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.

Editor

Jay Singh
Industrial Technology
Cal Poly State University
San Luis Obispo, CA 93407, USA
Phone: 805-756-2129, Email: singhjapr@gmail.edu

Editorial Advisory Board

JOURNAL OF APPLIED PACKAGING RESEARCH—Published quarterly—January, April, July and October by DEStech Publications, Inc., 439 North Duke Street, Lancaster, PA 17602-4967.

This journal is recommended by The National Institute of Packaging Handling and Logistics Engineers (www.niphle.org).

Indexed by Chemical Abstracts Service.

Indexed and abstracted by Pira International.

Subscriptions: Annual $299 (Print), $299 (Electronic) and $324 (Print and Electronic). Single copy price $89.50. Foreign subscriptions add $45 per year for postage.

(ISSN 1557-7244)

DES*tech* Publications, Inc.
439 North Duke Street, Lancaster, PA 17602-4967, U.S.A.

CONTENTS

Research

High Permeable Films Used for Modified Atmosphere Packaging Improve Quality and Shelf Life of Baby Corn

VANEE CHONHENCHOB[1,*], WANNEE CHINSIRIKUL[2],
ASIRA FUONGFUCHAT[2], SAOWAPA CHAIWONG[2],
KANCHANA BOONRUANG[1,2] and NOPPADON KERDDONFAG[2]
[1]*Department of Packaging Technology, Kasetsart University, Bangkok 10900, Thailand*
[2]*National Metal and Materials Technology Center, National Science and Technology Development Agency, Pathumthani 12120, Thailand*

ABSTRACT: This study presents the optimum controlled modified atmosphere (CA) conditions for baby corn and their applications in film selection for modified atmosphere packaging (MAP). The optimum CA condition for baby corn stored was shown to be 2–5 kPa O_2 + 5 kPa CO_2. Baby corn (~ 250 g) were packaged in the bags (15 cm × 23 cm) made of various films (PE-1, PE-2, PE-3, and PP) ranging in oxygen (OTR) and carbon dioxide (CO_2TR) transmission rates and permselectivities (PCO_2/PO_2, β) and stored at 5 and 10°C. Treatments were evaluated for changes in color, firmness, total soluble solids, weight loss, and percent decay. Shelf life was determined using sensory scores. The highest CO_2 accumulation was observed in the PP packages resulting in severe browning and fermentation. Low O_2 concentrations were observed in all packages. All of the high gas permeable films created equilibrium modified atmospheres. PE-3 (OTR = 16,826 $cm^3\ m^{-2}\ 24\ h^{-1}$; CO_2TR = 51,328 $cm^3\ m^{-2}\ 24\ h^{-1}$; $\beta = 3.05$) maintained a package atmosphere at 2 kPa O_2 + 4 kPa CO_2 as recommended in the CA study and maximized shelf life of baby corn stored at 10 and 5°C to 21 and 15 days, respectively, as compared to 6 days for PP packaged baby corn. Chilling injury occurred in baby corn packaged in PE-3 after storage at 5°C for 15 days.

INTRODUCTION

MODIFIED atmosphere packaging (MAP) is an effective method for prolonging the shelf life of intact and fresh-cut fruits and vegetables. MAP prolongs shelf life by reducing the respiration rate and metabolism of produce [1]. The establishment of the desired atmosphere in

---

[*] Author to whom correspondence should be addressed. Email: vanee.c@ku.ac.th

1557-7244/09/02 057-18

the package depends on various factors including the respiration rate of produce, the gas permeability of film, and the ambient environment [2]. MAP design involves the study of the optimum controlled atmosphere (CA) conditions. Desired modified atmosphere can be established by matching the film permeability with the produce respiration rate. The ratio of CO_2/O_2 permeability coefficient, defined as film permselectivity (β) is also one of the key controlling factors for MAP systems. Modifying film permselectivity effects the equilibrium gas composition of the MAP systems of produce [3]. The use of polymeric films to extend shelf life of fresh cut products through a modification of the atmospheric conditions has been reported in literature [4,5]. However, the use of these films is restricted due to a lack of commercially available polymeric films with a wide range of permseletivity. The polymer group at National Metal and Materials Technology Center (MTEC), Thailand has developed 'high gas permeable films' of polyethylene (PE) based resins with optimized optical and mechanical characteristics desirable for fresh produce packaging [6]. The primary objective for developing these 'high gas permeable films' includes both high oxygen transmission rate property and a wider range of permselectivity of the films over the available commercial films. The high gas permeable films ranging in O_2 and CO_2 transmission rates and permselectivity allow optimized equilibrium modified atmosphere (EMA) to be established inside the packages suitable for various tropical produce.

Baby corn is consumed worldwide. Demand for fresh-cut produce, including fresh baby corn, is increasing rapidly in the global market due to its convenience and fresh-like characteristics [7]. For baby corn, the postharvest and processing steps, such as husk removal and cutting, result in tissue damage. This physical damage leads to rapid changes in quality and limits the shelf life of fresh-cut produce [8]. Benefits of modified atmosphere packaging in maintaining quality and extending shelf life have been reported for various fresh-cut produce [1]. However, limited research is available for extending the shelf life of fresh baby corn.

The primary objectives of this study were to determine the optimum controlled atmosphere conditions for baby corn and to study the effect of high gas permeable films towards maintaining quality and extending shelf life of baby corn stored at 5°C and 10°C. The effect of O_2 and CO_2 transmission rates and permselectivity on establishing the equilibrium modified atmosphere conditions in the high gas permeable packages was also investigated.

MATERIALS AND METHODS

Baby Corn

Baby corn (*Zea mays* Linn., var. Zeba SG 17) was obtained from a commercial producer. This is the major variety currently being exported from Thailand. Husks were removed prior to use. Baby corn uniform in size (100 mm in length and 10 mm in diameter at the base) and free from defects, decay and browning was washed by immersion in a chlorine solution (150 ppm, 2 minutes) prior to CA or MAP storage.

Respiration Rate Measurement

Samples (150 g) were placed in 750-mL glass jars. Respiration rate was measured using an air flow-through system (150 $cm^3 min^{-1}$). Carbon dioxide production was measured during storage at 5, 10, and 20°C using an Agilent 6890 gas chromatograph (Agilent Technologies Inc., USA) equipped with a thermal conductivity detector (TCD) at 200°C and a Pora Plot Q column.

Controlled Atmosphere Storage of Baby Corn

Approximately 600 grams of baby corn was placed in the 3,200-mL glass jars and stored at 10°C. Each glass jar was closed with a rubber lid containing one inlet and one outlet tubes connected to the integrated CA controller system ICA 61 (International Controlled Atmosphere Ltd., UK). The system was connected to compressed oxygen and nitrogen tanks. Gas mixtures tested in this study were 2 kPa O_2 + 5CO_2, 2 kPa O_2 + 15CO_2, 5 kPa O_2 + 5CO_2, and 5 kPa O_2 + 15CO_2, compared to normal air. The gas mixtures compared in this study were obtained from the preliminary study in this laboratory where the O_2 and CO_2 tolerance and their effects on the quality of baby corn were assessed. Results from the tolerance study showed that baby corn stored at 0 and 5 kPa O_2 under normal CO_2 and 5 and 15 kPa CO_2 under normal oxygen conditions had the best quality throughout storage. Baby corn was sampled every 3 days for quality assessment. The optimum controlled atmosphere conditions were selected to use for MAP study. The experiments were carried out in triplicate.

Modified Atmosphere Packaging of Baby Corn

Two hundred and fifty grams of baby corn was packaged in the bags (15 cm × 23 cm) made of various polymeric films. The samples were stored at 5 and 10°C and randomly selected for quality assessment during storage. The experiments were done in triplicate. The polymeric films used in this study were 4 types: (a) polypropylene (PP, control) commonly used as packaging for baby corn, and (b) three films with an oxygen transmission rate (OTR) 3–4 times higher than that of PP. These three high oxygen permeability films, denoted as PE-1, PE-2, and PE-3 with increasing OTR, respectively, were the polyethylene (PE) based films, developed at National Metal and Materials Technology Center (MTEC), Thailand. These films were different in their microstructures or morphologies, as influenced by the film's thicknesses and compositions, resulting in different film permeability and permselectivity. Water vapor transmission rates (WVTR) of the films were tested using a water vapor permeation analyzer (Illinois Instruments, Inc., USA). The transmission rates of carbon dioxide (CO_2TR) and oxygen (OTR) were measured using a Permatran C 4/41 and Oxtran 2/21 (Modern Controls Inc., USA), respectively. The transmission rates values and permselectivity ($PCO_2/PO_2,\beta$) of the films along with their thicknesses are presented in Table 1.

In-package Gas Composition Measurement

Changes of gas composition in the package headspace was measured using an Agilent 6890 gas chromatograph (Agilent Technologies Inc., USA) equipped with a thermal detector (TCD) at 200°C. Molecular Seive 5A and Pora Plot Q columns were used to separate O_2 and CO_2. Helium at 6 mL/min was used as a carrier gas. Modified atmosphere conditions in the baby corn packages were measured every 24 hours.

Determination of Quality and Shelf Life of Baby Corn

Baby corn was evaluated for quality changes during storage. All ears of baby corn in each treatment were assessed for weight loss, percentage decay, browning index, and overall liking. Two ears of baby corn in each treatment were randomly selected to evaluate for changes in color, firmness, and total soluble solids (TSS) content. Percentage decay was cal-

Table 1. *Carbon Dioxide Transmission Rates (*CO_2*TR), Oxygen Transmission Rates (OTR), and Permselectivity* (β) *at 25°C and 0% Relative Humidity and Water Vapor Transmission Rates (WVTR) at 25°C and 100% Relative Humidity of the Polymeric Films Used in this Study.*

Film	**CO_2TR (cm^3 m^{-2} 24 h^{-1})**	**OTR (cm^3 m^{-2} 24 h^{-1})**	**PCO_2/PO_2 (β)**	**WVTR (kg m^{-2} 24 h^{-1}**	**Thickness (μm)**
PP	6,024	2,733	2.20	0.0035	33 ± 1
PE-1	25,411	7,073	3.59	0.0083	61 ± 2
PE-2	38,900	12,153	3.20	0.0134	29 ± 1
PE-3	51,328	16,826	3.05	0.0180	26 ± 1

culated as: Σ(Number of baby corn with decay/Total number of baby corn) × 100%. Browning index was calculated as: Σ(Browning level × Number of baby corn at the browning level)/Total number of baby corn in the treatment. Browning level was determined based on the percentage of the total area as: 1 = No browning; 2 = 1–25%; 3 = 26–50%; 4 = 51–75%; 5 = 76–100%. An untrained panel of 30 people evaluated the sensory quality of baby corn for appearance, color, texture, odor, and overall liking using a hedonic scale (9 = excellent, 7 = good, 5 = average, 3 = fair, 1 = poor). Changes in color of the tip and cob of baby corn were measured using a CR-3500 Minolta Colorimeter (Minolta Inc., Japan) in Hunter Lab values. Tip area was defined as the area covering the length down of 1.5 cm and cob area as the remaining surface of the baby corn ears. Firmness (Newton) of baby corn tip and cob was measured using a Lloyd testing machine (Lloyd Inc., U.K.) equipped with the Volodkevitch bite jaws on two ears of baby corn selected at random from each treatment. Total soluble solids content of baby corn was measured using a hand refractometer (Atago Inc., USA). Shelf life of baby corn was considered as finished when the overall liking scored below 5. Total plate counts, mold and yeast counts, and *Escherichia coli* (*E. coli*) were conducted during storage according to the Association of Official Agricultural Chemists (AOAC) methods to evaluate microbiological quality of baby corn [9].

Statistical Analysis

Data were analyzed for multiple comparisons by analysis of variance (ANOVA) with Duncan's Multiple Range Test (DMRT) at a significant level of $^*P \leq 0.05$ using SPSS for Windows, Version 12.0.

RESULTS AND DISCUSSION

Respiration Rates of Baby Corn

Respiration rate decreased rapidly during the first 12 hours and changed slightly throughout storage. The average respiration rates of baby corn stored at 5, 10 and 20°C and expressed as CO_2 production were 64, 110, and 163 mg CO_2 kg^{-1} hr^{-1}, respectively. Respiration rates increased with increasing temperature. According to the classification described by Weichmann (1997), baby corn is among vegetables with extremely high respiration rate (above 100 mg CO_2 kg^{-1} h^{-1} at 10°C). Other commodities in this class include broccoli, peas, and sweet corn [10]. These relatively high respiration rates caused important quality changes of baby corn after harvest.

Optimum Controlled Atmosphere Conditions of Baby Corn

Controlled atmosphere storage can reduce weight loss of baby corn as compared to the normal air condition. Storage in 2 kPa O_2 + 5 kPa CO_2 and 5 kPa O_2 + 5 kPa CO_2 conditions delayed browning in baby corn. Hunter L and a values were well correlated with browning index of baby corn, while b values were not different ($*P > 0.05$) among the treatments. There were no significant differences ($*P > 0.05$) in firmness of baby corn stored under controlled atmosphere and normal air conditions. However, the limiting factor of baby corn was shown to be cob and tip browning. Loss in firmness was unacceptable after browning occurred.

Table 2 summarizes the shelf life and the limiting factors of baby corn stored under CA conditions at 10°C. Baby corn stored under 2 kPa O_2 + 5 kPa CO_2 and 5 kPa O_2 + 5 kPa CO_2 maintained the best quality during storage. Shelf life of baby corn stored under these conditions was extended to 24 days from 18 days under the normal air condition. The results suggested that 5 kPa CO_2, when combined with reduced O_2 could extend the shelf life of baby corn as compared to that at 15 kPa CO_2. No significant differences were observed between 5 and 15% CO_2 when combined with normal air as shown in the CA study.

According to the results, the optimum CA condition for baby corn at 10°C was 2–5 kPa O_2 + 5 kPa CO_2. The composition of O_2 and CO_2 in this range was reported to be the optimum CA/MA for other vegetables such as artichokes, beans, cabbage, and celery [11] and fruits such as avocado, banana, lychee, and nectarine [12]. The results from the present

Table 2. *Shelf Life and Limiting Factors of Baby Corn Stored Under Different Controlled Atmosphere Conditions at 10°C.*

CA Condition	Shelf Life (days)	Limiting Factor
2 kPa O_2 + 5 kPa CO_2	24	Senescence (Browning)
2 kPa O_2 + 15 kPa CO_2	21	Senescence (Browning)
5 kPa O_2 + 5 kPa CO_2	24	Senescence (Browning)
5 kPa O_2 + 15 kPa CO_2	18	Senescence (Browning)
Air (control)	18	Senescence (Browning)

CA study suggested the potential success of modified atmosphere packaging for baby corn in high gas permeable packaging. Baby corn packaged in common plastic bags currently being used had a very limited shelf life (less than 7 days) due to anaerobic fermentation. This could also lead to an expanded package from excessive CO_2 accumulation.

Effect of Gas Permeability on Gas Composition in the Packages

Figure 1 shows the changes in gas composition in the packages of baby corn, as a function of time under the storage temperature of 5 and 10°C. Changes in gas composition within packages showed a very low O_2 concentration for all of them since baby corn has very high respira-

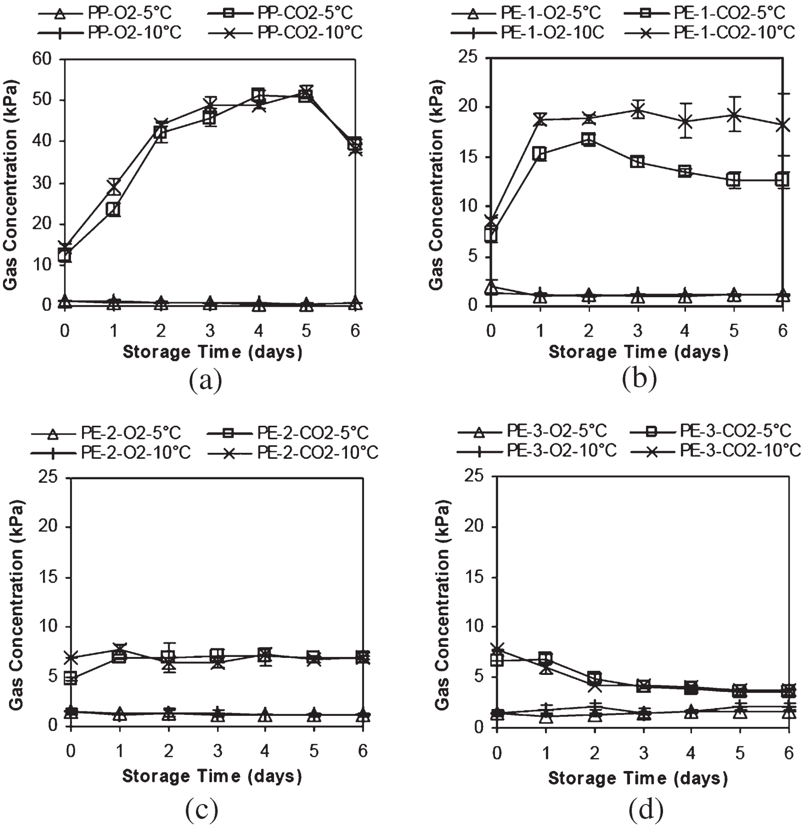


__Figure 1.__ Oxygen and carbon dioxide concentrations (kPa) in (a) PP, (b) PE-1, (c) PE-2, and (d) PE-3 packages of baby corn stored at 5 and 10°C.

tion rate. Carbon dioxide concentration depended on the film type. The highest accumulation of CO_2 was observed in the PP packages of baby corn, due to its lowest gas permeability and permselectivity. In the PP packaging systems, CO_2 concentration increased gradually after 2-day storage and decreased rapidly after 5-day storage when the shelf life ended.

