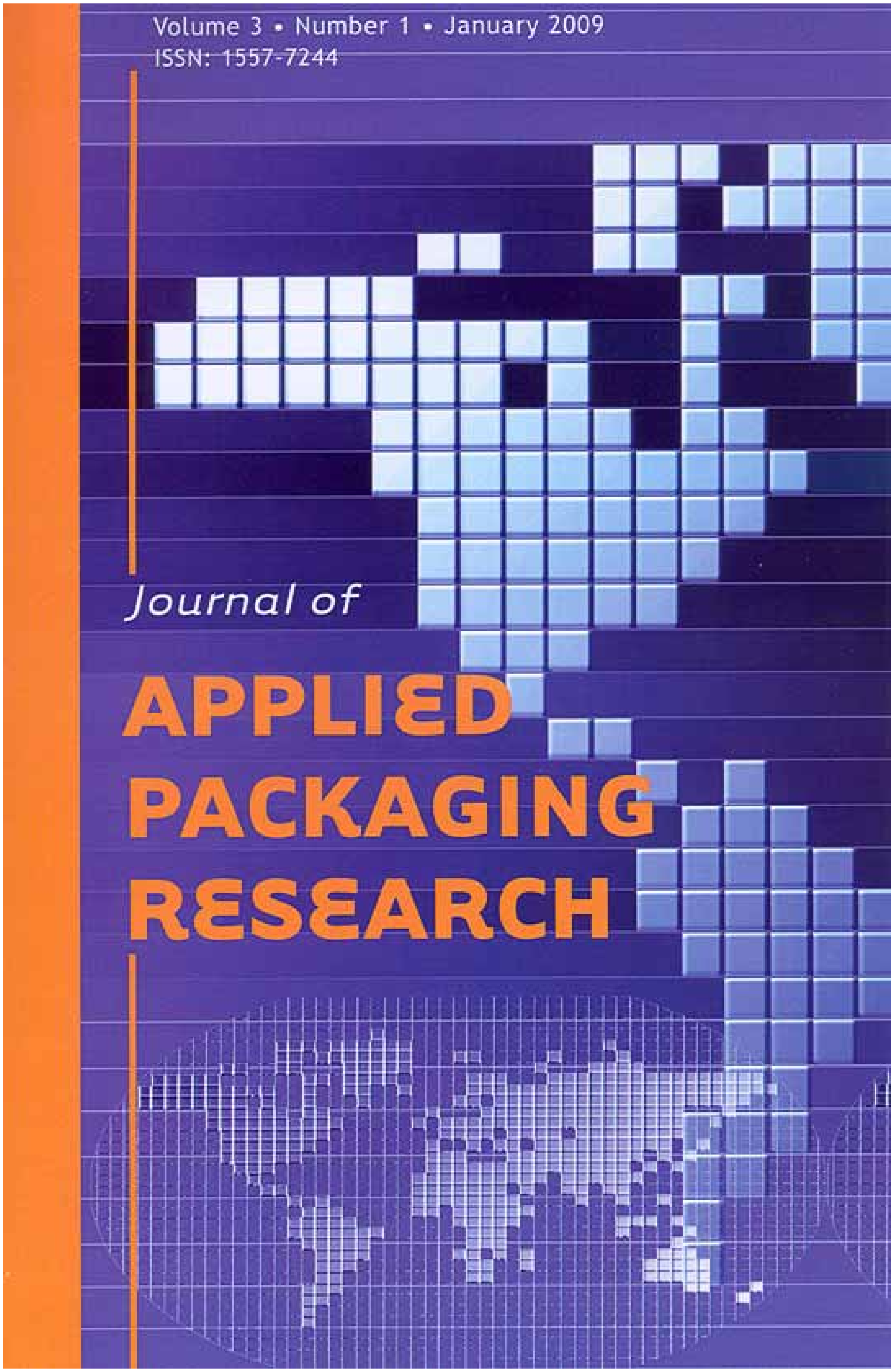

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Aim and Scope

The *Journal of Applied Packaging Research* is an international forum for the dissemination of research papers, review articles, tutorials and news about innovative or emerging technologies for the packaging industry. The journal is targeted towards the broad packaging community including packaging scientists and engineers in industry or academic research and development, food scientists and technologists, materials scientists, mechanical engineers, industrial and systems engineers, toxicologists, analytical chemists, environmental scientists, regulatory officers, and other professionals who are concerned with advances in the development and applications of packaging.

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CONTENTS

Research

Performance of Pre-Cut Lettuce Packaged in Biodegradable Film Formed on Commercial Vertical-Form-Fill-and-Seal Machines

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ABSTRACT: The purpose of this study was to determine the feasibility of using biodegradable films suitable for fresh-cut lettuce with commercial vertical-form-fill-and-seal packaging machines (VVFS) equipped with heat-sealing bars. Biodegradable high-density polyethylene (BHDPE) and polypropylene (BPP) films were tested. Commercial bags of pre-cut Romaine sealed in a polyethylene/oriented polypropylene (PE/OPP) bag formed on a VVFS machine were used as the control. All bags were held at 4.4°C, 80% RH and assessed for reduction in quality during storage per a commercial (in-house) standard utilized by a large pre-cut salad packer in Salinas, California.

When the biodegradable films were sealed with a VFFS machine equipped with a thermal-bar heat sealer, a 52.5% fail rate was observed due to the non-continuity of the seals. Leaks were found when bags were vacuum tested to 14 in Hg absolute for 15 seconds. However, a 45.5% fail rate was also observed for commercial bags made using the same VFFS machine, suggesting similar seal concerns for current industry film structures. Though an attempt was made to only store bags that were sealed properly, bags made from the biodegradable films sealed with the thermal-bar did not perform as well as the commercial packages and the shelf-life of the pre-cut Romaine was shortened. When biodegradable bags were sealed using a bar impulse sealer, hermetic seals were obtained. The Romaine stored in these bags had a similar rate of decay and level of pinking after 14 days storage as Romaine packaged in the commercial PE/OPP bags. These results indicate that the use of commercial impulse sealers, rather than thermal-bar heat sealers, would allow industry to utilize these biodegradable films for pre-cut lettuce mixes.

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INTRODUCTION

FRESH-CUT produce is defined as any fruit or vegetable that has been cut and packaged for consumer use without being cooked or prepared by means of heating (Mayen, 2005). Fresh-cut produce sales have increased in the last 20 years and were $1.9 billion in 2000 (Sexton, 2003). This increase in sales over the last 2 decades has lead to an increase in landfill waste, resource scarcity and consumer [environmental] concerns over the films being used to package these products (Sexton, 2003).

A traditional film currently used in the U.S. for fresh-cut produce is oriented polypropylene (PP), referred to as OPP (Del Nobile, 2007). In recent years, many bio-based and biodegradable films have been developed to serve the U.S. market but little if any data is available regarding their processing requirements or suitability for use with the commercial Vertical-Form-Fill-and-Seal (VFFS) packaging machines currently used to package fresh-cut produce. This has limited the adoption of bio-based and biodegradable films by the produce industry.

In the U.S., fresh-cut produce is typically processed on packaging lines where gravity is used to fill bags with product, and in-line, thermal-bar heat sealers are used to seal the bags. This set-up is commonly referred to as VFFS packaging line. To run on these lines, films must possess sufficient mechanical strength, low static charge and a controllable amount of slip (NIIR Board, 2002). Sufficient mechanical strength prevents elongation and tearing of the film. A low static charge reduces: the dust the film attracts, layer separation and jamming. Slip refers to the coefficient of friction of the film and film with insufficient slip may jam in the VFFS machine or elongate. If the film is too slippery, it may not feed properly (NIIR Board, 2002).

When a bag to hold pre-cut produce is formed and sealed, the seal must be continuous and strong. When sufficient heat is applied, molecular entanglement within the polymer chains produces a hermetic seal that resists rupture from both normal handling and the pressure changes that may occur during transportation. Heat seal integrity primarily depends on the pressure and temperature of the sealing jaws, and dwell time. Dwell time is the length of time, typically in milliseconds, that the sealing jaws are clamped on the plastic film. The temperature of the sealing jaws is determined by heat controllers and their accuracy is important. If a VFFS machine does not have sufficiently accurate temperature controllers, films with narrow sealing ranges may not seal correctly (NIIR Board, 2002).

The shelf life of pre-cut produce is dependent upon the degree of processing of the product, permeation characteristics of the packaging or lidding film, storage temperature, initial quality of the product (Ballantyne et al., 1988) and sharpness of cutting knives (Solica-Fortney and Martin-Belloso, 2003). Permeation refers to the rate at which gases, such as carbon dioxide and oxygen, move through a film and the films used in the pre-cut industry typically have widely varying permeation rates (Kader et al., 1989). When a film is correctly chosen, its use with a properly processed and stored pre-cut product can increase shelf life by 5 to 14 days (Ballantyne et al., 1988).

The equilibrium levels of oxygen and carbon dioxide that occur with a package are affected by a number of factors: the size of the package, the amount of product, the product itself, the temperature of storage, the length of storage and the type of film used. Film water vapor transmission may also be important when wilting of product or condensation within a package are considerations (Ballantyne et al., 1988).

Polyethylene/oriented-polypropylene films are typically used to package pre-cut produce. The films possess low moisture transmission rates, good mechanical stability, and have controllable slip, permeation and optical properties that are easily modified for specific applications (NIIR Board, 2002).

Curtzwiler et al. (2008) conducted research to characterize currently-available biodegradable films that could, potentially, be used to package pre-cut produce. It was concluded that a biodegradable high-density polyethylene (BHDPE) and a biodegradable polypropylene (BPP) were acceptable substitutes for the PE/OPP films currently used by the pre-cut industry as these biodegradable films had similar transmission rates for oxygen, carbon dioxide and water vapor as the PE/OPP films. Though previous studies characterized biodegradable films for their barrier and mechanical properties, at the time of this study, there was no published research evaluating the machinability of these films or their potential use with fresh-cut produce. The purpose of this study was therefore to determine the feasibility of using the BHDPE and BPP films with commercial vertical-form-fill-and-seal packaging machines (VFFS) equipped with thermal-bar heat sealers and used in the pre-cut lettuce industry.

MATERIALS AND METHODS

The industry standard film was a two-layer laminate consisting of polyethylene (PE) and oriented-polypropylene (OPP). It was supplied by a major pre-cut packager in Salinas, California and had an average thickness of 0.058 mm (Curtzwiler et al., 2008). The alternative films tested were a 0.051 mm biodegradable high-density polyethylene (BHDPE) and a 0.061 mm biodegradable polypropylene (BPP) both supplied by Bloomer Plastics (Bloomer, WI). The BHDPE and the BPP films were provided with or without anti-fog treatment, and one roll of BHDPE was provided with slip, but without anti-fog treatment.

Coefficient of Friction Testing

Before cutting samples, the machine direction was identified for each material using a Nikon 73231 optical microscope. The sample cutting surface and ruler were cleaned with alcohol before use, and latex gloves were worn during sample prep and testing to limit sample contamination. Three test runs were performed for each sample; on the third run the COF value was recorded. Five samples of each film type were tested for inside-to-inside, and five for outside-to-outside, COF. All samples were conditioned for a minimum of 40 hours prior to testing at 23°C, 50% relative humidity. Tests were conducted according to ASTM Standard D1894-06 (Anonymous, 2006) using a Model 32-50-00 Coefficient of Friction Tester (Testing Machines Inc., Ronkonkoma, New York). The manufacturer provided the angle interpretation table.

Machinability Testing Part I: Vertical-Form-Fill and Seal Packaging Lines

Films selected for this test included BPP without or without anti-fog treatment, BHDPE with or without anti-fog treatment, and BHDPE without anti-fog but with slip treatment. The line used for testing employed a Hayssen Ultima Twin-8-16HR (HayssenSandiacre, Duncan, SC) VFFS machine to package pre-cut Romaine lettuce. Each film was loaded onto the machine and run through a series of temperature and dwell time settings to determine suitability for use. Films were tested according to a modified ASTM 03078-02 (Anonymous, 2008) until packages made from the films had a seal that would maintain integrity for 15 seconds when tested under water at 14″ Hg absolute (Pak-Vac Leak Tester, Haug, Morgan Hill, CA), or until it was determined that the film would not produce a suitable seal using a VFFS machine equipped with a thermal-bar heat sealer, regardless of temperature and dwell time.

Shelf-life Study Part I: Bags Sealed Using VFFS Machinery

Shelf-life studies were conducted simultaneously at the packager in Salinas, California and California Polytechnic University (Cal Poly), San Luis Obispo, California. A total of 18 each of the PE/OPP, BPP with antifog, and BHDPE (no antifog but with slip) bags of pre-cut Romaine lettuce were used for the study. The PE/OPP and the BPP bags were leak tested before use with only bags passing the leak test being used in this study. The BHDPE bags were initially leak-tested but were found to have poor seal quality. Therefore, subsequent bags produced using BHDPE were simply removed from the packing line and used without being leak-tested.

All bags were stored at 4.4ºC, 80% RH for 14 days. Bags were weighed initially and after 7 and 14 days to determine moisture loss from the lettuce. Oxygen and CO_2 in the headspace of each bag was measured daily using a Model 6600 O_2/CO_2 Headspace Analyzer (Illinois Instruments, Johnsburg, IL.). Lettuce was assessed for pinking and decay daily based on color charts and a proprietary methodology developed by the packer in Salinas, California.

Shelf-life Study Part II: Impulse vs Thermal-bar Sealed Bags

In the first shelf-life study, the primary problem with the bags made from the biodegradable films was seal integrity. The VFFS machinery employed thermal bars to seal a laminated film (PE/OPP), whereas the biodegradable films were non-laminated polypropylene or high density polyethylene. Therefore, a second test was conducted in which biodegradable salad bags were hand-sealed at Cal Poly using an 18″, 600 watt impulse sealer (AIE-500, Fisher Scientific). The bags were made to the same dimensions, 25.4 × 22.2 cm, as the commercially-available PE/OPP bags.

