 44.15A. The samples of bun and bread weigh to  $\pm 0.2$  g and placed on the large sheet of smooth paper and cut into slices 2–3 mm thickness. The cut slices were dried on paper at room temperature until they were in approximate equilibrium with moisture of air usually 15–20 h would suffice. Dried slices and crumbs were weighed and percent moisture lost on air drying were calculated (A). The air dried bread and bun along with crumbs in were oven dried at 130°C for 1 h. The samples were cooled by using desiccators and the percentage of moisture loss in second stage were calculated (B). The total moisture was calculated by following equation.

$$\% \text{ Total moisture} = [A + (100 - A) \times B]/100$$

where A = % of moisture loss on air drying; B = % of moisture loss as determined by oven drying.

### Microbiological analysis

The samples along with appropriate control were analyzed for total bacterial count and yeast and mold count. Samples analyzed included bread and bun packed in oxygen scavenging films and in plane LLDPE including those samples kept without any packing materials.

Ten grams of sample was mixed with 90 mL of sterile saline ( $10^{-1}$  dilution). One milliliter of this mixture was transferred into 9 mL saline ( $10^{-2}$  dilution). One milliliter from each dilution was pour plated with plate count agar in duplicates, and 0.1 mL of the dilution was spread plated on to YMA (Yeast and mold agar plate). PCA plates were incubated at 37°C for 3 days and colonies were counted and recorded.

## RESULTS AND DISCUSSION

### Oxygen scavenging ability of the films

Oxygen scavenging ability of extruded films was measured in Portmap 2 Systech Instruments, Oxfordshire, UK is shown in Figure 1. Ascorbic acid and iron combination has absorbed 47.6 mL of oxygen and ascorbic acid and zinc combination has absorbed 37.4 mL in 750 h.

Based on the results, it is evident that the combinations of the iron + ascorbic acid and zinc + ascorbic acid have got good oxygen scavenging ability. But the degree of scavenging ability was found to vary with different combinations.

### Physicomechanical properties of oxygen scavenging films

#### Thickness

The thickness of film was measured at various points on film and the average standard deviations were taken. Table I shows the thickness measurement of various oxygen scavenging films. From the above table, the thickness of 2A [ascorbic acid + iron + LLDPE] and 3A [ascorbic acid + zinc + LLDPE] were found to be  $102 \pm 5$   $\mu\text{m}$ . Whereas the thickness of 100% LLDPE was found to be  $100 \pm 5$   $\mu\text{m}$ .

#### Tensile strength

Tensile strength was carried out using Instron (LR 5K) LLOYD, UK with load cell 100 kgf/kN. From Table I, it is evident that low tensile strength was noticed for cross direction compared to machine direction. It is also observed that in the machine direction, the tensile strength is low for 2A, 3A compared to LLDPE. In cross direction, the tensile strength of 3A was less and more for 2A compared to LLDPE. This was due to the disturbance of

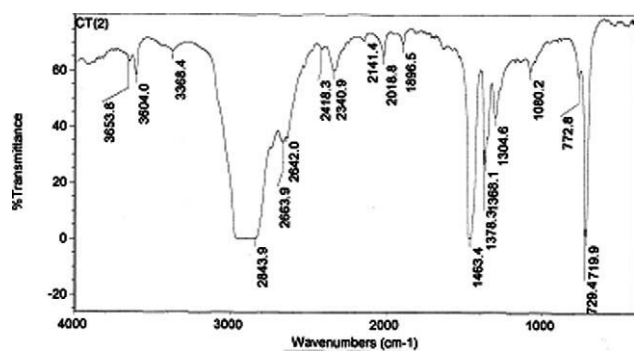


Figure 2 FTIR of virgin LLDPE film control (C).

crystallization nature by the addition of additives into pure LLDPE.