Equilibrium modified atmosphere (EMA) conditions summarized in Table 3, were achieved in all high gas permeable films. The compositions of O_2 and CO_2 at equilibrium in PE-1 were 1 kPa O_2 + 13 kPa CO_2 and 1 kPa O_2 + 17 kPa CO_2, at 5 and 10°C, respectively, while gas compositions were approximately 1 kPa O_2 + 7 kPa CO_2 in PE-2 and 2 kPa O_2 + 4 kPa CO_2 in PE-3 at both temperatures (Table 3). A slight increase in CO_2 concentration at equilibrium was shown in PE-2 and PE-3 stored at 10°C as compared to 5°C. This was most likely due to an interaction between the high gas permeability of the films and a high respiration rate of the baby corn.

Low O2 concentrations at equilibrium were observed in all types of the high gas permeable films. Highest O_2 and CO_2 transmission rates in PE-3 films were obtained by reducing the thickness of PE-1, resulting in increased O_2 and decreased CO_2 concentrations within the packages. When PE-3 was used, an O_2 concentration of 2 kPa gas obtained, higher than those observed in PE-1 and PE-2 packages. In the case of CO_2 concentration, the resulting value at equilibrium was 4 kPa which was lower than those for PE-1 and PE-2. It should be noted that all three PE-based films used in this study were PE blends with structural control through processing in order to achieve high gas permeable properties. Film thickness (Table 1) is also one of the key parameters affecting gas transmission rate. Overall results from the present study suggested that the equilibrium gas composition was affected by O_2 and CO_2 transmission rates and permselectivity of the films and the produce respiration rate. Hence, in-package gas composition was also dependent on produce weight and surface area of the bags.

Effect of Modified Atmosphere Conditions on Quality and Shelf life of Baby Corn

Color

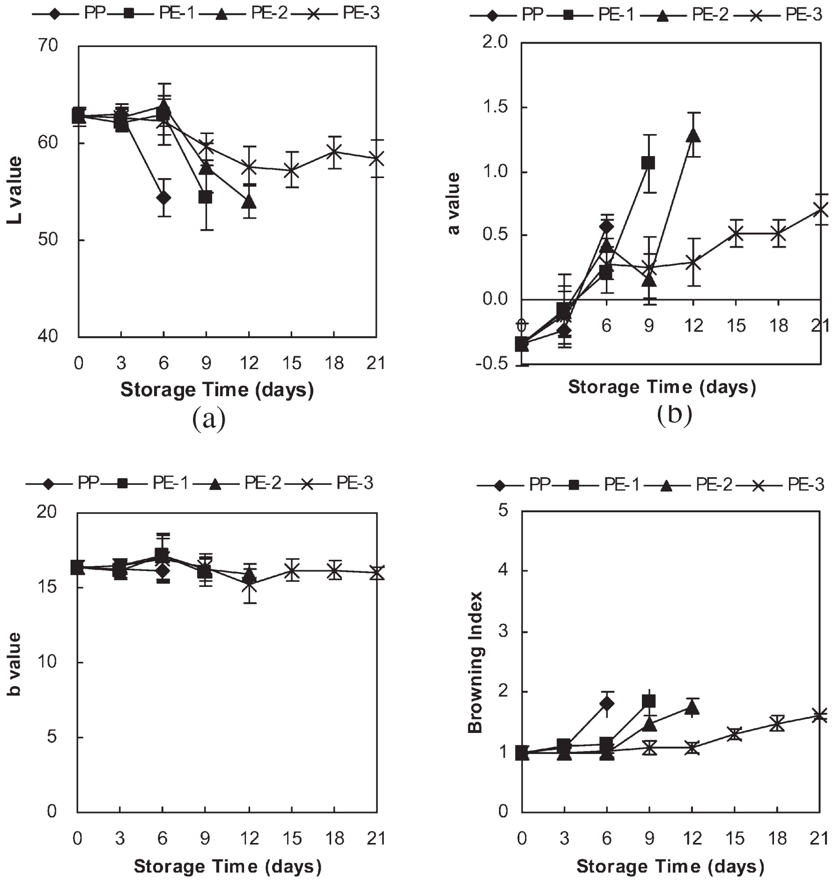
The color of baby corn changed from light yellow to light brown as the storage time increased. Baby corn packaged in PP had severe browning

Table 3. *Equilibrium Modified Atmospheres (EMA), Shelf Life, and Limiting Factors of Baby Corn in Various Packaging Films Stored at 5 and 10°C.*

	5°C			**10°C**		
Film	**EMA**	**Shelf Life (days)**	**Limiting Factor**	**EMA**	**Shelf Life (days)**	**Limiting Factor**
PP	–	6	CO_2 injury (Browning)	–	6	CO_2 injury (Browning)
PE-1	1 kPa O_2+13 kPa CO_2	9	Senescence (Browning)	1 kPa O_2+17 kPa CO_2	9	Senescence (Browning)
PE-2	1 kPa O_2+7 kPa CO_2	12	Senescence (Browning)	1 kPa O_2+7 kPa CO_2	12	Senescence (Browning)
PE-3	2 kPa O_2+4 kPa CO_2	15	Chilling injury (Browning)	2 kPa O_2+4 kPa CO_2	21	Visible mold

and showed the lowest L and the highest 'a' values and browning index. The results showed that PE-3 maintained acceptable color of baby corn throughout storage for 21 days. There were significant treatment effects (*P ≤ 0.05) on L and a values and browning index of baby corn. However, no significant treatment effects (*P > 0.05) on 'b' values were observed among baby corn in various film types.

L values of baby corn generally decreased after 6-day of storage [Figure 2(a)]. Browning in the tip area changed markedly and was more pronounced than that in the cob area. The tip browning was highly corre-



(a) (b)

(c) (d)

Figure 2. *Changes in L (a), a (b), and b (c) values of baby corn tips and browning index (d) of baby corn in various films during storage at 10°C. Vertical bars represent the mean ± SD.*

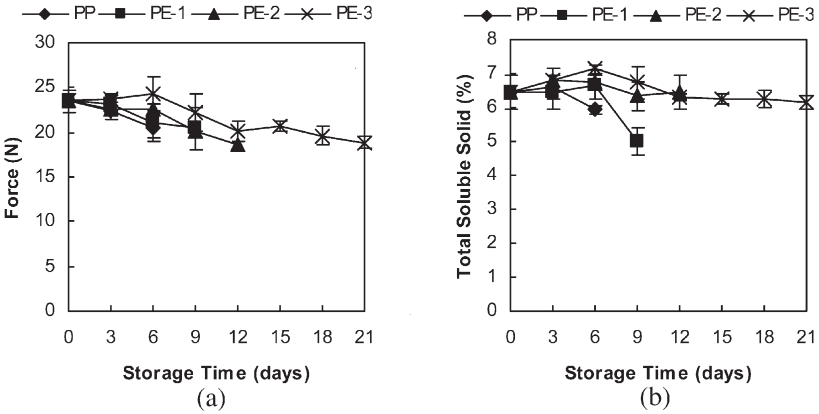
lated with the L values and color and overall liking scores. Baby corn in PP packages with significant (*P ≤ 0.05) lowest L values had severe browning caused by elevated CO_2 above the tolerance limit (15 kPa). The results corresponded to the finding of Ittanuvakin and Siriphanich [13], which reported that baby corn stored under 20–40 kPa CO_2 had a higher degree of browning than those stored in air. Ke and Saltveit [14] also reported that CO_2 injury caused brown strain disorder in iceberg lettuce. In our study, only slight changes in L values of baby corn were observed in PE-3 packages throughout storage. Figure 2(b) shows changes in ‘a’ values of baby corn tips during storage. A rapid increase of ‘a’ values of baby corn was observed in the PP packages. That increase of ‘a’ values was associated with a decrease of L values. ‘b’ values changed slightly throughout the storage interval and there was no significant treatment effect (*P > 0.05) [Figure 2(c)]. Values for ‘b’ changed slightly throughout the storage interval and there was no significant treatment effect (*P > 0.05). Results suggested that baby corn with a browning index higher than 2 was unacceptable [Figure 2(d)]. However, baby corn packaged in PE-3 had the browning index below 2 throughout storage. The results are consistent with the interpretation that the limiting factor of baby corn in PE-3 was not browning but decay. L and ‘a’ values, browning index, color score, and overall liking were highly correlated as shown in Table 4.

Firmness

Firmness of baby corn changed slightly during the first 6 days of storage and more rapidly until the end of storage [Figure 3(a)]. Highest (*P ≤ 0.05) loss of firmness was observed in the PP packages. Firmness of

Table 4. *Correlation Between Various Parameters of Baby Corn in Various Packaging Films Stored at 10°C.*

	Correlation	
Parameter	**Browning Index**	**Overall Liking**
L	–0.90	0.88
a	0.82	–0.87
b	–0.39	0.41
Browning Index	1	–0.87
Firmness–Tip	–0.81	0.92
Firmness–Cob	–0.67	0.89
TSS	–0.74	0.58



(a)

(b)

Figure 3. Changes in firmness of baby corn tips; (a) and total soluble solids of baby corn, (b) in various films during storage at 10°C. Vertical bars represent the mean ± SD (n = 6).

baby corn was highly correlated with L and 'a' values and sensory quality (Table 4). Changes in firmness of baby corn tips declined slightly more rapidly than did the cobs. Similar to the observations in the CA study, loss of firmness to an unacceptable level occurred after browning, which was the limiting factor of baby corn marketability.

Total Soluble Solids Content

Total soluble solids (TSS) content was higher in the cobs than in the tips [Figure 3(b)]. Baby corn in the high gas permeable films had higher TSS as compared to those in the PP packages. A previous study reported a rapid loss of sucrose in asparagus and broccoli stored in air [15]. Carlin et al. [16] also reported that modified atmospheres of 2–10 kPa O_2 and 10–40 kPa CO_2 retained sugar content in shredded carrot. Reduced O_2 and elevated CO_2 in high gas permeable packages delayed senescence process of fresh fruits and vegetables resulting in a delay of sucrose reduction in baby corn.

Weight Loss

Weight loss of baby corn in various packaging films during storage at 10°C ranged from 0.031–1.403%. The lowest ($*P \leq 0.05$) weight loss was observed in PE-3 packages, while the highest ($*P \leq 0.05$) weight loss was observed in PP and PE-1 packages. Percentage weight loss of baby corn in PE-3 packages at the end of shelf life (21 days) was as low as 0.113%. Weight loss of produce was ascribed to moisture loss through the films [17]. However, the results suggested that the modified atmospheres established in PE-3 and PE-2 may reduce weight loss, although the water vapor permeability of the films is higher than that of PE-1 and PP. This is most likely due to the reduced respiration rates of baby corn under the optimum modified atmospheres established in PE-3 and PE-2, resulting in less amount of water loss. In addition, the substantially high gas transmission rate of the films resulted in film shrinkage in PE-3 and PE-2. At this condition a free volume was depleted, which resulted in reduced time to reach steady-state, minimizing the water vapor loss. The results suggested that proper use of high gas permeable films may reduce weight loss of other produce. A further study on the effect of the high gas permeable films related to water vapor permeability on reducing weight loss is required for clearer explanation.

Sensory Quality and Shelf Life

Baby corn was subjected to sensory evaluation for appearance, color, odor, firmness, and overall liking (Figure 4). The highest (*P ≤ 0.05) scores for overall liking was observed in baby corn packaged in PE-3, followed by PE-2 and PE-1, respectively after storage for 9 days and 12 days. Baby corn packaged in PP was acceptable until the sixth day of

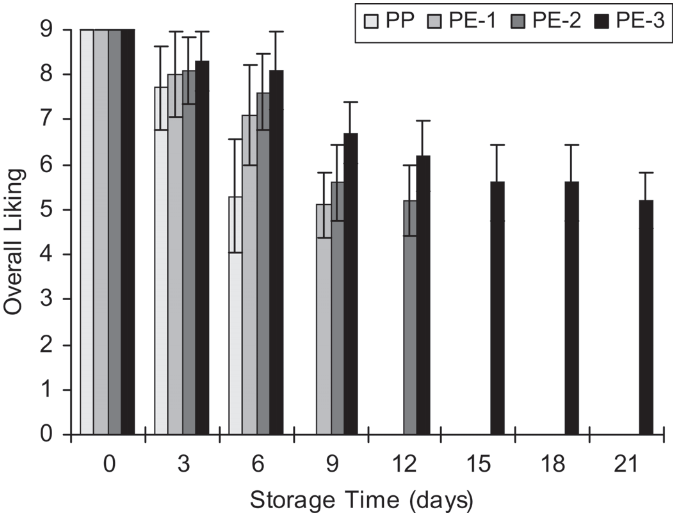


Figure 4. *Overall liking scores for baby corn in various films during storage at 10°C. Vertical bars represent the mean ± SD (n = 30). Means with the same letters are not significantly different (*P > 0.05) by Duncan's Multiple Range Test.*

storage. Primary indication of quality loss of baby corn in PP packages was browning attributed to CO_2 injury, whereas that in PE-3 stored at 5°C, PE-1, and PE-2 stored at 5 and 10°C was browning due to senescence. However, the limiting factor of baby corn in PE-3 packages stored at 10°C was mold growth. Percentage decay of baby corn in PE-3 packages at the end of shelf life was 12.56%. No decay was observed at the end of shelf life in any other treatments. Shelf life determined by overall liking scores below 5 of baby corn stored at 5 and 10°C is shown in Figure 5. The limiting factors for deterioration and shelf life of baby corn packaged in various films are presented in Table 3. Total plate counts and yeast and mold counts in all treatments during storage were in the range of 5–7 log CFU/g and 3–4 log CFU/g, respectively. *E. coli* was not found in all treatments. Mold growth was visible in baby corn packaged in PE-3 after 21 days of storage.

Shelf life of baby corn at 10°C was extended from 6 days in PP to 9, 12, and 21 days in PE-1, PE-2, and PE-3, respectively. The results showed that gas transmission rates and permselectivity of the high gas permeable films significantly ($^*P \leq 0.05$) affected the quality and shelf life of baby corn due to modified atmosphere conditions achieved in the packages. The results from MAP study were associated with the CA study. The highest shelf life was observed in PE-3, with a gas composition (2 kPa O_2 + 4 kPa CO_2) close to the recommended condition (2–5 kPa O_2 + 5 kPa CO_2) obtained from the CA study. The results from the CA study suggested that O_2 concentration below 2 kPa could cause anaerobic fermentation as occurred in the other treatments (PE-1, PE-2, and PP) at around 1 kPa. The results suggested a limitation of the films in creating low O_2 concentrations, which would not be desirable in several products other than for baby corn.

It should be noted that for high gas permeable films, CO_2 concentrations in the package atmospheres increased with decreasing film CO_2TR and with increasing permselectivity. However, in the case of PP films that possessed the lowest CO_2TR and lowest permselectivity ($\beta = 2.2$), unfavorably high CO_2 concentration of > 40 kPa was obtained after 2 days of storage. It is obvious that both gas transmission and permselectivity of the films should be taken into account when considering their combined effect on the resulting in-package atmosphere. Permselectivity is a ratio of CO_2 permeability and O_2 permeability of the films, which indicates the amounts of O_2 and CO_2 permeating through the films and the gas composition achieved at equilibrium. Commercial

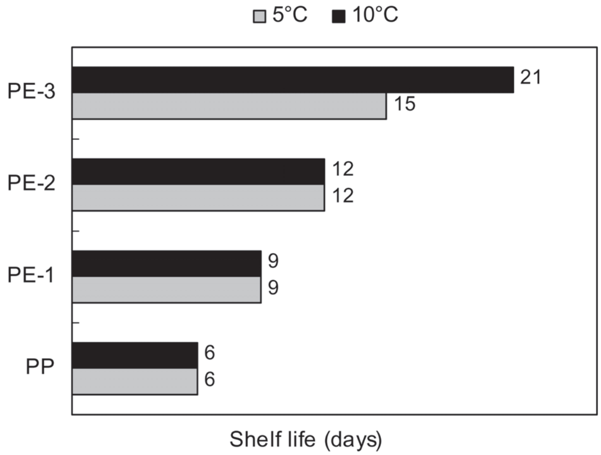


Figure 5. *Shelf life of baby corn in various films during storage at 5 and 10°C.*

films typically had permselectivities between 4–8 [3], while most fresh produce require permselectivity in other ranges. The results from the present study suggested that PE-3 with a permselectivity of 3.05 matched the respiration rates of baby corn, therefore, creating the equilibrium modified atmospheres close to the optimum conditions (2–5 kPa O_2 + 5 kPa CO_2). PE-1 and PE-2 with permselectivity slightly above 3 could also extend the shelf life of baby corn as compared to PP with permselectivity of 2.2. However, increased O_2 and CO_2 transmission in PE-1 and PE-2 were not sufficient to obtain enough O_2 and to eliminate CO_2 from the package atmospheres to avoid anaerobic fermentation for high respiring produce like baby corn. Changing permselectivity of the films would further allow desired equilibrium modified atmospheres to be established in the produce packaging systems.

Effect of Storage Temperature on Quality of Baby Corn

No difference (*P > 0.05) was shown in the gas composition of the baby corn in PE-2 and PE-3 packages stored at 5 and 10°C, while slightly higher CO_2 concentration was observed in PE-1 packages stored at 10°C. This is most likely due to relatively high respiration rates of baby corn. Similar trends in quality changes of baby corn stored at 5 and 10°C were shown. Shelf life of baby corn in PE-3 packages was 15 days at 5°C and 21 days at 10°C, while storage temperature did not affect the shelf life of baby corn in other treatments. Critical tissue softening and browning was observed in baby corn stored at 5°C due to chilling injury after storage for 15 days. Chilling injury should be taken into consideration when storing at 0–10°C. Several commodities are chilling sensitive and vary among cultivars [18]. Severe browning observed in baby corn in PP packages was due to CO_2 injury and was not significantly (*$P > 0.05$) temperature dependent. Hence, most of the results presented here were at the storage temperature of 10°C as this temperature gave the maximum shelf life of baby corn. In addition, this temperature is widely used in commercial practices and reflects the average fluctuating temperature in the supply chain. However, the optimum storage temperature of fresh produce depends on the cultivar and the shelf life required for the specific produce.

