THE IMPACT OF PACKING LINE PROCESSES ON GLOSS DEVELOPMENT, AND THE USE OF GLOSS AS AN INDICATOR OF COATING INTEGRITY AND QUALITY OF ‘RED DELICIOUS’ APPLES (*Malus domestica*)

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ABSTRACT

THE IMPACT OF PACKING LINE PROCESSES ON GLOSS DEVELOPMENT, AND THE USE OF GLOSS AS AN INDICATOR OF COATING INTEGRITY AND QUALITY OF 'RED DELICIOUS' APPLES (*Malus domestica*)

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Apples are coated with food grade waxes to maintain and improve quality, such as color and firmness, and to enhance gloss. In this study, a combination of simulated packing line cleaning and wax application processes were used to evaluate their efficiency in cleaning and developing gloss on 'Red Delicious' apples.

Commercially available alkaline and neutral detergents were applied in a pilot packing line while varying the temperature and dwell time in the dump tank (DT) together with the rinse-water pressure and dwell time on the washing brushes (WB) to simulate the cleaning process. A solvent extraction system quantifying surface residue after cleaning was developed to evaluate cleaning efficiency. The alkaline detergent was more effective in acquiring a cleaner surface than the neutral detergent. Increasing the temperature of the DT also resulted in a significantly cleaner surface.

Cleaned fruit were subsequently waxed with commercially available shellac fruit coatings at different viscosities; they were applied and "dried" in different environmental conditions of temperature and relative humidity (RH) to investigate the conditions conducive for gloss development and the maintenance of the quality attributes of weight and firmness. A non-destructive device for measuring gloss on curved surfaces was developed and correlated to the human perception of gloss. The wax viscosity with RH in the drying zone, and the duration of drying significantly affected the gloss. The inability to remove moisture from the apple surface in the drying zone under high RH conditions in the environment decreased the ensuing gloss as a result of dilution and therefore a decrease in the deposition of the coating thickness. Wax with higher viscosity “dried” under lower RH conditions produced significantly higher gloss fruit compared to a lower viscous formulation. Furthermore, the decay of gloss during storage was accompanied by decreasing attributes of weight and firmness as the uniformity and integrity of the wax coating deteriorated.

Respiration as influenced by the availability of O_2 was impacted by the barrier properties of the peel and storage temperature. Hand-coated shellac ‘Red Delicious’ apples were “dried” at 50 ^{o}C under two RH conditions of 25 and 60%, and stored at 4, 10 and 20 ^{o}C for a month. In this controlled system, it was determined that the drying treatments resulted in varied coating thickness, surface roughness and gloss. Surface gloss was determined to be directly related to coating thickness, and inversely to surface roughness through the use of micrographs. The transmission of respiratory gases through shellac was studied by bar-coating polyethylene film with different shellac wet thicknesses, 4 and 10 μm. The transmission rate of O_2 was higher at higher temperatures with a corresponding increase in respiration rate, production of CO_2 per g of fruit. The increase in respiration rate was accompanied by an increase in the loss of fruit weight. All of these increases were less pronounced in higher gloss fruit. In contrast the internal CO_2 concentration was higher for the higher gloss fruit. Coated fruit, compared to the uncoated, recorded lower respiration rates with significantly reduced gloss and weight loss.

KEYWORDS: ‘Red Delicious’ Apples, Packing Line, Fruit Detergent, Shellac Coating, Gloss, Microstructure, Quality, Respiration Rate, O_2 and CO_2 Transmission Rate.

"To make a great dream come true, the first requirement is a great capacity to dream; the second is persistence – a faith in the dream" – Hans Selye, MD

This is dedicated to my husband, **Charles Mawuse Ofori**, for his encouragement and incredible support through all of my dreams, desires and goals.

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CHAPTER 1

INTRODUCTION

1.1. Introduction

With the upsurge of diet and health awareness in recent years, consumers are opting for the healthy foods category under which fresh produce falls. However, appeal places a pivotal role in determining the quality (texture, taste, etc.) of the produce and the consumers' decision to purchase, let alone consume them. The fundamental appeal is derived from attributes like appearance, flavor, aroma and texture; these attributes have been used in many studies to measure the overall quality.

Even though the state of Michigan is ranked third in apple production in the United States, and apples are Michigan's largest fruit crop by volume [*1*], its 'Delicious' apples have experienced a loss in market share with a decline in sales in the last few years [*2*]. This trend has been attributed to the lack of appeal of the fruit to the consumer. Though the gloss of Michigan apples are deemed to be lower than that of Washington's and hence the relatively lower sales, it has been documented by horticulturists that 'Red Delicious' producers in the United States are faced with the pressure to provide optimum quality fruit after storage [*3*]. The problem is therefore not confined to Michigan only.

Prior to being delivered to the consumer after harvesting, apples are subjected to a number of postharvest processes including storage, washing, sorting, waxing, drying and packaging. The apple fruit is then consumed as fresh or processed for use in pies, sauce, juice and cider; the determinants of its uses are uniformity of color, size, and the absence/presence of defects amongst many others. Thus the perceived quality is the dictation factor in its end use.

Apples, pears, oranges and other produce are coated with food grade waxes; the applications of natural waxes such as shellac and carnauba on apples have been documented to improve the fruit appearance by imparting gloss in addition to retarding weight and firmness loss, and delaying ripening during storage [*4*]. A reduction in weight loss is the result of decreased transpiration, by means of the presence of a gaseous barrier derived from the applied coating, which translates to firmness retention. Thus coating fruit not only enhances appeal but also serves as a barrier to moisture loss resulting in weight loss control and firmness preservation, both of which are measures of quality and lead to enhanced preservation and extension of shelf life.

Shellac and carnauba coatings are most popular in the produce industry. Though carnauba wax provides a lesser gloss compared to shellac, the finish does not face the problems of whitening [*5*]. In view of this a lower gloss may compensate for the risk of higher gloss with the tendency of whitening in humid environments. However, it has been found by researchers that shellac coatings are excellent for dark ‘Red Delicious’ apples as it “impacts gloss, hides bruises and forms a modified atmosphere condition that tends to preserve firmness and prolong shelf life for this variety” [*6*]. To optimize the aesthetics and preservation qualities of the fruit, the surface of the fruit needs to be clean, warm and thoroughly dry prior to the application of the coating for a good finish.

1.2. Research Driver

This study was motivated by the need to evaluate and improve the appeal and quality of Michigan ‘Red Delicious’ by investigating the effect of the different factors on the packing line on gloss development. The appearance of the fruit which is mainly the result of genetic and environmental factors is enhanced by processes on the packing line, and the choice of ‘Red Delicious’ was because it is the most produced and consumed variety in the US and Michigan.

Many studies aimed at enhancing the quality of 'Delicious' have looked at factors such as firmness, soluble solids, internal ethylene concentration, and the development of coatings to enhance quality. These studies have related fruit coatings to quality attributes such as weight loss [*6, 7*], flesh firmness [*4, 5*], soluble sugar content [*4, 7*], coating permeability [*6, 8, 9*], volatiles concentration [*10-12*], internal atmospheres [*4, 6, 8*] and fruit respiration [*8, 10, 11*] but only a few [*5, 6, 11*] have directly correlated measurements of gloss to these attributes. Since applying coatings to fruit introduces a barrier to moisture with modification in the atmosphere of the fruit, changes in physiological activity occur. These changes, inclusive of respiration and transpiration, lead to texture preservation by means of reduced moisture and weight loss. Thus, gloss derived from applying coating has a direct impact on texture quality attributes, but the magnitude of the correlation will be dependent on the uniformity and integrity of the dried coating which is dictated to a large extent by waxing conditions, giving rise to the need to investigate the relationship between these.

The gloss of the skin embodies the overall appearance which is what attracts the consumer in the first place before intrinsic factors like flavor and firmness come into play; it is therefore critical for Michigan apples to meet the high quality standards in appearance. Gloss measurements to evaluate the quality of produce like apples is challenging because of the curvature of their surface. Most commercially available glossmeters are predominantly designed for flat surfaces and therefore require a device modification or destruction of the produce to successfully measure the gloss of curved surfaces. Very few studies [*13-17*] have addressed the challenge of measuring gloss of uneven curved surfaces, and the need to provide a solution to the problem cannot be over emphasized.

1.3. Goal and Objectives

The goal of this research was to improve the appearance and value of Michigan 'Red Delicious' by enhancing the gloss through optimized packing line processes. This was achieved by evaluating and altering packing line protocols, as well as correlating gloss to quality attributes of the fruit. A good wax finish for enhanced appeal requires that several factors on the packing line such as the washing, drying and application of the coating be defined and monitored. Thus the objective was to evaluate and improve the packing line to achieve the desired fruit finish and enhanced quality by:-

1. Validating a new method to measure gloss
2. Identifying the parameters on the washing section of the line that will ensure efficient cleaning of the fruit surface in preparation for further processing
3. Evaluating different wax application treatments on gloss development as a function of environmental conditions of temperature and relative humidity
    a. Studying gloss decay with storage
    b. Studying the effect of gloss on the quality attributes of firmness and weight loss during storage
4. Investigating the effect of coating on the peel barrier properties and analyzing the impact of coating integrity and gloss on the respiration rate of the fruit, and hence their influence on the loss or retention of quality
5. Correlating wax formulation to gloss and recommending formulations that work better in the humid Michigan environment
6. Verifying the findings from the pilot study on a commercial production line

1.4. Hypotheses

To achieve good gloss and consumer appeal, cleaning and wax application variables as well as environmental factors need to be optimized. Apples have a layer of natural wax together with debris, chemicals deposited on the fruit from sprays and other extraneous variables that have to be removed via efficient cleaning prior to packaging and consumption. Following cleaning, the fruit are coated with food grade waxes. Temperature and humidity affect the waxing, and according to packing house personnel, though no empirical data exist, the surface of the fruit needs to be clean, dry and warm for a good wax finish.

Hypotheses were set in place to be tested and aid decision making on the effect of packing line variables and environmental factors on the overall quality of 'Red Delicious'.

Hypothesis I – The impact of the packing line on surface cleanliness

A combination of different cleaning parameters will result in varying degrees of surface cleanliness. The use of fruit cleaner, increases in temperature of the dump tank, dwell time in both the dump tank and on the washing brushes, and rinse pressure on the packing line will improve surface cleanliness. These hypotheses were established to make statistical decisions to prove or disprove the above:

Null Hypothesis (Ho): Fruit cleaned by different washing treatments will have the same level of cleanliness.

Alternative Hypothesis (Ha): Different levels of cleanliness will be observed in fruit cleaned by different washing treatments.

Rationale

Fruit cleaners are formulated to cleanse the fruit from contaminants and other deposits, thus the use of appropriate agents will achieve the goal of a cleaned surface. Also most of the fungicides, pesticides and chemical sprays used on the field are water soluble and therefore longer contact with the cleaning system will increase dissolution. With efficient monitoring to avoid heat injury, temperature is well able to remove the natural wax on the fruit, and pressure is able to dislodge debris that may not easily be removed by the other washing factors considered.

Hypothesis II – The effects of coating formulation and environmental conditions on gloss

Cleaning fruit tends to remove the natural waxes from the surface of the fruit [*8*]. The use of fruit coatings not only replaces the function of the natural wax removed during washing, but imparts a sheen which enhances the aesthetic value of the fruit, thus improving visual appeal, quality and marketability. However environmental conditions of temperature and humidity affect the way the wax adheres to the fruit, markedly resulting in different levels of gloss. Gloss is affected by the type of coating formulation and its flow properties. These hypotheses were established to make statistical decisions to prove or disprove the above:

Null Hypothesis (Ho): The wax formulation, and the temperature and humidity conditions in which it is applied and dried in have no effects on the sheen of the fruit.

Alternative Hypothesis (Ha): The wax formulation, and the temperature and humidity conditions in which it is applied and dried in have effects on the sheen of the fruit.

Rationale

Fruit coatings containing volatiles, isopropanol and morpholine, in their formulation are susceptible to changes in their rheological profiles and hence their flow behavior. A viscous coating with reduced flow properties may inhibit uniform fruit surface coverage thus affecting coating integrity which influences gloss. The environmental conditions on the packing line influence the dew point. Warm conditions favor good curing of the wax, but if the surface temperature is at or below the dew point, condensation will form on the apple surface and interfere with wax application. High humidity conditions also pose the threat of washing off the applied wax which will lower the level of expected gloss and affect quality.