Bags of freshly-packaged pre-cut Romaine were obtained from the packer in Salinas, California and immediately transported on ice to Cal Poly. Eighteen bags were repacked into BPP bags (with no slip, no anti-fog) and 18 were repacked into BHDPE bags (with no slip, no anti-fog). The bags were opened and the lettuce was transferred in a laminar flow hood to prevent microbial contamination. Repacked bags were quickly transferred to a Smith Supervac GK180 vacuum/flush bag sealer. Bags were flushed with industrial-grade nitrogen to obtain a headspace volume and initial O_2/CO_2 levels comparable to those found in the bags of pre-cut Romaine obtained from the packer in Salinas. Control bags were not leak tested but all biodegradable bags were leak tested at 14″ Hg absolute for 15 seconds using a Test-A-Pack System (Carleton Technologies, Orchard Park, NY).

All bags were stored at 4.4ºC, 80% RH for 14 days. As before, the bags were weighed at 0, 7 and 14 days to assess weight loss from the product as affected by film type. Headspace O_2/CO_2 levels were measured daily. All lettuce was assessed daily for pinking and decay by observing the lettuce directly through the bags and after opening at 14 days. Only the "surface" lettuce was observed, i.e., the bags were not shaken during the observation process and only the lettuce closest to the film was evaluated, per the protocol developed by the Salinas packer. The biodegradable and control bags were leak tested again after 14 days storage. After 14 days, all lettuce bags were opened and assessed for off-odors and degree of sliminess. Sliminess was determined tactically.

Head Space Volume

Headspace was determined through water displacement. Each bag was submerged completely in water and the evacuated air was collected and measured. Five bags of each film type were tested.

Statisical Analysis

For the shelf-life studies, the researchers were normalized on-site to the proprietary procedures used by the Salinas, CA packer to assess and quantify pinking and decay. Pinking and decay were quantified for each study by 3 researchers who also, collectively, assessed sliminess and the presence or absence of off-odors. The experimental design was completely randomized with 18 replicates per treatment. All data was analyzed using the PROC GLM procedure of the Statistical Analysis Systems Ver. 9.1 software package (SAS Institute, Raleigh, NC). Where applicable means were separated using Duncan's Multiple Range Test ($\alpha = 0.05$).

RESULTS AND DISCUSSION

Coefficient of Friction Testing

The BPP films with and without anti-fog had coefficient of friction values that were the closest to the values obtained from representative industry films (Table 1). The BHDPE films with and without anti-fog had much higher coefficient of friction values than those obtained from the industry films. However, both types of biodegradable film had much higher COF values than the industry films tested.

Machinability: VFFS Packaging Lines

The BHDPE film without anti-fog was run on the VFFS machinery (Table 2), but the seals were of poor quality. Bags produced were used in shelf-life testing, but were not leak tested due to poor seal strength. The BHDPE film with anti-fog was not run due to the high COF of the film. The BPP film with slip was run on the packaging line with moderate success. During this trial run, ninety-percent of the BPP bags were thrown off the line before leak-testing due to obvious poor seals. A total of 80 bags of the BPP with slip were leak tested: 38 passed and 42 failed (52.5% fail rate). At this time, the control (commercial) bags were also leak tested. Out of 66 bags tested, 36 passed and 30 failed (45.5% fail rate). Regardless, all BPP and PE/OPP bags were leak tested and only those bags which passed where used in the study.

Due to the poor performance of the BPP with anti-fog, and the BHDPE with slip, films with similar or higher COF values were not

Table 1. *Characteristics of Films Tested on February 7, 2008.*

Film Type	Anti-fog Capability	Slip	COF[z] Inside to Inside	COF Outside to Outside
Garden Salad[y]	Yes	Yes	0.17	0.19
Caesar Salad[y]	Yes	Yes	0.24	0.33
Premium Romaine[y]	Yes	Yes	0.13	0.16
BPP	No	No	0.23	0.49
BPP	Yes	No	0.22	0.45
BHDPE	No	No	0.33	0.52
BHDPE	Yes	No	0.40	1.81

[y] Commercial packages from Salinas, CA packer containing respective pre-cut mixes.
BPP = biodegradable polypropylene
BHDPE = biodegradable high-density polyethylene

Table 2. *Vertical-form-fill and Seal Machine Parameters Used at a Packer in Salinas, CA.*

Setting Description	**BPP**^{z}	**BHDPE**
Front Jaw	345°F	295°F
Rear Jaw	340°F	295°F
Platen	310°F	280°F
System Speed		35 bags/min
Bag Length	125–130 mm	125–130 mm
Vibration-line feeders	1016	1016
Vibration-main	1790	1790
Target weight of product	255 g	255 g
Average weight	255.4 g	255.4 g
Regular Roller^{y}	30–130	30–120
Pull belts^{y}	20–120	20–120
Platen^{y}	120–347	114–345
Stager^{y}	120–359	120–359
Stripper^{y}	120–355	135–355
Jaw Close^{y}	170–340	170–340
Knife^{y}	240–320	200–300
Dwell time^{y}	7 ms^{x}	10 ms
Typical Seal Orientation	Outside-to-outside	Outside-to-outside

^{z}Film does not have anti-fog, but does have slip.
^{y}Based on a 360° cycle.
^{x}Millisecond.
BPP = biodegradable polypropylene
BHDPE = biodegradable high-density polyethylene

tested. The films not run were the BPP without anti-fog or slip, BHDPE with anti-fog but without slip and the BHDPE without anti-fog or slip.

Shelf-life Study Part I: Bags Sealed Using VFFS Machinery

In this study, bags were formed from biodegradable films which were sealed with a thermal-heat bar. The resulting seals were of poor quality, in spite of the fact that the BPP bags had been leak tested at the packer. During subsequent storage, the bags made from both biodegradable films did not perform as well those made from the commercial PE/OPP in maintaining the quality of pre-cut Romaine lettuce. Most of the lettuce in the biodegradable bags became unmarketable within 5 days after packing [data not shown].

Shelf-life Study Part II: Impulse vs Thermal-bar Sealed Bag

In this study, bags developed leaks during storage and handling at the

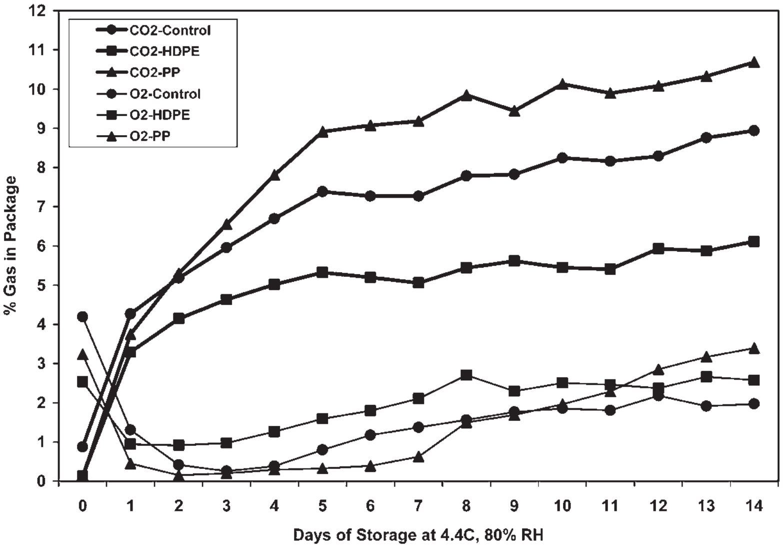

Figure 1. *Levels of oxygen and carbon dioxide in the headspace of bags containing pre-cut Romaine. Control = commercial polyethylene/oriented polypropylene laminate, HDPE = biodegradable high-density polyethylene, PP = biodegradable polypropylene.*

same rate (16.7%), regardless of the film or sealing machinery that was used. The levels of O_2 and CO_2 in PE/OPP bags were intermediate to those that developed in bags made with the biodegradable films (Figure 1). Overall, the levels of O_2 were lower and CO_2 higher in bags constructed from BPP. Lettuce in the PE/OPP commercial bags had the strongest smell after 14 days of storage but tended to have the least amount of sliming (Table 3).

There was an interaction between package film and storage duration on the amount of water lost from stored pre-cut Romaine (Figure 2).

Table 3. *Effect of packaging on the overall amount of sliming and development of off-odors from pre-cut Romaine lettuce during 14 days storage at 4.4°C, 80% RH.*

	Off-odor Level[z]	Slimy Product (% bags)
PE/OPP (Control)	3.7 a[y] Faint to moderate off-odor	13 b[x]
Biodegradable Polypropylene	3.1 b Faint odor	21 b
Biodegradable high-density PE	2.9 b Faint odor	53 a

[z] 1= fresh; 2 = no odor; 3 = faint off-odor; 4 = moderate off-odor; 5 = spoiled smell.
[y] Pr > F = 0.0551.
[x] Pr > F < 0.05.

However, the amount of water lost was minimal (≤ 0.25 g after 14 days) regardless of packaging.

The rates of decay for lettuce packaged in the PE/OPP bags or biodegradable PP bags were comparable (Figure 3), as were the number of bags which became unmarketable due to excessive decay of the lettuce during 14 days storage (Figure 4). In contrast, the lettuce stored in bags made from the biodegradable high-density PE decayed at a faster rate and to a greater extent than lettuce bagged in either the PE/OPP or biodegradable polypropylene bags. The number of HDPE bags that were unmarketable started to increase at day 3, the PP bags at day 12 and the control at day 13.

Lettuce stored in biodegradable high-density PE "pinked" at a faster rate (Figure 5) and to a greater extent (Figure 6) than lettuce bagged in either PE/OPP or biodegradable polypropylene. Romaine stored in the biodegradable PP bags tended to pink at a slower rate and at a later time than lettuce stored in the bags made from PE/OPP.

Overall, decay levels were lowest for pre-cut Romaine stored in the PE/OPP bags, but the overall level of pinking was the same for lettuce stored in either the PE/OPP or BPP bags (Table 4).

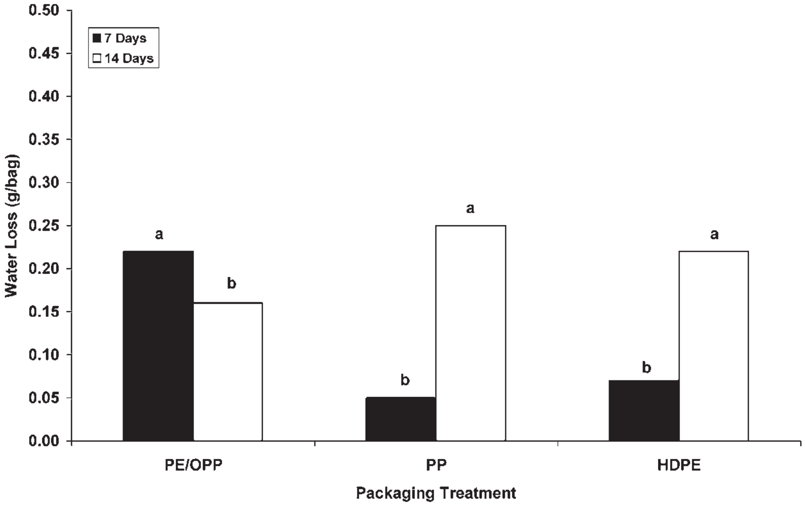

Figure 2. Weight loss from pre-cut Romaine lettuce during 14 days storage at 4.4°C, 80% RH. Comparisons should be made for values at a specific day of storage. Means separation determined using Duncan's Multiple Range Test, α = 0.05. Control = commercial polyethylene/oriented polypropylene laminate, HDPE = biodegradable high-density polyethylene, PP = biodegradable polypropylene.

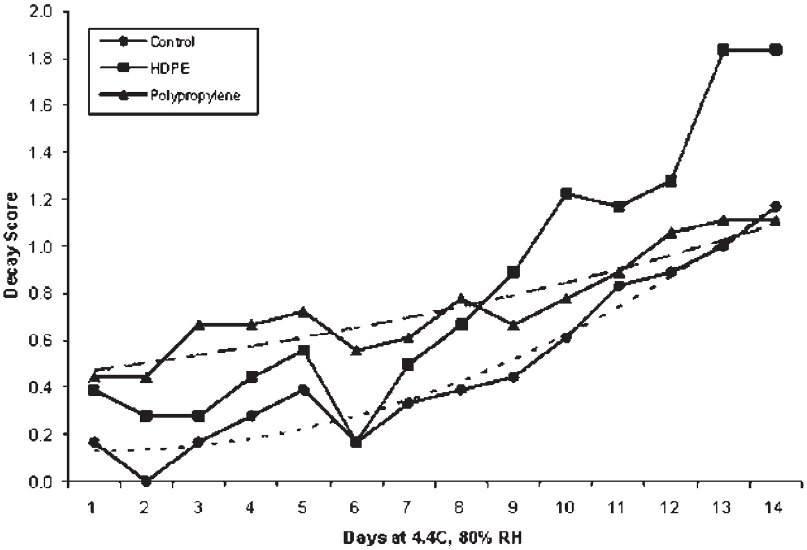

Figure 3. *Effect of packaging on the rate of decay of pre-cut Romaine lettuce during 14 days storage at 4.4°C, 80% R.H. "Best fit" hatched lines indicate the relative rates of decay of the Romaine packaged in biodegradable polypropylene vs. the commercial PE/OPP (control). HDPE = biodegradable high density polyethylene.*

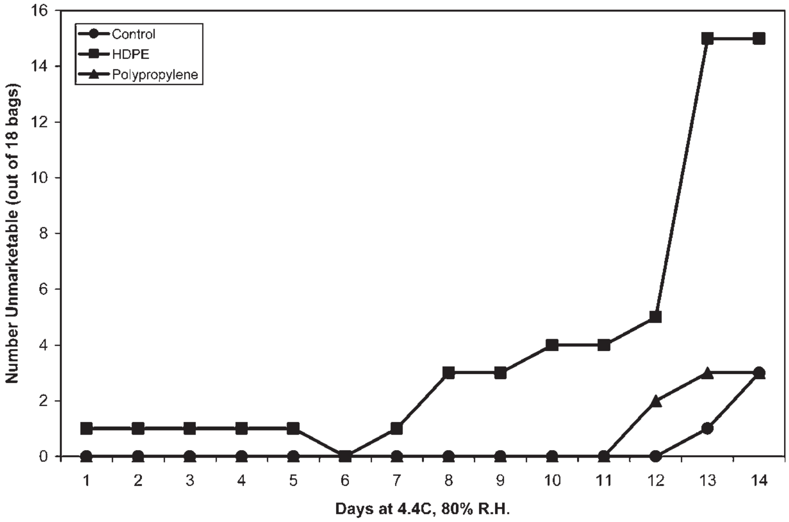

Figure 4. *Effect of packaging on cumulative decay as it relates to the number of bags of pre-cut Romaine lettuce which became unmarketable during 14 days storage at 4.4°C, 80% RH. 0 = None; 1 = total decay less than size of a quarter (4.62 cm^2); 2 = total area ≥ 4.62 cm^2 (failure). Control = commercial polyethylene/oriented polypropylene laminate, HDPE = biodegradable high-density polyethylene, PP = biodegradable polypropylene.*