CONCLUSIONS

Use of high gas permeable films could extend the shelf life of baby corn. In PE-3 packages stored at 10°C, the EMA of 2 kPa O_2 + 4 kPa CO_2 maintained acceptable quality of baby corn until 21 days. Whereas, storage of baby corn in PP film resulted in an accumulation of CO_2 which after 6 days of storage caused severe browning from CO_2 injury. Storage at 5°C caused tissue softening and chilling injury of baby corn after 15 days. The gas composition at equilibrium is dependent primarily on the interaction of gas transmission and permselectivity of the films and respiration rate of baby corn.

ACKNOWLEDGEMENTS

The authors would like to thank National Metal and Materials Technology Center and Postgraduate Education and Research Development Project in Postharvest Technology Kasetsart University for financial support.

REFERENCES

1. Gorny, J. R., Hess-Pierce, B., Cifuentes, R. A., Kader, A.A., Quality changes of fresh-cut pear slices as affected by controlled atmospheres and chemical preservatives, *Postharvest Biol. Technol.,* Vol. 24, 2002, pp. 271–278.
2. Lee, D. S., Haggar, P. E., Lee, J., Yam, K. L., Model for fresh produce respiration in modified atmosphere based on principles of enzyme kinetics, *J. Food Sci.,* 56, 1991, pp. 1580–1585.

3. Al-Ati, T., Hotchkiss, J. H., The role of packaging film permselectivity in modified atmosphere packaging, *J. Agric. Food Chem.,* 51, 2003, pp. 4133–4138.
4. Rizzo, V., Muratore, G. Effects of packaging on shelf life of fresh celery, *J. Food Eng.*, 90, 2009, pp. 124–128.
5. Kim, J. G., Luo, Y., Gross, K. C., Effect of package film on the quality of fresh-cut salad savoy, *Postharvest Biol. Technol.,* 32, 2004, pp. 99–107.
6. Chinsirikul, W., Fuongfuchat, A., Kerddonfag, N., Phibulkulsumrit S., Winotapun C., Khumsang, P., Preventing film blocking and optimizing haze properties of metallocene PE blown films through blending with rigid polymers. *Intern. Polymer Processing,* 23, 2008, pp. 152–160.
7. Gonzalez-Aguilar, G. A., Ruiz-Cruz, S., Cruz-Valenzuela, R., Rodiguez-Felix, A., Wang, C. Y., Physiological and quality changes of fresh-cut pineapple treated with anti-browning agents, *LWT - Food Sci. Technol.,* 37, 2004, pp. 369–376.
8. Agar, I. T., Massantini, R., Hess-Pierce, B., Kader, A. A., Postharvest CO2 and ethylene production and quality maintenance of fresh-cut kiwi fruit slices, *J. Food Sci.,* 64, 1999, pp. 433–440.
9. AOAC. 2000. *Official Method of Analysis, 17th ed.*, Washington, DC: Association of Official Analysis Chemists.
10. Weichmann, J. 1987. Low oxygen effects, *Postharvest Physiology of Vegetables,* New York, NY: Marcel Dekker, Inc.
11. Kader, A. A. 2001. *A summary of CA requirements and recommendations for fruits other than apples and pears: Postharvest Horticulture Series No. 22A.* University of California, Davis, CA.
12. Saltveit, M. E. 2001. *A summary of CA requirements and recommendations for vegetables: Postharvest Horticulture Series No. 22A.* University of California Davis, CA.
13. Ittanuvakin, S., Siriphanich, J., Effects of carbon dioxide on quality of baby corn (*Zea mays* Linn.) cv. Supersweet, *Kasetsart Journal (Natural Science),* 26, 1992, pp. 342–349.
14. Ke, D., Saltveit, M. E., Carbon dioxide-induced brown strain development as related to phenolic metabolism in iceberg lettuce, *J. Amer. Soc. Hort. Sci.,* 144, 1989, pp. 789–794.
15. Irving, D. E., Hurst, P. L., Respiration, soluble carbohydrates and enzymes of carbohydrate metabolism in tips of harvested asparagus spear, *Plant Sci.,* 94, 1993, pp. 89–97.
16. Carlin, F., Nguyen-the, C., Hilbert, G., Chambroy, Y., Modified atmosphere packaging of fresh ready-to-use grated carrots in polymeric films, *J. Food Sci.,* 55, 1990, pp. 1033–1038.
17. Simon, A., Gonzalez-Fandos, E., Tobar, V., Influence of washing and packaging on the sensory and microbiological quality of fresh peeled white asparagus, *J. of Food Sci.*, 69, 2004, pp. 6–12.
18. Watada, A., Qi, L., Quality of fresh-cut produce, *Postharvest Biol. Technol.,* 15, 1999, pp. 201–205.

Effects of Heat-seal Variables on Water Vapor Ingress in Small High-Barrier Pharmaceutical Blisters

LILLIAN LIU[1] and FRITZ YAMBRACH[2,*]
[1]*M.S., Dept. of Nutrition, Food Science & Packaging, San Jose State University, One Washington Square, San Jose, CA*
[2]*Ph.D, Director of Packaging, Department of Nutrition, Food Science & Packaging, San Jose State University, One Washington Square, San Jose, CA*

ABSTRACT: In this study, a two-factor (temperature and dwell-time), three-level design of experiment was conducted to evaluate the barrier performance of cold-form foil unit-dose blister packages stored at accelerated aging conditions (40°C at 75% RH) over a fourteen-week period. Because the packaging was formed from laminated webs of aluminum foil, PVC, and nylon, the evaluation concentrated on the heat-seal area formed between top and bottom foil layers. This evaluation included visual inspection (behavior of adhesive melt during sealing stage, thickness of heat seal layer) and physical examination (gross leak testing, seal strength testing) of the formed samples, as well as Karl Fischer titrimetric analysis of moisture ingress to assess barrier performance of the packages. Physical tests indicated that the samples were free from gross leaks, pinholes, flex-cracks or other compromises to the structural integrity of the packaging. Statistical analysis (ANOVA, $P < 0.05$) of the results indicated increasing seal strength across the samples was associated with increases in temperature (Table 3), but was not affected by changes in dwell-time. ANOVA conducted for moisture results indicated that there was no significant difference across the different process variables (i.e., temperature-dwell time combinations).

The practical significance of the results lies in the flexibility awarded to manufacturers in scaling processes for larger operations, as well as in adjusting process conditions as appropriate for specific product formulations (e.g., capability to lower processing temperatures for product formulations that are sensitive to heat). Although moisture testing was conducted over a 14-week period, limited resources prevented the study from extending to a more ideal 24-week study period.

---

[*] Author to whom correspondence should be addressed. Email: Fritz.Yambrach@sjsu.edu

Journal of Applied Packaging Research, Vol. 3, No. 2—April 2009

1557-7244/09/02 075-16

1. INTRODUCTION

THE development of products which are contained in very small packages and are very sensitive to moisture has broadened the methodology to include transmission testing of the seal area for flexible and semi-flexible packages. It is widely accepted that aluminum foil (of thickness > 25 microns) functions as an absolute barrier against moisture. An aluminum blister made from foil thickness greater than 25 microns is thus practically impermeable, with the exception of the blister's heat-seal area. These ultra high-barrier packages are examples of where current industry test methodologies require higher levels of sensitivity.

The moisture barrier of a package is simply viewed as the amount of water vapor that will permeate through a given area of a material and the amount of water vapor that will travel through the corresponding heat-seal area. The largest component to transmission is usually the material's permeability and the surface area of the package.

For items such as pouches and lidded blisters, the heat-seal of the formed package is an area where the main concerns have been physical strength and hermetic integrity of the seal. The general assumption is that if there is hermetic integrity, the seal is good. In many ways this is correct because if there is an intact seal it will be a barrier to the outside environment. A poorly-formed (weak) seal and a well-formed (strong) seal will both have hermetic integrity, but is the WVTR of both seals necessarily equal?

A heat-seal is a diffusion bond between two thermoplastic layers of materials. The area which is designated as the seal area is normally heated by a bar and the heat transfer from that bar softens, then melts the thermoplastic. Pressure exerted by the bar on the seal area is the mechanism that causes the heat-seal layer from both layers to come into contact and when semi-solid, they flow together creating an area where both layers are joined. The ideal seal will then be an even mix of the two layers and be twice as thick as a single heat-seal layer. The possible problem areas that could cause a weak or non-hermetic seal are:

1. Too much heat, causing the heat-seal thermoplastic to become very liquid and flow out of the seal area.
2. Too much pressure, which will cause the seal material to flow out of the seal area leaving a thinner seal layer.
3. Too little heat, not melting the thermoplastic sufficiently to allow the

two layers to flow into each other, giving a weak and possibly incomplete hermetic seal.

The relationships between temperature, duration, and pressure variables in the heat-seal process are well understood and highly controlled for products—such as food, pharmaceutical drugs, sterile medical devices—where seal integrity is essential to product quality. While there can be variability in seal strength, hermetic integrity of a seal either exists or not, and lack of it corresponds with increased product degradation as a result of exposure to the outside environment. The usual route of heat-seal testing requires that hermetic integrity be established first, followed by tensile testing to quantify seal strength.

This investigation will look at the relationships between heat-sealing variables as they affect thickness of the formed heat-seal and subsequent moisture ingress. Potential differences in thickness in the seal area can pose serious adverse effects upon the overall WVTR of the package and significantly shorten the shelf-life for moisture-sensitive products, such as those found in the biotechnology industry.

2. REVIEW OF LITERATURE

Since package integrity and barrier performance are critical elements to product stability and shelf-life, much scientific research has been focused on seal quality and barrier performance of flexible materials. Richtsmeir [1] evaluated bond quality and seal integrity of flexible packages and medical devices using non-destructive ultrasound frequencies ranging from 5 to 300 MHz. This technology allowed features as small as 5 to 10 microns in size to be imaged within a wide variety of materials used within the medical device sector, including adhesives, solders, laminate foils, polymer films, as well as molded plastics, metals, ceramics and composites. Ozguler [2,3] used an ultrasonic “Backscattered Amplitude Integral (BAI)” method to detect major defects such as non-bonding, wrinkles, and bubbles distributed within the seal area of flexible food packages, such as snack bags. Although he showed this technique to have potential applications in real-time quality control for production lines, the method is dependent upon manual data collection for development of calibration curves unique to each material being inspected. Frazier [4], in turn, utilized a RF-based technique to examine seal quality of food packages used in retort applications; the study results showed the RF sampling technique to have improved detection rates for seal defects relative to Ozguler's BAI method for channel defects 15 microns in size or smaller.

Other research efforts have concentrated on destructive methods to evaluate seal quality, such as the gross leak testing and seal-strength testing examined by Tonrey [5] for unit-dose pharmaceutical packaging. While use of the traditional bell-jar-and-colored-dye method provided a qualitative test for detecting gross leaks in a package, the technique destroyed both package and contained product; Tonrey advocated the use of seal strength testing as a quantitative alternative that destroyed the packaging, but not necessarily the packaged product. Foil-based packaging was examined by destructive means in studies performed by Auslander [6,7] in which three test methods were employed to detect defects—vacuum-dye, seal strength, and pressurized ammonia vapor. Study results showed pressurized ammonia vapor to be the most sensitive when used with foil packages. Besides pressurized ammonia vapor, leak detection systems based on mass spectrometers calibrated for helium as a tracer gas have also been utilized to confirm the integrity of pharmaceutical and food packaging. Helium is non-toxic, chemically-inert, and its low concentration in air (5 to 50 ppm depending on atmospheric pressure) allows the gas molecules to be easily detected and quantified to an extremely sensitive level (1×10^{-10} gm/sec). Despite its wide acceptance in evaluating package integrity for microelectronics, as well as rigid containers (such as rubber-stoppered vials) for the pharmaceutical industry [8,9,10], helium-based leak detection tests—which can be destructive (packaging samples are pressurized internally with helium in a pressurized "burst test") or non-destructive (packaging samples are enclosed within a vacuum chamber filled with helium)—have been shown to be inappropriate to measure the hermeticity of organic, adhesive bondings [11]. Although Pascall [12] utilized pressure differential and peel strength approaches for seal evaluation, his research involved semi-rigid packaging trays made from PET/EVOH/PP laminates (no aluminum foil) for food applications. Pascall's results showed the pressure differential equipment used in the study had detection limits of 40 microns for channel leaks 6 mm in length and 15 microns for pinhole defects. Gibson [13], on the other hand, did evaluate the seal quality of packaging made from laminated foil materials, but the study was intent upon comparing the efficacy of infrared- vs. ultraviolet-based illumination systems on commercial production lines. Yet another approach to evaluating seal quality was proposed by Sasaki [14], where high frequency, high voltage techniques were applied to flexible packages; study results showed this method to be effective for identifying defects as small as 5 microns.

There is also much scientific research dedicated to moisture analysis techniques and development of permeability models for various barrier materials, but more recent studies appear to be directed towards applications in the coatings industry [15,16]. Permeability research that does focus on flexible materials, however, tends to center on food applications [17,18,19] and less on applications in pharmaceutical packaging, where products are more sensitive to moisture ingress.

While there have been many approaches to evaluating seal quality and moisture permeability of barrier materials presented in the scientific literature, R&D activities within the pharmaceutical industry are often considered proprietary to each company and rarely published in the scientific literature. No articles were identified by the authors to include a combination of visual inspection, physical examination, and moisture analysis testing specifically for foil-based, unit-dose pharmaceutical packaging. The collective use of these methods is believed to be a more comprehensive and interdisciplinary approach and is the basis for the barrier performance study presented below.

3. METHODS

In this study, a two-factor (temperature and dwell-time), three-level design of experiment (DOE) was conducted to evaluate the barrier performance of cold-form foil unit-dose blister packages stored at accelerated aging conditions (40°C at 75% RH) over a fourteen-week period. Two replicate sets—Group A and Group B—were included in this study. Each group A and B were made from identical materials on the same equipment. Group A was used to test for cross-cut thickness and Group B was used for peel testing. Because the packaging was formed from laminated webs of aluminum foil, PVC, and nylon, the evaluation concentrated on the heat-seal area formed between top and bottom foil layers. This evaluation included visual inspection (behavior of adhesive melt during sealing stage, thickness of heat seal layer) and physical examination (gross leak testing, seal strength testing) of the formed samples. In addition, Karl Fischer titrimetric analysis of moisture ingress was performed to assess barrier performance of the packages.

The unit-dose blister package is made from top and bottom webs of laminated aluminum foil, with all foil layers of thickness greater than 25 microns. Since, theoretically, foil layers of 25 micron thickness or greater prevent any moisture from permeating through the foil, any observed moisture ingress is assumed to be the result of permeation through the heat-seal area of the formed samples. Barrier performance for this study is thus associated with the quality of the heat-seal, which is formed between the heat-seal coating and the PVC layer of the top and bottom web materials, respectively.

Although heat-sealing processes involve three machine variables—temperature, dwell-time, and sealing pressure—it was determined that the study would focus on the first two variables, as sealing pressure is not applied during the cooling phase for this package design and subsequently has less of an influence upon the resulting seal quality. Also, in practice, pressure is held constant and often dictated by the packaging equipment. The three levels—"high," "middle," and "low"—for each process factor were selected as 190°C, 170°C, 150°C and 1.5 sec, 1.2 sec, and 1.0 sec for temperature and dwell time variables, respectively (Table 1).

A placebo (e.g., desiccant) was packaged within the blisters and served to maintain an environment inside the blister cavity of $< 5\%$ RH, while the environmental storage chamber created an external environment of 75% RH for the blister samples. This difference in moisture levels across the packaging barrier served to create the driving force for moisture ingress. Because of its ability to be produced into a tablet form small enough to fit comfortably within the blister cavity (Figure 1), as well as absorb moisture in environments up to 105°C, a molded silica gel was ultimately selected as the desiccant for use in the study (Table 2).

Table 1. *Summary of Experimental/Variable Process Combinations.*

Process Combination Group	Temp (°C)	Dwell Time (sec)
Low-Low	150	1.0
Low-Middle	150	1.2
Low-High	150	1.5
Middle-Low	170	1.0
Middle-Middle	170	1.2
Middle-High	170	1.5
High-Low	190	1.0
High-Middle	190	1.2
High-High	190	1.5

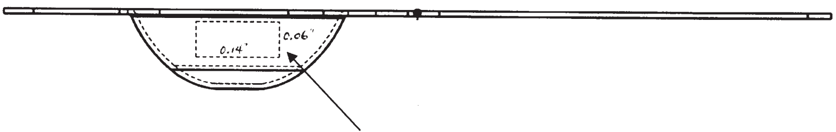
Table 2. *Materials Summary.*

Materials	Product/Part No.	Lot No.	Manufacturer
Desiccant	MultiForms® Molded Silica Gel Part No. 07176	M657980	MultiSorb Technologies, Inc. (Buffalo, NY)
Foil-Laminate, Top Web	Alcan Product Code 10126	103391	Alcan Packaging, Pharma Center (Shelbyvile, KY)
Foil-Laminate, Bottom Web	Alcan Product Code 90256	103709	Alcan Packaging, Pharma Center (Shelbyville, KY)

When preparing the samples, the desiccant was allowed to equilibrate at ambient temperature of 23°C within a sealed enclosure surrounding the filling and sealing stations of the packaging equipment, and clean dry air (0% ± 2% RH) was allowed to circulate within. Because the desiccant was supplied by the manufacturer with moisture content less than 2.0% (w/w), a 24-hour period within the enclosure was deemed sufficient to equilibrate the desiccant with the enclosure environment.