Hypothesis III – The influence of coating on the diffusion of respiratory gases

The resistance of the fruit skin to respiratory gases and water vapor is altered with the application of commercially formulated coatings, which form a barrier to the passage of gases through the peel. These hypotheses were established to make statistical decisions to prove or disprove the above:

Null Hypothesis (Ho): The respiration rate of fruit is not influenced by the application of coatings.

Alternative Hypothesis (Ha): The respiration rate of fruit is influenced by the application of coatings.

Rationale

Coated fruit in comparison to uncoated fruit have reduced respiration rates and water loss from the fruit as a result of changes in physiological activity. The transfer of gases between the fruit and the atmosphere may decrease as a result of the combined effect of reduced fruit respiration and the coating barrier properties. Also resistance of the peel to respiratory gases can change in response to environmental conditions of temperature and relative humidity. Peel resistance is also highly influenced by the integrity of the coating layer and its characteristics. According to

Banks *et al.* [*18*], it is the manner in which the coating adheres to the peel, loosely or tightly, and also the proportion of the pores in the peel that are covered and blocked by the coating that determines the respiratory response of the fruit to the coating.

1.5. Research Plan

In order to achieve the goal of this research, the work was divided into 4 phases: I) Surface Preparation; II) Pre-Waxing; III) Wax application; and IV) Packing Line Recommendations. The work plan of the phases is detailed in Figure 1.1.

Phase I

In this phase apples were treated with combinations of the different factors listed in the 'Phase I' section in Figure 1.1.

1. A full factorial design was used to assess the effect of each factor and their 2-way interactions on surface cleanliness prior to wax application. A method to extract the residue remaining on the fruit after the application of each specific washing treatment was developed as a means to quantify and evaluate the degree of cleanliness that resulted from subjecting the fruit to different combinations of washing treatments.
2. Surface dryness is critical in achieving a good coating finish. Coated fruit were dried for 90 or 300 s at 50 ^{o}C in either 25 or 60% RH; the purpose of varying the drying time and humidity was to simulate different lengths of drying tunnels and climatic conditions respectively. A method for quantifying surface dryness, by means of quantifying the moisture left on the apple surface after drying, to enable a correlation with resultant gloss was developed. The surface temperature of the apples emerging from the different drying conditions was also measured.

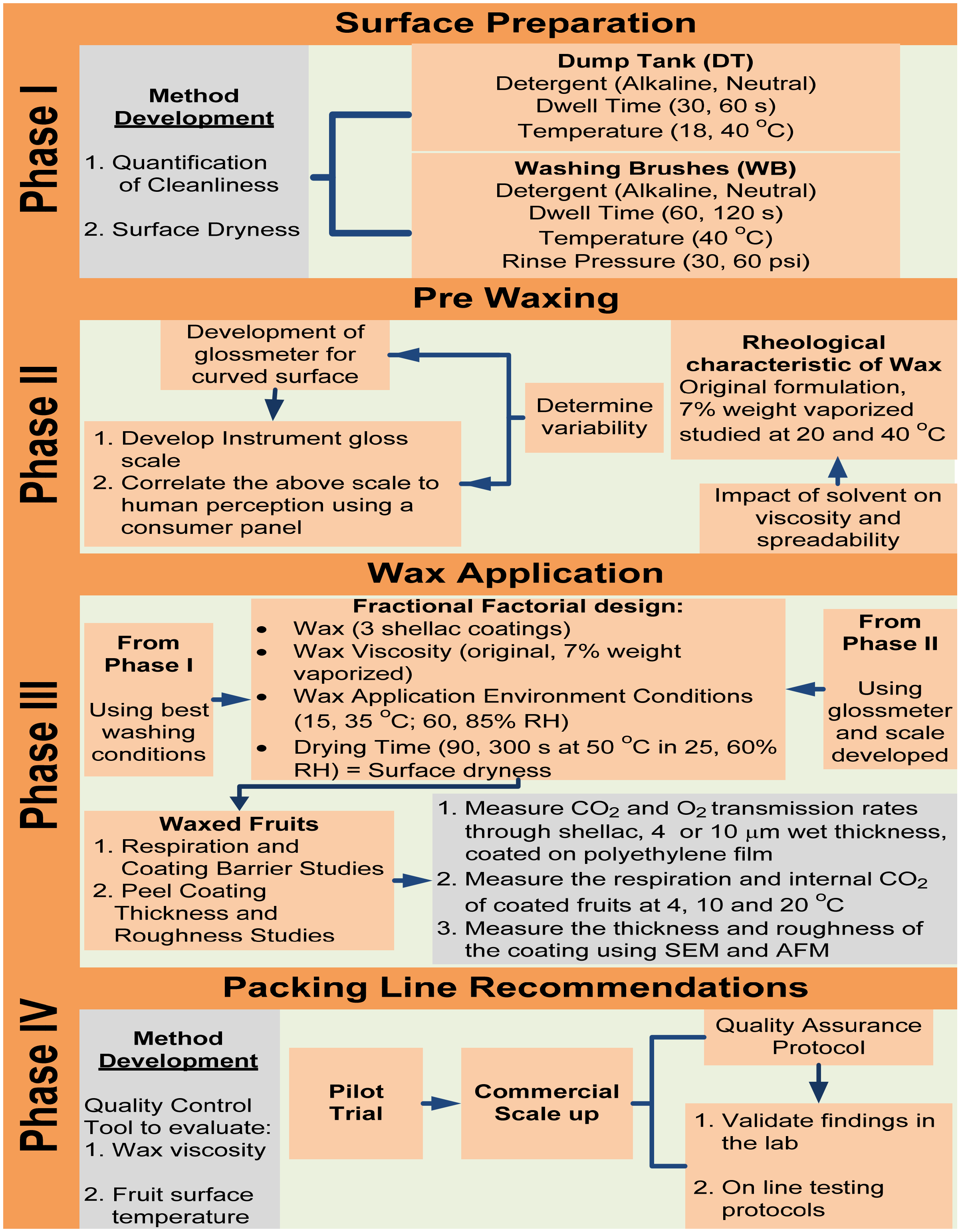

Figure 1.1. Work plan of the study

For the interpretation of the references to color in this and all other figures, the reader is referred to the electronic version of this dissertation.

Phase II

This phase together with Phase I was required to lay the foundation for Phase III; this included developing a method to measure surface dryness, studying the rheological profile of the shellac coatings to be used, and developing a device for measuring the gloss on curved surfaces.

1. Washed and dried fruit from Phase I were waxed with shellac coatings in Phase III. Gloss, from the application of wax, needed to be measured to enable a correlation with waxing application conditions. Since most of the commercial glossmeters are designed for measuring gloss on flat surfaces, a customized gloss device with the capability of non-destructively measuring the gloss on the curved surface of apples was designed and built. It was important that the gloss measured by the new device could be perceived by the human senses, and therefore required the use of a consumer panel to establish a correlation between the instrument's measurements and the human perception of gloss.
2. Shellac fruit coatings have a volatile solvent base, composed of isopropanol and morpholine. Evaporation of these solvents concentrates the solid content which increases viscosity. An increase in viscosity which impedes flow may affect coating spreadability on the fruit surface. Rheological profiling of the waxes was conducted to enable a correlation between the wax flow properties and the resulting gloss.

Phase III

Fruit emerging from the best identified washing treatment in Phase I were used in this phase.

1. The shellac waxes were applied in 15 or 35 ^{o}C, and 60 or 85% RH. Following waxing, the fruit were dried for either 90 or 300 s at 50 ^{o}C in 25 or 65% RH. The coated fruit were stored

at 4 ^{o}C in 95% RH regular atmosphere for 8 weeks and monitored for gloss, weight and flesh firmness changes fortnightly.

2. Both coated and uncoated fruit were stored at 4, 10 and 20 ^{o}C for respiration studies; investigations included the effect of the presence of a coating layer, coating application conditions by means of gloss levels, and storage temperature on the rate of respiration.
3. Coatings create a barrier to respiratory gases. The O_2 and CO_2 barrier properties of the coatings were studied by measuring their transmission rates through coatings of different thicknesses at 10, 15 and 20 ^{o}C using polyethylene film as the carrier.
4. In an effort to better understand the effect of coating on gloss and the barrier properties of the peel, with their corresponding effects on changes in weight, firmness and respiration rates, microstructural studies were conducted. The roughness and thickness characteristic of the coated peel that evolved from using varied amounts of coating were studied using the Atomic Force Microscope (AFM) and Scanning Electron Microscope (SEM) respectively.

Phase IV

A scale up was carried out on a commercial packing line. The findings of the pilot trials in the laboratory were adapted to a packing line at one of the packing houses in Michigan. At the completion of the scale up a ‘guideline for good practices for achieving good coating finish’ document was made available to packers for their reference.

1.6. Overview

Chapter 2 of this document is the literature review. Chapter 3 elaborates on the effect of washing fruit, using different cleaning treatments, on the surface cleanliness of the fruit, as well as the design of a customized non-destructive gloss device. The effect of waxing conditions on gloss, weight and firmness; and the changes in respiration rates with coating and varied storage temperatures are discussed in chapters 4 and 5 respectively. Chapter 6 summarizes and concludes the study.

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CHAPTER 2

LITERATURE REVIEW

2.1. The Apple Fruit

The US is one of the four key apple producers in the world, coming second to China [*1*] but third to the EU-27 in 2009 [*2*]. Michigan is the third largest apple producer in the US after Washington and New York; it produced 12% of US apples in 2009 compared to 54 and 14% from Washington and New York respectively. Its production was down 4% from 2007 and 3% from 2006 as a result of frost and hail damage. However, its production was up 6% from 2008 achieving record highs above historic levels [*3*]. Apples are Michigan's most valuable fruit crop and the largest by volume, representing over half of the state's total fruit production [*4*]. Out of the hundreds of commercial apple varieties, the most prevalent dominating the US and Michigan orchards is the 'Red Delicious', followed closely by the 'Golden Delicious'. However, the 'Gala' and 'Fuji' apples are rapidly gaining market share [*1, 5*].

With the development of new products and technologies, in the last decade, that result in reduction in product losses and open avenues for new markets, the apple industry worldwide is faced with intense competition. These factors resulted in the saturation of the supply chain, seen in the scenario where fruits from the previous year have to be dumped because of the onset of the harvest of new fruits. There is however capacity to increase utilization of fruits especially in the geographical areas deemed not to meet the minimum requirements of fruit consumption [*6*]. Regional and national discussions on the need to increase fruit and vegetable consumption to curb future public health issues resulting from low produce consumption have been ongoing [*6, 7*]. Efforts have been made to identify the underlying factors contributing to the low fruit consumption among populations.

A recent Swiss surveyed 5062 visitors to a local food exhibition and showed that apple consumption trends are influenced by both gender and age. Out of the 14.3% of the consumers that ate more than 6 apples per week, 10% were under the age of 40, 20% between 50 – 70years and over 40% above the age of 70 [*8*]. Péneau *et al.* [*8*] attributed this trend to the possibility of the older generation doing so out of tradition when there were fewer options. Other studies have also shown the general significant low consumption rate of fresh produce by the younger generation [*9*]. The percentage of men consuming less than an apple a day was double that of 10% by women [*8*]; a similar trend was reported by Thompson *et al.* [*9*]. Consumer education on health benefits from consumption trends will not only promote health and reduce waste, but will also open avenues for unexploited capacity in the potential growth of the fruit industry. Nutritional quality has been categorized as one of the attributes used to judge freshness and quality but only a minority make a purchase decision on the basis of nutritional profile [*10*]. A 2006 study on consumers' perception of apple freshness by the same Swiss team [*8*] revealed that not much importance was attributed to nutritional value. However in the cases where it did, age and consumption rate as well as gender influenced the consideration of nutritional quality. More importance was given to derived nutrient with an increase in the consumers' age and in consumers eating at least 5 apples per week. Females also attributed more importance to nutritional value than males.

More emphasis on changes in consumer attitudes to consumption than modifications to supply [*6*] may sound like the best solution to increasing fruit utilization. Though a glossy appearance like in many fresh produce may not necessarily have a parallel to quality, it is used to judge apple quality [*11, 12*]. Modern consumers are reported to demand impeccable appearance which comprises the intensity and uniformity of skin color and blemish-free fruits [*13*]. Michigan 'Red Delicious' apples have suffered a loss of market share and reduced sales as a result of perceived lower quality in comparison to other cultivars of the crop and the same cultivar from different regions [*14*]. Therefore how does the industry change the consumer consumption trend if the fruit is not appealing enough to influence their purchasing intent, let alone consumption of it?