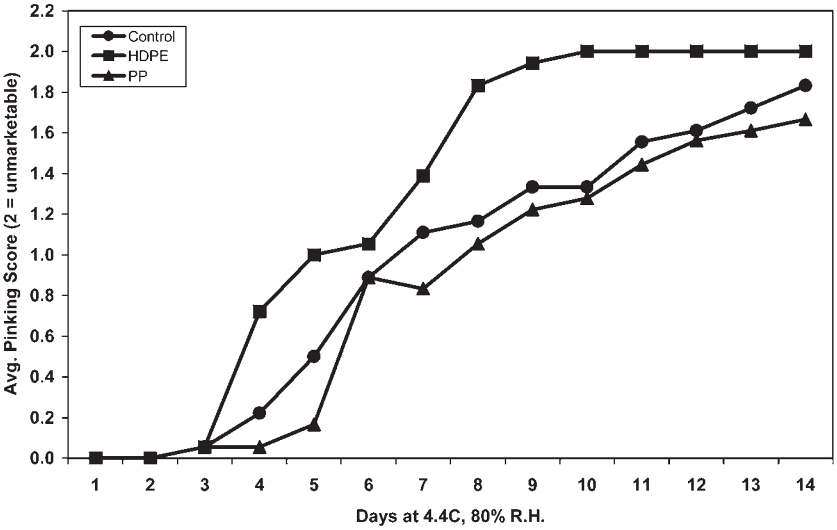

Figure 5. *Effect of packaging on the rate of pinking of pre-cut Romaine lettuce during 14 days storage at 4.4°C, 80% RH. 0 = None; 1 = some pinking but marketable; 2 = unmarketable. Control = commercial polyethylene/oriented polypropylene laminate, HDPE = biodegradable high-density polyethylene, PP = biodegradable polypropylene.*

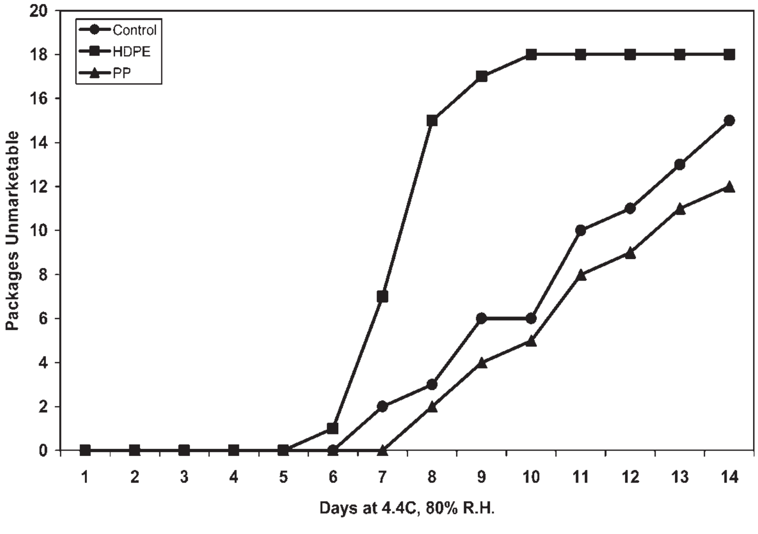

Figure 6. *Effect of packaging on cumulative pinking as it relates to the number of bags of pre-cut Romaine lettuce which became unmarketable during 14 days storage at 4.4°C, 80% RH. 0 = None; 1 = some pinking but marketable; 2 = unmarketable. Control = commercial polyethylene/oriented polypropylene laminate, HDPE = biodegradable high-density polyethylene, PP = biodegradable polypropylene.*

Table 4. *Effect of packaging on the overall amount of decay and pinking of pre-cut Romaine lettuce during 14 days storage at 4.4°C, 80% RH.*

Film Type	**Decay Level**^{z}	**Pinking Level**^{y}
PE/OPP (Control)	0.50 b	0.95 b
Biodegradable Polypropylene	0.75 a	0.84 b
Biodegradable high-density PE	0.82 a	1.29 a

^{z}0 = None; 1 = total decay less than size of a quarter (4.62 cm^2); 2 = total area ≥ quarter (failure).
^{y}0 = None; 1 = some pinking but marketable; 2 = unmarketable.

DISCUSSION

Biodegradable polypropylene and high-density polyethylene required the use of an impulse sealer and increased dwell time to obtain satisfactory closure of bags formed from the films. The biodegradable polypropylene performed well in maintaining the quality of pre-cut Romaine lettuce stored for 14 days at 4.4°C, 80% R.H., resulting in a shelf-life comparable to that of commercially-packaged, pre-cut Romaine. The results indicate that the use of biodegradable bags for the pre-cut salad industry is feasible if care is taken that seal integrity is insured. Subsequent investigation should assess the impact of using impulse sealers in VFFS machinery on product thru-put. The results likewise indicate that improvements are needed to broaden the heat-seal temperature range of biodegradable films for ease of running on commercial VFFS machinery. This study also indicates a need for biodegradable laminated films compatible with commercial VFFS packaging lines.

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Effect of Transport Vibration on Quality of Minimally Processed and Packaged Fresh-cut Cantaloupe

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ABSTRACT: This study was undertaken to determine the quality of packaged fresh cut cantaloupe subjected to transport vibration after treating with various anti-browning agents. Cantaloupe (*Cucumis melo*) pieces were dipped in two anti-browning solutions: Treatment-A (2% ascorbic acid + 1% calcium chloride + 0.5% citric acid) and Treatment-B (3% NatureSeal™) for 2 minutes and packaged in bio-based clamshell containers and vibrated for 60 minutes (ASTM 4169, Truck assurance level II). Vibration of cut-cantaloupe packaged in sample containers had a positive effect on the flavor and overall liking. The overall liking scores were higher than 6(slightly like) on a hedonic scale of 1–9 for all samples except for vibrated-Treatment-A. The texture of vibrated cut-cantaloupe deteriorated with time, which was partially supported by firmness values obtained using a Kramer shear press. Treatment-B cut-cantaloupe subjected to vibration performed better than Treatment-A for appearance, flavor, texture and overall acceptability. No off-odor, sliminess or mold growth was observed in any of the samples during 10-day storage at 5°C ± 0.3°C. The sensory panel examined the final quality of fresh-cut fruit for several quality parameters at day 1, 4, 7 and 10 after being vibrated and stored in bio-based packaging made from Poly (lactide) (PLA) polymer. These findings are significant for quality preservation of cut-cantaloupe during transportation and distribution channels.

1.0 INTRODUCTION

FRESH cut fruits (FCF) are increasingly becoming popular in the marketplace. The FCF is a $300 million industry, projected to reach $1 billion by 2010 (IFPA, 2004). With growing health concerns, consumers are resorting to more nutritional options in their diet, such as FCF (IFPA, 2004). It is known that fresh cut fruits are more perishable than intact

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fruits (Watada et al., 1996) as a result of chemical and physical stresses during processing, handling and storage. At undesirable temperature, humidity, atmosphere and sanitary conditions can deteriorate product quality (Watada et al., 1996) mainly in appearance, flavor and purging at the bottom of the package. Respiration rates of FCF are normally higher than intact fruit and increases with temperature (Watada et al., 1996). Therefore it is recommended that FCF should be stored at lower temperatures unless there is a risk of chilling injuries. Another, important component which can affect FCF quality are transportation abuses. It is well known that whole fruits like mango, banana, tangerine and papaya get bruised and quality deteriorates more rapidly. Such spoilage is dependent on road conditions and type of trucks used to transport fruits (Chonhenchob and Singh, 2006). However consumers expect to buy cut fruits fresh and without any defects at a grocery store (Watada and Qi, 1999).Therefore, one of the on-going challenges is to delay browning and extend the shelf life of highly perishable FCF. This can be achieved by processing cut fruits with anti-browning solutions prior to packaging and distribution. Several studies have reported that ascorbic acid in combination with calcium chloride ($CaCl_2$) is an effective anti-browning agent (Chonhenchob and Singh, 2005). A commercially available anti-browning agent NatureSeal™ is a calcium ascorbate powder used extensively in the fresh cut industry. Ascorbic acid functions as reducing agent to deter surface browning (Whitaker, 1994) and $CaCl_2$ treatment provides tissue firming and has been reported to reduce browning (Drake and Spayd, 1983; Hopfinger et al., 1984). Also, non-biodegradable rigid containers made from PET (Polyethyleneterephthalate) and PS (Polystyrene) contribute substantially towards solid waste in a landfill. To combat this issue biodegradable rigid containers made from a PLA (Poly lactic acid) are increasingly becoming popular in the fresh cut fruit industry. Thus the objective of this study was to determine the effect of transport vibration and anti-browning agents on the sensorial attributes of fresh-cut cantaloupe packaged in bio-based plastic containers.

MATERIALS AND METHODS

Fresh Cut Processing

Whole cantaloupe was purchased from the local supermarket.The whole cantaloupe was washed and dipped in a commercial sanitizer-Fruit & Vegetable Wash (SC Johnson Professional, Sturtevant, WI) (100-ppm chlorine) for 5 minutes. Following which they were stored in 5°C ± 0.3°C walk in chamber for a period of 12 hours prior to cutting. Once cantaloupes equilibrated to the desirable temperature, they were cut in 22°C ± 4°C environment. After removing seeds and peel, the cantaloupes were cut into 1-inch cubes using a sharp stainless steel knife cleaned in 100 pm chlorine solution (Figure 1) Cantaloupe pieces were dipped in two anti-browning solutions: Treatment-A (2% ascorbic acid + 1% calcium chloride + 0.5% citric acid) and Treatment-B (3% NatureSeal™ containing calcium ascorbate) for 2 minutes. Following which 180 ± 5 grams of cantaloupe pieces were packaged in bio-based clamshell containers (19.1 × 16.5 × 4.4 centimeters) made from poly (lactide) (PLA) (Figure 2). Twelve cantaloupe filled containers were packaged in corrugated fiberboard boxes (C-flute; FEFCO 0306 AB)(Dimensions- Lid: 51.4 × 41.3 × 5.7 cm ; Base: 48.9 × 40.6 × 15.6 cm) in 2 layers, 6 containers per layer, which were subjected to random vibration for 60 minutes to represent a 500 mile trip during distribution,

Figure 1. *Minimally processed fresh cut cantaloupe (size: 1 in^3) before transport vibration.*

Figure 2. *Cantaloupe filled containers packaged in corrugated box.*

(ASTM 4169, Truck assurance level II) on a vibration table (Lansmont Model 10000-10, Inc, Monterey, CA, USA) as shown in Figure 3. The cantaloupe filled containers were stored at 5°C ± 0.3°C for 12 hours before further evaluation and testing. These tested samples of PLA containers with fresh cut fruit were compared to "control" samples that had not been vibrated but were packaged and stored under identical conditions for the same period of time as the tested packages (Figure 4).

Sensory Evaluation

Each set of treated samples had a non-vibrated control container containing cut cantaloupe for comparison with vibrated and treated samples. Appearance, flavor, texture and overall liking of cut cantaloupe were evaluated by an eight-member panel on a hedonic scale of 1–9 hedonic (9 = Like extremely, 7 = like moderately, 5 = neither like nor dislike, 3 = dislike moderately, 1 = dislike extremely) for at day 1, 4, 7 and 10. A score of 6 was determined as the limit of marketability. Each panelist was provided with 4 samples (Table 1) in 2 oz cups labeled with random numbers. The test setup is shown in Figures 5 and 6.

Figure 3. *Cantaloupe filled containers packaged in corrugated boxes subjected to random vibration.*

Figure 4. *Comparison of control versus minimally processed fresh cut cantaloupe after transport vibration.*

Figure 5. *Tray setup presented to sensory panelist.*

Figure 6. *Sensory panelist booth for testing fresh cut cantaloupe.*

Table 1. *Sensory Evaluation Samples Provided to Panelists.*

Samples	Description
Control A	Non-vibrated and Treatment A
Tested A	Vibrated and Treatment A
Control B	Non-vibrated and Treatment B
Tested B	Vibrated and Treatment B

Instrumental Texture Analysis

A Kramer shear press (Model FTA-300, FTC, Stering, VA) was used to determine flesh firmness at 1, 4, 7 and 10 days to compare it with texture scores from sensory evaluation. A sample holder (6.6 × 6.6 × 6.4 cm) was loaded with 60 grams of cut cantaloupe cubes. Upon placing the sample holder in the test cell 10 movable blades were lowered at 20 cm/min, compressing the cut samples to a distance of 10.2 cm. Following which the force required to compress test sample was recorded.

Statistical Analysis

The collected data was analyzed using statistical software Minitab 13.1 (Minitab Inc, State College, PA, USA). Analysis of variance was performed on sensory and firmness data and the means were separated using Fisher's LSD at significance level of $p \leq 0.05$.