Samples were generated on a Körber Medipak (Clearwater, FL) Model CP-2 form-fill-seal blister packaging machine, and material for the top and bottom foil-laminate webs was supplied by Alcan Packaging's Pharma Center (Shelbyville, KY). Formed samples were visually-inspected for gross defects and labeled according to their respective process temperature and dwell-time conditions. Samples were then selected at random for ($t = 0$) moisture testing, visual inspection under microscope, as well as physical evaluation of the heat-seal area. The remaining samples were placed in storage at accelerated aging conditions of 40°C at 75% RH; samples selected at random from this group were subsequently tested for moisture content at $t = 4$, 8, and 14 weeks.

When performing visual inspections on the ($t = 0$) samples, the pri-



Ideal location for placement of desiccant with approximate diameter of 0.14-inches and depth of 0.06-inches.

Figure 1. *Side-View of Blister Package and Placement of Desiccant.*

Table 3. *Analysis of Variance (ANOVA) for Seal Strength Testing Results, Group B.*

(Values in lbf)	**150°C**	**170°C**	**190°C**	**Σ(Row)**	**$[\Sigma(\text{Row})]^2$**
1.0 sec	3.60	4.00	4.45	12.04	144.90
1.2 sec	3.52	4.36	4.25	12.14	147.26
1.5 sec	3.60	3.95	4.46	12.01	144.18
Σ(Column)	10.72	12.30	13.16	36.18	436.34
$[\Sigma(\text{Column})]^2$	114.92	151.35	173.12		
$\Sigma(X^2)$		146.60			
$\Sigma(X)$		36.18			
$[\Sigma(X)]^2)$		1308.99			
Correction Factor (CF)		145.44			
Sum of Squares (SS)					
Total SS		1.15			
Temp/Column SS		1.02			
Dwell/Row SS		0.00			

ANOVA Table for Group B

Source of Variation	**Degrees of Freedom**	**Sum of Squares (SS)**	**Mean Square (MS)**	**F-ratio, calculated**	**F-ratio (table, $P < 0.05$)**	**F-ratio (table, $P < 0.01$)**	**F-ratio$_{(\text{calc})}$ > F-ratio$_{(\text{table})}$?**
Temperature	2	1.020	0.510	15.738	6.94	18.00	Y, $P < 0.05$
Dwell Time	2	0.003	0.001	0.046			N
Interation (error)	4	0.130	0.032				
Total	**8**	**1.152**					

mary objective was to obtain qualitative data on the adhesive melt behavior and thickness of the heat-seal area for the blister samples. The hope was that this information would detect any unusual or undesired behavior of the adhesive during sealing, and could be used to help substantiate any unusual observations in corresponding moisture content results. Visual inspections were also expected to confirm that no pinholes, flex-cracks, or other compromises to the integrity of the blister samples were generated during the forming process. Magnified inspection (100×) using a Leitz Ergolux microscope was performed on samples selected at random from each of the nine process combinations; the Spot Insight imaging system (v.3.2.6) from Diagnostic Instruments, Inc. (Sterling Heights, MI) was used to capture top-down and cross-sectional images of melt behavior and thickness, respectively, of the adhesive layer for each sample.

Physical evaluation of the (t = 0) samples involved destructive test methods to assess integrity of the formed blisters and quality of their respective heat-seal layers. To ensure there were no gross leaks in the samples, a water-bath vacuum leak test (based on *ASTM F2338, Standard Test Method for Nondestructive Detection of Leaks in Packages by Vacuum Decay Method*) was performed on five samples selected at random from each of the nine variable conditions. Samples were submerged within a Pack-Vac Leak Detector from Haug Quality Equipment (Morgan Hill, CA) for approximately 60 seconds under a vacuum pressure of 18 inches Hg. Samples were removed from the chamber, gently towel-dried, and then allowed to air-dry at ambient conditions for 30 minutes before a razor blade was used to open the blister cavities to inspect for leaks.

Peel testing (*ASTM F88, Standard Test Method for Seal Strength of Flexible Barrier Materials*) was then performed on (t = 0) samples to obtain a relative comparison of the quality of the heat-seals across the variable process conditions. For this evaluation, peel samples were prepared from scrap material leftover from the blister-forming process, and a Model AG-IS Precision Universal Tester from Shimadzu Scientific Instruments (Columbia, MD) was used to perform the seal strength analysis. Four trials per variable process condition were tested on web material saved from Group B for a total of 36 trials.

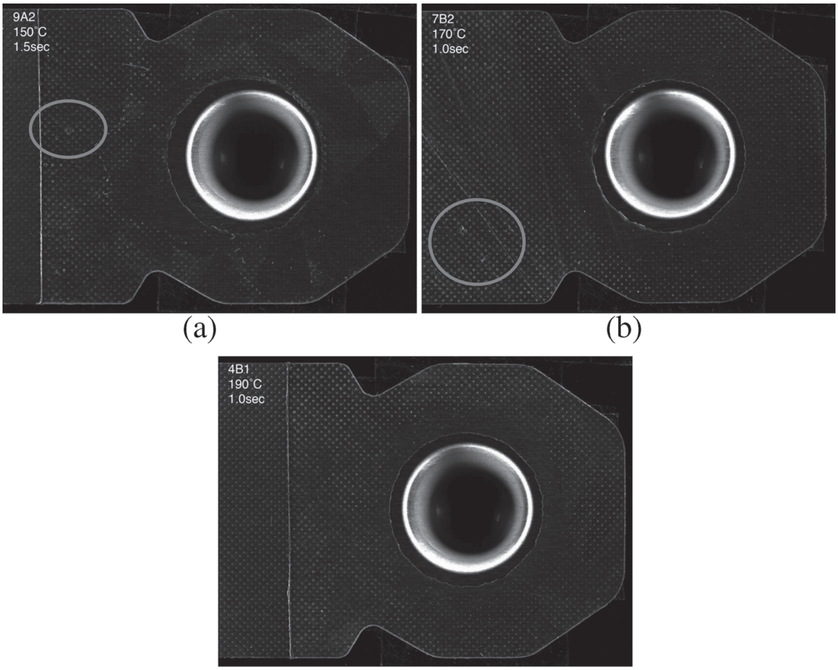
3.1 Sensitivity of Moisture Detection

Results from the visual inspections and physical assessments were expected to confirm that the samples did not contain any gross leaks or other defects in the heat-seals of the blisters, and allow the study to then concentrate on measuring moisture content of the samples held in storage. Historical data for previous-generation packaging established a WVTR of (2.4×10^{-4} g H_20/g product * 24h * %RH). At such low levels of moisture ingress, gravimetric methods would not be sensitive enough for measuring moisture content of the samples. Karl Fischer titration is a "wet chemistry" analytical method used to detect moisture in gasses, liquids, or solids. It is a proven and relatively inexpensive method that is highly selective for water, and it is favored for its accuracy to detect moisture (< 100 ppm) in a relatively short amount of time. The Karl Fischer determination is based on the reduction of iodine (I_2) to hydrogen iodide (HI) by sulfur dioxide (SO_2) in the presence of water. The presence of water in a sample to be analyzed initiates a series of reactions which results in the presence of free I_2, measured potentiometrically at the endpoint of the reaction. The Karl Fischer titration method has been incorporated in several pharmacopoeias around the world, and has also been adopted as an ASTM standard. For these reasons, this method was employed for moisture analysis for this study. Samples were held at accelerated aging conditions of 40°C and 75% RH per International Conference on Harmonization (ICH) guidelines, and moisture content of the samples was analyzed at $t = 0$, 4, 8, and 14 weeks. Limited resources prevented the study from extending to a more ideal 24-week study period.

4. RESULTS

4.1 Seal Inspection

As expected, microscopic inspection confirmed that the quality of the blister samples was consistent—no pinholes, flex-cracks or other gross defects in the formed packages were observed. In looking at the melt behavior of the adhesive, no channel leaks were detected, but slight inclusions and/or solitary air bubbles (Figure 2) were observed on occasion for the 150°C samples (both Groups A & B) and the 1.0 second samples (Group B only). It should be noted, however, that due to the extensive amount of time required in preparation of the samples, only two samples per variable condition (total of 18 samples each per Groups A and B) were subjected to this examination. While no recurring patterns of inclusions or air bubbles can be definitively asserted in the adhesive melt be-



(a) (b)

(c)

Figure 2. *Images comparing adhesive melt behavior for: (a) 150°C, (b) 170°C and (c) 190°C samples (Air bubbles and/or inclusions circled in red; no air bubbles or inclusions found in samples from 190°C process group).*

havior across the variable process conditions, it is not surprising that the low temperature/low dwell-time process combinations demonstrated slightly lower consistency in the quality of the heat-seals.

4.2 Cross-Sectional Thickness

When measuring for thickness of the heat-seal layer, a total of seventeen cross-sectional images (100× magnification) were taken per sample across the width dimension of the blister (Figure 3). The heat-seal layer was defined as the region sandwiched between top and bottom foil layers, and average thicknesses ranged from 69.5 microns to 73.1 microns, with corresponding standard deviations ranging between 0.8 and 1.2. While analysis of variance (ANOVA) indicated no significant difference in thickness existed across the variable conditions, it should be highlighted that only a single sample per variable process combination, from Group A only, was used in this analysis. This, again, was due to the time-intensive set-up work required in preparing the samples for evaluation.

4.3 Seal Integrity and Strength

Results for the water-bath vacuum-leak test corroborated the findings from the visual inspections above; no gross leaks were found in the tested samples. Seal strength results confirmed that the top and bottom webs had bonded fully across the heat-seal regions of the samples, although stress-strain graphs from the testing indicated "optimal" seals were achieved more consistently under the 150°C and 170°C process conditions. Test results also showed an average seal strength of 3.57 lbf, 4.10 lbf, and 4.39 lbf for the 150°C, 170°C, and 190°C samples, respectively. Statistical analysis (ANOVA, $P < 0.05$) of the results indicated increasing seal strength across the samples was associated with increases in temperature (Table 3), but was not affected by changes in dwell-time.

4.4. Moisture Content

With confidence in the integrity of the formed blister samples and the quality of their respective seals from the above physical test results, subsequent moisture content results (Figure 4) were analyzed to determine if any relationships could be established between moisture ingress and the temperature/dwell-time processing conditions. First, a two-tailed

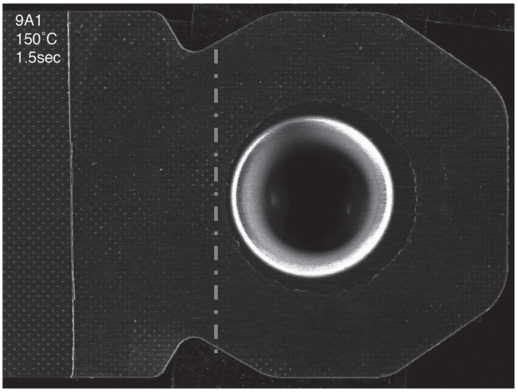


Figure 3. *Approximate Location of Cross-Sectional Image Capture for Heat-Seal Thickness Measurements (17 images captured across width dimension of each sample).*

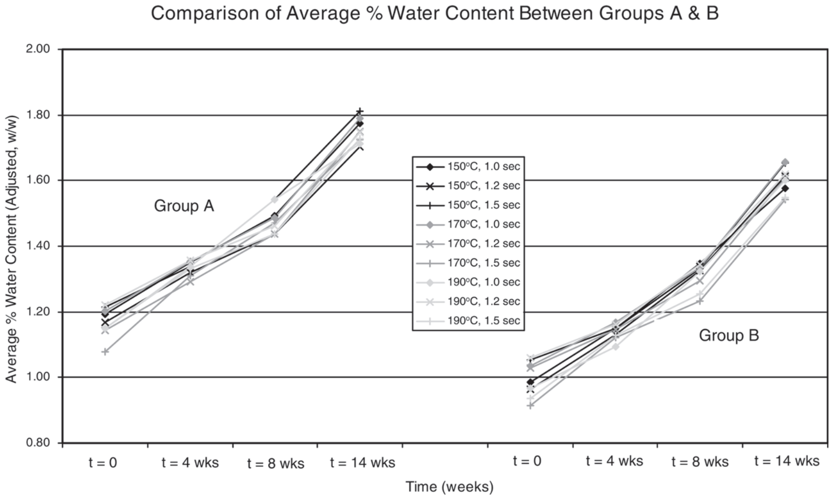


Figure 4. *Graphical Comparison of Moisture Content Results for Groups A & B at* t *= 0, 4, 8, and 14 weeks.*

t-test indicated there was no statistical difference between results for Groups A and B. Further evaluation using Analysis of Variance (ANOVA) indicated there was no significant difference in moisture content across the variable process conditions.

5. CONCLUSION

While the above study—a two-factor, 3-level design of experiment (DOE)—is a common experimental design, use of the highly-sensitive Karl Fischer titrimetric method for moisture analysis serves to highlight the increasing levels of barrier performance found in unit-dose packaging for today's pharmaceutical products. In this study, the heat-seal of a cold-form foil unit-dose blister package design was evaluated for WVTR across varying conditions of process temperature and dwell-time. Results were shown to produce unit-dose blister packages with fully-formed heat-seals of consistent quality. Thus hermetic integrity across the varying process conditions was established, but what was the effect upon WVTR? Although analysis of moisture results for the blister samples determined there was no effect on barrier performance from temperature or dwell-time process conditions, peel testing of the samples indicated that processing temperature did play a direct role in seal strength.

Table 4. *Moisture Content Results for Groups A & B at t=0, 4, 8, and 14 weeks.*

		Group A					**Group B**				
		Average, % Water Content, Adjusted (w/w)				**Rate of Moistrue Ingress (“slpoe”)**	**Average, % Water Content, Adjusted (w/w)**				**Rate of Moistrue Ingress (“slpoe”)**
Temp (°C)	**Dwell Time (sec)**	**t = 0**	**t = 4 wks**	**t = 8 wks**	**t = 14 wks**		**t = 0**	**t = 4 wks**	**t = 8 wks**	**t = 14 wks**	
150	1.0	1.19	1.35	1.49	1.78	0.006	0.99	1.15	1.35	1.58	0.006
150	1.2	1.17	1.32	1.44	1.70	0.005	0.96	1.13	1.33	1.61	0.007
150	1.5	1.21	1.34	1.54	1.81	0.006	1.6	1.15	1.33	1.65	0.006
170	1.0	1.20	1.35	1.49	179	0.006	1.03	1.17	1.34	1.66	0.006
170	1.2	1.14	1.29	1.44	1.75	0.006	1.03	1.15	1.30	1.61	0.006
170	1.5	1.08	1.31	1.47	1.72	0.007	0.91	1.12	1.23	1.54	0.006
190	1.0	1.15	1.34	1.54	1.71	0.006	0.97	1.09	1.32	1.60	0.006
190	1.2	1.22	1.36	1.46	1.75	0.005	1.06	1.16	1.35	1.62	0.006
190	1.5	1.15	1.33	1.44	1.73	0.006+	0.94	1.13	1.26	1.55	0.006

These findings are significant because the implications enable future development efforts to capitalize on a wider range of seal process parameters. It also awards the capability to lower process temperatures during packaging for product formulations and/or small fill doses that are temperature-sensitive without compromising hermetic integrity or barrier performance for similar types of unit-dose blister packages. In addition, having a wide range in process temperature conditions which yields no difference in barrier performance can help to transition processes established for development activities into larger-scale packaging operations, where controlling for drift in process temperature can often pose a challenge.

While the Karl Fischer method is not a novel technique for moisture analysis, it offers a practical, reliable and cost-effective solution for the barrier-evaluation challenges faced by packaging engineers across the pharmaceutical/biotechnology, medical device, food and other industries where barrier-protection is a key element for product quality

ACKNOWLEDGEMENTS

Special thanks are extended to Scot Cheu, Dan Miller, Priti Durbha, and Marcus Hinkson for generously sharing their knowledge of moisture analysis methods and techniques, as well as for providing the necessary equipment and resources to support this study. Special thanks are also extended to David Shimmin at NanoCraft Electron Microscopy (Mountain View, CA) for his expertise in microscopic analysis and imaging.

REFERENCES (as cited in narrative)

1. Richtsmeier J, Kessler L. Inspecting bond quality and seal integrity in medical devices using acoustic micro imaging (AMI). *Proceedings from the Materials & Processes for Medical Devices Conference, September 8–10, 2003 (Anaheim, CA)* 2003; pp. 236–239.
2. Ozguler A, Morris SA, O'Brien Jr WD. Evaluation of defects in the seal region of food packages using the ultrasonic contrast descriptor, BAI. *Packaging Technology and Science* 1999; 12, pp.161–171.
3. Ozguler A, Morris SA, O'Brien Jr WD. Ultrasonic monitoring of the seal quality in flexible food packages. *Polymer Engineering & Science* 2001; 41(5), pp.830–839.
4. Frazier CH, Tian Q, Ozguler A, Morris SA, O'Brien Jr WD. High contrast ultrasound images of defects in food package seals. *IEEE Transactions on Ultrasonics, Ferroelectrics & Frequency Control* 2000; 47, pp. 530–539.
5. Tonrey JF. Non-destructive leak testing of blister and strip packaging for pharmaceuticals. *Packaging Technology* 1984; 14, pp. 58–59.