Over 60% of all Michigan apples are processed; in 2008 about 73% of the apples were processed, up 7 and 9% from 2007 and 2006 respectively but down 16% in 2009 as a result of a better growing season and larger sized fruits [*3*]. The increase in the amount used for value-added processed products from previous years was the result of weather damaged and under-sized fruits. While Michigan apples are the main source for apple sauce, fresh and processed fresh-cut slices and apple cider [*5*], fresh apples have a higher economic value than those sold for processing into value-added products. The fresh market generated $0.355/lb in comparison to $0.135/lb for that sold for processing in 2008, whereas in 2009 it generated $0.215/lb and $0.07/lb respectively [*3*]; the lower pricing in 2009 was the result of the abundance of fruits. Therefore the economic need is to increase the proportion of the sale of fresh Michigan apples to increase its economic value and the revenue generated from it.

'Red Delicious' has received great attention from producing and exporting areas of the world. In addition to a high percentage of red coloration for red apples, most guidelines dictate optimum pre- and post-storage appearance and intrinsic qualities for highest grade fruits [*15*]. According to Fellman *et al.* [*15*] there is the need to improve the industry's capacity to produce, store and market optimal quality fruits all year round in order to fortify the consumer's confidence in the 'Red Delicious' apple. Though the initial purchase may have been made on the basis of appearance, good edible quality is what assures the consumers' satisfaction, confidence and repeated purchases [*16*]. Therefore the need for apples to meet the high quality standards in flavor and texture besides appearance cannot be overemphasized.

Just as research in orchard management, postharvest handling and storage have allowed the production and availability of quality fruits all year round, it is believed that the processing used in Michigan can be optimized through research to produce glossy apples that will compete with those from other regions. This study aimed at improving the visual quality of Michigan 'Red Delicious' to the same level as those harvested and packaged in Washington, and in so doing gain market shares. Improving visual appeal mainly achieved by enhancing gloss with fruit coatings comes with added benefits of quality preservation. This is discussed further in this document. This study also addresses the need to evaluate and quantify gloss to facilitate its use as an indicator of fruit quality.

2.2. What is Gloss?

The appearance of both inanimate and animate objects is crucial to humans, and specular gloss (light distribution by the commodity) is widely used to measure the quality of a surface [*17*]. Gloss is used to describe the appearance of a material and is basically the interaction of light with the surface of interest. It therefore describes a surface's ability to reflect light in the specular direction. The topography of a surface, the angle of the incident light and the refractive index of the surface affect gloss; it is the superposition of these factors that influence surface appearance [*17, 18*].

Gloss is a geometric attribute that arises from the spatial distribution of light from an object. It is the attribute of surfaces that is responsible for the perception of a shiny or lustrous appearance, and its perception is highly dependent on how light is distributed by an object. According to Hunter *et al.* [*18*], gloss, as associated with specular reflection, varies from one surface to the other by:

(i) the amount of light, relative to the beam, reflected in the specular direction

(ii) the pattern and degree to which the reflected spreads away from the specular direction

(iii) how varying the specular angle changes the specular reflection.

The perception of gloss, according to the same team [*18*], is a combination of six sensations, not necessarily in equal proportions:

(i) ***Specular gloss*** occurs in medium-gloss surfaces like those of book paper, paint and plastics. It is the shininess and brilliance of highlights, usually measured at 45^{o} to the normal.

(ii) ***Sheen*** is the shininess, viewed at almost grazing angles of 85^{o}, of low-gloss (matte) surfaces like that of paper and paint.

(iii) ***Contrast gloss*** or ***luster*** is the contrast between specularly reflecting areas and other areas. Low gloss surfaces of textile fiber, yarn and cloth, newsprint, bond paper, diffuse-finish metals, fur and hair are examples of surfaces that exhibit this visual criterion.

(iv) ***Absence-of-bloom gloss*** is the absence of a haze or milky appearance adjacent to specularly reflected beams. This occurs in high- and semigloss surfaces in which specularly reflected highlights may be seen.

(v) ***Distinctness-of-image gloss,*** which is the distinctness and sharpness of mirror images, occurs in all types of high-gloss surfaces in which mirror images may be seen.

(vi) ***Surface-uniformity gloss*** deals with the freedom from visual surface non-uniformities such as texture which diminishes the intensity of gloss. It can occur in all types of medium-to-high gloss surfaces with the absence of texture or markings from which the position of a surface may be identified.

2.2.1. How Surface Characteristics Dictate Gloss

When evaluating visual properties, the gloss of the surface is one of the most important properties considered; gloss measurements are a routine in assessing quality. Specular gloss is an important factor in the estimation of the quality of smooth and rough surfaces [*17*]. The amount of light reflected, when light encounters an object, is largely dependent on the nature of the surface, whether it is smooth or rough. Reflected light, perceived as specular reflection, is responsible for gloss of an optically smooth surface. The lack of a glossy reflection is indication of a rough surface; light is scattered in many directions when it encounters a rough or textured surface. The intensity of the specular reflection is therefore weaker, and the irradiance distribution is shorter and wider as the surface roughness increases. Also, all of the specularly reflected light may therefore not be collected by the photodetector leading to lower readings [*17, 18*]. In summary, light distributions determine the perception of our judgment of an object [*18*].

2.2.2. The Measurement of Gloss

Appearance evaluation, which used to be an art, has in recent years become a science. Appearance measurements are made to obtain numbers that represents the way objects look. This crosses a broad range of industries, from food to automobile to clothing and furniture, where consumers will judge appearance and link it to what they deem as quality. Appearance measurements span from the developmental stages (research and development) to production (quality control of the raw products, during processing and the finished product) to the end use (performance evaluation) [*18*]. The first step in designing instruments that can measure what the human eye perceives is to recognize the relationship between appearance and the specific optical phenomena. The four main optical phenomena are recognized as diffuse reflection, specular reflection, diffuse transmission and regular transmission [*18*].

Visual sense is complicated and experience plays a major role in the evaluation of what is observed. Nerve impulses, on encountering light patterns, are complexly sorted for the identification of objects, movements, etc. "Patterns of light entering the eye are the stimuli on which appearance judgments are based" [*18*]. At a tender age, using other senses for confirmation, one tends to develop the abilities for visual discrimination using the eyes and brain. There is a certainty for branding an object as dirty or clean, glossy or matte, fresh or stale, without necessarily being aware of the optical processes that led to the judgment; these evaluations are done with very little conscious analyses of the specific optical attributes that lead to the decision [*18*].

Whereas the eye can distinguish between a low gloss reading due to a poor image reflecting quality of the object and that as a result of surface curvature deflecting the specular reflection, an instrument cannot. Though the eye is more versatile in assessing numerous geometric factors simultaneously, it is subjective with observer variability. An instrument is better able to provide repeatable evaluations than the unaided eye. Also, an instrument possesses the ability to output geometric measurements with numbers whereas the eyes lack that ability to numerically express attributes though it is a sensitive comparator. Most gloss devices are based on the detection of specular gloss which is the ability of a surface to reflect light specularly [*17*], but the design of instruments to make similar inferences as humans requires the knowledge of the behavior of light and its interactions with objects. An investigation of the human response to light and the decisions on the attributes of an object, defines the proper scale to use for appearance-measuring instruments, once an established correlation with visual perception is made [*18*]. Of the varying current universal methods for measuring gloss, each method for a specific class of surfaces was designed with correlations between visual appraisals and instrumental values of gloss. Each optically unique product requires its own unique method for measuring gloss which is the reason for the varied American Society for Testing and Materials (ASTM) angular (20, 45, 60, 75 and 85^{O}) methods employed in gloss instrumentation to meet unique application requirements. Low angle methods are known to better distinguish high-gloss specimen while the high angle methods are best known for use on low-gloss materials [*18*].

Two categories of instruments are employed in appearance measurements. Physical analysis instruments like the spectrophotometer measure the physical properties of the light distributed by the object, while psychophysical analysis instruments such as glossmeters and colorimetric spectrophotometers are designed with information about how the observer perceives the distributed light. The latter's measurements are correlated to the human perception of the attribute of interest. Both spectral and geometric considerations are made in the design of such instruments. Spectral properties are most important in designing color-measuring devices but for geometric devices (gloss, haze measurements) it is the angular and directional dimensions of the instruments that are important. However, both properties must be efficiently controlled for the accuracy and reproducibility of measurements [*18*]. Appearance-measuring instruments like the glossmeters are designed with a light source, placement for the object to be measured, a light receiver, and a signal measurement device. For glossmeters, the photodetector (light receiver) for the specular reflection is placed at the same but opposite angle to that of the incident beam. Light sources used in appearance-measuring devices include the incandescent, fluorescent, xenon arc and laser. The incandescent source is the most common because of its low cost, ability to control output with a power control, ability to produce a steady intensity output, among other compelling factors [*18*].

The judgment of food qualities is greatly influenced by the perception of factors such as gloss, oiliness, stickiness and softness. Using conventional glossmeters for most foods require a destructive preparation of the surface for measurements, because the need for an extended flat surface makes it impossible to measure gloss of the invariably curved and uneven surfaces of food efficiently [*19*]. Measurement of reflectance with respect to the angular properties of the incident and reflected can be made with a goniophotometer. A goniophotometer measures the quantity of light reflected or transmitted as a function of the angle of incidence, providing information about the spatial distribution and therefore geometric attributes [*18, 19*]. Goniophotometric properties – changes in the intensity of the specularly reflected as a function of the incident and viewing angles – give rise to the perception of gloss of curved surfaces [*19*].

2.3. Postharvest Handling and Storage

Apples are harvested commercially with a range of different maturities depending on it immediate end use. Once the fruits leave the orchard they are either packed immediately for the consumer, or stored in refrigerated or controlled atmosphere (CA) for future processing. Improper handling and storage aggravate defects that may already be present or initiate deterioration, both of which lead to the loss of quality.

Produce like apples that are not field-packed upon harvesting, are picked into field containers and transported to packing houses for storage and further processing [*20*]. Figure 2.1 depicts the typical unit operations used in an apple packing house for delivery to the consumer.