RESULTS AND DISCUSSION

Sensory Evaluation

a) Appearance

Fresh cut cantaloupes stored in PLA containers and subjected to their respective treatments were evaluated for the attributes mentioned in the methods section. The appearance of cantaloupe showed a general trend of deterioration with time (Figure 7). One day after processing and vibration there was no significant difference in appearance (Figure 7) between cut cantaloupe processed with Treatment A and Treatment B. However, cut cantaloupe 'Control B' (6.63a) appeared to be better than 'Control A' (6.25a), 'Tested A' (6.25a) and 'Tested B' (6.25a) (Figure 7) at day 1. Even though 'appearance' deteriorated with time (Figure 7) it

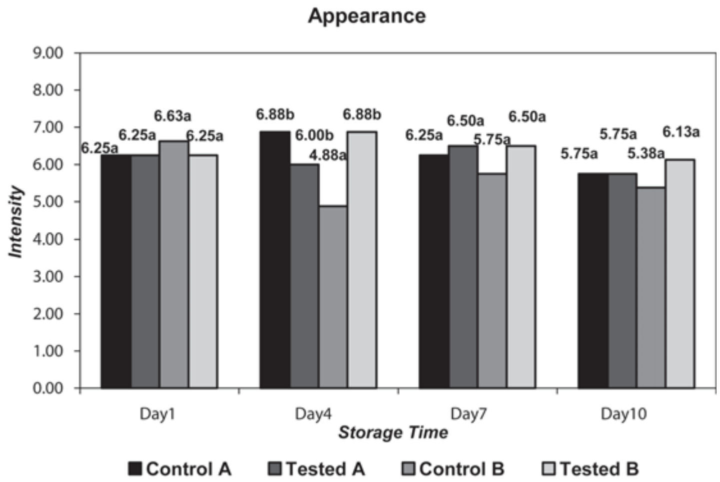

Figure 7. *Sensory scores for fresh cut cantaloupe appearance over storage period. 1 = Dislike extremely; 9 = Like extremely. *Mean scores with different letters are significantly different.*

was observed that the appearance scores for 'Tested B'(6.13a) samples were higher than 'Control A'(5.75a), 'Tested A'(5.75a) and 'Control B'(5.38a) by day 10. This indicates that fresh cut cantaloupe treated with Natureseal™ and subjected to vibration had the best appearance over a period of 10 days.

b) Flavor

It was observed that the day 1 flavor scores compared to Day 4 had lower hedonic scores for all the treatments except for 'Tested B' (7.0a) at day 1. Also, at day 1, 4 and 7 'Tested B' cantaloupe samples were rated to have better flavor scores than its 'Control B' samples (Figure 8). Similarly at day 1 and 7 'Tested B' samples had higher flavor scores than 'Control A' (Figure 8). This is possibly due to ripening of cut cantaloupe as a result of higher respiration rate and ethylene induced ripening during storage. Ethylene production as a consequence of cutting has been observed in tomato (Lee et al., 1970), strawberry (Abeles et al., 1992) and papaya (Paull and Chen, 1997) leading to accelerated ripening. Similarly, cantaloupe has shown high ethylene release upon cutting (Hoffmann and Yang, 1982). Also, several cut fruits have shown higher respiration rates than whole fruits (Watada et al, 1990; Cantwell, 1992) leading to shorter shelf life. Therefore accelerated ripening due to increased ethylene production and respiration rate can explain better 'fla-

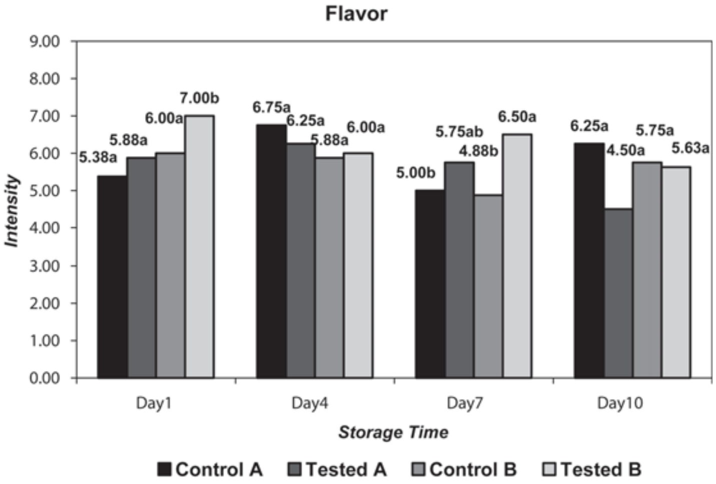

Figure 8. *Sensory scores for fresh cut cantaloupe flavor over storage period. 1 = Dislike extremely; 9 = Like extremely. *Mean scores with different letters are significantly different.*

vor' scores at day 1, 4 and 7 for the samples which were exposed to mechanical vibration. This indicates that vibration during distribution has some positive effects in enhancing flavor of fresh cut cantaloupes. However at the end of the study 'Control A' samples had the highest flavor scores (Figure 8).

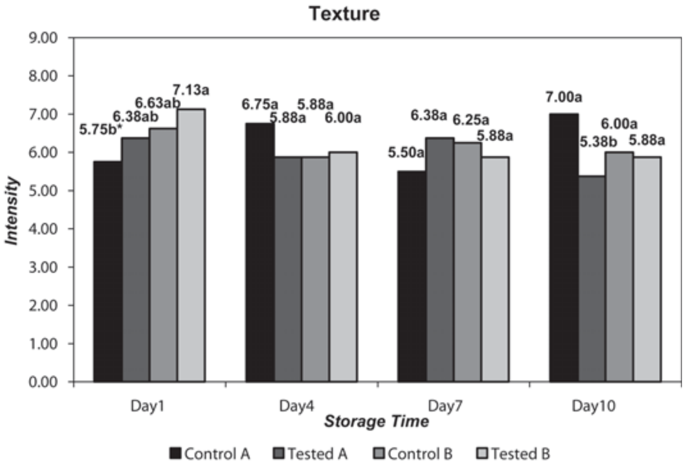

Figure 9. *Sensory scores for fresh cut cantaloupe texture over storage period. 1 = Dislike extremely; 9 = Like extremely. *Mean scores with different letters are significantly different.*

c) Texture

Overall the texture of all the sample treatments deteriorated with time except for ‘Control A’ samples (Figure 9). ‘Control A’ samples were rated with the highest texture scores (6.75) at day 4 and day 10 (7.0) compared to day 1 samples (5.75) (Figure 9). Initially ‘Tested B’ had the best texture scores but it deteriorated with time from a score of 7.13 at day 1 to 5.88 at day 10. Where as, ‘Control A’ sample had the best texture (7.0) at day 10 compared to the remaining treatments. The texture of ‘Tested A’ and ‘Control B’ was observed to be similar at day 1, 4 and 7 (Figure 9). Therefore, it can be concluded that at the end of the study the ‘Control A’ samples (7.0a) were observed to have the best texture followed by ‘Control B’(6.0a), ‘Tested B’(5.88a) and ‘Tested A’(5.38b) samples by day 10.

d) Overall Liking

Initially it was observed that ‘Tested B’ samples had the highest overall liking score of 7.0 at day 1 followed by ‘Control B’ (6.5ab), ‘Tested A’ (5.75ab) and ‘Control A’ (5.63b) (Figure 10). At day 4 ‘Control A’ samples were rated to have the highest overall score of 7.13. ‘Tested A’ samples were preferred over ‘Control A’ samples at day 7. Similarly, ‘Tested B’ samples had higher overall scores than ‘Control A’ at day 1, 4, 7 and 10 (Figure 10). This indicates that there can be a positive effect

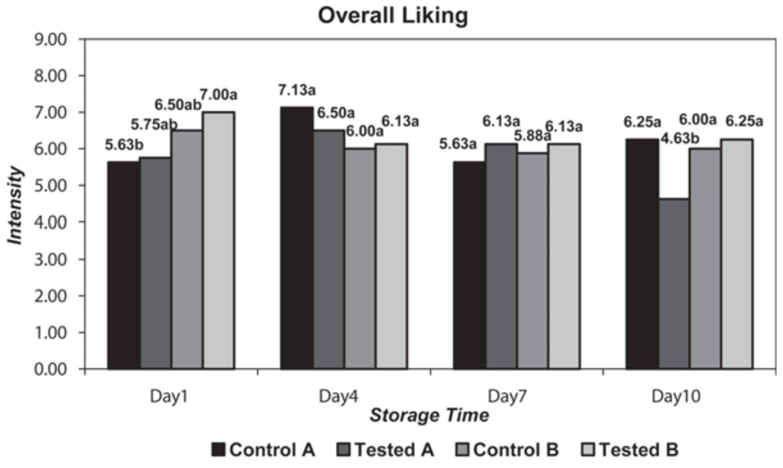

Figure 10. *Sensory scores for fresh cut cantaloupe overall liking over storage period. 1= Dislike extremely; 9= Like extremely. *Mean scores with different letters are significantly different.*

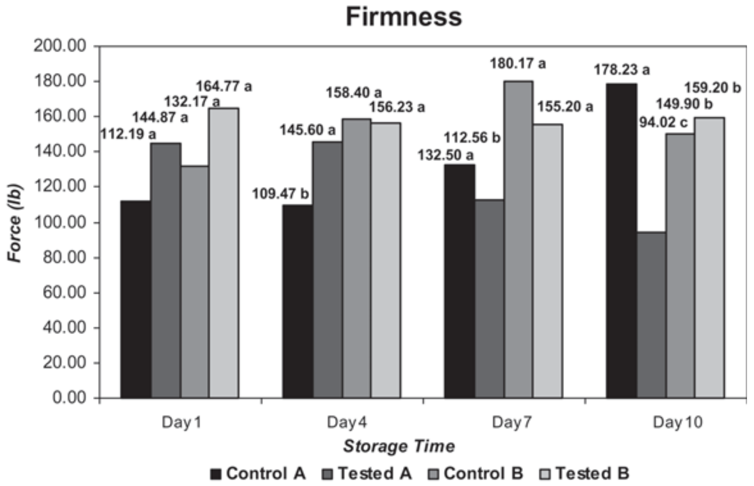

*Figure 11. Kramer firmness of fresh cut cantaloupe over storage period. *Mean scores with different letters are significantly different.*

of vibration during distribution on the overall acceptability of a sample at a particular storage time.

Firmness Measurements

There was no direct correlation between firmness measurement and texture scores from the sensory panelists. However by day 10 the firmness values for ‘Control A’ sample (178.23 lb) was observed to be the highest followed by ‘Tested B’ (159.20b), ‘Control B’ (149.20b), and ‘Tested A’ (94.02c).Comparing this trend to texture scores as observed at day 10 by the panelists (Figure 9), it can be seen that cut fruit firmness is related to its texture during storage. Also, the firmness of the cut cantaloupe was observed to be somewhat decreasing from day 1 to day 10 for ‘Tested A’ and ‘Tested B’ samples (Figure 11), showing that there is a distinct effect of vibrational forces on firmness during transportation. Thus, softening of cantaloupe flesh can be expected as these samples are subjected to vibrational forces during the transportation. It was interesting to find that vibration tested cut cantaloupe with ‘Treatment B’ (159.20 lb) had higher firmness values than ‘Treatment A’ (94.02 lb) at day 10. This is indicative that commercially available anti-browning solution Natureseal™ will perform better in preserving cut fruit texture in a transport environment.

CONCLUSION

Vibration of fresh cut-cantaloupe packaged in sample containers had a positive effect on the flavor and overall acceptability. The overall liking scores were higher than a hedonic score of 6 (like slightly) for all samples except for vibrated-Treatment-A. The texture of vibrated cut-cantaloupe deteriorated with time. There was some evidence of correlation between by firmness values and texture scores at day 10 (Figures 9 and 10), which shows that flesh firmness and texture scores at the end of the storage period is better in control samples compared to vibration tested samples. Thus, it can be said that vibrational forces during transportation as an effect on texture quality of cut cantaloupe. Treatment-B cut-cantaloupe subjected to vibration performed better than Treatment-A for appearance, flavor, texture and overall acceptability. No sliminess or mold growth was observed in any of the samples during 10-day storage. Finally, these findings are significant for quality preservation of cut-cantaloupe during transportation and distribution channels.

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Effect of Truck Vibration during Transport on Damage to Fresh Produce Shipments in Thailand

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ABSTRACT: The increase in global trade allows packaged products to be shipped across borders using inter-modal transportation. Trucks are still the most prevalent mode for surface shipments and time sensitive transport perishable products such as fresh produce. This study focused on measuring the transportation environment in truck shipments from various packing houses to major retail distribution centers in Thailand and then the subsequent distribution to regional stores in smaller trucks. Test measurements were compared to test methods used in North America and Europe. This study compared the quality of cabbage, lettuce, plums and pears after being shipped in truck transport by quantifying the level of bruises and cuts on fruit.

1.0 INTRODUCTION

THE effects of shock and vibration caused during shipping can result in serious product damage. Fresh produce (fruits and vegetables) are extremely sensitive to any physical or climatic changes after being harvested and during transportation and handling from the fields to ultimate purchased by the consumer at a retail store. Shipping and handling can cause various forms of bruises and cuts on the fresh fruit or vegetable which compromises its quality, aesthetic appeal and reduces its economic value to the grower and retailer. There are various modes of transportation in the growing international trade that requires inter-modal shipments over land, sea and air. However, trucks are still the most prev-

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alent mode for surface shipments and time sensitive transport perishable products such as fresh produce. This is also attributed to the fact that trucks are used in most cases as the first mode of transportation from the field or packing house to the retail distribution center.

Distribution data is therefore a significant factor in designing optimum product package systems. The dynamic levels measured in actual shipments can also be used to develop test methods to better simulate the shipping environment and avoid product quality loss or value at sales.

The primary objective of this study was to measure and analyze the distribution environment during truck transportation of fresh produce shipments in Thailand. The distribution measurement was focused on the major fresh produce regions to retail distribution centers and then downstream distribution to retail stores. In addition the amount of damage to the produce was also measured. Four different fresh produce items including cabbage, lettuce, pears and plums were shipped and monitored. In addition the measured vibration levels were compared to vibration data used in western countries to simulate truck transport environments.