#### Heat seal strength

The heat seal strength was conducted by using T peel specimen on an Instron (LR 5K) with load cell 100 kgf/1KN, LLOYD, UK. Both machine direction as well as cross direction tests were performed. It was found that the heat seal strength in machine direction was good for LLDPE compared to 2A and 3A. In cross direction, seal strength of almost all the scavengers (containing additives) decreases.

#### Impact strength

Impact strength was performed using Elmendorf tearing and impact tester, Thwing Albert Instrument Co. Philadelphia. The results of impact strength are shown in Table I. It was found that there was no significant change in impact strength in oxygen scavenging films in comparison to control LLDPE with the values from 15 to 15.3.

#### Tear strength

From Table I, it is clearly shown that the tear strength of oxygen scavenger filled LLDPE films were low compared to virgin LLDPE. The reason for

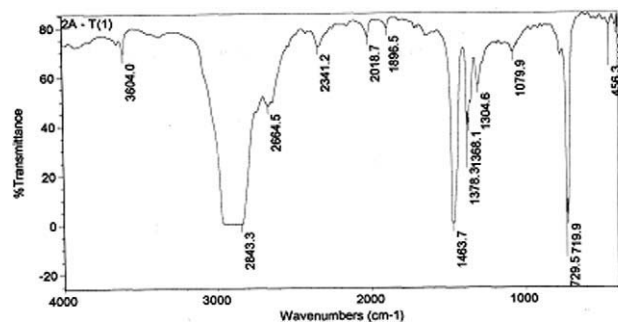


Figure 3 FTIR of ascorbic acid and iron incorporated LLDPE film (2A).

the behavior of oxygen scavenger is the weakening of the intermolecular force because of the addition of metallic scavengers such as iron and zinc.

#### WVTR

From Table I, it is evident that the WVTR of 2A (17.2) and 3A (17.4) were lower than the pure LLDPE (20 g/m<sup>2</sup> 100 guage/day). The difference in WVTR of oxygen scavenging film with pure LLDPE may be because of rearrangement of molecules after combining oxygen scavenger chemical with the pure LLDPE.

#### OTR

OTR of the films was measured using Permeability cell, Model CS-135:319, Custom Scientific Instruments, NJ. From Table I, it is evident that the OTR of pure LLDPE was higher than oxygen scavenger film (2A and 3A). The combination of oxygen scavenger 3A has got less OTR, that is, 15106, whereas LLDPE got OTR of 18252. The less OTR of oxygen scavenger film may be due to rearrangement of molecules because of the addition of oxygen scavenging chemicals into LLDPE.

#### FTIR

FTIR was taken by Nicolet 5700 FTIR, Thermo Electron Corporation. FTIR spectroscopy was used to analyze the presence of oxygen scavenging chemicals in the extruded film. The chemical used for oxygen scavenging were in minute quantity (i.e., ascorbic acid, iron, zinc) compared to main substrate LLDPE (99.6%), and hence difficult to analyze the chemicals by FTIR.

The FTIR spectra obtained for LLDPE control film and oxygen scavenging films are shown in Figures 2–4. FTIR spectrum of LLDPE shows the intensities of two peaks at 2925 and 2843 cm<sup>-1</sup> assigned to the —CH<sub>2</sub>— or —CH<sub>3</sub> asymmetric and symmetric

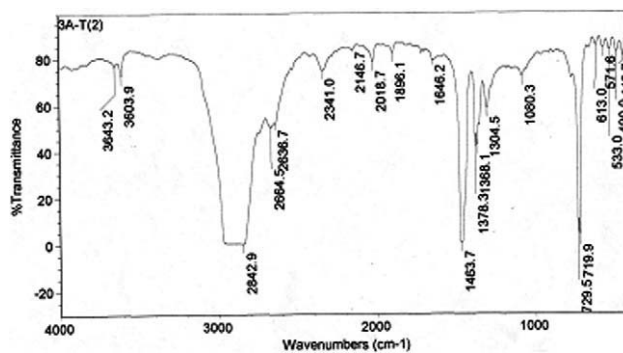


Figure 4 FTIR of ascorbic acid and zinc incorporated LLDPE film (3A).