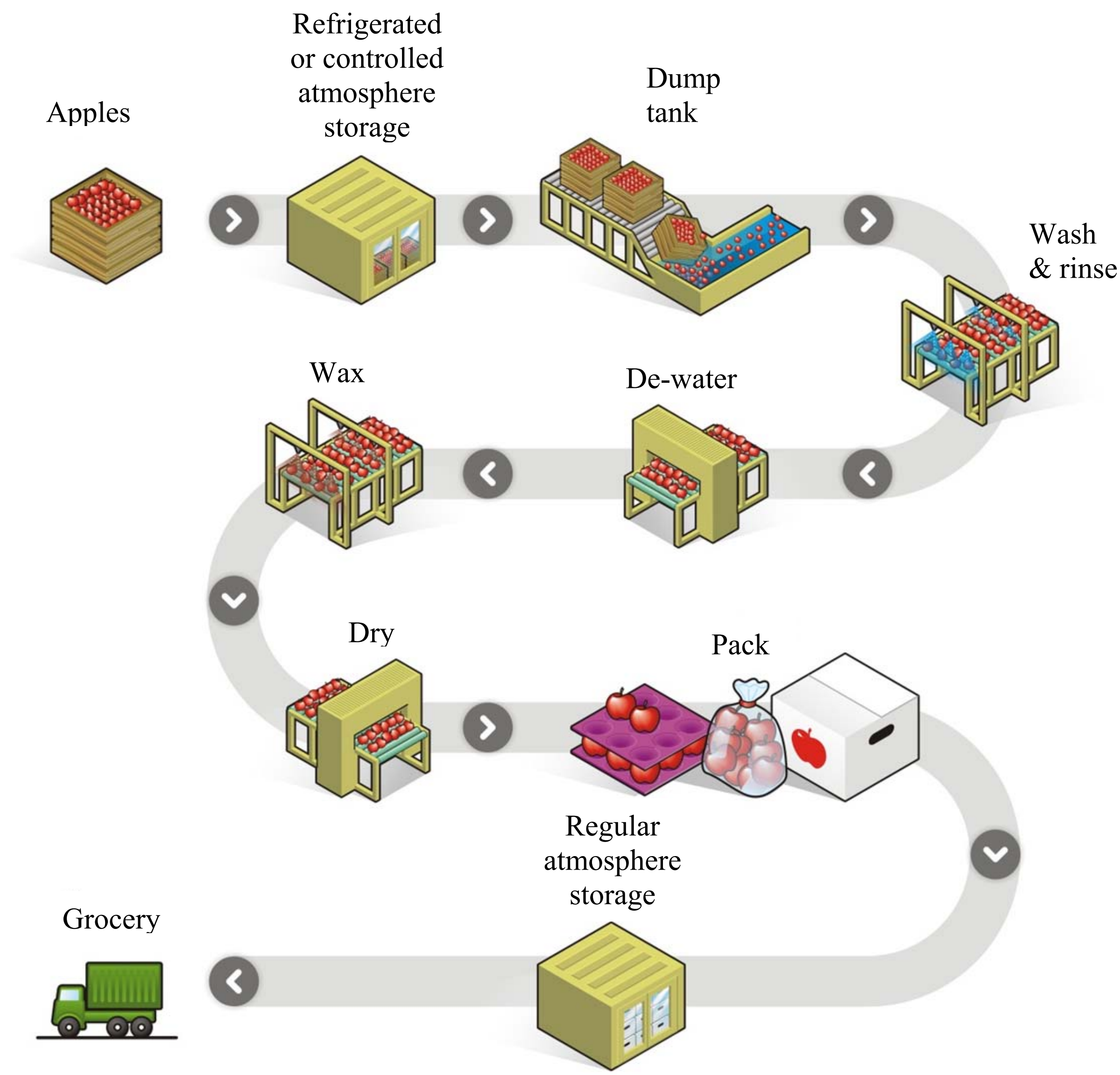

Figure 2.1. A schematic layout of a typical commercial apple packing line

Apples are the predominant horticultural crop stored under CA with about 60% of its production stored in CA for prolonged storage [*13, 21*]. In CA storage the atmospheric composition is usually altered by reducing oxygen and/or raising CO_2. This is a way to prolong the storage life of fresh produce by retarding ripening through reduced ethylene production or action [*13*]. The recommended standard CA storage for 'Delicious' is 0.7 - 2% O_2 and 2% CO_2 in 0 - 1°C. A relatively high humidity of 90 - 95% is recommended to prevent water loss and shriveling [*13, 21*]. An optimized CA storage will therefore retard respiration of the produce and allow longer storage of seasonal crops.

Packing houses in Washington State clean and pre-size fruits prior to CA storage as observed in a recent visit. Cleaning prior to storage ensures the removal of chemical residues, field debris and microbes (to an extent), and therefore reduces the potential decay by spoilage agents during storage. This also provides an added benefit of minimizing the contamination of the facility. However since the natural occurring waxy layer is partially removed during cleaning, the fruits are more prone to rapid water loss and are therefore stored for shorter periods in CA than fruits that are not cleaned prior to CA storage. Pre-sizing enables the packers to sort the fruits according to their end use, and also provides a means of controlling size and weight uniformity when the fruits are ready for further processing for the consumer. The practice of pre-sizing reduces down-time during processing since a bin of uniformly sized fruits minimizes differences in product quality and will invariably be directed to the same packaging and distribution line.

Coming out of refrigerated or CA storage, fruits are introduced onto the packing line where they are cleaned, washed and sorted prior to packaging. First the fruits are delivered from the field bins to the packing line which is accomplished by either dry bin dumps or water dumps into a dump tank [*20*]. In dry bin dumping, padding is secured over the lid and the fruits delivered through a controlled flow opening, whereas as the name suggests in water dumping the fruits are delivered directly into water. Submerging the bins allows the fruits to float readily and freely in the water which serves to protect the fruits from impact damage [*20*]. Since dump tank water accumulates fungal spores and bacteria which can infect postharvest produce wounds, sanitation of the dump tank is important. Chlorine and water ozonization are frequently used to control decay-causing organisms. Detergent brush washes followed by clean water rinses are used to remove soil and other contaminants from the surface of the produce [*20*].

After washing, apples are dewatered and dried either by drying tunnels or heavy duty industrial propeller fans to prepare the surface for waxing. Thorough cleaning and sufficient dewatering are important to obtain a good wax finish. With no interference from dirt and other extraneous materials on the surface, a good wax adhesion/setting can be achieved. However, a good wax finish is the interaction of a clean, dry, and warm fruit. In my communication with numerous apple fruit specialists, it was conveyed that the use of warm water in the dump tank raises the temperature of the skin in preparation for waxing. Warm fruit minimize moisture condensation on the surface of the fruit as they are introduced into a warm waxer. The excess water on the fruit surface dilutes the applied coating, resulting in low quality sheen. Using a heated dump tank not only warms the fruit in preparation for waxing but also aids in the cleaning of the fruit surface. Therefore, the importance of having clean-warm-dry fruits entering the waxing section of the packing line is paramount.

Currently no standard detection systems for the degree of cleanliness exist though there are several complex methods for the determination of the presence of residual crop fungicides and their extraction [*22*]. Most of these methods are, however, destructive, comprising fruit homogenization, and pre-concentration and extraction of the residues using organic solvents. Ethyl acetate [*23, 24*], acetone [*25*], acetone with methanol [*26*], and methylene chloride [*27*] are some of the solvent systems used in the extraction. In the research by Ong *et al.* [*28*] using chlorinated and ozone washes is the only non-destructive procedure reported, to the author's knowledge, that evaluates residual chemicals on whole fresh apples after they have been cleaned by various treatments. Based on the information garnered from the above mentioned studies, we developed a method to evaluate the level of cleanliness after subjecting the fruits to different combinations of washing treatments. The method which utilizes a solvent extraction system by rinsing the fruit surface with isopropyl alcohol is described in detail in Chapter 3.

Waxes derived from natural sources are applied to the fruits, after cleaning, to help reinstate some of the properties of the removed natural wax. Applied waxes need to be dried – usually by use of convection dryers – to achieve the proper wax properties [*20*]. In the industry either cold or hot air is used to achieve drying [*29*], with hot air commonly used for water-based wax emulsions [*20*]. Drake *et al.* [*29*] investigated the effect of the temperature used in drying wax on apple quality and found that cold dried (32 ^{O}F) 'Gold Delicious' apples tended to be firmer than those that were hot dried (140 ^{O}F), and also tended to loss less weight over the 90 days of refrigerated storage. In the case of 'Red Delicious', firmness was not affected by the temperature of the wax dryer, and though the waxed fruits dried with the hot method tended to lose weight the weight loss was not economically significant. Following drying, waxed fruits are either manually or mechanically sized and sorted, after which they are either hand or mechanically packaged. Pallets of packaged fruits are temporarily cold-stored [*20*] while awaiting pickup and delivery to a consumer point of purchase.

On delivery to the consumer, a decision on the end use of the produce is made on the basis of quality judgment from the appearance without necessarily having tasted it. Thus the applied coating serving as an edible package not only has to serve the ultimate function of preservation but also clearly communicate 'quality' by virtue of an attractive visage.

2.4. What is Fruit Quality?

Quality is the degree of excellence, and in the case of produce, the absence of defects [*16, 30*]. The role of texture, appearance and flavor in the consumers' perception of freshness and quality has been documented by several studies [*8*]. Ninety six percent of consumers surveyed by Zind [*31*] indicated intrinsic attributes of taste, freshness and ripeness as the most important selection criterion, in comparison to 94% who indicated that appearance and the condition of the product was more important in their selection criterion [*32*]. Thus in grading produce, intrinsic quality goes hand-in-hand with visual quality.

The goal of production, handling, storage and distribution is to provide consumer satisfaction and is critical to product quality [*6, 30*]. For red colored apples, quality is based on the intensity and characteristics of the red skin - their appearance [*33*]. However, the complete reliance on appearance to judge quality sacrifices critical attributes like firmness, color and aroma. After successfully jumping the hurdle of appearance, the consumers' acceptability and preference of apples are highly influenced by price [*6*] and prior experience with firmness [*34*] which is said to be the consumers' primary edible quality factor contributing to their choice of fruit. An European study by Péneau *et al.* [*8*] reported that the age of consumers and the frequency of consumption of apples had an influence on the perception of freshness based on appearance; consumers eating less than 2 apples per week gave more importance to appearance, and the importance attached to appearance decreased with increasing age. The understanding of the consumers' perception and preferences of apples and produce in general requires the integration of economics, marketing, psychology, postharvest and sensory science [*6*].

Quality is used frequently in the post-harvest world but according to Shewfelt [*30*] it is rarely defined and there are as many concepts as there are many perspectives on postharvest handling. The quality of produce changes from harvest time to consumption, and the relative importance and meaning of quality changes at each stage of production [*30, 35*]. Both subjective and objective factors define quality depending on the party of interest. Culture, economics, psychological, ethical and religious views influence the subjective while the organoleptic and physicochemical characteristics and food safety influence the objective. All these together create a wide and varying concept of quality [*36*]. The producers' view of quality is good appearance with minimal defects in a high yielding, disease resistant, easily harvested commodity with good shipping qualities. To market distributors, appearance is paramount to quality while attaching importance to firmness and long storability. Appearance, firmness, good flavor and nutritive value are the attributes the consumer uses to judge quality [*16*]. The limiting factor in delivering quality produce to the consumer is therefore a lack of appreciation of the different perspectives of quality which stems from orientation. Product-oriented quality which is the focus of postharvest researchers, producers and handlers considers the intrinsic attributes of the produce such as taste and firmness whilst consumer-orientation, which is the focus of consumers, marketers and economists, considers the needs and wants of the consumer [*30*]. It is the merger of both product- and consumer-orientation of quality that will produce optimum produce quality. This study aimed at achieving both.

To summarize, the overall quality of the fruits is greatly influenced by the preharvest conditions, the stage of maturity at harvesting, the harvesting methods and postharvest handling [*16, 37, 38*]. Quality results from a complex interaction of preharvest factors spanning from cultivar and rootstock genotype, mineral nutrition during growth, irrigation, and canopy manipulations to crop rotations [*38*]. According to Crisosto *et al.* [*38*] the selection of the correct genotype for specific growing conditions will ensure maximum postharvest quality. A mature fruit is one that has completed natural growth and development, and is the “stage at which a commodity has reached a sufficient stage of development that after harvesting and postharvest handling, its quality will be at least the minimum acceptable to the ultimate consumer” [*39*]. It is therefore recommended to pick fruits at optimum maturity for a greater chance at ensuring maximized fruit quality from harvest to consumption, while keeping in mind that poor temperature management reduces the quality and maximum potential shelf life of produce [*16, 40*]. Fruits harvested too early or too late in the season are more susceptible to physiological disorders and have shorter storage life than those picked at optimum maturity –e.g. fruits picked immature may not fully ripen and those picked over-mature tend to be softer, easily damaged and more susceptible to senescent breakdown [*13*].

The quality of fresh produce is also limited by the internal gas concentrations; the ease with which gases diffuse through the peel of the produce plays a vital role in the preservation of quality [*41*]. When the supply of O_2 required for respiration and the escape of CO_2, a bi-product of respiration, are blocked, the interior O_2 and CO_2 are reduced and raised respectively. This alteration in interior gases results in variation in product quality [*41*], and therefore makes it important to maintain internal gas concentrations within limits, irrespective of whatever processes are applied to the whole fruit in preparation for marketing and to improve consumer appeal, that do not cause unacceptable changes in quality.

Since quality in the produce industry is complex and its concept varies with different markets and stages in the supply chain, certain controls and measures are set in place to ensure the consistency in delivery of quality commodities from farm to fork.

2.4.1. Quality Assurance and Control

Dealing with apples which lack uniformity of color in some varieties, size, shape, and general organoleptic properties pose a challenge for quality control and assurance. Another challenge is the different concepts of quality in different markets. Quality assurance is the process designed for correct implementation of specific steps to ensure that quality control measures are met, while quality control is the process designed to ensure the delivery of adequate quality through product evaluation. Quality assurance and control protocols are set in place to monitor, evaluate and ensure that quality standards are met and also to guarantee the continued delivery of quality fruits to the consumer.