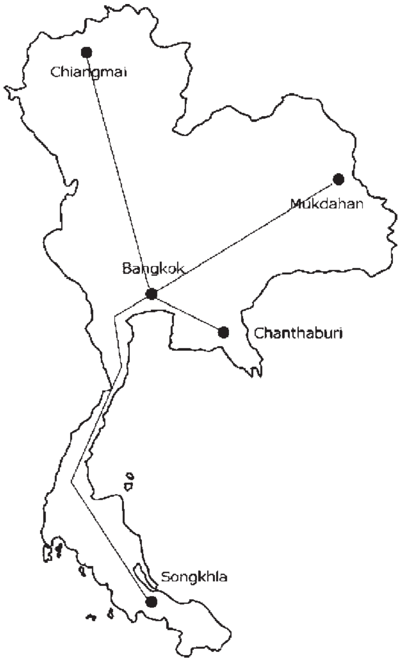

Figure 1. *Thailand map indicating truck transportation routes investigated.*

Table 1. *Truck Shipment Routes Studied.*

Route		Distance (km)
Truck	National Highway (NH)	
Bangkok-Chiang Mai (North)	1	700
Bangkok-Mukdahan (North-East)	2	642
Bangkok-Chanthaburi (East)	3	245
Bangkok-Songkhla (South)	4	950

Thailand is the world's leading fruits and vegetables producer and exporter. The primary cause of produce loss is physical damage, attributed to vibration forces during handling and transportation. The most common method for shipping produce in many countries, including Thailand is truck transportation. Reusable plastic containers (RPC) are commonly used to package the produce for domestic shipping. The data in this study represents the major routes of produce distribution in Thailand from the growers to the distribution centers, and then onto retailers (Figure 1 and Table 1).

2.0 VIBRATION DATA MEASUREMENT AND ANALYSIS

The study measured vibration levels in various truck shipments from various regions of Thailand. A more comprehensive paper on this topic has been prepared that discusses both truck and rail shipments. The Shock and Vibration Environmental Recorder (SAVER) Model 3X90 developed by Lansmont, Corp. (Monterey, CA, USA) was used to quantify vibration levels (Figure 2). The SAVER consists of a piezoelectric

Figure 2. *SAVER model 3X90 (Lansmont, Corp.).*

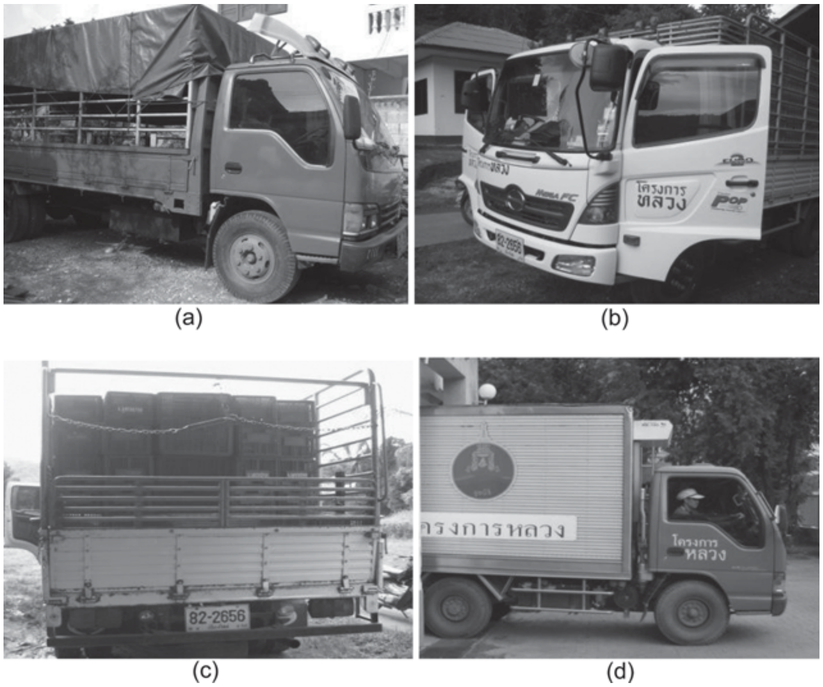

Figure 3. *Various trucks used in the vibration measurement study (a)–(d).*

tri-axial accelerometer, and is a battery powered instrument that measures shock (impact/drop), vibration, temperature, and humidity conditions that occur during shipping and handling. The recorders were mounted on platform base, at the rear floor position. This location produces the highest vertical vibration measurement.

Figure 3 shows the different types of leaf spring trailers used in the different sections of fresh produce distribution. Figure 4 shows the different road conditions that packaged fresh produce is shipped on from the farm to the final retail shelves.

To measure the vibration levels in the various sections of the distribution environment, the following settings were used on the SAVER's for the present study:

- Minimum time triggered sampling: 10 min
- Trigger threshold level: 2.4 G
- Minimum sampling rate: 500 samples per second
- Minimum recording window: 2.048 sec
- Sample size: 1024

2.1 Data Collection

The truck shipments in this study were conducted on the major distribution routes in the country. The details of the truck shipments routes are described in Table 2. The speeds of the measured trucks were in the range of 30–90 km/h, where the average speed on the good road conditions was 80–90 km/h, while on the poor road conditions (mostly two-lane roads) was 30–40 km/h. Truck shipment routes measured in this study are shown in Figure 1. The vibration data was analyzed using the SaverXware software (Lansmont Corporation, Monterey, CA, USA).

2.2 Data Analysis

The recorded acceleration amplitudes in the random vibration were analyzed as a function of frequency to determine the power density (PD)

Figure 4. *Various road conditions measured in this study.*

Table 2. *The Details of Produce Shipments Studied.*

Origin	Destination	Average Distance (km)	Average Speed (km/h)	Truck	Load (kg)
Collecting Centers, Chiang Mai	Packing House, Chiang Mai	80	50	6-wheel, unrefrigerated, 2 m × 4.8 m × 1.9 m	7,200
Packing House, Chiang Mai	Distribution Center, Bangkok	700	70	6-wheel, refrigerated, 2.3 m × 7.1 m × 2.2 m	15,000
Distribution Center, Bangkok	Retailers, Bangkok	15	60	4-wheel, refrigerated, 1.7 m × 4.7 m × 2.2 m	5,000

levels. The average PD within a narrow band of frequencies of the spectrum is calculated as follows:

$$PD = \frac{1}{BW} \sum_{i=1}^{n} (RMS\ G_i^2) / N$$

where RMS G_i is the root mean square acceleration value measured in g within a bandwidth (BW) of frequencies, and N is the number of instants sampled. The corresponding *PD* levels are then plotted against the frequency of the bandwidth to develop the power spectrum density (PSD) plot.

The PSD plot is the variation of the vibration magnitudes as a function of frequencies. Data are presented from 1 to 100 Hz since this frequency band is the most accountable in causing package/product damage during transportation. Average power density spectra of the transport vibration measurement studied were computed and are presented similar to previous studies[1].

2.3 Produce Damage

Vibration during truck shipments has been known to be a critical cause of produce damage and loss due to bruising and cutting. Effect of vibration on mechanical damage of several fruits and vegetables such as apples[2], pears[3], peach[4] has been studied before in different regions. The effect of vibration levels and road conditions on damage to packaged tangerines during truck shipments in Thailand was also investigated by Jarimopas et al.[5] The produce distribution network for the Royal Project represented in this study is shown in Figure 5. The Royal Project has a large and extensive network of growers in the mountain areas in the northern region of Thailand. There are a number of fruit and vegetable products under the Royal Project ranging from tropical to temperate produce. The trucks used for produce pick-up from the collecting centers in the mountain areas to the packing house in Chiang Mai were six-wheel non-refrigerated trucks. To transport produce from the packing house in Chiang Mai to the distribution center in Bangkok, six-wheel refrigerated trucks were used. Produce was transported to the retailers by the 4-wheel or 6-wheel refrigerated trucks. All trucks used were a leaf-spring suspension type.

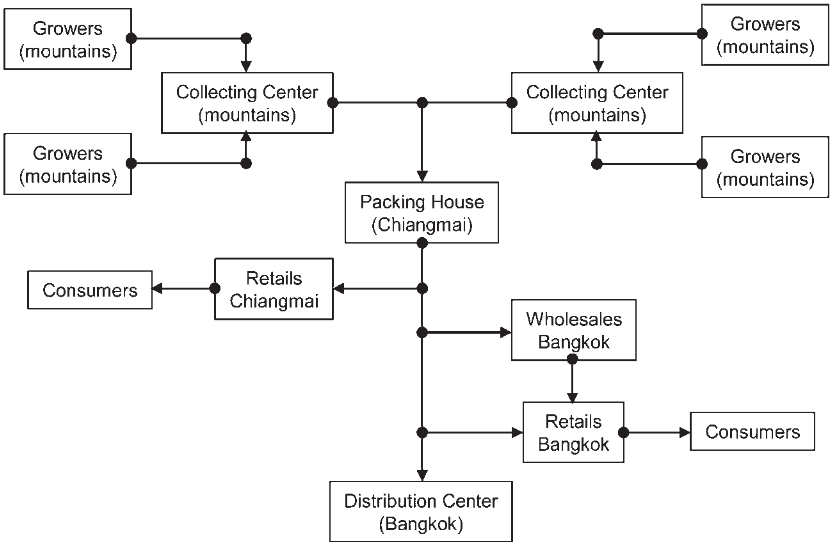

Figure 5. *The produce distribution network measured in this study.*

The primary objectives of this study were as follows:

1. To measure the vibration levels in the truck shipments of the major produce distribution routes in Thailand.
2. To measure the damage levels attributed to transportation and handling to various produce (cabbage, lettuce, plums and pear).
3. To compare highway vibration levels to inner city transportation from distribution centers to retail stores.

3.0 RESULTS AND DISCUSSION

3.1 The Truck Vibration Measurement

The average PSD plot developed for Thailand truck shipments in all axes is shown in Figure 6. The data for lateral and longitudinal vibrations is also shown. Since produce items vary in shape from spherical to elliptic, the damage caused to each produce item is a combination of vibration movement in all three orientations and rotational effects caused to the individual fruit, accompanied with dynamic compression. Figure 7 shows the vibration power density spectrums measured from smaller inner city trailers used to move produce from distribution centers to retail stores. These data spectrums were compared to composite vibration

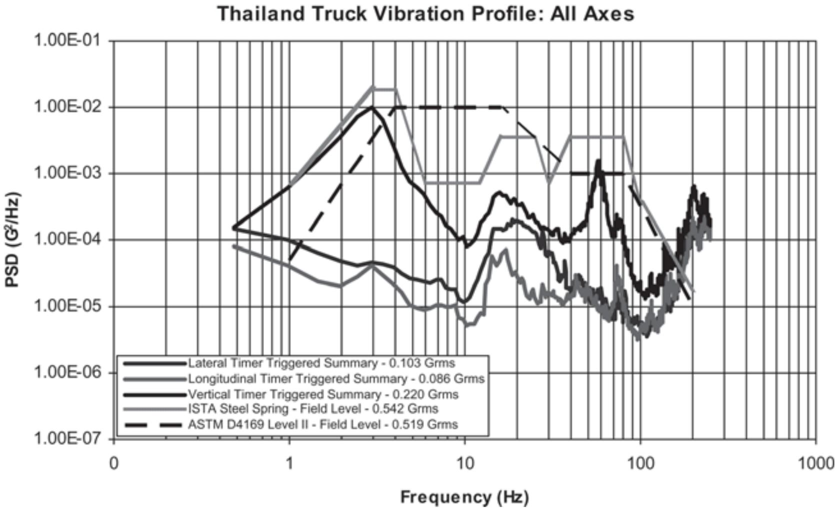

Figure 6. *PSD plot for truck vibration in Thailand in all axes compared with ASTM and ISTA.*

spectrums for vertical vibration that are used for vibration testing and recommended by the American Society of Testing and Materials International[6] (ASTM) and the International Safe Transit Association[7] (ISTA).

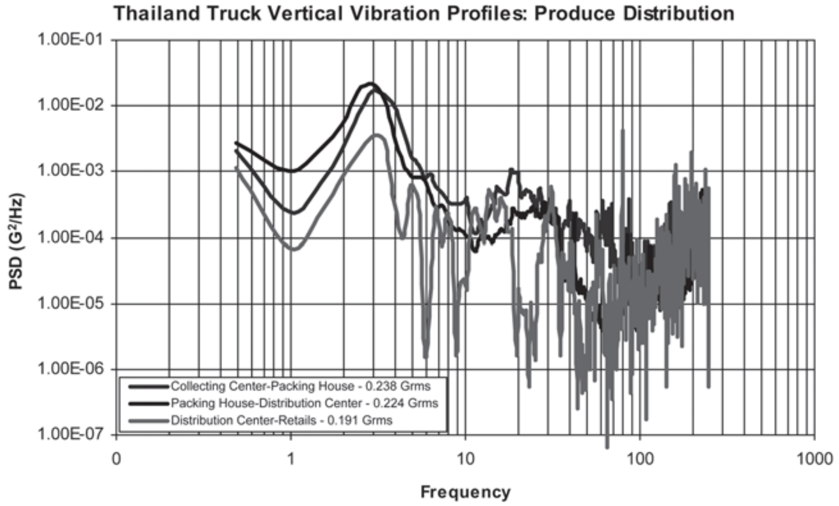

Figure 7. *PSD plot for vertical vibration of different truck shipments for produce distribution in Thailand.*

The results in Figure 6 and 7 show that the measured vertical vibration levels for highway shipments were similar to that of ASTM and ISTA in the low-frequency range (1–5 Hz). However in the higher frequency range the vertical vibration levels for truck highway transport are much lower. These higher frequencies represent the structural response of the trailer and effect of speed. Vibration levels measured in the smaller vehicles from distribution center (IDC) to retail stores are generally higher in the higher frequency regions (Figure 7). The highest vibration levels occurred from the field to packing house (PH), followed by PH to DC's. The lowest overall vibration levels occurred between DC's to retail stores. Table 3 shows Grms levels for the vertical, lateral and longitudinal orientations.