**TABLE II**  
Sensory Score Card for Bun

S. No	Day	Bun							
		Softness				Taste			
		C	D	2A	3A	C	D	2A	3A
1	First day	4	4	4	4	4	4	4	4
2	Second day	4	3	4	4	3	3	4	4
3	Third day	3	1	4	4	1	1	4	4
4	Fourth day	1	1	3	3	1	1	4	4
5	Fifth day	1	1	3	3	1	1	3	3
6	Sixth day	1	1	3	3	1	1	3	3

C, product packed in LLDPE film; D, product without any packaging; 2A, product packed in ascorbic acid and iron incorporated LLDPE film; 3A, product packed in ascorbic acid and zinc incorporated LLDPE film.

Score key: poor, 1; fair, 2; good, 3; better, 4; best, 5.

stretching vibration and the two peaks at 1463 and 1378  $\text{cm}^{-1}$  assigned to asymmetric deformation vibration of  $-\text{CH}_2-$  and  $-\text{CH}_3$  groups. The absorption peak at 729  $\text{cm}^{-1}$  represents the deformation vibration in  $-(\text{CH}_2)_n$ .

### Shelf life studies

Sensory analysis was carried out by a panel with a score of 5–3 with acceptable conditions and 2–1 with poor acceptability for evaluating the softness and taste of bun/bread. Based on the results presented in Tables II and III for bun and bread, it is clear that bun packed in oxygen scavenging film showed acceptability upto 6 days with respect to softness and taste. Whereas, bread packed in oxygen scavenging film showed its acceptability of 5 days with respect to softness and taste.

### Texture analysis

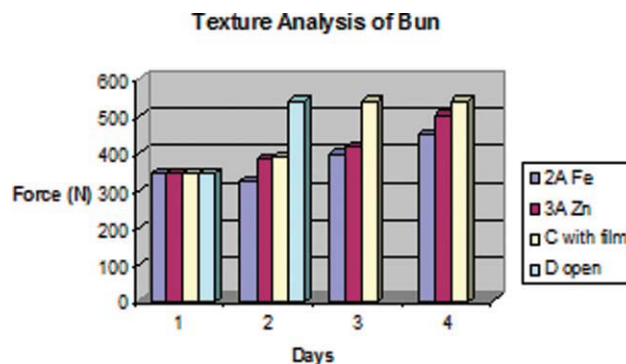
The texture analysis of bun as indicated in Figure 5 was shown marginal increase in scavenging films

**TABLE III**  
Sensory Score Card for Bread

S. No	Day	Bread							
		Softness				Taste			
		C	D	2A	3A	C	D	2A	3A
1	First day	4	4	4	4	4	4	4	4
2	Second day	3	2	4	4	3	2	4	4
3	Third day	1	1	4	4	1	1	4	4
4	Fourth day	1	1	3	3	1	1	3	3
5	Fifth day	1	1	3	3	1	1	3	3
6	Sixth day	1	1	2	2	1	1	2	2

C, product packed in LLDPE film; D, product without any packaging; 2A, product packed in ascorbic acid and iron incorporated LLDPE film; 3A, product packed in ascorbic acid and zinc incorporated LLDPE film.

Score key: poor, 1; fair, 2; good, 3; better, 4; best, 5.