Quality control starts in the field and continues through the supply chain to the end user with each step having the potential to either maintain or reduce quality [*10, 16*]. Very few postharvest procedures can improve quality which makes it essential that a commodity is harvested when it will provide maximum quality, and that harvesting is carefully carried out to minimize injuries to ensure the maintenance of the quality at harvest [*16*]. Hazard analysis and critical control points (HACCP) is fundamental and forms the basis of the majority of control systems enforced [*10*]. It is used to identify and assess potential hazards and risks, and to establish process steps (critical control points) to control, minimize and eliminate the occurrence of such hazards and risks.

Both manual and automated systems are used on most packing lines for quality management. Hand-sorting is usually used to separate products on the basis of color, size, surface blemishes and grade. Small fruits and vegetables require more sorting decisions than larger commodities; the use of systems that turn and rotate eases the sorting decision as they provide surface visibility from all angles. In manual grading, however, it is important to adequately train workers and minimize monotony and fatigue for consistency and accuracy [*10, 20*]. Automated systems include sorting of fruits by weight or dimension, and detection and elimination of defective units using size sensors and electronic imaging and/or light reflectance systems respectively. The latter is also used for color sorting. X-rays or light transmittance are used for the detection of internal defects [*16, 20*], and non-destructive testing for internal quality of texture and flavor are also monitored [*10*]. The units which do not meet the set standards are diverted onto another lane where they are graded accordingly.

Final sorting prior to packaging, as a quality control measure that quality standards have been met, is practiced in some facilities [*20*]. Packaging, an important quality management step, is accomplished either manually or mechanically. It is critical that a commodity is correctly labeled with variety, size and grade, and packed into the right containers. To meet legal grades and quality standards, the product and packaging are inspected before final padding and closure of the package [*20*]. Governmental agencies also play a role in the assurance of quality with their primary role being the assurance of the delivery of pest- and disease-free products to both local and international markets, thus ensuring food safety which is the primary non-commercial product assurance [*10*].

Other quality control measures prior to packaging include frequent changing of the water in the dump tank to minimize the buildup of microbial contaminants and ensure efficient cleaning not only in preparation for waxing but also for the assurance of food safety. Using warm water in the dump tank to help raise the surface temperature of the fruit is another control measure to facilitate the efficient interaction of the wax with the fruit surface for a good coating. Frequent cleaning of the waxing brushes is also practiced to prevent the drying of the wax in the brushes which renders them hard and inflexible not only causing injury to the fruit but also inhibits the ability to spread the coating evenly. Wear and tear on brushes is closely monitored to facilitate timely replacements to help prevent commodity injury from worn out brushes.

With measures in place for quality assurance and control, quality attributes are evaluated to verify that standards have been met.

2.5. Quality Attributes and their Evaluation

An attribute is a quality or characteristic of the commodity in question. These characteristics usually exhibit changeable properties and several methods have been designed for their measurement and evaluation. The ease of measurement and evaluation varies with the accumulated knowledge base for its properties and also in the range in which it is measured. Quality attributes are categorized as extrinsic and intrinsic, with the extrinsic factors playing a critical role on the judgment of intrinsic factors. The extrinsic quality factors are mainly, but not limited to, appearance (color, size, gloss, surface blemishes). Texture (firmness, juiciness) and flavor (aroma and taste - sweetness, sourness) form the basis of intrinsic quality factors. Various equipment or techniques exist for quantifying these attributes to enable an efficient analysis of their effect on the perception of produce quality. Due to the high potential of individual units of the same crop being different in quality attributes, several fruits are required for measurement to minimize the variations that may arise. Table 2.1 lists some of the research and/or commercial methods used for quantifying quality attributes.

Gloss, weight, and firmness preservation are the quality attributes of interest in this study. Gloss, which is a measure of the shiny fruit surface, is derived from the application of wax. The wax layer creates a barrier to gases and its characteristics determine the level of gloss. The gloss level and barrier properties are highly dependent on the thickness and uniformity of the layer.

Table 2.1. Methods for quantifying quality attributes

Attribute*		**Equipment/Method(s)**	**Reference(s)**
Appearance			
	Color	Chromameter	[*42*], [*33*]
	Gloss/Sheen	Reflectometer Glossmeter	[*43*], [*44*]
	Defects/Disorders	Visual inspection	[*42*], [*44*]
Texture			
	Firmness/Crispiness	Penetrometer	[*15*], [*33*]
		Acoustical Firmness Sensor	[*34*]
		Texture Analyzer	[*37*], [*45*]
Aroma/Volatiles			
	Ethylene	Gas chromatography	[*15*], [*41*]
	O_2 consumed	Gas chromatography	[*43*], [*37*]
	CO_2 produced	Gas chromatography	[*42*], [*43*], [*41*]
	Ethanol content	Gas chromatograph	[*43*], [*44*]
Taste			
	Total soluble solids	Refractometer	[*42*], [*43*]
		Near Infra-Red	[*34*]
	Titratable acidity	Titration with NaOH pH meter	[*33*], [*45*]

*All of these attributes in some studies were also evaluated by a consumer panel

The barrier directly influences the uptake and release of respiratory gases and water vapor; a thicker and uniform coating layer provides a higher barrier compared to a thinner and an uneven coated surface. The rate of respiration and transpiration affect the maintenance of weight and firmness; high respiration and transpiration rates result in a rapid loss of weight and firmness.

It is therefore important to evaluate peel permeability to better understand respiration rates to facilitate a correlation to changes in gloss, which will also be correlated to weight and firmness.

2.5.1. Fruit Respiration and Peel Permeability

Respiration is a metabolic process which involves the oxidative break down of organic reserves to provide energy and carbon skeletons for biochemical processes. The oxidative break down of the stored organic substances results in the formation of simpler molecules such as CO_2 and H_2O with the release of energy [*10, 46*]. Glucose, a common substrate for respiration, if completely oxidized is represented as

$$C_6H_{12}O_6 + 6O_2 \longrightarrow 6CO_2 + 6H_2O + \text{Energy}$$

Respiration is influenced by both intrinsic and extrinsic factors. The type of commodity and the stage of maturity are two of the intrinsic factors that affect respiration. Climacteric commodities have high respiration rates at the onset of development which declines until the climacteric rise occurs with ripening or senescence. On the other hand non-climacterics exhibit high respiration rates early in development but the rate declines steadily during maturation. Temperature is the most important extrinsic factor affecting respiration. Enzymatic denaturation may occur at high temperatures resulting in reduced respiration, and at low temperatures physiological injury may occur, leading to an increase in respiration [*46*]. Oxygen and CO_2 concentration as well as physical stress such as wounding are other extrinsic factors that affect respiration. A reduction in O_2 to less than 10% (by volume) is what is required to achieve a reduction in respiration. However, this reduction is temperature dependent such that when temperature is lowered the oxygen requirement is reduced [*10, 46*].

Respiration rate is an excellent indicator of ongoing metabolism and therefore provides a useful tool to estimate the quality profile [*10*]. Three common methods are used in the determination of respiration rates: the static or closed; flow-through or flushed; and the permeable systems [*46*]. In the closed system, changes in O_2 and CO_2 concentrations in a gas tight container, of known volume, are measured and used to estimate respiration rate. In the flow-through system, a gas mixture flows at a constant rate through an impermeable container. Generally it is the absolute difference in O_2 and CO_2 concentrations between the inlet and outlet when the system is in steady state that is used to calculate respiration rates. In the permeable system, steady state O_2 and CO_2 concentrations in a package of known permeability and dimensions are measured. Respiration in this system is estimated based on mass balance and the other measured variables [*46*].

Atmospheric O_2 must pass through the peel of the fruits to the interior for normal respiration and CO_2 produced by respiration must also escape through the peel to be discharged from the interior of the fruit to minimize variations in quality [*41*]. This phenomenon is highly influenced by the peel permeability, which is altered early in processing through washing and waxing and results in changes in the normal concentration of internal respiratory gases [*41, 47*] The changes in the peel barrier properties occur in two ways [*41*]:-

- Diffusion through openings such as the lenticels, stomata, stem scars and injuries in the peel
- Classic permeance – dissolution of a gas into a barrier on the high concentration side, diffusing through the barrier and exiting on the low concentration side.

Permeation through a membrane or coating is the diffusion of a permeant through the layer which is then sorbed/desorbed from/into the external/internal environment [*48*]. The diffusion and solubility properties therefore determine the mass transfer behavior of a material which is expressed as the permeability coefficient (P). The permeability coefficient is therefore a combination of the effects of the diffusion (D) and solubility (S) coefficients, and determines the barrier properties for the material in question [*49*]:

$$P = D \times S \quad (1)$$

D describes how fast the molecules of the permeant move through a unit area of the membrane while S is the quantity of molecules sorbed by the membrane. At steady state, the former is a function of temperature and independent of concentration; this behavior is described as Fickian. Henry's law of solubility is what describes S, which is also temperature-dependent and holds in situations where there is no pemeant-polymer interactions at low concentrations and low partial pressure [*48, 49*].

The permeability coefficient is defined as the quantity of permeant dissolved and sorbed / desorbed per unit area, per unit time at a specified pressure gradient and through a specified thickness:

$$P = \frac{ql}{At\Delta p} \quad (2)$$

Where P = Permeability Coefficient

q = Quantity of Permeant

l = Thickness of Medium

A = Surface Area

t = Temperature of the Environment

Δp = Pressure gradient

Because of the high tolerance of 'Red Delicious' (the dominant cultivar used in the development of fruit coatings) to high gas barriers, apple coatings have focused on development of visual gloss with little on the needs that arise from the effects of high barrier to gaseous exchange [*43*]. It is well documented that coating applied to the fruit surface serving as a protective layer may also serve as a barrier to gaseous exchanges with the atmosphere [*41, 43,* *47*]; as a result respiration may become unaerobic with undesirable changes [*20, 43*]. On the basis of this, investigations have been conducted on the changes in the internal atmosphere of fruits with waxing and the development of new coatings with improved barrier properties [*41, 43, 50, 51*].

As expected, coatings with lower permeabilities result in low internal O_2 and high internal CO_2 [*43, 50, 52*]. These reports confirm a 1953 study on the effect of skin coating on the behavior of apples in storage by Trout *et al.* [*53*]. Trout and his team reported that "coating increased the resistance of the skin to gaseous diffusion and thus greatly reduced the internal oxygen concentration, increased the internal carbon dioxide concentration, reduced respiration rate and retarded ripening changes by varying degrees". Gaseous diffusion occurs through the pores and the cuticle of the fruit peel. The application of surface coatings increase the resistance to gaseous exchange by covering the cuticle and pores of the skin which culminates in decreases in the transmission rates of gases between the internal and external atmospheres [*54*]. Thus surface coatings, through these effects, have the tendency to modify the composition of the internal atmosphere, suppress the respiration rate and reduce transpiration in fruits [*54*].

A recent study by Hagenmaier [*41*] showed that high barrier coatings not only caused significant decreases and increases in internal O_2 and CO_2 respectively, but also showed much higher variations in the internal gas concentration of different individual fruits with the same coating in comparison to different individual non-coated fruits. This is a classic indication of higher variation in the quality of fruits coated with high barrier waxes. The variability in the individual fruit response to a coating treatment is due to the inherent variability in the fruit skin resistance to gas diffusion and hence respiration rate [*54*]. The variation in the proportion of blocked pores, by coating, as well at the extent to which the coating adheres to the fruit surface, especially along the edges of the pores, account for differing fruit response to the same coating treatment. A loosely adhering coating offers the opportunity for gaseous exchange in the space between the fruit and the coating, whereas the opposite applies in the situation of a tightly adhering coating. Essentially coating a pore (blockage) eliminates its contribution to gaseous exchange [*54*]. The extent of internal atmospheric changes depend on the storage temperature, thickness and type of wax used, the viscosity at which it is applied and also the variety, peel anatomy and condition of the fruit [*52, 53, 55*]. The wax characteristics are important as these factors influence the blocking of pores on the peel to gases [*56*]. Viscosity, a controllable factor is particularly important since flow properties influence the distribution and coverage of coating on the fruit surface. It therefore affects the thickness of the coating layer and hence the barrier properties which influence quality.