3.2 The Produce Distribution Study

In addition to the vibration data, the different produce was also inspected for physical damage due to cuts and bruises. A bruise was identified if it measured more than 1 cm^2 and a cut was identified to be 1 cm long with a minimum depth of 5 mm. These levels are generally known to make the sale of fresh produce ineffective at the desired retail price. The data and results from inspecting damaged produce (Figure 8) is presented in Table 4. The produce received at the retailers from the distribution center had the least damage, corresponding to the lowest vibration levels (Figure 7). Higher percentage and intensity of produce damage was observed at the packing house as compared to those at the distribution center. This was due to poor road conditions between the collecting center and the packing house. Bruising and cutting were shown to be the

Table 3. *Average Grms Levels for Different Truck Shipments for Produce Distribution.*

Spectrum	Route	Orientation	Level (Grms)
1	Collecting Centers—	Vertical	0.238
2	Packing House	Lateral	0.106
3		Longitudinal	0.061
4	Packing House—	Vertical	0.224
5	Distribution Center	Lateral	0.079
6		Longitudinal	0.050
7	Distribution Center—	Vertical	0.191
8	Retailers	Lateral	0.072
9		Longitudinal	0.054

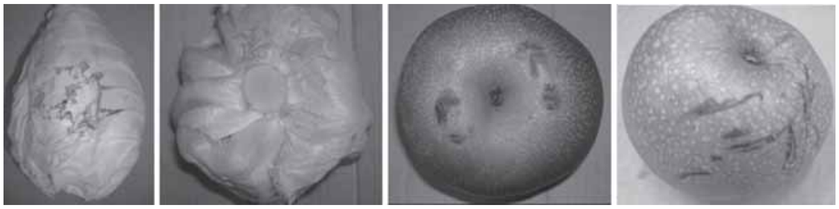

Figure 8. *Physical damage of selected produce after truck transportation.*

primary causes of damage. Mechanical injury is a known cause of rapid deterioration of produce. This is a significant parameter in produce packaging design. It is therefore critical to develop better produce protective solutions for post-harvest handling of produce to reduce the amount of downstream packaging materials, and reduce the burden of packaging materials in today's requirements to reduce packaging and be more sustainable. Reduction in damage delivers more product with less packaging in the entire supply chain.

4.0 CONCLUSIONS

- Vibration levels measured in truck shipments in Thailand showed the highest levels in vertical direction, followed by lateral and longitudinal orientations.
- The highest level of vibration occurred from the grower to packing houses, due to the poor road conditions.
- The highest level of damage occurred between the fruits and vegetables being transported to the packing houses.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the financial support and in-

Table 4. *Percentages Loss of Various Produce After Actual Shipments.*

	Percentages Damage		
Produce	**At Packing House**	**At Distribution Center**	**At Retailers**
Head lettuce	45	30	10
Cabbage	50	40	15
Chinese pear	39	29	21
Chinese plum	15	10	5

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Enhancement of Oxygen Barrier with Polymer Clay Nanocomposite Coatings on Polypropylene Treated with Atmospheric Pressure Plasma

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ABSTRACT: Production of polymer clay nanocomposite (PCN) materials using hydrophobic polymers such as polyolefins can be difficult due to hydrophilic nature of clay. Nano-composite coatings bonded to polyolefins may offer a simpler method of achieving higher barrier properties for these materials. Atmospheric pressure plasma (APP) offers opportunities for modifying surface tension and thus enhancing bonding of coatings to polyolefins.

Mixtures consisting of synthetic layered silicate in polyvinyl alcohol (PVOH) were applied to atmospheric pressure plasma treated, semi-rigid polypropylene (PP) samples. Atmospheric pressure plasma treated (50/50 air/nitrogen) and coated samples were characterized by atomic force microscopy (AFM), scanning electron microscope (SEM) and wide angle diffraction (WAXRD). Results showed that APP treatments enhance surface roughness, which contributed to enhanced bonding of coatings. Wide angle diffraction confirmed exfoliation of Laponite nano-particles on coated samples. Due to the exfoliation of clay platelets and good adhesion, oxygen transmission rate (OTR) values of PP were reduced from 150 to 10 cc/m^2/day when coated with PCN solution containing 50 wt% clay. This work demonstrates potential for PCN coatings to enhance barrier performance of PP.

INTRODUCTION

POLYMER clay nanocomposites (PCN) have been widely studied as property enhancers for polymers due to their superior properties such as mechanical strength [1,2], thermal stability [3,4] and barrier

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properties [5,6]. Enhancing oxygen barrier has been achieved by maximizing the path length required for oxygen to pass, which is referred to as a tortuous path. Clay nanoparticles mixed into polymers helps to provide such a tortuous path. Gas molecules enter at one surface, diffuse through the interior of a packaging film and finally exit at the other side. Gas molecules diffuse into amorphous regions of polymers, which have lower chain packing densities than crystalline regions. Small intermolecular pores introduced by the random entanglements of polymer chain in amorphous regions allow gas molecules to diffuse through films [7]. In the case of polyethylene (PE), the size of this small intermolecular pore is below 1 nm above the glass transition temperature (T_g) and the number of these pores is estimated to be about 10×10^{21}/cc. Van der waals diameters for nitrogen, oxygen, and carbon dioxide are 0.402, 0.375, and 0.414 nm, respectively [8]. Diffusion rates of the oxygen gas molecules through PE film are relatively high. Thus, increasing crystallinity is one of the most effective ways to reduce diffusion rates of gas molecules [18]. Highly dispersed clay nanoparticles can mimic increased polymer crystallinity resulting in greater gas barrier properties.

Dispersion of layered silicates in a polymer matrix can be classified as non-intercalated, intercalated and exfoliated. Non-intercalated structures occur when polymer chains cannot move into the intergallery region between nanoparticles. This is undesirable for barrier properties because nanoparticles essentially clump together and behave as fewer larger particles that do not effectively increase diffusion path length. Intercalated structures are formed when there is some penetration of polymer chains into the intergallery with fixed spacing. Exfoliated structures involve complete separation of particles into random arrangements (Figure 1) [9,10]. Fully Exfoliated structures are the ideal arrangement of clay platelets. Exfoliation offers superior physical, mechanical and

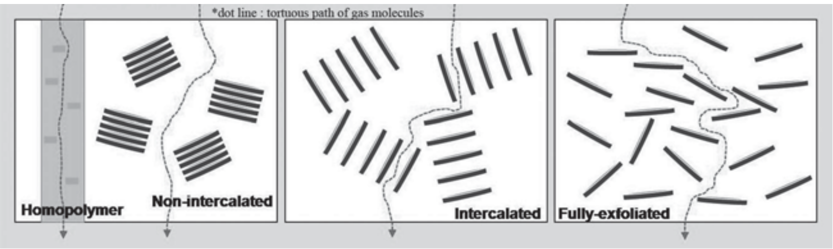

Figure 1. *Typical structures of layered silicate in polymer matrix; (a) Non-intercalated, (b) Intercalated and (c) Exfoliated structure.*

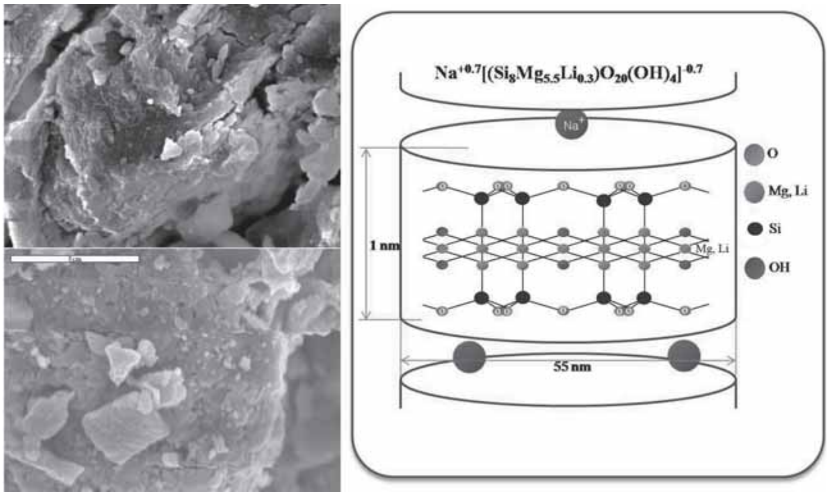

Figure 2. *SEM micrographs and structure of Laponite JS.*

barrier properties. Exfoliation requires relatively high shear forces and modification of interlayer to separate particles completely [11,12].

Once fully-exfoliated structures are obtained, maintaining the exfoliated state without aggregation is another challenge. Additionally, obtaining fully-exfoliated PCN in non-polar polyolefin polymers is difficult because silicate based clay is polar and therefore, incompatible with non-polar polymers such as polyolefins. Research using organo-modified clay has been focused on mitigating this limitation [13–15].

In this study, Laponite JS (Southern Clay Products, Louisville, KY) in polyvinyl alcohol were used to produce PCN coatings. Laponite JS is a synthetic layered fluorosilicate modified with an inorganic polyphosphate dispersing agent. Figure 2 shows SEM micrographs and the structure of Laponite JS. This clay is hectorite, prepared in a reaction between Mg, Li, and Na-silicate salts, which results in partially crystalline, mono-dispersed powder with particle sizes of about 0.92 nm thick by 25 nm diameter (aspect ratio of 25–35), bulk density of 950 kg/m^3 and specific surface area of about 300 cm^2/g [9]. Laponite JS hydrates and disperses in water to give virtually clear and colorless colloidal sols of low viscosity. Therefore, concentrations of Laponite colloidal solutions of up to 18 wt% can be stored for about one month.

Polyvinyl alcohol (PVOH) is a water-soluble polymer that is known to be non-toxic, biodegradable, and "generally recognized as safe"

(GRAS) by the United States Food and Drug Administration (FDA). PVOH has been used as a coating material, stabilizer, polarizing film, sizing agent, adhesive, drug delivery system, contact lens material, basic bio-material with expanding applications in nano-technology [16].

When protected from water, PVOH offers excellent oxygen barrier performance for packaging materials by itself. However, polyethylene vinyl alcohol copolymer (EVOH) is most often in packaging applications in multilayer packaging film structures. Excellent oxygen barrier properties can be attributed to high crystallinity and large cohesive energy caused by highly polar hydrogen bonding. PCNs involving clay nano-particles in PVOH have been studied [17–22]. While wet, PVOH nano-composites demonstrate excellent particle dispersion in gels. This state represents a true nano-scale organic-inorganic hybrid material. However, a drying process causes portions clay layers to aggregate. Steric constraints of PVOH impede complete aggregation of clay, allowing various degrees of dispersion depending on the processing methods used for the exfoliation and the nature of clay such as type of clay, aspect ratio, and cation exchange capacity (CEC) in the PCN system [22].

Nano-composite preparation techniques are designed to create amorphous domains with uniformly distributed mineral layers, but preparation of PVOH/clay nanocomposites from solutions is difficult because of aggregation of clay [23]. To prevent aggregation, Yeun, et al. [17] prepared PVOH/Saponite nano-composites with various clay concentrations successfully. Oxygen permeability values were reported to decrease with increasing clay loading within 0–10 wt%, while optical properties remained nearly constant.

A major limitation of PVOH as a nano-composite matrix polymer is the polar nature of hydroxyl (–OH) groups that make PVOH hydrophilic and soluble in water. Small amounts of water, particularly relative humidity above about 35%, plasticize PVOH resulting in dramatic loss of oxygen barrier properties [32,33]. A variety of techniques used to reduce oxygen permeability under high relative humidity have been developed by several research groups. One of the most common is sandwiching the water-sensitive polymer between hydrophobic layers such as polypropylene (PP) and/or polyethylene (PE) [24,25]. However, laminated structures are expensive and may suffer from poor flexibility.

Polypropylene is an attractive packaging material because it is light, cheap and offers good overall mechanical properties. However, PP suffers from low oxygen barrier properties. Improvement of barrier properties of PP without creating expensive laminate structures may enhance the usefulness of the polymer. Therefore, this work focused on development of techniques to produce and apply nano-based barrier coatings to PP.

Critical to this work was enhancement of bonding between the hydrophobic substrate and the hydrophilic PVOH/Laponite coating. Modification of the surface of PP was required to achieve strong adhesion. In general, the adhesion property at the interface is determined from micro interactions such as intermolecular van der waals force, hydrogen bonding, electrical properties, acid-base chemistry and mechanical effects such as dispersion and anchoring. Various techniques have been proposed to modify the surface of polymeric materials such as corona discharge, ultra-violet, plasma and chemical modification [26–29,35]. Plasma treatment is growing in popularity due to its flexibility and uniformity [29,35].

Plasma is an ionized gas, which is the fourth state of matter and constitutes more than 99% of the universe. Plasma is created by applying energy to a gas in order to reorganize the electronic structure of the species and to produce excited species and ions. Plasma usually can be classified by the type of energy supply and the amounts of energy transferred to the plasma and the resulting properties of the plasma change, in terms of electronic density and temperature. Recently, atmospheric pressure plasma (APP) has become commercially available [30–31,34]. In this work, APP treatments were used to enhance bonding of coatings.

MATERIALS AND METHODS

Materials

Polymer clay nano-composite solution was made with synthetic layered silicate known as Laponite JS (Southern Clay Products, Louisville, KY). To evaluate effects of molecular weight (MW) and degree of saponification (DS) on coating adhesion and oxygen barrier, PVOH provided by Celanese Company (Dallas, TX) and Scientific Polymer Product Co. (Ontario, NY) with different properties were used (Table 2). Organic ammonia chloride (OAC), [2-(Acryloyloxy)ethyl]-trimethyl ammonium chloride was used for intergallery modification.

Preparation of PCN solutions

An OAC buffer solution of 1 wt% relative to clay was added into a corresponding amount of deionized water at 40°C with pH 10. The solution was premixed for 2–3 minutes with a magnetic stirrer. Laponite JS was added to this solution ("clay solution"). Prepared clay solutions were placed at room temperature for about one day to obtain good ion-exchange in the intergallery of clay platelets.

Polymer stock solutions were prepared by dissolving PVOH in deionized water at elevated temperature and stirring with magnetic stirrer for at least 6 hours. Prepared PVOH stock solution was added to clay solutions and mixed for 1 hour with a magnetic stirrer and then mixed with a high shear mixer (KadyMill-L, Kady® International, Scarborough, ME). Prepared PCN solutions were placed in a hood at ambient temperature for 3–10 hours depending on viscosity in order to allow entrained air bubbles to dissipate.

Coating Samples after APP Treatment

Atmospheric pressure plasma treatments were performed using a Dyne-A-Myte VCP (Enercon Industries Co., Menomonee Falls, WI). Air and nitrogen gas (about 50/50) were used to create plasma. Controllable variables when treating surfaces were flow rate of nitrogen gas and distance between APP applicator tip and sample surface. Table 1 lists specific conditions used in this study. Surfaces of rigid PP were spin coated with 20g PCN solution at fixed spin-coating parameters involv-

Table 1. *Specific Conditions of APP Treatment.*

Condition No.	N_2 Flow Rate (l/min)	WD (mm)	Treatment Times	Belt Speed
APP-Cond01	25	20	4	20
APP-Cond02	20	20	4	20
APP-Cond03	18	20	4	20
APP-Cond04	15	20	4	20
APP-Cond05	12	20	4	20
APP-Cond06	20	30	4	20
APP-Cond07	20	25	4	20
APP-Cond08	20	20	4	20
APP-Cond09	20	10	4	20
APP-Cond10	20	5	4	20

Table 2. *Specific Data of PVOH Samples.*

Grade	DS	MW	Company
Celvol107	99.3% +	31,000–50,000	Celanese Company
Celvol425	95.5–96.5%	120,000–150,000	Celanese Company
PVOH-PH	88%	120,000–150,000	Scientific Polymer Product Co.