6. Auslander DE, Gilbert SG. Hermetic packaging of drugs: optimized sealing of foil pouches. *Package Development & Systems* 1977; 7(2), pp. 20–22.
7. Auslander DE. Hermetic packaging of drugs: optimized sealing of foil pouches. *Package Development & System*s 1978; 8(5), pp. 20–22.
8. Kirsch LE, Nguyen L, Gerth, R. Pharmaceutical container/closure integrity. III: Validation of the helium leak rate method for rigid pharmaceutical containers. *Journal of Pharmaceutical Science & Technology* 1997; 51(5), pp. 203–207.
9. Kirsch LE, Nguyen L, Moeckly C.S. Pharmaceutical container/closure integrity. I: Mass spectrometry-based helium leak rate detection for rubber-stoppered glass vials. *Journal of Pharmaceutical Science & Technology* 1997; 51(5), pp. 187–94.
10. Kirsch LE, Nguyen L, Moeckly CS, Gerth R. Pharmaceutical container/closure integrity. II: The relationship between microbial ingress and helium leak rates in rubber-stoppered glass vials. *Journal of Pharmaceutical Science & Technology* 1997; 51(5), pp. 195–202.
11. Veyriè D, Roux F, Tetelin, A, Pellet, C. Hermeticity tests on organically sealed micropackages using FTIR spectroscopy. *Proceedings of SPIE Micromachining and Microfabrication Process Technology XI* 2006; 6109, pp. 6111–31.
12. Pascall MA. Evaluation of a laboratory-scale pressure differential (force/decay) system for non-destructive leak detection of flexible and semi-rigid packaging. *Packaging Technology & Science* 2002; 15, pp. 197–208, DOI:10.1002/pts.582.
13. Gibson TG. A system for inspection of package seal integrity. *Proceedings of SPIE—International Society for Optical Engineering* 1995; 2597, pp. 37–39.
14. Sasaki H, Kamimura K. Pinhole inspection machine for sealed packages: for detection of pinholes of 0.5μm or below. *Packaging Technology & Science* 1997; 10, pp. 109–118.
15. Houston TE. Methods for the determination of water in coatings. *Metal Finishing* 1997; October, pp. 36–38.
16. Sangaj NS, Malshe VC. Permeability of polymers in protective organic coatings. *Progress in Organic Coatings* 2003; 50, pp. 28–39.
17. Czeremuszkin G, Latrèche M, Wertheimer MR Permeation through defects in transparent barrier-coated plastic films. *44th Annual Technical Conference Proceedings for the Society of Vacuum Coaters—Philadelphia (April 21–26)* 2001, pp. 178–183.
18. Tolhurst LJ, Carroad PA, Henderson JM. Correlation between high and low speed WVTR tests in packaging. *Package Development & Systems* 1979, 9(5), pp. 17–20.
19. Steven MD, Hotchkiss JH. Comparison of flat film to total package water vapour transmission rates for several commercial food wraps. *Packaging Technology & Scienc*e 2002; 15, pp. 17–27.

Flexography Printing Performance of PLA Film

J. JACOBSON[1], M. KEIF[2,*], X. RONG[2], J. SINGH[3] and K. VORST[3]
[1]*Undergraduate Assistant, Graphic Communication, Cal Poly State University, San Luis Obispo, CA*
[2]*Professors, Graphic Communication, Cal Poly State University, San Luis Obispo, CA*
[3]*Professors, Industrial Technology, Cal Poly State University, San Luis Obispo, California, USA*

ABSTRACT: During the past decade polylactide acid (PLA) polymer has been the subject of numerous researches aimed at comparing it with traditional petroleum based polymers for many packaging applications. PLA is biodegradable and derived from agricultural by-products such as corn starch or other starch-rich substances like maize, sugar or wheat. While PLA is currently being used in many packaging applications with well documented performance, little work has been done comparing printing processes and performance. This study presents PLA printing performance and sustainability findings using the common flexography printing process. Various analytical methods were used to evaluate performance and provide recommendations for optimized printing on PLA as compared to PET, oriented PP and oriented PS. Results of this study found that PLA films were comparable in printability and runnability to standard petroleum based flexible packaging films.

1.0 INTRODUCTION

SINCE 1960, the annual generation of municipal solid waste (MSW) has increased more than 65 percent to 251.3 million tons in 2006 [1]. By 2006 material recovery of MSW through recycling and composting accounted for over 32.5 percent of all waste generated, an increase of nearly 83 percent since 1960 [1]. Containers and packaging accounted for nearly 32 percent of all products generated in the MSW in 2006. Plastics which ranked fourth after paper (33.9%), yard trimmings (12.9%), and food scraps (12.4%) accounted for 11.7 percent of the 251 million tons of MSW generated in 2006 [1]. More than 10 percent of all plastic

---

[*] Author to whom correspondence should be addressed. Email: mkeif@calpoly.edu

1557-7244/09/02 091-14

containers and packaging, comprising of soft drink, milk and water bottles, were recycled with milk bottles accounting for 31 percent of all bottles [1].

Due to the increasing environmental consciousness of consumers and corporations over the past decade, biodegradable polymers have received ever increasing attention. Amongst commercially available biodegradable polymers are NatureWorks™ Polylactide (PLA), Nodax, Eastar Bio, and Biomax. Biodegradable polymers provide a potential solution to a wide range of environmental concerns typically associated with conventional polymers such as greenhouse gas emissions and sustainability. PLA is derived from lactic acid and has been received well by the medical and packaging industry in recent years. PLA is manufactured from annually renewable sources such as corn and is promoted as being recyclable and compostable.

PLA has been researched internationally for its adaptability to practical applications such as in medical devices and packaging in comparison to traditional petroleum based polymers. Due to its ability to be hydrolyzed, PLA has been studied for use in bio-absorbable medical devices. In de Braekt et al. studied its application for suturing material [2], Bos et al., Laitinen et al., and Matsusue et al. researched its application for surgical implants [3,4,5] and Bodmeier et al., Conti et al., Omelczuh and McGinity, and Suzuki and Price [6,7,8,9] studied its promise in the drug-delivery systems application.

Auras et al. provided an overview of PLA as packaging materials by discussing its physical, optical, rheological, processing, mechanical, solubility, barrier, and degradation properties [10]. Sinclair et al. provided a similar report in their paper on polylactic acid as a commodity packaging plastic [11]. Auras et al. compared food service containers made with oriented PLA to those manufactured using PET and OPS by quantifying their physical, mechanical, barrier and compatibility properties [12,13]. Martino et al. in their research on processing and mechanical characterization of plasticized films for food packaging reported mechanical properties of PLA films for different plasticizer concentrations and preparation conditions [14]. Results of a study involving characterization of L-polylactide and L-polylactide-polycarprolactone co-polymers for use in cheese packaging applications were reported by Plackett et al [15].

The purpose of this study was to research the runnability and printability of PLA and to discover some key considerations when printing on PLA with the flexographic process. There has not been a great deal of published research on PLA—especially on printability. Green Bay Packaging (Green Bay, Wis.) has been working with printability of PLA for more than five years and has performed numerous tests regarding the surface energy, runnability, and printability of PLA [16]. In recent testing Green Bay Packaging is using their soon-to-be patented treated PLA with film surface conditions of no lower than 52 dynes [16]. This allows the polymer to securely anchor any ink process. Green Bay ensures that the surface energy of the film is high enough so that any print engine and most inks will not have problems printing. The company has found during these tests that the key to successful anchorage of ink to the PLA is high surface energy of the film. If a company wants to add a varnish for extra protection of the image, UV varnish by Sun Chemical (item # RCMSV0482232) is recommended [16]. The UV over-varnish doesn't contain silicone, and therefore can biodegrade and compost easily. Green Bay Packaging has not experienced problems with the runnability of PLA. According to them PLA has good stiffness and should run well on any press—the rigidity helps with registration, trapping, and tension on line [16].

2.0 MATERIALS AND METHODS

2.1 Ink Adhesion

An ink adhesion test was preformed using a lab ink proofer prior to running the polymer films on press. The surface energy of PLA was tested using Accudyne test solution swabs to sample the film. A large difference between the dyne level of surface energy in a material and the dyne level of surface tension in ink, most often results in better print quality. The optimal surface energy of the substrate is dependent on the ink system but is typically above thirty-eight dynes. The surface energy of the film should be higher than the surface tension of ink because it is more practical and economical for a printer to change the surface tension of a material than to change the inks on their press. Surface-treated film (corona or plasma treated) creates higher surface energy polymers. Most water-based inks have a surface tension of about 36 dynes. The water-based inks used in this experiment where designed for PET and PP plastic films, and therefore leads to better ink adhesion for those particular films. Inks formulated specifically for PLA were uncommon at the time. A draw-down test with the anilox-roll hand proofer was used to test ink adhesion to the film. Once blown-dry, a crinkle test determined that the adhesion was acceptable to print on press.

Prior to running the film on the press, a test target was created in Adobe Illustrator to produce a plate and is shown in Figure 1. The image contains vector and raster images, a solid strip, tick marks an eighth inch apart along the lateral edge, slur targets, multiple point sizes, regular and reverse print, 1 through 100 percent density patches, and some gradient strips. The file was RIPed through Esko's Cyrel Digital Imager (CDI) Spark System using Esko's Suite 7 workflow. The plate was set at 150 line screen ruling, 68 degree angle using a circular dot shape. The vertical distortion scaling was set to 96.751 percent. The CDI system was calibrated prior to output with a focus search, stain test, and midtone density test.

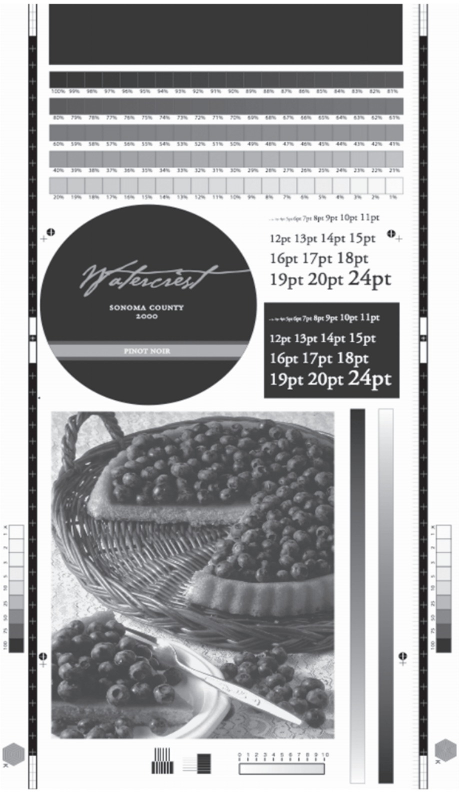


Figure 1. *Test target used to produce a plate.*

The plate was initially placed into the Dupont Cyrel FAST Exposure Unit for 23 seconds for the back exposure. The carbon masked plate material was positioned into the CDI, in which a laser then began to ablate the mask creating a negative image area. Following the setting of the floor and relief through the initial exposure, the plate was polymerized by exposing the emulsion side using a main light source for eight minutes. The plate was then carried to the Cyrel FAST Dupont Processor to remove the unexposed photopolymer prior to detack and post-exposure. Prior to the plate running on the press, a BetaFlex334 system was used to measure the dot area patches on the plate. The finished plate was mounted on a 96 tooth, 1/8 CP cylinder using the Mark Andy Conversource PM-160.

2.2 Runnability

Conventional methods were performed to setup the press. There were many variables controlled during the press run. Many Flexo variables are hard to control because they involve manual deck settings, which makes them more prone to error. Constants that can be controlled include: viscosity, pH, anilox roll, speed, dryer, and tension. Plate and anilox and Plate to impression are difficult to repeat precisely on a Mark Andy 2200 because of the manual deck settings. Environmental Inks and Coatings Film Ink III system was shipped at a viscosity of 25–30 seconds on a #2 Zahn cup with a pH of 9.0–9.3. Prior to running the ink on press, a #2 Zahn cup was used to measure the viscosity of the ink. It measured at 50.9 seconds, which is higher than normal. Too low pH levels usually result in an ink-transferring problem. The uncut ink measured at a pH level of 9.44. The desired level is between 9–9.3 for the Film III Ink System on a Flexographic press. However, the average range for water-based inks is between 8–9.5, therefore the pH was within the tolerance level.

Water-based inks are more difficult to control on the press. Their viscosity and pH levels can change with time on the press. As the press continues to run, the amine may evaporate and the pH level decreases and the viscosity increases. However, the run was so short that the viscosity and pH remained constant throughout the pressrun. Environmental Inks recommended an addition of under ten percent of an ammonia substitute every half hour. The press only ran an hour for about 700 feet of film.

To maximize repeatability and consistency, the impression was controlled using check gauges that had the same diameter of the pitch surface being used. The impression was adjusted by hand and varied with polymer thickness, which makes it difficult to replicate exactly. Therefore, it contributes to the reason for running all the plastics on the same day, to try to reduce the variables. The anilox roll was also constant. A 2.48 volume, 600 line count, 60°, Harper roll was chosen for the Mark Andy 2200 seven-inch Flexographic press. The anilox roll was selected based on the 150 resolution of the plate to achieve appropriate cell count. The press speed was set at 50 feet per minute, and the dryer was positioned at stage 3, which was approximately 167° F. The speed and dryer settings were chosen because there are challenges with film inks drying on the plastic with lower heats and faster speeds on an in-line press. The tension was set at 20 psi.

The five films were run on the Mark Andy press using water-based inks with relative ease. The order in which the webs of film were spliced in were: white PLA, clear PLA, PET, OPP, and OPS. A little over a hundred feet of each material were used. Some of the visual potential runnability problems that occurred on press included problems running the OPS and having dirty print. The OPS was very brittle and broke easily on the in-line press. There were two web breaks when running OPS. And lastly, the print became increasingly dirty throughout the press run. There were many predictions to the reason for the dirty print that are discussed later.

2.3 Printability

The tests conducted to determine printability were: dot area, tone reproduction, optical/reflective density, specular gloss, dot shape, visual tests, rub resistance, adhesion, and tensile strength. The dot area and tone reproduction of the printed material were determined with a BetaFlex334 system. The tonal patches on each film were measured in increments of five, from one to a hundred. An Xrite 528 Spectrodensitometer was used to measure the densities of a solid black area on each film. The Novo-glass Statistical Glossmeter (Rhopoint 60 degree angle model) determined the specular gloss of each film using the same solid black area. Horizontal and vertical readings were taken. The ImageXpert system was used to determine the roundness of the five percent dots on each film. A digital image was captured of the five percent dots using SonyXCD-X710 video camera. The dot roundness was defined by the ratio of the circumference of a circle with the same average radius to the perimeter length of the dot. Some other digital images were captured for visual comparisons, using the same video camera system. They include samples of bridging at five percent dot and samples of font type quality.

3.0 RESULTS AND DISCUSSION

3.1 Dot Gain

There was no substantial difference in the trend of dot gain in the curve across the different films. The largest dot gain occurred between 35 and 55 for all of the films. The white PLA film had a 29.3 percent dot gain at 40 percent dot. The clear PLA film had a 24.7 percent dot gain at 40 percent dot. The PET film had a 31.1 percent dot gain at 40 percent dot. The OPP film had a 28.9 percent dot gain at 40 percent dot. The OPS film had a 27.9 percent dot gain at 40 percent dot. The plastic film with the highest dot gain was PET with 31.1 percent at 40 percent dot. Figure 2 below shows the dot gain results. The dot area of the printed material was determined with the Xrite 528 system. The density patches on each film were measured in increments of five, from one to a hundred.

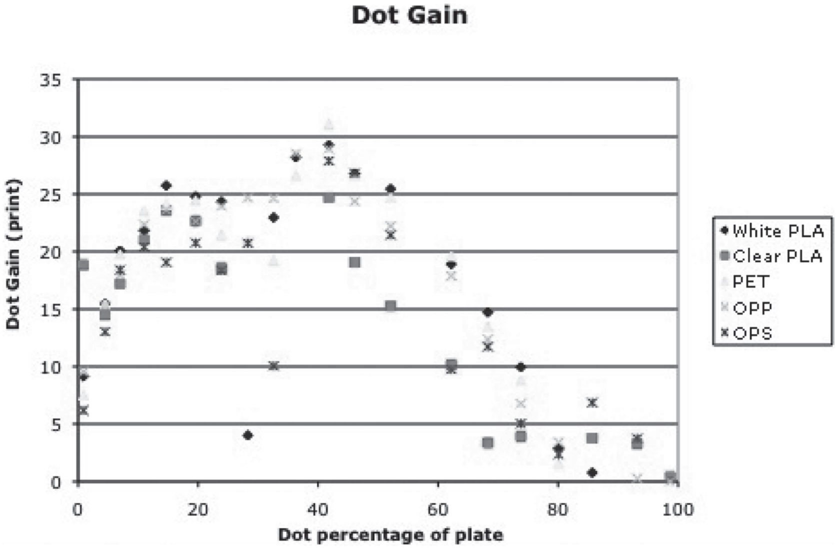


Figure 2. *Dot gain results.*

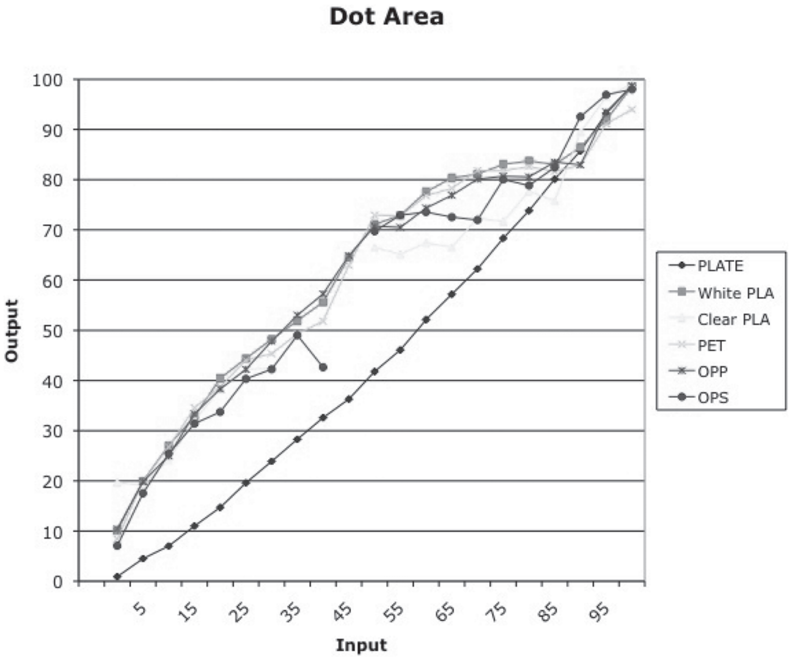


Figure 3. *Tone reproduction of printed material.*

3.2 Tone Reproduction

There was no substantial difference in the trend of the tone reproduction curves across the different films. However, all of the clear films experienced difficulties while being read by the instrument, as shown by the jagged lines in Figure 3. Therefore, it was hard to compare which plastic film had the best tone reproduction. The white PLA appears to have the smoothest curve, which makes it easier to compensate for dot gain in prepress.