Coatings can create a modified atmosphere similar to CA which may change in response to environmental temperature and relative humidity, and a combined effect of fruit respiration and coating permeability [*50*], thus having a direct impact on produce quality. The effects of storage temperature and humidity have been shown over and over again to affect fruit quality. High humidity will not prevent moisture loss if the temperature of the produce is not near that of the air temperature [*55*], and it also increases the permeability of shellac-based coating to O_2 and CO_2 [*57*]. Low temperature storage not only extends the shelf life of temperate fruits but also protects non-appearance quality attributes such as texture, nutrition, aroma and flavor [*55*]. Studies by Hall *et al.* [*58*] report that the temperature and the maturity of the fruit are the most important factors that influence the storability of coated fruits.

The first few days after coating are said to be the most critical period, where a rise in internal carbon dioxide concentration was observed. Many other studies have also shown the changes in storage temperature and humidity on the significant demand for respiratory O_2 indicating how environmental conditions affect fruit respiration and coating permeability [*50, 59*]. Outside the range of proper temperature maintenance, the loss in apple firmness is concomitant with an increase in skin color indicating a direct effect of temperature on the physiological activities of ripening and changes in firmness [*55*]. Only 7% of apples are held at the recommended and required temperature storage at the retail level; 97% of the fruits experience temperature above the recommended range making them victims of the greatest temperature abuse [*55, 60*]. However, the robust nature of the fruit reduces the rate of quality loss unlike produce like strawberries which perish rapidly in abused temperature conditions. The rate of mass loss depends to a great extent on the nature of the peel surface of the commodity type and cultivar [*55*].

2.5.2. Firmness and Weight

Firmness is the primary edible quality factor for consumer preferences and acceptance of fruits [*34, 61*]. Loss of firmness as a result of softening is characterized by fruit ripening [*62*]. During ripening, firmness declines partly as a result of cell wall disassembly and degradation of the polysaccharides of which it is composed [*63, 64*] due to enzymatic activity of polysaccharide-modifying enzymes secreted into the cell wall from the symplast [*64*]. It has been extensively documented that these enzymes including cellulase and pectinase are mostly hydrolases cleaving their primary substrate using water, their secondary substrate [*62, 65*]. The cell wall provides rigidity and adhesion of intercellular structure, and the extent of its modification has a direct bearing on the degree of textural changes. Overall, the declining strength of the cell wall and intercellular connections will determine the firmness and textual changes of a fruit [*64*]. The cell wall is composed of a network of polysaccharides, proteins and some phenolics. The wall polysaccharides are categorized as pectins, hemicelluloses and cellulose [*65*], and though the component varies among species the generally composition is of equal amounts to about one-third of the dry weight, while structural proteins make up only about 1 to 10% of the dry weight [*64*]. Pectins however form about half of the polymeric content of the cell wall [*64*]. Pectin is a major constituent of the middle lamella and contributes to cell adhesion [*61, 62*]. The components of the cell wall are linked by numerous bonds including hydrogen bonds, ionic calcium bridges and ester bonds [*64, 65*]. The degradation of cell wall polymers collapse these adhesive bonds leading to loss in firmness [*62*].

The cell wall is highly hydrated with dissolved solutes, ions and soluble proteins including enzymes [*64, 65*] and the decline in firmness with ripening is also attributed to reduction in tugor pressure. This is the result of water loss by the fruit and accumulation of osmotic fluids in the apoplast, which together reduces the expansionary pressure on the walls cumulating in textural changes [*64, 66*]. Depolymerization of xyloglucans and the solubilization and depolymerization of polyuronides [*62, 64*] also result in the loss of tissue firmness and ripening of fruits. This is due to a loss in intercellular contact as the cell wall is more open and hydrated causing softening of the fruit tissues [*64*].

Relative humidity is crucial in determining moisture loss, and it has been shown in the case of apples that low RH more than temperature is the main cause of deterioration [*67*]. The rate of water loss from a commodity is dependent on the water vapor pressure deficit which is the difference between the actual vapor pressure and the saturated vapor pressure [*55*]. Reduced water loss prevents shriveling and textural changes that lead to a loss in weight and firmness. In a study on mangoes, a climacteric fruit exhibiting rapid ripening after harvest just like apples, the firmness of coated fruits was found to be significantly different from that of the uncoated [*51*]. Coated mangoes required 7.0 – 5.3 N to compress 2 mm compared to the significantly different 2.8 N needed to compress the same distance for uncoated fruits. The coating, by virtue of reinforcing the moisture barrier properties, impacted physiological changes in the cell wall which translated to the retention of firmness. Meng *et al.* [*63*] in a study on peaches also found firmness to decrease rapidly at higher storage temperature. Fruits stored at 10^{o}C softened rapidly with a significant decrease in firmness compared to 5^{o}C storage with an effective maintenance of firmness.

2.5.3. Gloss

The subject of gloss has been discussed in detail in section 2.2, but this sub-section on gloss will focus more on the application of waxes to achieve a glossy surface and also relate the discussion in section 2.2 to the quality attributes of weight, firmness and respiration.

Coating apples impacts gloss, the shine on the fruit surface that gives the fruit an attractive appearance. The application of coatings to apples is an essential unit operation in the packing of the fruit as it not only enhances visual appeal and quality perception, but it also improves preservation as discussed in the previous section. Shellac derived from the secretions of an insect, *Tachardia lacca* [*68*], and carnauba from the leaves of the *Cerifera* palm [*69*] are the two commonly used waxes in the fruit industry. Since glossy surfaces are considered attractive, fruit waxes are formulated to be glossy [*19*]. A shiny surface may not be valued for all produce but is valued for apples [*11, 12*] and is beneficial for its sale [*56*]. Apples are assessed for quality based on surface texture, color, translucence and gloss – as part of its visual structure [*19*].

Surface gloss, a measure of specular reflectance is dependent on the light source and its intensity, as well as the surface characteristics of the commodity [*12, 70, 71*]. It is one of the main visual attributes that influence the evaluation and grading of fresh produce [*71*], though a good shiny looking commodity may not necessarily have a direct correlation to freshness and quality, and therefore doesn't guarantee a tasty and crisp product. Aside from the intensity of the light source and surface morphology of the commodity, gloss is also dependent on the uniformity and thickness of the coating. Applying a viscous wax or voluminous amounts increases the layer of coating which translates to higher gloss. However a viscous coating with reduced flow properties may not provide an even distribution and coverage on the fruit and the inconsistencies in the coating layer may translate into lower gloss.

To make objective evaluations of gloss levels to successfully correlate it to visual appeal and quality, physical measurements are required. Gloss measurements, as discussed in section 2.2.2, are often made by projecting a light beam at a specified angle onto a surface and tracking the amount of reflected light [*70*]. Numerous devices exist for measuring gloss but most are designed for flat surfaces which would mean a modification to the device to efficiently and non-destructively measure the gloss of uneven and curved surfaces like those found in fresh produce as was reported in recent studies by Bai and his team [*43, 72*].

Commercial glossmeters designed for curved and uneven surfaces have evolved in recent years but there is limited data on their efficiency of measuring gloss of fruits and vegetables [*70*]. Research by Nussinovitch *et al.* [*12*] report the successful construction of a device capable of measuring gloss on a variety of produce including oranges, bananas, onions, eggplants and tomatoes. Mizrach and his team [*70*] also recently developed an automated device for measuring the gloss of apples, with a repeatability error of 16% for 90% of their measurements. This automated device was initially used to measure gloss of the apples used in this study, but because of the complexity of the mechanical automation, a new but simpler device operating on similar principles to that of Mizrach *et al.* [*70*] was constructed in the laboratory of Dr. Lu. Details on the features and operation of the prototype gloss device are given in Chapters 3 and 4.

Although the coating barrier, by impacting physiological activity, is able to slow down the deterioration process, changes in the degree of gloss are observed with prolonged storage and associated with changes in other quality attributes. An example of this is the observation that decreased gloss of banana peel indicated a loss of weight during storage [*73*]. This observation is attributed to the fact that gloss is related to surface composition and morphology, and thus a change in morphology results in changes in surface gloss [*71*]. Studies over the years like that of Hagenmaier *et al.* [*47*] report the decay of gloss from shellac with storage. The same authors investigated the ethanol content (associated with off flavors) with waxing and found that shellac coated fruits had higher ethanol content and this is a sacrifice of flavor for appearance. Bai *et al.* [*56*] also report the decay of gloss from six different waxes over a 4-week testing period. Investigations by Fellman *et al.* [*74*] on the effect of refrigerated storage on the changes in appearance of waxed 'Delicious' fruits showed that cold drying of wax caused greater whiting than hot drying for both shellac and carnauba. The incidence of shellac whiting on cold dried apples was observed to decrease with a month of refrigerated storage but increased after 2 months. A similar pattern was observed in the hot dried fruit with a relatively smaller amount of wax coating. The authors suggest that "a naturally occurring cuticular component of apples influences the whiting reaction of shellac waxes". A ripening period (ambient temperature storage) of 8 days, however, totally eliminated the incidence of whiting. This they explained was the replacement of a plasticizer in the shellac formula by natural substances, and a decrease in natural wax synthesis of waxed fruits. They recommend holding waxed apples in refrigerated storage for less than one month after packaging to minimize whiting.

2.6. Waxes and Packaging

The practice of enrobing fruits and vegetables with edible coatings was reported in the twelfth and thirteenth centuries in China where wax was used on oranges and lemons. Although the mechanism of preservation was unknown to the Chinese back then, the process was sustained due to the realized shelf life extension of the coated compared to the uncoated [*75*]. It wasn't until the 1930s that hot-melt paraffin became commercialized for use as edible coatings on apples and pears [*75*]. Edible packages are mostly films composed of a polymer base and serve to function in the same way as inedible packages. As the name suggests an edible film aside from providing packaging functionalities is considered safe for human consumption. Edible films are found in a variety of food categories including wax coatings for fruits and vegetables, casings for sausages and chocolate coatings for confectionaries. They are also very common in the pharmaceutical industry in enteric protective coatings and shells for capsules.

2.6.1. Natural Wax Coating

Natural waxes by virtue of their numerous useful properties have been used by mankind for centuries. Waxes in nature function to protect plants from destructive environmental influences such as microbes, drying and chemicals. They have been found to be highly resistant to chemical and biological degradation, and hence its use in food protection [*76*]. Natural waxes on fruit surfaces function to reduce the loss of moisture and provide water repelling properties to the fruit, which are important to retain chemicals that are applied to the skin [*77*].

Natural waxes are small crystals on the surface of the fruit and the chemical composition of the wax determines the shape of the crystals [*77*]. The crystals are highly impervious to water vapor and gaseous exchanges with the barrier properties highly dependent on the intercrystalline lattice; tightly packed crystals are less permeable to gases in comparison to a loosely packed lattice [*78*]. Figure 2.2 below depicts the characteristic crystals on apples. Light reflection and scattering by the waxy crystals on the fruit surface is responsible for the perceived surface appearance [*77*]. This principle is the same as that on which the perception of gloss from applied waxes work.

With growth and maturity the natural wax on fruit increases, but its quality and quantity changes during postharvest [*77, 79*]. There are however a few varieties that have been investigated to record an initial increase in surface wax during storage but decreases after about the 3rd month in storage. These apple varieties include the 'Calville Blanc', 'Cox's Orange' and the 'Dougherty' [*79*].