1) High DS: 99.3 +.
2) Fully saponified: 93–98.
3) Intermediate saponified: 91–93.
4) Partially saponified: 88.

ing a two-step process involving first, 500 rpm for 1.5 min, followed by 700 rpm for 30s. The spin coater was a Model Laurell WS-400B-6NPP/LITE (Laurell Technologies Co., North Wales, PA). All Samples were dried at 25°C in a vacuum desiccator.

Characterizations

Morphological effects of APP surface treatments were analyzed using scanning electron microscopy (SEM). Specimens were cut into 40 × 10 mm^2 pieces and washed with acetone. Surfaces were sputter coated with gold-palladium alloy (Au-Pd) for 30 + 30s. Morphologies of samples were observed at the magnification of 11,000 using a Model JSM-6400SEM (Jeol Ltd., Tokyo, Japan).

Micrographs of submicron-detailed surfaces were obtained under ambient conditions using atomic force microscopy (AFM) (Digital Instruments Dimension 3100,Veeco Instruments., Plainview, NY) by contact mode.

Oxygen transmission rates of all uncoated and PCN-coated samples were measured in accordance with the procedure described in ASTM D-3985 using a Model OX-TRAN 2/20MH (Mocon Corporation, Minneapolis, MN). Permeation cell area was 50 cm^2.

Degree of exfoliation was measured by analyzing changes in intergallery spacing of clay platelets. The change in spacing was measured using an wide angle diffraction (WAXRD) (Philips XRD APD 3720 powder diffractometer, Philips Electronics, Mahwah, NJ) with a rotation anode and CuKα radiation ($\lambda = 1.54056$ Å; scanning range from 1.01° to 69.99°). Clay powder was mounted on a sample holder with a large cavity and a smooth surface was obtained by pressing the particles with a glass plate. Samples for PCN solution were prepared by spin-coating using a silicon wafer as a substrate.

RESULTS AND DISCUSSION

Surface Modification by Atmospheric Pressure Plasma Treatment

Degree of surface modification caused by APP is determined by several important factors such as plasma energy source, which can be classified regarding their excitation mode, exposed area energy density, and collision time and intensity of the electron energy source [34]. In this research, air and nitrogen gas were used as a plasma source and the flow rate of nitrogen gas and distance between tip and sample were controlled to investigate effects of APP treatment on surface modification as well as resulting oxygen barrier property. Resulting SEM micrographs of APP treated and untreated PP at 6k magnification are shown in Figure 3(a)–(d). Enhanced roughness was observed microscopically depending on the APP treatment parameters. Less surface texture was observed for working distances (WD) at 3 cm or greater as shown in SEM micrograph [Figure 3(b)] and this can be identified in Root Mean Square (RMS or Rq) value analysis shown in Figure 8.

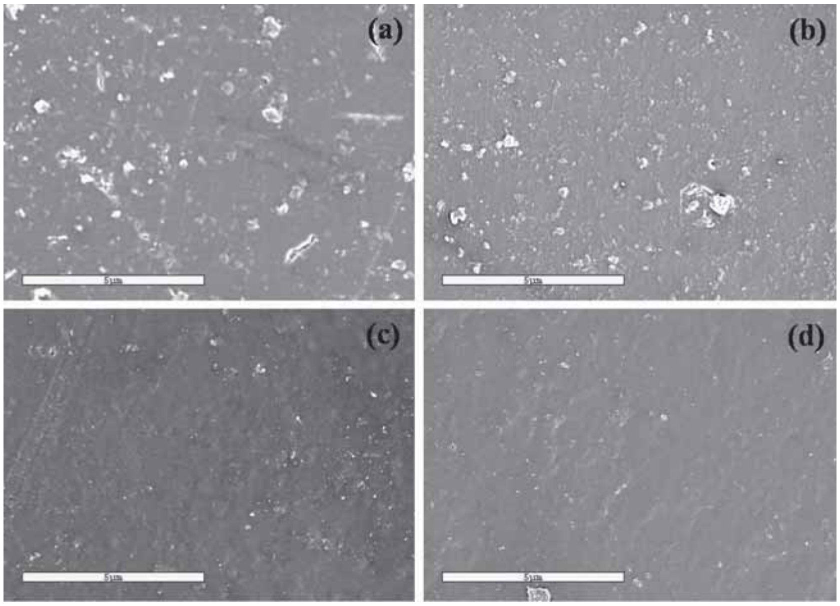

Figure 3. *SEM micrographs taken at 6000× of untreated surface (a) and APP-treated surfaces by APP-Cond06 (b), APP-Cond01 (c) and APP-Cond09 (d).*

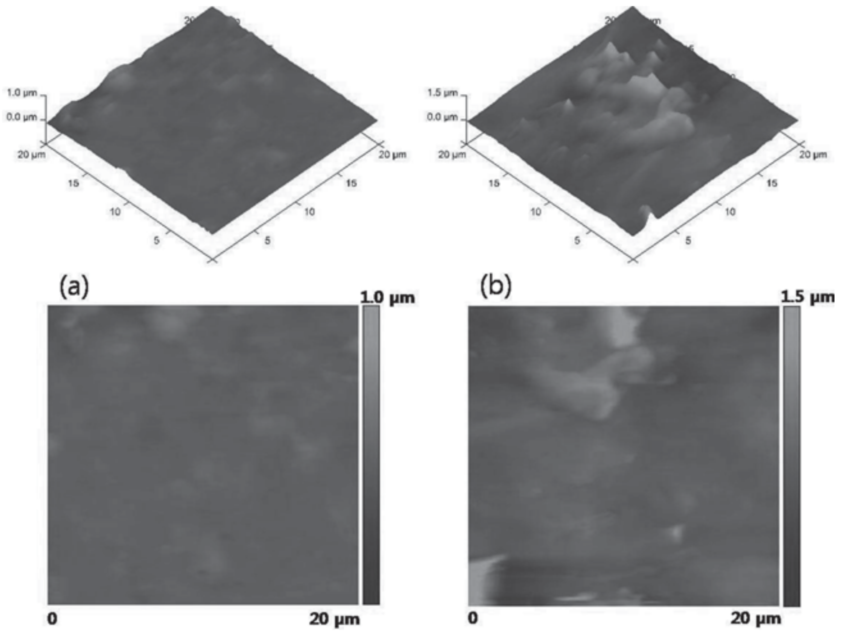

Figure 4. *3D surfaces and heights taken by AFM (contact mode) on a 20 x 20 µm^2 of untreated surface (a) and APP-treated surface by APP-Cond09 (b).*

Surface topography of nitrogen plasma PP substrate measured by AFM on a 20 × 20 µm^2 area is shown in Figure 4. Non-treated surfaces in Figure 4(a) appeared to be flat and smooth while APP treated surfaces appear to have surface contours [Figure 4(b)]. Figure 5 and 6 show effects of APP parameters on surface roughness. These figures are 3-dimensional heights taken by AFM on a 50 × 50 µm^2 area of (a) untreated surface and (b) APP treated by App-Cond01 to (k) App treated by App-Cond10 as listed in Table 1. More surface contours were observed in (c)–(e) and (h)–(j) when comparing others.

For quantitative analysis of surface roughness of these topographical images, R_a, R_{max} and RMS values taken by AFM were used. R_a and RMS are both representations of surface roughness, but each is calculated differently. R_a is calculated as average roughness (Equation 1) of measured microscopic peaks and valleys. RMS is calculated as the root mean square of measured microscopic peaks and valleys (Equation 2). Each value uses the same peak and valley dimension measurements. A single large peak or flaw within the microscopic surface texture will affect the RMS value more than the R_a value. R_{max} is defined as a maximum height

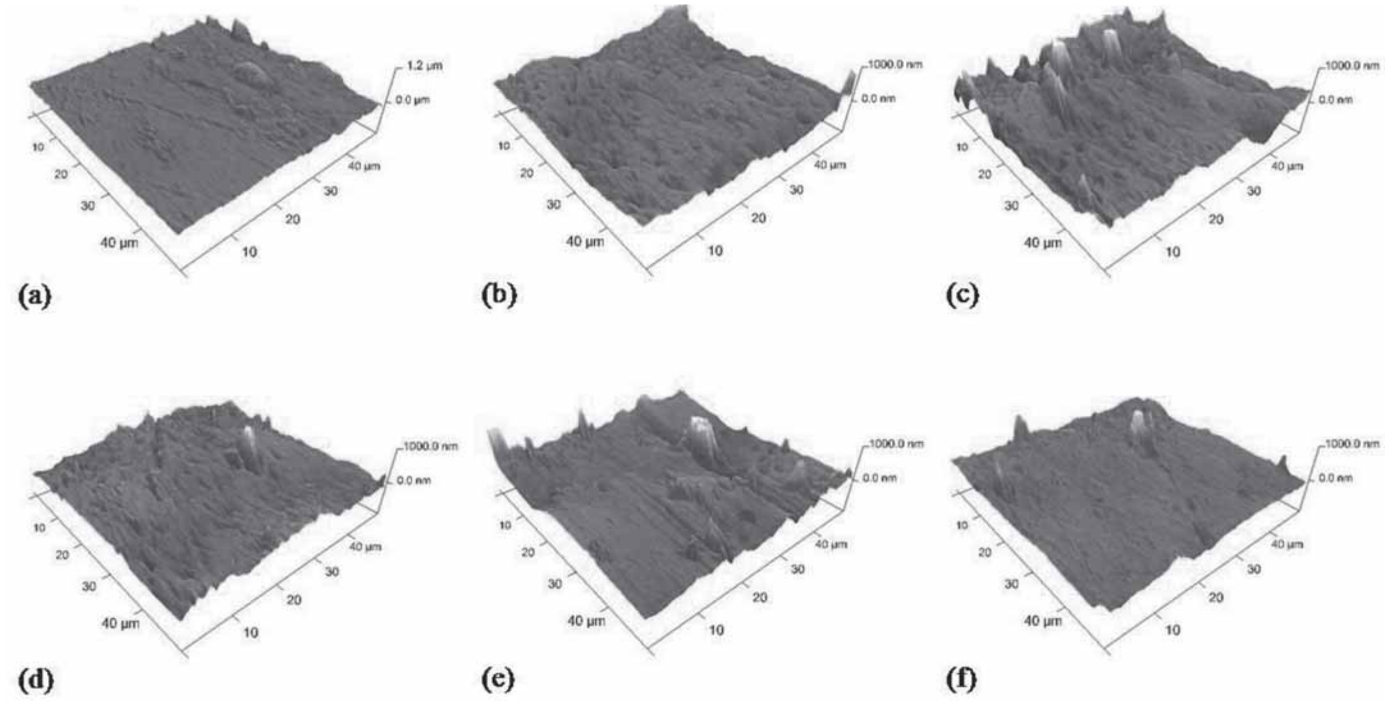

Figure 5. *Influence of N_2 flow rate; 3D surface images taken by AFM (contact mode) on a 50 x 50 μm^2 of untreated surface (a) and APP-treated surface by APP-Cond01 (b), 02 (c), 03 (d), 04 (e) and 05 (f).*

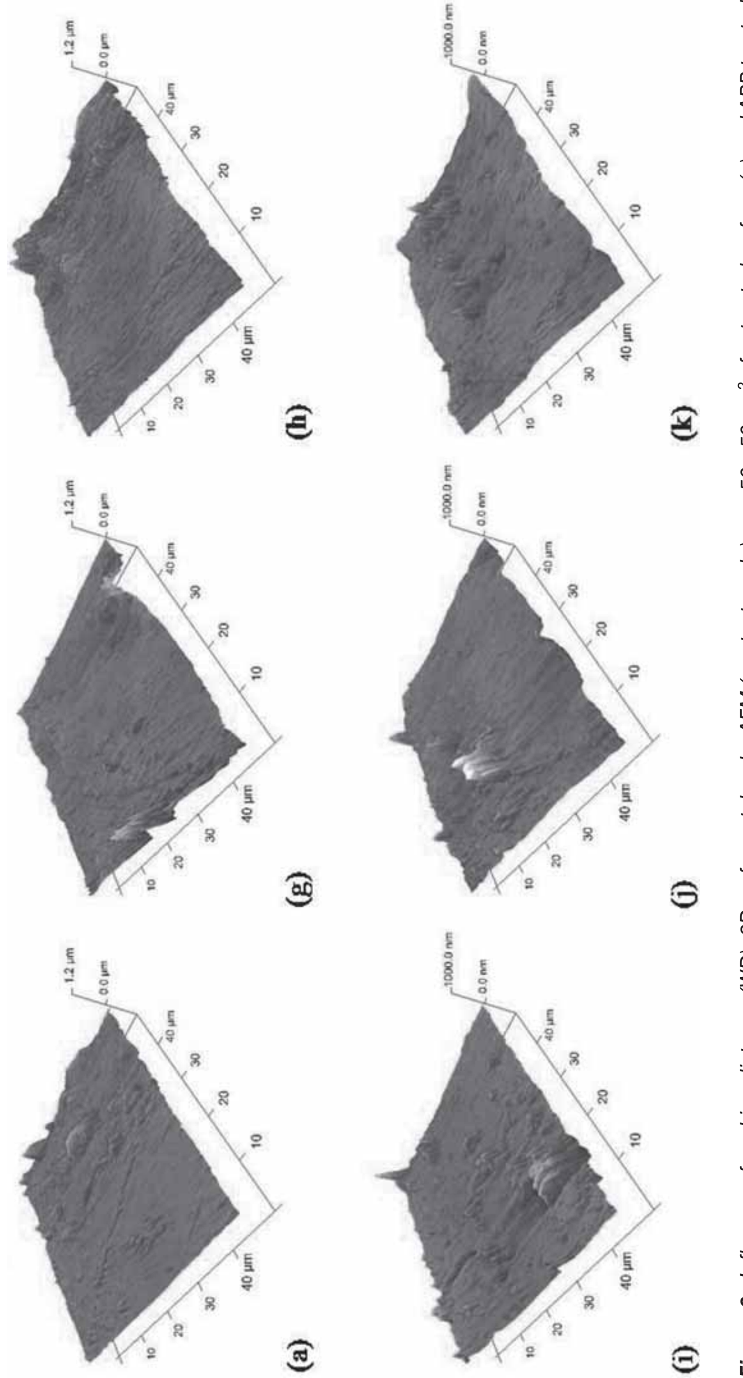

Figure 6. *Influence of working distance (WD); 3D surfaces taken by AFM (contact mode) on a 50 x 50 µm^2 of untreated surface (a) and APP-treated surface by APP-Cond06 (g), 07 (h), 08 (i), 09 (j) and 10 (k).*

representative of a difference between a highest point and a lowest point [34–36].

$$R_a = \left(\frac{1}{L}\right)\int_0^L |Z(x)|\, dx \tag{1}$$

$$R_q = \left[\left(\frac{1}{L}\right)\int_0^L (Z(x)^2\, dx)\right]^{1/2} \tag{2}$$

Where,

L = evaluation length,
$Z(x)$ = the profile height function

The largest RMS value of 132 nm was obtained at 18 l/min nitrogen (Figure 7). No additional roughness was measured at greater nitrogen flow rates. Therefore, 18 l/min nitrogen was used for APP treatments for this study. Increased surface roughness by APP has been attributed to surface activation involving grafts of active chemical functions onto the surface [34]. Working distance was the distance between the plasma jet tip and the sample. WD was determined to be critical for effectiveness. Figure 8 shows increased surface roughness at WD of 20 mm.