3.3 Optical/Reflective Density

The FIRST density standard is 1.4 for black ink on film products using narrow web. All of the densities printed had higher densities than the standard. The chosen anilox roll was not optimized for the desired ink density and lay down. The white PLA film had the highest density over the other films. The clear PLA has the highest density over the clear film. PLA films may achieve higher densities than other films.

Table 1. *The Xrite 528 Spectrodensitometer was used to Measure the Densities of a Solid Black Area on Each Film.*

Films	White PLA	Clear PLA	PET	OPP	OPS
Densities	1.68	1.71	1.56	1.68	1.66
	1.78	1.71	1.61	1.61	1.63
	1.70	1.66	1.60	1.73	1.59
	1.70	1.66	1.61	1.65	1.68
Average	**1.72**	**1.68**	**1.60**	**1.67**	**1.64**
Standard Deviation	**0.04**	**0.03**	**0.02**	**0.05**	**0.04**

3.4 Dyne Levels

The tests showed that the natural dyne level of clear PLA is about 38 and the white PLA had a dyne level of about 36. The other films had similar dynes levels: PET had about 39 dynes, OPP had about 37 dynes, and OPS had about 37 dynes.

3.5 Type Quality

Type Quality Images were captured for visual comparisons, using the ImageXpert system. The results for type quality were in the following order from best to worst: white PLA, PET, OPP, clear PLA and OPS films. The images are shown in Figure 4.

3.6 Dot Shape

The ImageXpert system was used to determine the roundness of the five percent dots on each film. The dot roundness was defined by the ratio of the circumference of a circle with the same average radius to the perimeter length of the dot. The five percent dot was chosen to compare

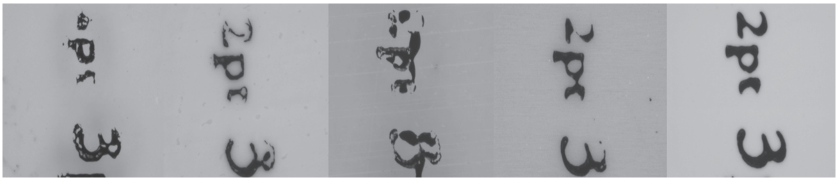


Figure 4. *Type quality images.*

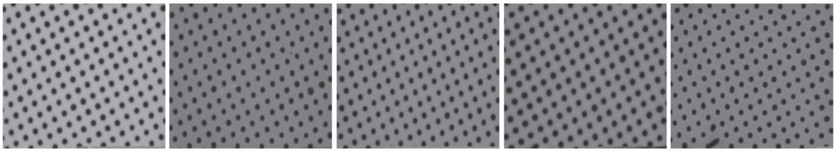


Figure 5. *Digital images of the five percent dots.*

the roundness. The ideal number to achieve is one. The PET film is the closest to achieving this standard. The PET film also has the highest dyne level, which may correspond to being the closest to one.

3.7 Specular Gloss

The Novo-glass Statistical Glossmeter (Rhopoint 60 degree angle model) determined the specular gloss of each film using the same solid black area. Horizontal and vertical readings were taken as shown in table 3 below. With no lamination, high gloss is more desired to achieve a better print appearance. The clear PLA has the highest gloss compared to all of the films, and has the highest gloss compared to the other clear film. The white PLA has the highest gloss compared to the other white films.

3.8 Rub Resistance

The TMI Ink Rub Tester determined the rub resistance of each material. The two by four inch and two and a half by six inch strips were cut and taped onto the base and test block. The four-pound test block was placed on top of the base. The settings were adjusted to 100 cycles at 42 cycles per minute.

A visual test was performed on the rub test samples. The clear PLA film had the poorest rub resistance, closely followed by the white PLA film. The OPS film had the next worse rub resistance. The OPP and PET films had the best rub resistance.

Table 2. *Dot Roundness Results*

White PLA	Clear PLA	PET	OPP	OPS
0.62	0.62	0.82	0.57	0.53

Table 3. *Specular Gloss Results.*

Gloss	White PLA	Clear PLA	PET	OPP	OPS
Horizontal	57.65	65.70	58.60	46.88	56.80
Vertical	62.60	75.38	46.38	48.18	62.18
Average	**60.13**	**70.54**	**52.49**	**47.53**	**59.49**

3.9 Ink Adhesion

Scotch 3M Premuim Grade Transparent Cellophane 610 Tape was used to perform the adhesion test. A small two-inch piece of tape was placed in approximately the same spot on every film and immediately pulled off after slightly patting it down. And lastly, the Testometric CX M350-5KN system measured the elasticity, force, and breaking point of each film. An ASTM slitter cut one by eleven inch strips prior to being placed into the machine.

A visual test was performed on the ink adhesion samples. The white PLA film had the poorest ink adhesion, closely followed by the clear PLA film. The OPS film had the next worse ink adhesion. The OPP and PET films had the best ink adhesion. The OPP film had the best adhesion.

3.10 Runnability

Some of the potential runnability problems that occurred on press included problems running the OPS and having dirty print. This test was run on a narrow-web in-line press. In commercial applications, these materials would often be run on a wide-web CI press, offering better tension control and reduced potential for web breaks.

Table 4. *Tensile Strength Results.*

	Force @ Peak (N)	Elongation @ Break (mm)	Time (sec)
White PLA	151.01	50.06	0.50
Clear PLA	173.70	41.38	0.41
PET	106.86	207.92	2.08
OPP	106.12	122.69	1.23
OPS	105.56	5.65	0.06

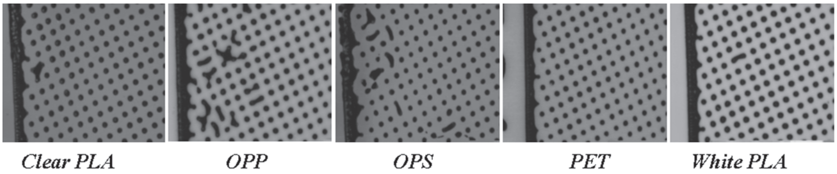


Figure 6. Images were captured for visual comparisons, using the ImageXpert system. The samples of the bridging are in order of CPLA, OPP, OPS, PET, WPLA.

3.11 Tensile Strength

Tensile strength measures the force required to pull a substrate to the point of when it breaks. The Testometric CX M350-5KN system measured the elasticity, force, and breaking point of each film. An ASTM sample cutter cut one by eleven inch strips prior to being placed into the machine. The speed of the machine was 100 mm/min. The OPS used the least force to break, which indicates that this film has the worse tensile strength. This was observed on press with the two web breaks. The clear PLA has the best tensile strength, and least likely to stretch and distort on press.

3.12 Dirty Print

The print became increasingly dirtier throughout the press run. There was an increased incidence of dot bridging the longer the press was running. The order the films ran on press were: white PLA, clear PLA, PET,

***Table 5.** Summary of Findings. (1= best/highest to 5=worst/least).*

Gloss	White PLA	Clear PLA	PET	OPP	OPS
Dyne	4	2	1	3	3
Dot Gain	4	1	5	3	2
Tone Reproduction	1	4	3	2	5
Density	1	2	5	3	4
Type Quality	1	4	2	3	5
Dot Shape	2	2	1	3	4
Specular Gloss	2	1	4	5	3
Rub Resistance	4	5	1	2	3
Ink Adhesion	4	5	1	2	3
Tensile Strength	2	1	3	4	5
Dirty Print	1	2	3	4	5
Average Score	**2.36**	**2.64**	**2.64**	**3.09**	**3.82**

OPP, and OPS. The OPS film seemed to have the most bridging. There were many predictions to the reason for the dirty print. Some articles have suggested that it could be caused from the plate, ink, anilox roll, and the doctor blade. In this research, the only other variable that is time sensitive is the pH and viscosity. The recommended viscosity was 25–30 seconds with a pH of 9.0–9.3. The ink ran on press with a higher viscosity and pH than was recommended. Since the pH level starts to decrease and viscosity increase the longer it is run on press, an ink-transferring problem usually occurs leading to increased incidence of bridging. The pH and viscosity were only controlled at the beginning of the press run; therefore it cannot be determined in this study. More research would need to be performed to confirm this relationship.

4.0 CONCLUSIONS

According to this summary of findings chart, it seems that the white and clear PLA films are most comparable to the PET film. Even though the ink was formulated for PP and PET, the white and clear PLA outperformed OPP in the majority of the printability and runnability tests. The OPS film performed the worst compared to the other plastic films. The white PLA film outperformed the PET film, which was also white. The clear PLA film performed equally as well as the PET film. If the PLA films used custom formulated ink, they would have likely outperformed all of the films. NatureWorks recommends using Akzo Nobel's Hydrokett3000 or Hydrofilm 4000 water-base inks for good ink adhesion.

Given time, PLA may replace some of the most common plastic films used in the food industry. It is difficult for a new film to break into a market that has twenty or more years of established film lines. Advancements are continuously being made to the structure of PLA to enable the plastic to be used in more applications. The PLA films are already ideal for many of the same applications other petroleum-derived films are used for today. They can be used for pressure sensitive labels, shrink sleeves, cut and stack labels, laminates, and more. PLA films can be produced both as mono-layer or may be co-extruded, with cast or blown film extrusion methods presently being the most common. The popularity of sustainable films will continue to increase as companies understand the savings of using annually renewable resources. There is an obvious momentum in the use of PLA, and it will continue to become increasingly competitive with traditional petroleum-based films in the future.

5.0 REFERENCES

1. United States Environmental Protection Agency, “Municipal Solid Waste Generation, Recycling, and Disposal in the United States: Facts and Figures for 2006,” United States Environmental Protection Agency, Solid Waste and Emergency Response (5306P), Washington, DC, 2006.
2. In de Braekt, M.M., van Alphen, F.A., Kuijpers-Jagtman, A.M. and Maltha, J.C., 1992. Wound healing and wound contraction after palatal surgery and implantation of poly-(-lactic) acid membranes in beagle dogs. *Journal of Oral and Maxillofacial Surgery* **50**, pp. 359–366.
3. Bos, R.R., Rozema, F.R., Boering, G., Nijenhuis, A.J., Pennings, A.J., Verwey, A.B., Nieuwenhuis, P. and Jansen, H.W., 1991. Degradation of and tissue reaction to biodegradable poly (-lactide) for use as internal fixation of fractures: a study in rats. *Biomaterials* **12**, pp. 32–36.
4. Laitinen, O., Tormala, P., Taurio, R., Skutnabb, K., Saarelainen, K., Iivonen, T. and Vainionpaa, S., 1992. Mechanical properties of biodegradable ligament augmentation device of poly(-lactide) in vitro and in vivo. *Biomaterials* **13**, pp. 1012–1016.
5. Matsusue, Y., Yamamuro, T., Oka, M., Shikinami, Y., Hyon, S.H. and Ikada, Y., 1992. In vitro and in vivo studies on bioabsorbable ultra-strength poly(-lactide) rods. *Journal of Biomedical Materials Research* **26**, pp. 1553–1567.
6. Bodmeier, R., Oh, K.H. and Chen, H., 1989. Effect of the addition of low molecular weight poly (d,l-lactide) on drug release from biodegradable poly (d,l-lactide) drug delivery systems. *International Journal of Pharmaceutics* **51**, pp. 1–8.
7. Conti, B., Pavanetto, F. and Genta, I., 1992. Use of polylactic acid for the preparation of microparticulate drug delivery systems. *Journal of Microencapsulation* **9**, pp. 153–166.
8. Omelczuh, M.O. and McGinity, J.W., 1992. Influence of polymer glass transition temperature and molecular weight on drug release from tablets containing poly-(-lactic acid). *Pharmaceutical Research* **9**, pp. 26–32.
9. Suzuki, K. and Price, J.C., 1985. Microencapsulation and dissolution properties of a neuroleptic in a biodegradable polymer poly-racemic lactide. *Journal of Pharmaceutical Sciences* **74**, pp. 21–24.
10. Auras, R., Harte, B. and Selke, S., 2004. An overview of polylactides as packaging materials. *Macromolecular Bioscience Journal* **4**, 835–864.
11. Sinclair R.G., Jane, J. and Huang S. J., 1996. The case of polylactic acid as a commodity packaging plastic. ACS Symposium, Anaheim, California, ETATS-UNIS (02/04/1995), vol. 33, no 5 (22 ref.), pp. 585–598.
12. Auras, R., Singh, S. P. And Singh, J., 2005. Evaluation of oriented poly(lactide) polymers vs. existing PET and oriented PS for fresh food service containers. *Packaging Technology and Science* **18**, 207–216.
13. Auras, R., Singh, S. P. And Singh, J., 2006. Performance evaluation of PLA against existing PET and PS containers. *Journal of Testing and Evaluation,* Vol. 34, No.6.
14. Martino, V. P., Ruseckaite, R. A. And Jimenez, A., 2005. Processing and mechanical characterization of plasticized poly (lactic acid) films for food packaging. 8th *Polymers for Advanced Technologies International Symposium,* Budapest, Hungary, 13–16 September 2005
15. Plackett, D. V., Holm, V. K., Johansen, P., Ndoni, S., Nielsen, P. V., Sipilainen-Malm, T., Sodergard, A. and Verstichel, S., 2005. Characterization of l-polylactide and l-polylactide-polycaprolactone co-polymer films for use in cheese-packaging applications. *Packaging Technology and Science,* Vol. 19, Issue 1, pp 1–24.
16. Anonymous, 2004. Natureworks PLA to compete in the no-look label market. http://www.labelsandlabeling.com/scripts/publish/headlines.asp?code=cnw&key=846&language=en&PageNo=5&pressYr=2004, accessed 24 July, 2006.

Design—An Opportunity in Reducing Corrugated Fiberboard Carbon Footprint

J. SINGH[1,*], R. KISCH[2], J. CHHUN[3] and E. OLSEN[1]
[1]*Associate Professor, Industrial Technology, Cal Poly State University, California*
[2]*President, Container Solutions International Incorporated*
[3]*Research Assistant, Industrial Technology, Cal Poly State University, California*

ABSTRACT: Telescoping Half Slotted Containers (HSC) and Diagonal Corner Bliss style containers are popular choices for packaging agricultural products such as apples, pears, citrus, potatoes, garlic and most vegetables. This study evaluated two unique corrugated container designs, Kisch Full Circle Tray (FCT) and Single V Kisch Bliss, which are both viable designs available to replace the presently used styles of boxes for produce distribution. This paper presents the compression strength results of Telescoping HSC containers as compared to two possible replacements and the Diagonal Corner Bliss designs when stored under standard, refrigerated and tropical conditions. It also compares the material savings and the lifecycle environmental impacts for the three designs against the Telescoping HSC design. Comparing the average overall peak forces, across all three environmental conditions of the Telescoping HSC boxes to that of the three designs, it was concluded that the Kisch FCT boxes were approximately 17% weaker, while providing material savings of over 14%; the Diagonal Corner Bliss boxes were approximately 9% weaker, while providing material savings of almost 22% and the Single V Kisch Bliss boxes were approximately 14% stronger, while providing material savings of over 19%. Savings in material ranging from 14 to 22% for the three designs tested, translates into significant energy savings, relative optimization of natural resources, reductions in green house gas emissions and relative minimization of waste water and solid waste generated during production in comparison to the Telescoping HSC style boxes.

1.0 INTRODUCTION

INFLUENCED by numerous demographic trends, including declining household size, rising income levels and the changing consumption habits, consumption of fresh produce has been favorably effected in re-

---

[*] Author to whom correspondence should be addressed. Email: jasingh@calpoly.edu

1557-7244/09/02 105-14

cent decades [1]. As a result, unprocessed fresh foods such as vegetables and fruits are being included at an increasing rate in diets all over the world. As the variety of fresh produce in terms of number, form and quality has increased, so has the packaging to help move these commodities through the marketing channels. The packaging conceivably contains different types, sizes, grades or maturity of produce and is available in many forms such as sacks and nets, wooden crates, corrugated fiberboard boxes, plastic crates, etc.

Volumes of fresh produce imported into the U.S. increased 43.1% between 1999 and 2006, with the vegetables share increasing by 32.2% and that of fruits by 19.6% for the same duration [2]. Imports of vegetables accounted for 17% of the total U.S. supply (production plus imports) whereas imported fruit accounted for 38% [2]. Between 1999 and 2006 a majority of the fresh vegetables were imported from Mexico (65%) and fresh fruits from Latin America (92%) [2].