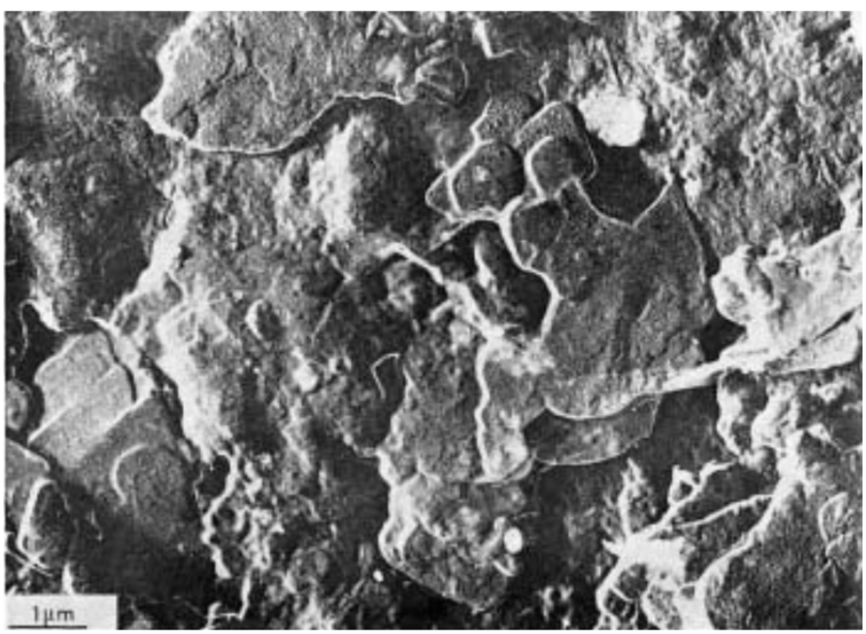

Figure 2.2. Electron micrograph of the surface of apple fruit showing platelet wax crystals.
Source: Kolattukudy [*77*]

Natural waxes contain mostly straight-chain carboxylic acids and alcohols in the region of *ca.* $C_{12} - C_{36}$ [*76*], and apple surface waxes are reported to be composed of hydrocarbons, alcohols, fatty acids, ursolic acid and α-farnesene [*79*]. Increases in the various components were associated with a corresponding increase in surface wax content. The main hydrocarbons in a study on 'Granny Smith', 'Dougherty' and 'Sturmer' cultivars are recorded to be *n*-nonacosane and *n*-heptacosane. Alcohols from C_{14} to C_{28} were also reported with *n*-tetracosanol, *n*-octacosanol and *n*-heptacosanol being the most abundant [*79*].

Earlier studies by Morice *et al.* [*79*] report the additional findings of numerous earlier studies indicating an increase in the fatty acid fraction of several apple cultivars during storage. Of the three cultivars studied by Morice *et al.* [*79*] it was found that with the exception of the 'Sturmer', the linolenic acid content of the 'Granny Smith' and the 'Dougherty' increased with storage. The linoleic acid in the Dougherty' and 'Sturmer' showed no trend during storage but increased in the 'Granny Smith'. There was however a decline in the levels of oleic acid with storage for all cultivars. The amount of stearic acid also increased for all cultivars, in contrast to plamitic acid in the 'Granny Smith' and the 'Dougherty'. The higher saturated acids components showed little changes during storage and the ursolic acid component was reported to increase with the total wax content.

One of the major differences between the natural and commercial wax is the absence of ursolic acid which is the major cyclic component of apple fruit wax [*77*]. Commercial waxes are, however, able to provide similar functions of the natural wax, which leads to a discussion on the details of their composition and functionalities.

2.6.2. Coatings as Edible Packaging

It is estimated that 25 to 80% of fresh produce go to waste due to spoilage as reported by various authorities [*80*], but with the understanding of the respiratory mechanism of produce, several techniques have been successfully developed to extend their shelf life [*75*]. Control and modified atmosphere storages are examples of techniques that have been successfully used to minimize quantity and quality losses in fresh produce. The use of edible coatings as an enrobing layer on fresh produce not only provides a semipermeable barrier to gases and water vapor, but also serves an alternative to modified atmosphere storage. It therefore modifies and controls the internal atmosphere resulting in an extension in shelf life [*75, 80*] through reduced water loss and respiratory activity.

Edible films serve as barrier to gases and water vapor by being positioned either on the surface of food as protective coating, or within the food to separate components with different water activities thereby preserving the textural properties of each component [*81*]. Edible coatings also function to improve the structural integrity of food products like that of pizza topping during distribution, and also provide physiological and physical protection to products like fruits and vegetables which are susceptible to injury during processing and transportation [*80*]. Incorporation of food additives such as antioxidants, antimicrobials, among many others into edible packages allow localized and controlled release rate of these additives into the packaged product. The two main advantages of coating fruit have been identified by Banks *et al.* [*54*] as:

1. Coatings by virtue of being in close contact with the fruit surface have the same temperature as the fruit surface. This eliminates condensation, arising from high humidity conditions, which causes rotting and is limiting to the application of MA packaging technologies.

2. Coatings serve as a form of packaging and can reduce deterioration. This holds an advantage in markets which are green-conscious in minimizing food packaging waste [81].

According to Donhowe and Fennema [81], hydrocolloids, lipids, and composites are the three main categories of edible films and coatings with their possible functions listed in Table 2.2. Hydrocolloid films possess good barrier to oxygen, carbon dioxide and lipids, but not to water vapor due to their hydrophilic nature. Films and coatings from hydrocolloids can be either carbohydrate- (natural or chemically modified starches, gums, etc.) or protein- (gelatin, casein, gluten, etc.) based. Water solubility of hydrocolloid films become advantageous when the film will be consumed with a product heated prior to consumption [81].

Films composed of lipids are often used as barriers to water vapor, and as gloss additives to products. Lipid films as stand-alone are limited because of the lack of sufficient structural durability; they are therefore used in conjunction with supporting matrixes [81]. Composite films are composed of both hydrocolloids and lipids, and combine the advantages of each while minimizing the distinctive disadvantages of each. They can exist as a bilayer or a conglomerate where the components are interspersed throughout the coating [81].

Just like any product edible packages are not without the negatives. According to Park [75] consumers tend to be wary of commodities for which they detect waxy coatings and therefore suggests the developments of coating that impart minimal detection of a waxy taste. This wariness stems from the association of waxes with non-food uses [72]. Low O_2 and high CO_2 disorders as a result of the modification in the internal atmosphere have been linked to edible coatings. This observation makes it vital for the identification of edible coatings that produce a favorable modified internal atmosphere with desired quality. This can be achieved by selecting the appropriate coating that gives the selective desired internal gas composition specific for a product [*75*] thereby ensuring the achievement of the possible optimal positive benefits derived from using edible coatings. The gas permeation properties of the coating, internal gas compositions of the coated fruits, and the coating effect on quality changes must be studied to effectively select the appropriate coating for a specific produce [*75*].

Table 2.2. Possible Uses of Edible Films and Coatings

USE	**APPROPRIATE TYPES OF FILM**
Retard moisture migration	Lipid, composite[a]
Retard gas migration	Hydrocolloid, lipid, or composite
Retard oil and fat migration	Hydrocolloid
Retard solute migration	Hydrocolloid, lipid, or composite
Improve structural integrity or handling properties	Hydrocolloid, lipid, or composite
Retain volatile flavor compounds	Hydrocolloid, lipid, or composite
Convey food additives	Hydrocolloid, lipid, or composite

[a]A composite film consists of lipid and hydrocolloid components combined to form a bilayer or conglomerate.
Source: Donhowe and Fennema [*81*]

Since the package is what attracts the consumer and it's the consumer's first point of interaction with a product, it is not enough in the fruit industry for edible coatings to successfully serve the function of preservation. It is highly important that it is also able to efficiently communicate "quality" to the consumer. Waxes have been applied to tree fruits for generations and in recent times coating fruits prior to marketing has become standard practice with the 'Delicious' being the key variety for the development of coatings [*43*]. An emphasis on coating development is controlled gas exchange with the attempt to create a modified atmosphere within the fruit to delay ripening and senescence similar to the more costly CA storage [*80*]. Experiments on storage and fruit weight loss of waxed and unwaxed apples conducted by Drake

[*29*] have shown that waxing is important in reducing weight losses when fruits are stored for longer periods. Thompson *et al.* [*20*] report about one-third reduction in water loss by waxing fresh produce. These reports also showed that equally good quality attributes were derived from using either shellac or carnauba. The amount of coating, however, applied to fruits is negligible compared to the natural wax present [*77*]. Studies on the amount of wax deposited on apples using 5 different apple coating formulations by Kolattukudy [*77*] showed that the amount added by the waxing process was insignificant such that the increase over that naturally present was negligible. The amount of wax (ppm) on the untreated fruit was 994 which reduced to 973 when washed. The application of wax, after washing the fruit, only increased it by 3units to 978.

Several studies have also been conducted on the coating of apples with natural occurring waxes to achieve an improvement in the visual appeal and marketability through an extension of shelf life with the appropriate internal atmosphere modification and maintenance of desired quality attributes. An earlier study of commercially available coatings reports shellac-coated fruits to have a low permeability to gases with the lowest and highest internal O_2 and CO_2 respectively [*50, 57*], and also a significantly less weight loss compared to other treatments [*50*]. Shellac of all the coatings provided the most gloss with the least quality changes; this depicts the direct relationship between gloss, respiration rate and changes in weight and firmness. Carnauba wax without the problems of whiting of the finish provided a lesser gloss compared to shellac [*72*]. In view of this a lower gloss may compensate for the risk of higher gloss and whitening in humid environments. However, it has been found by horticulturists that shellac coating is excellent for dark Red Delicious apples as it “impacts gloss, hides bruises and forms a modified atmosphere condition that tends to preserve firmness and prolong shelf life for this variety” [*43*].

2.6.3. Rheological profile of edible fruit coating

Gloss and related quality attributes are linked to the wax viscosity as discussed in previous sections, 2.5.1 and 2.5.3. Waxes have different physical states ranging from the solid to the liquid form, and different chemical properties ranging from the homogenous pure state to the multiphase emulsion system. The differences in the various properties indicate different flow behaviors. For the focus of this research it was important to study the rheological properties of the apple coatings by determining the flow behaviors and spreadabilty in relation to its interaction with the surface of the fruit under different simulated waxing conditions.

Rheology deals with the science of the deformation and flow of matter, and is the study of the behavior of materials under applied stress or strain. All materials have rheological properties which can differ for materials within the same product category. It's the rheological properties that govern the behavior of materials in multiphase systems and in processes like spraying, spreadability, and adhering, just to mention a few [*82, 83*]. Flow properties are described by a fluid model which is a mathematical equation derived from fitting experimental data to a statistical curve. Fluid models are either described as Newtonian or non-Newtonian characterized mainly by their relationship between shear stress and shear rate.

The shear stress is the force required to maintain velocity/flow of the coating per unit area, and the shear rate is the velocity relative to the available distance of flow. The behavior that is independent of time exhibiting a linear relationship between stress and strain and has no yield stress is described as Newtonian. All other fluids not showing the ideal viscous behavior are termed non-Newtonian [*82, 83*]. Rheological data conforming to Newtonian flow show the relationship between viscosity, shear stress and shear rate as [*82*]:-

$$\sigma = \mu \dot{\gamma} \qquad (1)$$

Where σ = shear stress (Pa)

μ = absolute viscosity (Pa.s)

$\dot{\gamma}$ = shear rate (s^{-1})

The power law model has been successfully used to describe the behavior of majority of non-Newtonian fluids [*82*]:-

$$\sigma = K \dot{\gamma}^{n} \qquad (2)$$

Where K = consistency coefficient ($Pa.s^n$)

n = dimensionless flow behavior constant

$\dot{\gamma}$ = shear rate (s^{-1})

When n=1 the power model becomes a Newtonian model and K = μ.

In the case n > 1, the flow behavior is described as shear-thickening or dilatant. The flow is considered shear-thinning or pseudoplastic when 0 < n < 1.

Viscosity modeled after the power law is described as the apparent viscosity (η):

$$\eta = \frac{\sigma}{\dot{\gamma}} = \frac{K \dot{\gamma}^{n}}{\dot{\gamma}} = K \dot{\gamma}^{n-1} \qquad (3)$$

Apparent viscosity is dependent on the numeric value of K and n, and changes with shear rate [*82*]. Non-linearity can therefore be said to be an indication of changing viscosity with shear rate and point to non-Newtonian behavior.

According to Steffe *et al.* [*82*], the power law model is appropriate for most non-Newtonian flow and provides a good estimation for the flow behavior of materials with a significant yield stress. However for highly viscous materials with substantially large yield stresses more complex rheological models such as the Bingham, Herschel-Bulkley and Casson equations may be used. Materials described by the Newton and power law models have rheological properties that are unaffected by the mechanical effects of the systems used and are thus considered as time-independent [*82*]. A classic example is seen in the case of measuring the viscosity of water which remains constant irrespective of the duration of stirring. A weak gel in the other scenario may record a reduction in its consistency with mixing as a result of the destruction of certain structures within its formulation. Time-dependent fluids are classified as thinning (thixotropic) or thickening (rheopectic) with time [*82*].

Numerous devices are available for determining the flow behavior of materials. Rheometers measure rheological properties whereas viscometers are more limited to measuring only viscosity. The most common instruments capable of measuring fundamental rheological properties are categorized as the rotational and tube types; the former includes the parallel plate, cone and plate, concentric cylinder and mixer; and the latter includes the glass capillary, high pressure capillary and pipe. Rotational systems are more suited to measure time-dependent behavior as the tube system permits only one passage of the material through it [*83*].