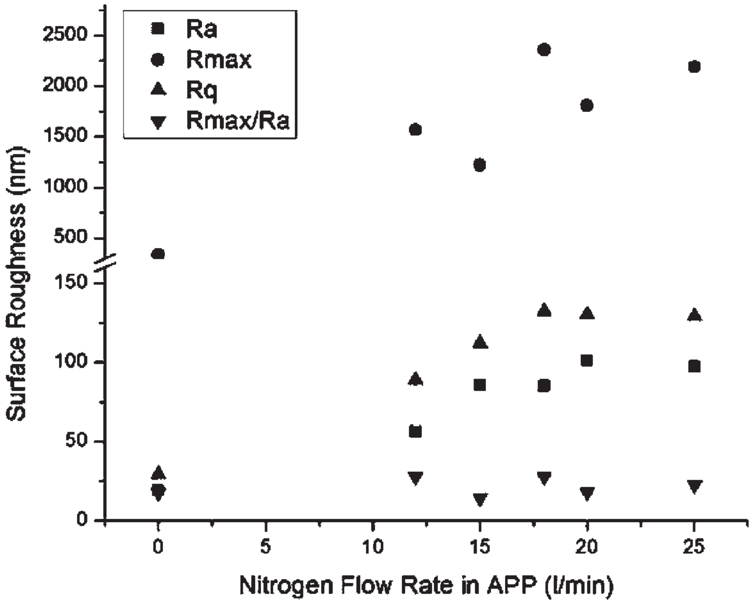

Figure 7. Influence of N_2 flow rate on surface roughness.

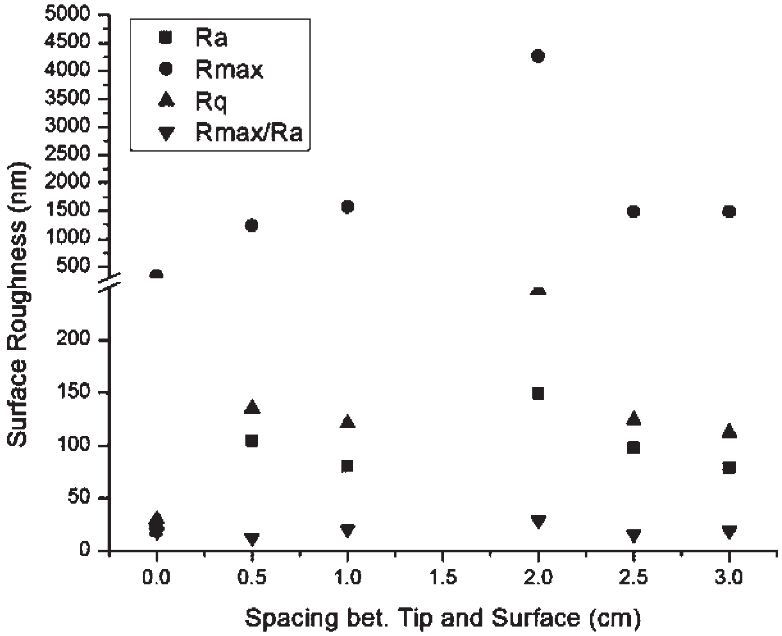

Figure 8. *Influence of WD on surface roughness.*

Clay Exfoliation

Degree of exfoliation depends on how platelets are modified and how well the intergallery is opened. Because of the small intergallery distance, high shear processes are needed to achieve exfoliation. Each platelet peels away from a stacked clay layer via the process of exfoliation by high shear in the polymer matrix. Wide angle x-ray diffraction pattern of pure Laponite JS is shown in Figure 9. As a silicate material, Laponite exhibits low angle peaks, much like natural montmorillonite clay. Two high angle peaks were also observed for Laponites JS at about 48 degrees and 62 degrees. Therefore, according to Bragg's law ($d = n\lambda/2\sin\theta$), $d = 1 \times 1.54/2 \sin(48/2) = 1.89$ Å and d-spacing for 62 degree $= 1 \times 1.54/2 \sin(62/2) = 1.50$ Å. These d-spacings should correspond to the lattice spacings of the crystalline unit cells. The two typical peaks of Laponite JS at 48 and 62 degrees disappeared on curves in Figure 9 for exfoliated LP20PVOH, abbreviated as 'LP20PVOH-EX' because of the exfoliation of the silicate or high disorder of the clay platelets [39]. However, a sharp strong peak at 48 degrees was shown at the unexfoliated

LP20PVOH, abbreviated as 'LP20PVOH-UE', meaning silicate layers were not delaminated nor dispersed.

XRD patterns of PCN showed no peak between 40 and 70 degrees and signal intensities showed a broad hump between 60 and 63 degree for LP20PVOH, LP30PVOH and LP50PVOH. This can be explained by exfoliated silicate layers of the clay mineral showing characteristic XRD pattern of amorphous material when platelets are randomly dispersed. This pattern is a characteristic XRD pattern of a clay-exfoliated type of polymer-clay nanocomposite [15]. The more clay added in PCN, the more distinctive the hump observed by XRD. However, if clay platelets fully exfoliated, d-spacing cannot be detected by XRD because d-spacing would become too large.

Oxygen Transmission Rate (OTR)

Polyvinyl alcohol as a coating material for food packaging materials to reduce oxygen permeability has several advantages in terms of its structural properties. Large inter- and intramolecular cohesive energy resulting from highly polar hydrogen bonding provides PVOH with excellent oxygen barrier properties at low relative humidites. Due to a combination of strong hydrogen bonding and high degree of crystallinity, PVOH has lower OTR values when compared to polyethylene terephthalate (PET) [40]. Clay platelets with large surface area and high aspect ratio were expected to show increased oxygen barrier

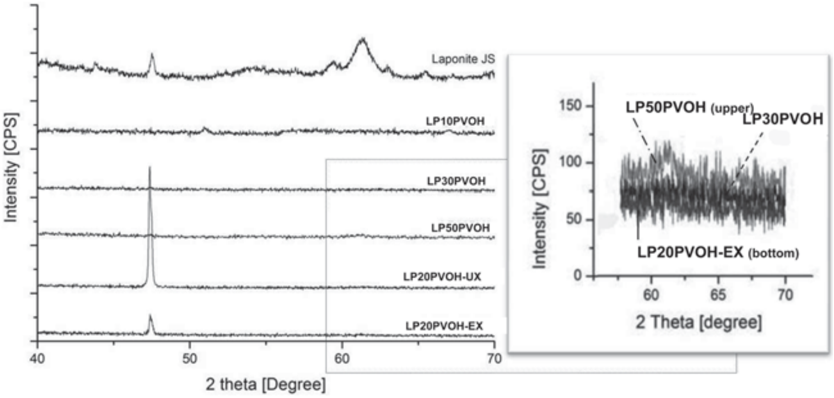

Figure 9. *WAXRD patterns of pure Laponite JS powder and PCNs with exfoliated silicate layers in PVOH matrix.*

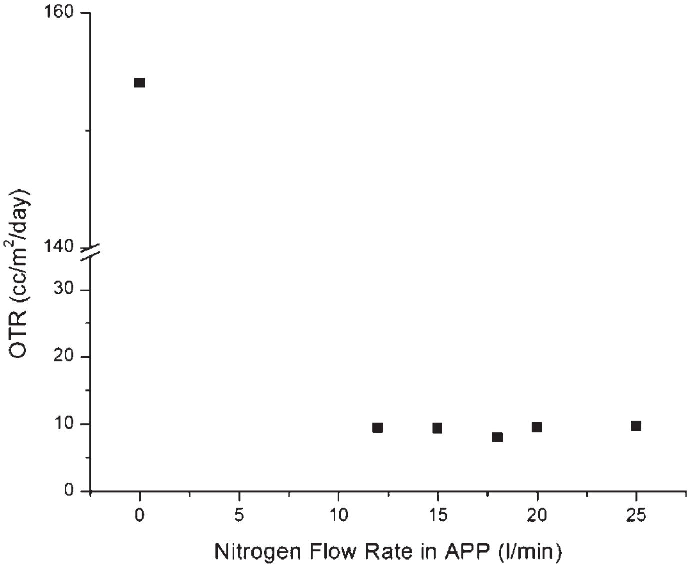

Figure 10. *Effect of N_2 flow rate on OTR.*

properties in much the same way as increasing crystallinity of the polymer [41,42]. Lowering OTR values usually can be achieved by virtue of greater tortuous paths for diffusants in fully exfoliated structures. Figures 10 and 11 illustrate effects of parameters of APP treatment on OTR values. Similar tendencies were observed as a result of surface roughness. Figure 10 illustrates effects of nitrogen gas flow rate on the OTR values. Figure 10 shows that flow rate of nitrogen gas did not affect OTR, which was consistently in the range of about 10 cc/m^2/day. Figure 11 shows effects of working distance on surface roughness. Working distance was shown to be critical. Poor results were seen when the plasma head was too close or too far away from test specimen (Figure 11).

Once high degrees of exfoliation were obtained, oxygen barrier properties were dominated by adhesion strength between PCN-coated layer and the surface of substrate. Therefore, it was found that quality of surface treatment plays a significant role in determining extent of barrier achieved with PCN barrier coatings.

Figure 12 indicates OTR was a function of clay concentration. Greatest barrier properties indicated by lowest OTR values were obtained at 50 wt% of clay in dried films. No significant difference in OTR values were observed between 20 to 40 wt% of clay concentration. Addition-

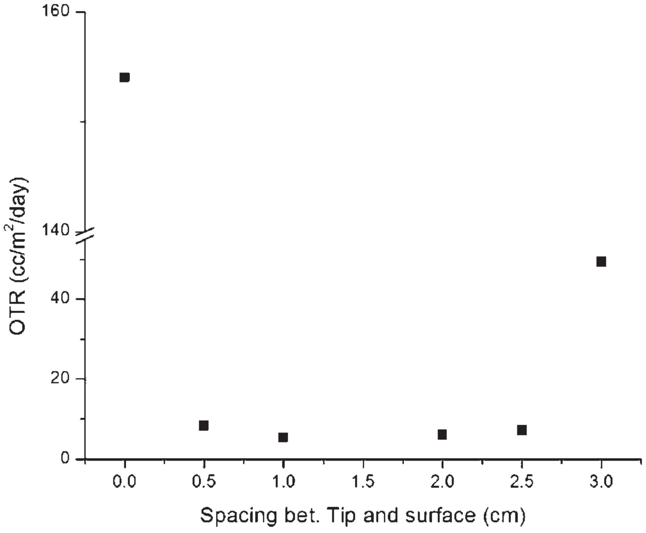

Figure 11. *Effect of the WD on OTR.*

ally, dramatic increases in OTR values were observed for loadings in excess of 50 wt%. This may be due to high degrees of aggregation of clay resulting in reduced effective surface area per unit concentration as well as lower degrees of exfoliation caused by steric interference [13,37,38].

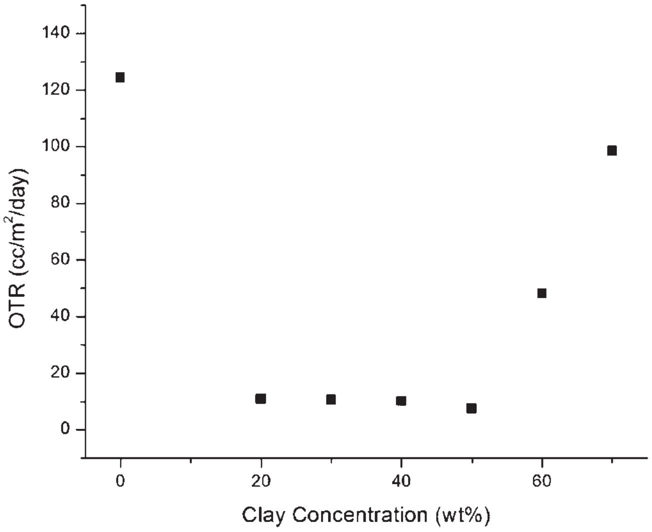

Figure 12. *Dependence of clay content on OTR.*

Comparisons of two different PVOH sources with high (Celenese) and lower DS (Scientific Polymer Product Co.) without clay showed that higher DS provided better barrier properties than low DS PVOH.

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Table 5. Comparison of state-of-the-art matrix resins with VPSP/BMI copolymers.

Resin System	Core Temp. (DSC peak)	Char Yield, %
Epoxy (MY720)	235	30
C379: H795 = 1.4	285	53

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