Exports of fresh produce from the U.S. showed a mixed pattern by volume with an increase of 10.6% overall, decrease of 2.6% for vegetables and an increase of 16.3% for fruits for the same duration [3]. Exports of vegetables accounted for 7.9% of the total U.S. production whereas exported fruit accounted for 27.8% [3]. Between 1999 and 2006 a majority of the fresh vegetables (67%) and fresh fruits (53%) were exported to Canada [3]. Figure 1 reflects the U.S. fresh produce import and export volumes between 1999 and 2006.

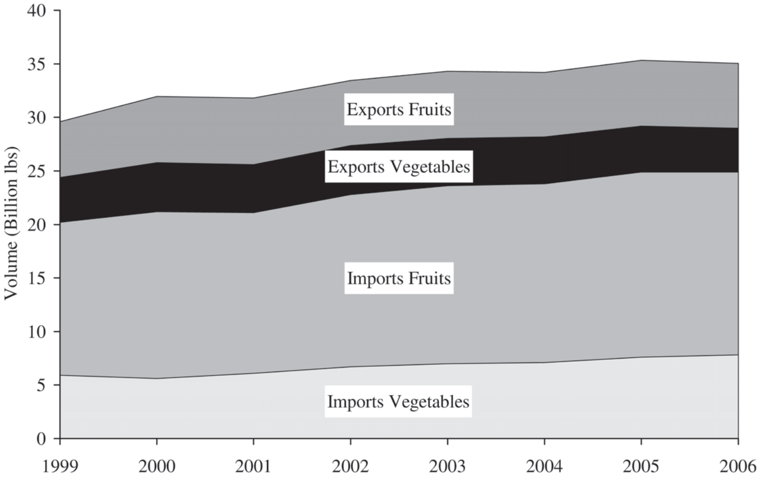


Figure 1. *U.S. Fresh Produce Import/Export Volumes 1999–2006 [2,3].*

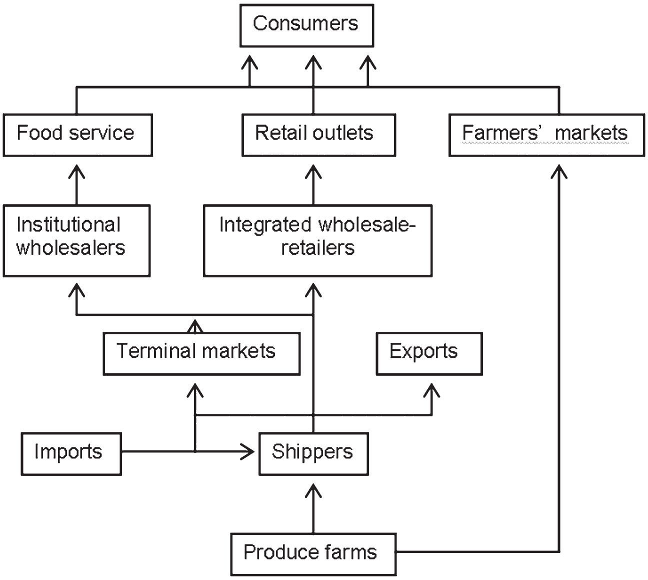


Figure 2. *U.S. Fresh Fruit and Vegetable Marketing Channels [5].*

Fresh produce typically follows expedited handling when moving through the supply chain due to its restrictive shelf life. From the farm production facilities to reach the consumers, the produce experiences multiple handling in the marketing channels. These marketing channels have evolved considerably since the late 1980's when fresh produce markets were more fragmented and most transactions occurred between the produce grower-shippers and wholesalers on a day-to-day basis based on varying market prices and quality levels [4]. Figure 2 shows the typical fresh fruit and vegetable marketing channels in practice today [5]. Some of the key drivers changing the fresh produce distribution include new competitors/rules such as mass merchandisers, European players and online food shopping; increasing buying power from upstream industry consolidation and new supply chain oriented procurement models; and changing consumers with higher incomes and an increasing interest in healthfulness.

Though, the vast improvements in the marketing channels have improved the efficiency of the passage of fresh produce from farm to fork, it needs to be provided adequate protection from distribution hazards experienced during transportation and warehousing. Corrugated shippers have adapted well with fresh produce by providing the desired key functions such as containment, protection and communication and as such are the most popular choice. It has been proposed that more than two thirds of the world's retailed commodities are packed and transported in corrugated packaging [6].

Worldwide demand for corrugated board has been increasing rapidly. Worldwide corrugated production increased 4.5% between 2006 and 2007 with a production of 42,285 million square meters in 2007 [7]. During the same period U.S. experienced a decline of 2.1% in the corrugated production, with a production of 8,938 million square meters in 2007 [7]. Figure 3 illustrates the percentage change in global corrugated production between 2006 and 2007.

Corrugated board packaging is specifically engineered to maximize performance and merchandizing impact throughout the supply chain while minimizing material and its carbon footprint. A few key developments towards this include recycling, use of environmentally-friendly inks, decreased formaldehyde use, and the practice of source reduction. The corrugated industry claims to use over 60% renewable energy from bio-fuels for fiber-based material production and of including 43% recycled content for corrugated board manufacturing [8]. Constant innovations in the area of corrugated shipper designs helps achieve this to a great extent by using lesser material while providing adequate protection to the product.

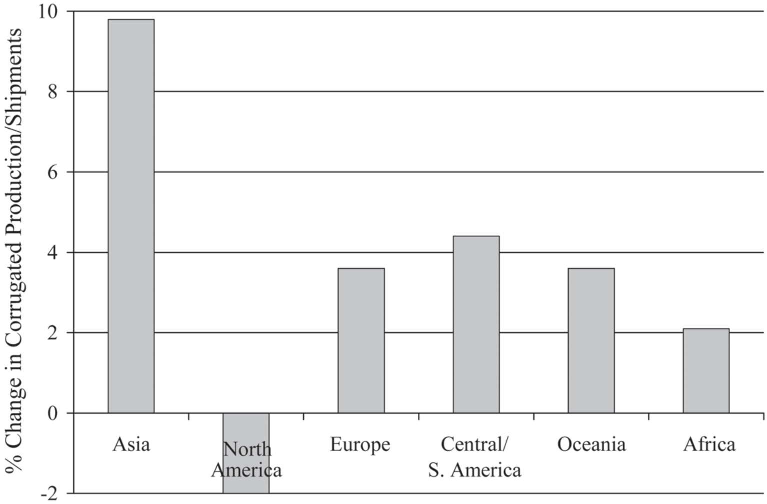


Figure 3. Percentage Change in Global Corrugated Production 2006–2007 [7].

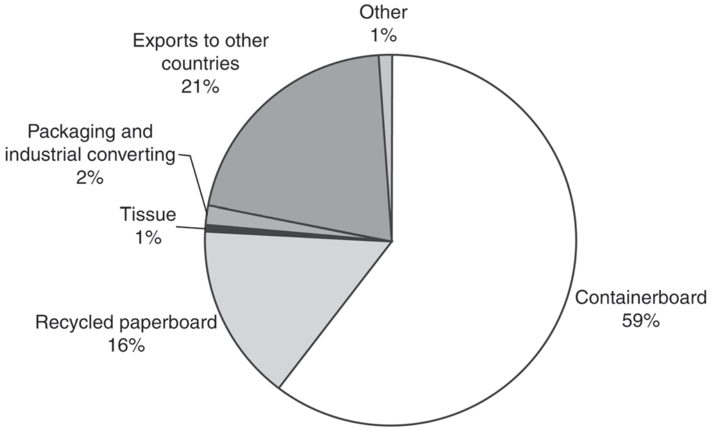


Figure 4. *End of Life Treatment of Old Corrugated Containers (2006).*

Essentially made from renewable resources, corrugated board is made from natural and environmentally sustainable materials which are recovered and recycled more than any other packaging substrate. Due to a nearly 11 percent rise in net exports, recovery of old corrugated containers (OCC) rose 2.0 percent in 2006 to 25.2 million tons [9]. During the same time, U.S. containerboard consumption rose 1.7 percent [9]. As a result, the OCC recovery rate increased to 76.4 percent in 2006 from a revised 76.1 percent in 2005 [9]. Figure 4 shows the end use of recycled OCC for 2006.

The transportation and warehousing hazards faced commonly by corrugated shippers include compression, shock, vibration, temperature, creep and humidity among others. Due to its high strength to low weight ratio corrugated packaging is poised as the leading choice for transport packaging in the United States. By some estimates corrugated packaging is used to package approximately 90% of all products for retail distribution in the United States [10]. The popularity of corrugated packaging also stems from the fact that it is practical, useful, economical, renewable and recyclable [10]. It is also a substrate that can be custom designed and provides excellent merchandising appeal through printing on box panels.

The three most commonly used styles of corrugated boxes for fresh-produce application are (Figure 5):

- *Slotted boxes:* generally made from one piece of corrugated or solid fiberboard. E.g. Regular Slotted Containers (FEFCO 0201).

- *Telescoping boxes:* usually consist of separate top and bottom that fit over each other or a separate body. E.g. Full Telescope Half Slotted Container (FEFCO 0320).
- *Rigid/Bliss boxes:* the three pieces of this style of box includes two identical end panels and a body that folds to form the two side panels, an unbroken bottom and the top. E.g. Bliss Style Container (FEFCO 0606).

FEFCO (European Federation of Corrugated Board Manufacturers) codes are an official system to substitute long and complicated verbal descriptions of fiberboard case and packaging constructions with simple symbols internationally understood by all, regardless of language and other differences [11].

This research involved redesign of corrugated shippers commonly used for fresh produce and evaluated their compression strengths under three common environmental conditions. Evaluation was also conducted in terms of life cycle inventory (LCI) calculations to quantify the material use, energy use, environmental discharges, and wastes associated with each stage of the four box designs over their life cycle. New unique replacement designs, Kisch Full Circle Tray (FCT) for the Telescoping Half Slotted Containers (HSC) style and the Single V Kisch Bliss for the Diagonal Corner Bliss style, were studied.

The scope of the research was:

1. To compare the compression strength of Telescoping HSC boxes with the two replacement designs and the Diagonal Corner Bliss style boxes when stored under standard, refrigerated and tropical conditions.
2. To compare the material savings and calculate the lifecycle environ-

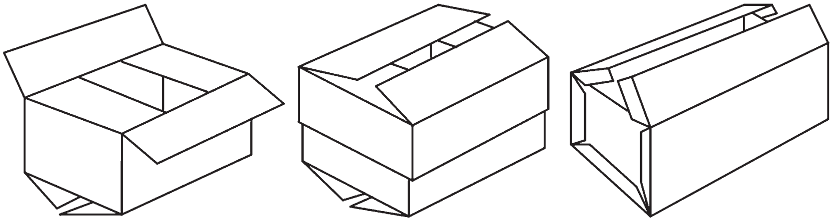
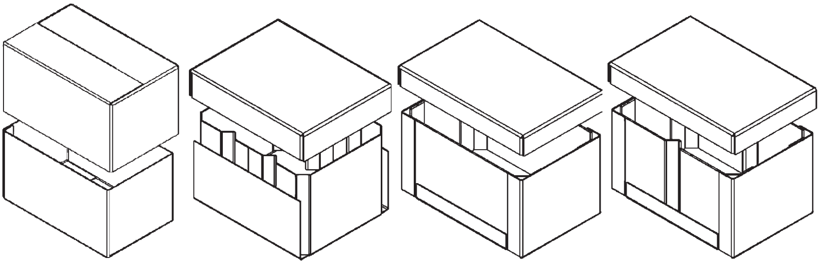


Figure 5. *Common Styles of Boxes used for Fresh Produce.*



Telescoping HSC **Kisch FCT** **Diagonal Corner Bliss** **Single V Kisch Bliss**

Figure 6. *Box Designs Evaluated in the Study.*

mental impact for the two replacement designs and the Diagonal Corner Bliss style with the Telescoping HSC design.

2.0 MATERIALS AND METHOD

2.1 Corrugated Board

C-flute corrugated board was used in the construction of both the bases and lids for all four designs (discussed in item 2.2). The lids were made with lower grade corrugated fiberboard with a basis weight of 17/15C/17 kg/ 92.9 sq. m. (38/33C/38 lb/ 1000 sq. ft.) as compared to the bases, 25/18C/25 kg/ 92.9 sq. m. (56/40C/56 lb/ 1000 sq. ft.), as is common industry practice.

2.2 Container Designs

Four designs for the corrugated produce containers were constructed using ArtiosCAD software and the Premium Line 1930 model of the Kongsberg table (Esko Graphics, Ludlow, Massachusetts, USA). The designs included Telescoping HSC, Kisch FCT, Diagonal Corner Bliss and Single V Kisch Bliss and are shown in Figure 6. All boxes were constructed to have the same internal volume of approximately 0.03 cu. m. (0.93 cu. ft.).

Table 1 reports the total area of the corrugated fiberboard used to construct the bases and lids for the four designs used in this study. It also reports the material savings for the two replacement designs and the Diagonal Corner Bliss style as compared to the Telescoping HSC box.

Table 1. *Total Area and Material Savings per Box Compared to Telescoping HSC Design.*

Box Style	Total Area, sq. m. (sq. ft.)	Area Saving
Telescoping HSC	1.16 (12.51)	—
Kisch FCT	1.00 (10.73)	14.24%
Diagonal Corner Bliss	0.91 (9.79)	21.79%
Single V Kisch Bliss	0.94 (10.08)	19.44%

2.3 Box Conditioning

Corrugated boxes are considerably prone to fluctuations in moisture content and compression strength values are typically based on the ambient relative humidity exposure [12]. Prior to all testing the boxes were conditioned at three environmental conditions in accordance to ASTM D4332 for 72 hours [13]. The three conditions selected were standard [23 ± 1°C (73.4 ± 2°F) and 50 ± 2% relative humidity], refrigerated storage [5 ± 2°C (41 ± 4°F) and 85 ± 5% relative humidity] and tropical [40 ± 2°C (104 ± 4°F) and 90 ± 5% relative humidity]. Five replicate tests were performed for all environmental conditions and the styles of boxes.

2.4 Box Compression Strength Testing

ASTM D 642 (Standard Test Method for Determining Compressive Resistance of Shipping Containers, Components, and Unit Loads) was used to test the compression strength [14]. This procedure is commonly used for measuring the ability of the container to resist external compressive loads applied to its faces, to diagonally opposite edges, or to corners. This test method is also used to compare the characteristics of a given design of container with a standard, or to compare the characteristics of containers differing in construction. This test method is related to TAPPI T 804 om-02 [15]. The tests were conducted using a fixed platen arrangement on a Lansmont compression tester Model 152-30K (Lansmont Corporation, Monterey, CA, USA), with a platen speed of 1.3 cm/minute (0.5 in/minute) and a pre-load of 22.68 kg (50 lb) for zero-deflection in accordance with the standard.

2.5 Lifecycle Environmental Impact Calculations

All environmental impact estimates were made using the Environmental Defense Fund Paper Calculator [15]. The information provided by this website is based on publicly available national averages and the research conducted by the Paper Task Force, a peer reviewed study of the lifecycle environmental impacts of paper production and disposal [16]. All calculations were based on the material usage for the four designs and a recycled content percentage of 43% [8]. Unbleached corrugated, as used to create all boxes for this research, was used as the identified paper type in the calculator.

3.0 RESULTS AND DISCUSSION

3.1 Box Compression Strength Testing

The compression test results are reported in Table 2. The values reported are averages for five replicate tests performed for each box style and conditioning environment. Figure 7 reflects the data in terms of percentage difference in force and deflection values for the two replacement designs and the Diagonal Corner Bliss style as benchmarked against the Telescoping HSC design.

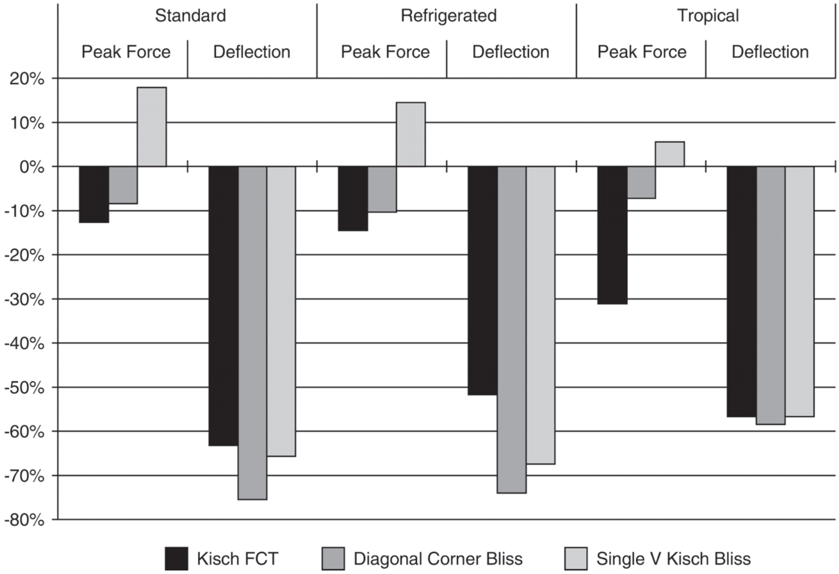


Figure 7. *Percentage Difference in Compression Test Values as Compared to Telescoping HSC.*

Table 2. *Compression Test Results.*

Box Style	**Standard**		**Refrigerated**		**Tropical**	
	Peak Force, kgf (lbf)	**Deflection, cm (in)**	**Peak Force, kgf (lbf)**	**Deflection, cm (in)**	**Peak Force, kgf (lbf)**	**Deflection, cm (in)**
Telescoping HSC	858.38 (1892.40)	1.67 (0.656)	533.70 (1176.60)	1.56 (0.616)	479.08 (1056.20)	1.43 (0.564)
Kisch FCT	762.49 (1681.00)	1.02 (0.402)	466.39 (1028.22)	1.03 (0.406)	365.58 (805.96)	0.91 (0.360)
Diagonal Corner Bliss	792.06 (1746.20)	0.95 (0.374)	483.71 (1066.40)	0.90 (0.354)	446.95 (985.36)	0.90 (0.356)
Single V Kisch Bliss	1045.89 (2305.80)	1.01 (0.396)	624.51 (1376.80)	0.93 (0.368)	507.57 (1119.00)	0.91 (0.360)

A shipper such as any of those tested, is likely to undergo compressive forces while exposed to the three climatic environments used for conditioning in this study. Comparing the average overall peak forces across all three environmental conditions of the Telescoping HSC boxes to that of the two new designs and the Diagonal Corner Bliss boxes, it was observed that:

- The Kisch FCT boxes were approximately 17% weaker, while providing material savings of over 14%.
- The Diagonal Corner Bliss boxes were approximately 9% weaker, while providing material savings of almost 22%.
- The Single V Kisch Bliss boxes were approximately 14% stronger, while providing material savings of over 19%.