**Figure 5** Texture analysis of bun. C: product packed in LLDPE film; D: product without any packaging; 2A: product packed in ascorbic acid and iron incorporated LLDPE film; 3A: product packed in ascorbic acid and zinc incorporated LLDPE film. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

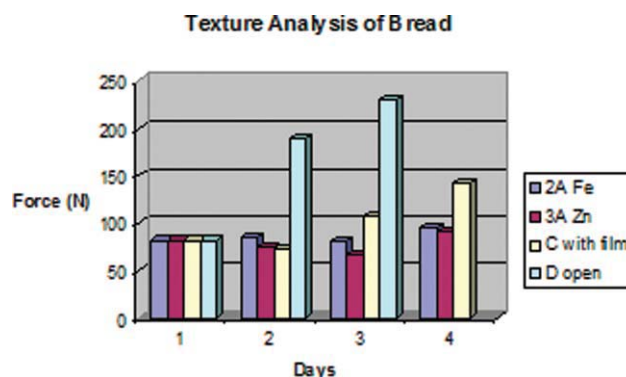
ranging from 29.8% and 45.8% in 2A and 3A, respectively, in 4 days; whereas, the increase in texture was high in control sample with 56.8 in 4 days and 57% in open within 2 days itself.

Similarly, the textural analysis of bread as shown in Figure 6, there was a marginal change of 17% and 11% in 2A and 3A, respectively, and change in C and D with 72% in 4 days and 180% in 3 days, respectively.

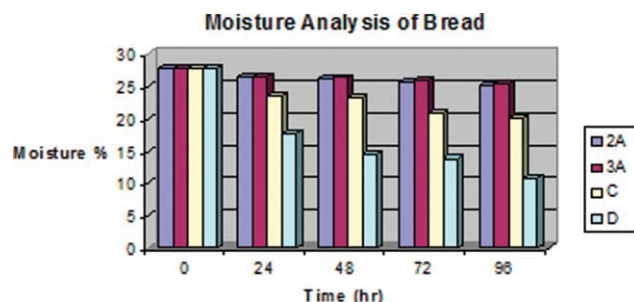
### Moisture analysis

The loss of moisture in bun was marginal of 11% and 6.4% in oxygen scavenger film of 2A and 3A, respectively, in 5 days. Moisture loss of bun in plain polyethylene pouches was 2–3% more than the buns packed in oxygen scavenger films reaching to 21.6% in 5 days. The moisture loss of unpacked buns was very high (42.4%) resulting into its dryness in 5 days Figure 7.

Similarly, it is clear from the above graph, that there was a marginal moisture loss of 9.5% and 8.3%



**Figure 6** Texture analysis of bread. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 7** Moisture analysis of bread. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

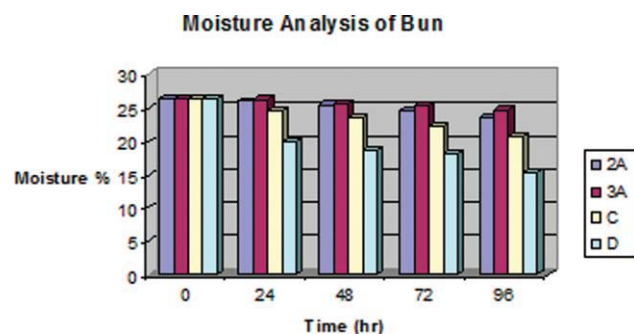
in bread in oxygen scavenger films 2A and 3A, respectively, in 5 days. Whereas the moisture loss in plain polyethylene pouches was three times higher than bread in oxygen scavenger films (27.7%), which was not desirable. Bread sample without any packaging was shown loss of moisture, 61.9% in 5 days resulting into dryness.

Moisture content of bread sample D (without any packing film) after fifth day was calculated as 10.6%, it is very less for a product like bread. Less than 10% of moisture levels in products are generally considered as dehydrated products. The sample packed with LLDPE got a moisture content of 20.1% after the fifth day, this was also lesser than 2A and 3A. The maximum moisture content was found in sample 3A, that is, 25.5% followed by 2A, that is, 25.1% after the fifth day of experiment.

From the above moisture analysis, it was evident that the samples packed in 2A and 3A retained sufficient moisture level because of its less WVTR (Figure 8).

### Microbiological analysis of bun

From the results of microbiological analysis of bun, we can see that CFU/g of TPC in case of 3A is nil, 2A is 10, and is very much lower than control sample C (packed in plain LLDPE) and D (sample with-



**Figure 8** Moisture analysis of bun. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

**TABLE IV**  
Microbiological Analysis of Bun

	TPC/gm (CFU/g)	Yeast (CFU/g)	Mold (CFU/g)
2A	10	–	–
3A	–	–	–
D	70	$1 \times 10^2$	$2 \times 10^2$
C	20	–	–

C, product packed in LLDPE film; D, product without any packaging; 2A, product packed in ascorbic acid and iron incorporated LLDPE film; 3A, product packed in ascorbic acid and zinc incorporated LLDPE film.

out any packing), that is, D is 70 CFU/g and C is 20 CFU/g. Yeast and mold growth were only found in sample D. The level of TPC/g in sample 2A was within the limit (Table IV).

### Microbiological analysis of bread

The results of microbiological analysis of bread indicated that CFU/g of TPC in case of 2A is 41 and 3A is 40 (Table V), which was very much lower than the control sample D and C. That is, D was having  $4 \times 10^2$  TPC/g, and in C, it was  $2.16 \times 10^4$ . As far as the growth of yeast and mold are concerned, only 3A and C got the growth of yeast, that is, 3A was 20 CFU/g and C was  $1.12 \times 10^4$  CFU/g. The mold growth was only observed in the control sample C, that is,  $1.2 \times 10^3$  CFU/g. As per the literature, TPC/g of 2A, 3A, and yeast growth in 3A were within the prescribed limit.

It is evident that the bread/bun packed in oxygen scavenging films has got very minute quantity of microbial growth which is under the prescribed limit. That is, the life of bread packed in oxygen scavenging film was extended from 2 to 5 days; it is basically because of less oxygen availability and control of moisture inside the package.

The field of oxygen scavenging using plastics is still largely under development, even though the use of sachets, labels, and closure liners is well established. The introduction of new technologies will depend upon the drives revealed in the food and packaging industries. Another factor of importance

**TABLE V**  
Microbiological Analysis of Bread

Samples	TPC/gm (CFU/g)	Yeast (CFU/g)	Mold (CFU/g)
2A	41	–	–
3A	40	20	–
D	$4 \times 10^2$	–	–
C	$2.16 \times 10^4$	$1.12 \times 10^4$	$1.2 \times 10^3$

C, product packed in LLDPE film; D, product without any packaging; 2A, product packed in ascorbic acid and iron incorporated LLDPE film; 3A, product packed in ascorbic acid and zinc incorporated LLDPE film.



will be the need to achieve current or better quality levels as packaging is changed, especially when newer materials are used.

Regulation by both food authorities and those caring for the environment will also have major impacts. A variety of scavenging systems have already been approved in Japan, United States, and European countries, among others. The expected amendment of the Food Packaging Directive by the European Commission in 2005 to address active packaging will make the future path. The potential for reduction in the complexity of multilayer plastics structures and the reduction in rigid packaging use following the use of oxygen scavenging may be expected to have favorable outcomes for the environment, as long as there are not adverse impacts in the manufacturing.

### CONCLUSIONS

The study was carried out to find out the possibilities for developing oxygen scavenging films and their use in active packaging system. Some of the good chemical combination was obtained in making the oxygen scavenging films like ascorbic acid and iron and ascorbic acid and zinc.<sup>15–18</sup> The effect of scavenging ability in films is lesser than that of chemicals placed in sachets. By using the films, one can avoid the accidental consumption of oxygen scavenging sachets by the consumer. Oxygen scavenging films with ascorbic acid and iron and ascorbic acid and zinc are found to be effective in extending shelf life of food products like bread and bun. For optimizing the chemical combinations and the conditions for developing oxygen scavenging films needs an in depth study, it was beyond the scope of this investigation work because of the paucity of time.<sup>19–22</sup>

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