It may be noted that the deflection, which is indicative of the side and bottom bulging of the boxes under compression, was considerably lower for the two replacement designs and the Diagonal Corner Bliss style as compared to that for the Telescoping HSC boxes (Figure 6). The lower peak deflection values are to be expected due to the reinforcing V columns incorporated in the new designs as well as the Diagonal Corner Bliss style boxes.

3.2 Lifecycle Environmental Impact Calculations

Table 3 reports the results of the lifecycle environmental impact calculations for all four styles of boxes. All new designs provide distinct advantage in terms of all quantified LCI values due to material savings in the designs.

Savings in material ranging from 14 to 22% for the new designs and the Diagonal Corner Bliss style tested, translates into significant energy savings, relative optimization of natural resources, reductions in green house gas emissions and relative minimization of waste water and solid waste generated during production in comparison to the Telescoping HSC style boxes.

4.0 CONCLUSIONS

Though the popularity of the Telescoping HSC and Diagonal Corner Bliss style boxes with the produce industry stems from excellent protection and superior stacking strength as compared to slotted boxes, this

Table 3. *Environmental Impact Comparison.*

Box Style	Material Used (tons)	Wood Use (tons)	Total Energy (million BTU's)	Greenhouse Gases, kg (lbs) CO_2 equivalent	Wastewater, cu. m. (gallons)	Solid Waste, kg (lb)
Telescoping HSC	1	2	24	2111 (4654)	27 (7069)	605 (1333)
Kisch FCT	0.86	1	21	1812 (3994)	23 (6065)	518 (1143)
Diagonal Corner Bliss	0.78	1	19	1651 (3640)	21 (5528)	473 (1042)
Single V Kisch Bliss	0.81	1	19	1702 (3752)	22 (5698)	487 (1074)

study shows that the alternate designs proposed can provide adequate, if not better, stacking strength while using considerably lesser material in their construction. The Kisch FCT and the Single V Kisch Bliss box designs as well as an ergonomic rotary corrugated forming machine for the Kisch FCT containers have either been patented or are patent pending.

- *Strength, resilience and sturdiness:* While the Kisch FCT and the Diagonal Corner Bliss boxes provided somewhat lower resistance to compression forces as compared to the Telescoping HSC boxes, the Single V Kisch Bliss boxes proved to be superior in comparison. Considerable decrease in peak deflection values for the new designs as well as the Diagonal Corner Bliss boxes was observed in comparison to the Telescoping HSC boxes.
- *Saving in material—commercial and environmental benefits:* agricultural products such as apples, pears, citrus, potatoes, garlic and most vegetables are currently packed into Telescoping HSC and Diagonal Corner Bliss style containers. The estimated production of these boxes is in the hundreds of millions in the US [17]. This presents a new opportunity to create considerable savings by converting to any of the new style boxes studied in this research. Saving in material translates into significant energy savings, relative optimization of natural resources, reductions in green house gas emissions and relative minimization of waste water and solid waste generated during production.

The Kisch FCT and the Single V Kisch Bliss boxes when configured with an integral divider (Figure 8) would nearly guarantee no bottom

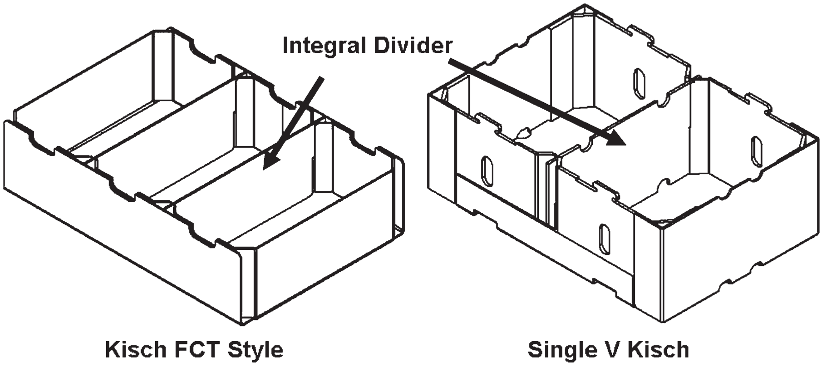


Figure 8. *Proposed Redesigns with Integral Dividers for Bulge Protection.*

bulge, thereby offering total protection for the shipping of fruit and vegetables, unlike the Telescoping HSC which typically reflects bottom bulge failure which is directly related to fruit and vegetable damage.

REFERENCES

1. R. Cook, The U.S. Fresh Produce Industry: An Industry in Transition, Chapter 2 in Postharvest Technology of Horticultural Crops, Adel A. Kader (eds.), University of California Division of Agriculture and Natural Resources, Publication 3311, 2001, pp. 5–30.
2. Produce Marketing Association, Fact Sheet, 2007, Fresh Produce Imports into U.S., http://www.pma.com, accessed May 12, 2008.
3. Produce Marketing Association, Fact Sheet, 2007, Fresh Produce Exports from U.S., http://www.pma.com, accessed May 12, 2008.
4. C. Dimitri, A. Tegene, P. R. Kaufman, U.S. Fresh Produce Markets—Marketing Channels, Trade Practices, and Retail Pricing Behavior, Agricultural Economic Report Number 825, United States Department of Agriculture, September 2003.
5. E. W. McLaughlin, G. M. Green, K. Park, Changing Distribution Patterns in the U.S. Fresh Produce Industry: Mid/Late-70s to Mid/Late-90s, E.B. 99-03, June 1999, Department of Agricultural, Resource, and Managerial Economics, College of Agriculture and Life Sciences, Cornell University, Ithaca, NY.
6. Confederation of Paper Industries, About Corrugated Packaging, http://www.ppic.org.uk/info/corrugated/aboutcorrugated.htm, accessed August 13, 2008.
7. International Corrugated Case Association, Worldwide Corrugated Packaging Industry Statistics Report—Corrugated Statistics for 1st Quarter 2007, http://www.aiccbox.org/pdf/2007_Statistics_Qtr1.pdf, accessed August 12, 2008.
8. International Corrugated Packaging Foundation, Corrugated Curricula—Course Materials on Corrugated Packaging, http://www.icpfbox.org/, accessed July 4, 2008.
9. Corrugated Packaging Alliance, Recycling Statistics, http://www.corrugated.org/Recycling/RecyclingStats.aspx, accessed August 5, 2008.
10. International Corrugated Packaging Foundation, The Facts about Corrugated Sustainability, Circulated to Packaging Programs in the US, January 2008.
11. FEFCO-ESBO Code, http://www.fefco.org/index.php?id=226, accessed January 16, 2009.
12. Singh, J., Singh, S. P., Bainbridge, P. Olsen, E. “Variability in Compression Strength and Deflection of Corrugated Containers as a Function of Positioning, Operators, and Climatic Conditions,” Journal of Applied Packaging Research, Vol. 2, No. 2, December 2007.
13. ASTM D4332-01(2006), Standard Practice for Conditioning Containers, Packages, or Packaging Components for Testing, Vol. 15.10, American Society of Testing and Materials, West Conshohocken, PA, USA, 2007.
14. ASTM D642, Standard Test Method for Determining Compressive Resistance of Shipping Containers, Components, and Unit Loads, Vol. 15.10, American Society of Testing and Materials, West Conshohocken, PA, USA, 2007.
15. T 804 om-06, Compression Test of Fiberboard Shipping Containers, 2006-2007 TAPPI Test Methods, Technical Association of the Pulp and Paper Industry, South Norcross, GA 30092.
16. Environmental Defense Fund, Lifecycle Environmental Impact Calculator, http://www.papercalculator.org., accessed April 26, 2008.
17. Corrugated Packaging Alliance, Recycling Statistics, http://www.corrugated.org/Recycling/RecyclingStats.aspx, accessed April 5, 2008.

Book Review: Embrace the Business of Sustainability

JOHN SPINK, Ph. D.
Assistant Professor, School of Criminal Justice, Michigan State University

***Green to Gold: How Smart Companies Use Environmental Strategy to Innovate, Create Value, and Build Competitive Advantage,* by Daniel C. Esty and Andrew S. Winston (New Haven: Yale University Press, 2006)**

Green to Gold is one of the most direct and applicable resources to help businesses and researchers to embrace the business of sustainability and to understand the underlying business drivers. The 2006 book provides insights that are evidenced in a wide range of successful commercial innovations by manufacturers who anticipated the risk of higher business costs and the opportunity of serving a sustainability sensitive consumer. As is discussed in this book, these manufacturers have gained a competitive advantage in volume, revenue, and profit.

THE CONCEPT

In *Green to Gold,* the authors take a business approach to environmental sustainability with a focus on providing insight, evidence, in-depth case studies, extensive interviews, and practical corporate strategic implementation tools. Probably most refreshing and valuable is the review of failures. The book presents a structured way for a company or research group to strategically and competitively "go green." Esty and Winston have a deep and broad perspective on the core drivers of environmental sustainability, based on their positions in academia, to leading projects for the Environmental Protection Agency (EPA) to consulting for the biggest companies. Make no mistake; this is a business

---

[*] Author to whom correspondence should be addressed. Email:SpinkJ@msu.edu

1557-7244/09/02 119-03

About the Author

John Spink, MSU Packaging PhD and Certified Packaging Professional, is an Assistant Professor in the School of Criminal Justice at Michigan State University where he is also the Director of the Packaging for Food and Product Protection (P-FAPP) Initiative Associate Director of the Anti-Counterfeiting and Product Protection Program (A-CAPPP). His work has included developing and teaching the graduate courses *Packaging for Food Safety, Anti-Counterfeit and Product Protection,* the packaging module of *Food Protection and Defense*, and the future of sustainability module of *Environmental Enforcement.* He has participated on the Steering Committee for Wal-Mart's Packaging Sustainable Value Network since 2005 and has participated for the last year on the Agriculture/ Food/Seafood network. Email: *SpinkJ@msu.edu.*

strategy book not an environmental manifesto.

THE CONTENT

The authors base their book structure on business strategy. For example, they draw frequently on the competitive strategy concepts of Michael Porter, who is widely regarded as the godfather of corporate strategy. Companies need to seek a competitive advantage by: (1) lower costs versus the competition and (2) differentiate products and services. An example from outside the book is Wal-mart investing in energy efficient tires and hybrid on-highway diesel engines. With Wal-mart owning the largest private truck fleet in America, competitors do not need to implement these innovations but they will be at a cost dis-advantage to Wal-mart. This competitive advantage is THE root business driver of sustainable sustainability.

The authors stress the sustainability priority must be (1) price, (2) performance, and then (3) green. This concept includes an emphasis on integrated thinking and it is constantly stressed to consider "What's really happening at the interface of business and the environment." For businesses, there are valuable quotes from recognized business leaders and strong sound bytes such as "The environment is not a fringe issue."

The book includes case studies and interviews that support that there is pressure on (1) natural resources in a growing and modernizing world, and (2) growing stakeholders care about the environment.

THE INSIGHT

For businesses or packaging research institutions, *Green to Gold* stresses the need for an understanding of the business issues and drivers. "Go Green" is the effect not the cause. There is a need to understand the underlying drivers combined with insight on what currently, and in the future, gets implemented. For example, a section of the book it titled "Correlation is not Causation." Why is a specific research project important and correlated with long-term strategic business need? Will your current research trajectory hit the long-term target?

To stay on the cutting edge you need to know where the cutting edge is. Do you clearly understand the business and environmental drivers? "Meet a need that actually exists" and "Don't get caught up in the technology and forget to make the business case." In some cases, such as reduced damage or extended shelf life, counter-intuitively more packaging could be the best environmental action! In "map the stakeholders and get them involved" it is introduced that the focus may (needs to?) shift from materials science to consumer behavior and the behavioral sciences. Regarding packaging technology innovation, a question may be "how far is far enough" versus just better use of current materials.

IN PRACTICE

Green to Gold helps businesses and research groups understand the broader competitive strategy aspects of environmental sustainability, which will lead to the selection of projects and investments that meet a current and future need.

There is continued emphasis that companies need to seek thought leaders and engage with innovative institutions. The thought leaders are more than physical or environmental scientists, but the visionaries who combine the evolving consumer needs with the business drivers with the potential of science.

GUIDE TO AUTHORS

1. Manuscripts shall be sent electronically to the editor, Dr. Jay Singh at singhjapr@gmail.com using Microsoft Word in an IBM/PC format. If electronic submission is not possible, three paper copies of double-spaced manuscripts may be sent to Jay Singh, Industrial Technology, Cal Poly State University, San Luis Obispo, CA 93407. (Telephone 805-756-2129). Manuscripts should normally be limited to the space equivalent of 6,000 words. The editor may waive this requirement on special occasions. As a guideline, each page of a double-spaced manuscript contains about 300 words. Include on a separate title page the names, affiliations, and addresses of all the authors, and identify one author as the corresponding author. Because communication between the editor and the authors will be electronic, the email address of the corresponding author is required. Papers under review, accepted for publication, or published elsewhere in journals are normally not accepted for publication in the *Journal of Applied Packaging Research.* Papers published as proceedings of conferences are welcomed.
2. Article titles should be brief, followed by the author's name(s), affiliation, address, country, and postal code (zip) of author(s). Indicate to whom correspondence and proofs should be sent, including telephone and fax numbers and e-mail address.
3. Include a 250-word abstract and 5–6 keywords.
4. If electronic art files are not supplied, submit three copies of camera-ready drawings and glossy photographs. Drawings should be uniformly sized, if possible, and planned for 50% reduction. Art that is sent electronically should be saved in either a .tif or .JPEG format for superior reproduction. All illustrations of any kind must be numbered and mentioned in the text. Captions for illustrations should all be typed on a separate sheet(s) and should be understandable without reference to the text.
5. DEStech uses a numbered reference system consisting of two elements: a numbered list of all references and (in the text itself) numbers in brackets that correspond to the list. At the end of your article, please supply a numbered list of all references (books, journals, web sites etc.). References on the list should be in the form given below. In the text write the number in brackets corresponding to the reference on the list. Place the number in brackets inside the final period of the sentence cited by the reference. Here is an example [2].

   Journal: 1. Halpin, J. C., "article title", *J. Cellular Plastics,* Vol. 3, No. 2, 1997, pp. 432–435.
   Book: 2. Kececioglu, D. B. and F.-B. Sun. 2002. *Burn-In Testing: Its Quantification and Optimization,* Lancaster, PA: DEStech Publications, Inc.

6. Tables. Number consecutively and insert closest to where first mentioned in text or type on a numbered, separate page. Please use Arabic numerals and supply a heading. Column headings should be self explanatory and carry units. (See example at right.)
7. Units & Abbreviations. SI units should be used. English units or other equivalents should appear in parentheses if necessary.

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

8. Symbols. A list of symbols used and their meanings should be included.
9. Page proofs. Authors will receive page proofs by E-mail. Proof pages will be in a .PDF file, which can be read by Acrobat Reader. Corrections on proof pages should be limited to the correction of errors. Authors should print out pages that require corrections and mark the corrections on the printed pages. Pages with corrections should be returned by FAX (717-509-6100) or mail to the publisher (DEStech Publications, Inc., 439 North Duke Street, Lancaster, PA 17602, USA). If authors cannot handle proofs in a .PDF file format, please notify the editor, Jay Singh at singhjapr@gmail.com.
10. Index terms. With proof pages authors will receive a form for listing key words that will appear in the index. Please fill out this form with index terms and return it to the publisher.
11. Copyright Information. All original journal articles are copyrighted in the name of DEStech Publications, Inc. All original articles accepted for publication must be accompanied by a signed copyright transfer agreement available from the journal editor. Previously copyrighted material used in an article can be published with the written permission of the copyright holder (see #14 below).
12. Headings. Your article should be structured with unnumbered headings. Normally two headings are used as follows:

    Main Subhead: DESIGN OF A MICROWAVE INSTALLATION
    Secondary Subhead: Principle of the Design Method

    If further subordination is required, please limit to no more than one (*Third Subhead*).

13. Equations. Number equations with Arabic numbers enclosed in parentheses at the right-hand margin. Type superscripts and subscripts clearly above or below the baseline, or mark them with a caret. Be sure that all symbols, letters, and numbers are distinguishable (e.g., "oh" or zero, one or lowercase "el," "vee" or Greek nu).
14. Permissions. The author of a paper is responsible for obtaining releases for the use of copyrighted figures, tables, or excerpts longer than 200 words used in his/her paper. Copyright releases are permissions to reprint previously copyrighted material. Releases must be obtained from the copyright holder, which is usually a publisher. Forms for copyright release will be sent by the editor to authors on request.

General: *The Journal of Applied Packaging Research* and DEStech Publications, Inc. are not responsible for the views expressed by individual contributors in articles published in the journal.