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CHAPTER 3

The Effects of Packing Line Washing Treatments and Waxing Conditions on the Surface Cleanliness and the Gloss Development of 'Red Delicious' Apples (*Malus domestica*)[1]

[1]To be submitted to Postharvest Biology and Technology

3.1. Introduction

Apples are a popular produce to which a number of postharvest practices are applied. These include controlled atmosphere (CA) storage, washing, sorting, waxing, drying and packaging prior to being delivered to the consumer. Apples have to be cleaned in preparation for waxing. The deposition of numerous elements including soil, and chemicals used to control pests and diseases caused by fungus, which not only have to be removed for health reasons, may interfere with the way the applied coatings interact with the fruit peel for an appealing coverage.

As practiced in the industry, waxes derived from natural sources are applied to cleaned fruit to improve appearance and quality. Appearance through the impartation of gloss and quality through replacement of the removed natural wax to control transpiration, the reduction of water loss, delayed ripening and senescence, and retention of color (Drake et al., 1990; Bai et al., 2003; Cisneros-Zevallos et al., 2005). A variety of materials are used in the formulation of commercial coatings which include but is not limited to lipids, proteins, carbohydrates, plasticizers and solvents like alcohols and water. The different properties of these materials provide a wide range of possible behavior of coating systems (Cisneros-Zevallos et al., 2005). In addition, the type of fruit and coating, coating thickness and permeability, fruit-coating surface coverage, and environmental conditions such as temperature and humidity play a significant role in the performance of the coating system (Paull, 1999; Cisneros-Zevallos et al., 2005).

Like all other tree fruits, apples are prone to pre- and postharvest physiological disorders that require that the fruit be treated while in the field and prior to storage – e.g. preharvest application of calcium to control bitter pit and drenching with diphenylamine (DPA) to control superficial scald in storage (Watkins et al., 2004). The use of fungicides in drench tanks is also practiced to reduce the survival and activity of *Botrytis cinerea* and *Penicillium expansum*, the agents of the two most important postharvest wound diseases, grey and blue mold respectively (PTRIC, 2002). Apples also have a film of natural wax which increases in thickness with growth and maturity (Kolattukudy, 2003) and serves as a function of protection against pests and fungi in addition to prevention of water loss. The residues that have to be removed from the skin for efficient cleaning are therefore predominantly chemical sprays, natural waxes, pest larvae and eggs, dirt and other external matter that may have been deposited at any time prior to packaging.

Once on the packing line, the objective is to clean the surface of the residue outlined earlier through chemical and mechanical actions. Detergent brush washes followed by clean water rinse are used to remove soil and other contaminants from the surface of the fruit (Thompson et al., 2002). Some commercial apple packing houses use cold water wash-rinse systems, while others use heated water and detergent to clean their surface prior to wax application. It is essential that cleaning treatments do not damage the apples but rather maintain or enhance quality. In view of this, the effects of hot water in the washing system on the quality of fruits have been described by Bai et al. (2006); Spotts et al.(2006); Hansen et al. (2006); and Neven et al. (2006). These serial studies documented that a wash temperature of 50 ^{o}C caused thermal fruit injury to pears (Bai et al., 2006) but not to apples (Hansen et al., 2006; Neven et al., 2006); high pressure sprays (400kPa and greater) contributed significantly to the removal and hatching of arthropod eggs on apple surfaces and did not cause any external or internal disorders (Neven et al., 2006); and the use of either soft or firm brushes did not affect apple quality (Hansen et al., 2006) unlike in pears with a more sensitive peel where the use of firm brushes caused friction discoloration (Bai et al., 2006). Chlorine and water ozonization are also frequently used to control decay-causing microbes.

Surface gloss is one of the many quality grading attributes detected by visual perception and is therefore used to describe the appearance of a material (Jha et al., 2002; Silvennoinen et al., 2008). It is "a function of spectral reflectance, and is dependent on source and intensity of the coincident light" (Jha et al., 2002), signifying the interaction of light with the surface of interest. A rough surface has a matt appearance, compared to a smooth surface which is glossy, as a result of reduced intensity of the specular reflectance (Silvennoinen et al., 2008). Therefore a uniformly coated fruit with an intact smoother coating will have higher gloss as a result of the intensity of the reflection, and also have decreased gaseous exchange as a result of the reinforced peel barrier. Whereas surface gloss may not be valued for certain commodities, it adds value to apples (Szczesniak, 1983; Nussinovitch et al., 1996) increasing consumer appeal and economic value (Bai et al., 2002a).

Environmental conditions of temperature and relative humidity (RH) mainly during the dewatering and drying process may influence the interface between the apple surface and the applied coating, compromising the interaction between the two. Studies on the effects of RH on apples have often focused on the relationship between coating permeability and storage RH, with the resultant effect on quality (Hagenmaier et al., 1991; Bai et al., 2003; Cisneros-Zevallos et al., 2005). Baldwin et al. (1995) and Bai et al. (2002a) also studied the effect of temperature during storage on the quality attributes of fruits. None to our knowledge have studied the effect of temperature and RH conditions during coating application on gloss development. It was therefore deemed necessary to investigate the effect of environmental temperature and RH on the waxing process and ensuing apple quality.

Several other studies have also looked at enhancing gloss and other quality attributes such as firmness, total soluble solids, titratable acidity and controlled respiration through the use of new wax formulations. A zein formulation by Bai et al. (2003) improved the quality of the fruit and depending on the concentration of ingredients used, provided comparable results to commercial shellac formulation. An earlier study by the same research group also documented the effectiveness of experimental polyvinyl acetate, starch and carnauba-polysaccharide coatings. These new wax formulations usually involved varied amounts of the different components which changed the solid content. For example, Bai et al. (2002b) studied the effect of 19% shellac against a 13.3% with 3% whey protein isolate or 9.5% with 8.3% carnauba. An experimental formulation of candelilla wax was also included in their study. AvenaBustillos et al. (1997) also studied the effect of an edible caseinate-acetylated monoglyceride coating, at different solid contents of the major components, on the quality of produce. Varying solid content directly affects the viscosity and flow properties of the coatings which directly influences the coating characteristics by virtue of the spreadability on the fruit and its resultant effect on fruit quality such as firmness and gloss.

The purpose of this study was to determine the washing line conditions that result in optimum fruit surface cleanliness and 'dryness' for an efficient wax application and a resultant appealing gloss. There is a need to evaluate the efficiency of the different cleaning treatments used but currently no standard detection systems for the degree of cleanliness exist though there are several complex methods for the determination of the presence of residual crop fungicides and their extraction (García-Reyes et al., 2006). Most of these methods are however destructive, comprising fruit homogenization, and pre-concentration and extraction of the residues using organic solvents (Di Muccio et al., 1999; Saad et al., 2004). Research by Ong et al. (1996) using chlorinated and ozone washes is the only non-destructive procedure reported to the author's knowledge that evaluates residual chemicals on whole fresh apples after they have been cleaned by various treatments. Based on the information garnered, we developed a non-destructive method for assessing the efficiency of the cleaning treatments used in this study, detailed in section 3.2.4, to allow the selection of the most effective cleaning treatment for waxing evaluation.

The ability to efficiently measure gloss for the evaluation of the waxing process has been a challenge since most commercial glossmeters are not designed for uneven curved surfaces like that of apples. The quest for the evaluation of the gloss and quality of fresh produce has been on the rise in recent years, and many studies have developed non-destructive but laborious techniques to determine the quality indices of produce (Jha et al., 2004). To the author's knowledge it was not until a little over a decade ago that Nussinovitch et al. (1996) and Ward et al. (1996) reported the construction of the first device capable of non-destructively measuring gloss on non-flat surfaces. Non-destructive measurement is a desired feature as this will make possible the evaluation of the same fruit unit for subsequent measurements during a study. It is also a desirable feature on a packing line as a quality control tool since it minimizes the number of fruit destroyed from quality testing and allows evaluated fruit to be packaged (i.e. if deemed to meet quality standards) for revenue. These challenges led to the development of a non-destructive gloss device by the research group for the evaluation of gloss that developed on apples from using a simulated packing line wax application process.

This study was to evaluate the effect of surface treatments in achieving fruit surface cleanliness in preparation for an efficient wax application and hence an appealing gloss. Investigating the effects of the wax application process conditions on surface gloss development was another objective for which a non-destructive gloss device was developed to enable gloss measurements of curvatures like that of apples.

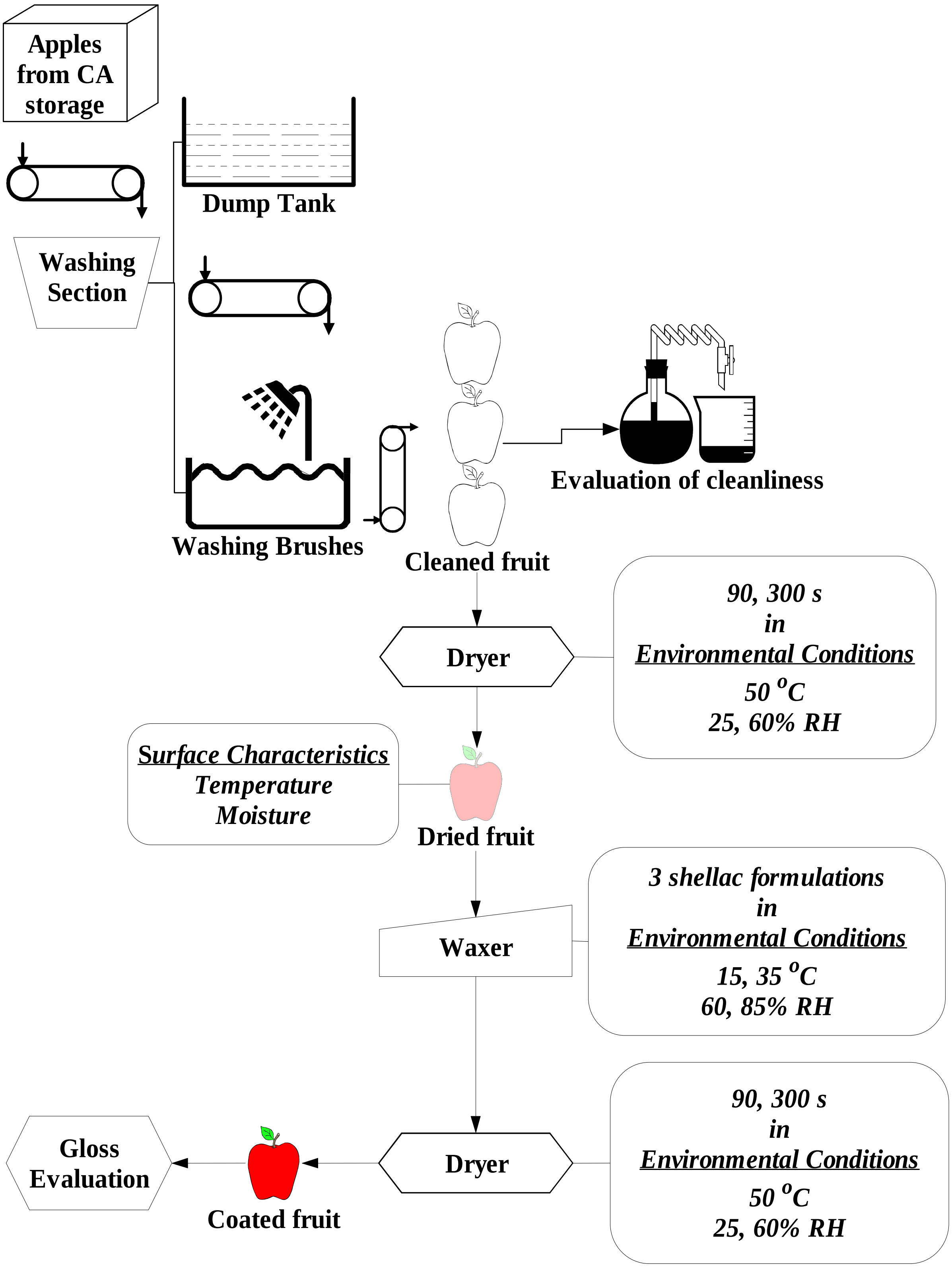


Figure 3.1. Flow diagram for the evaluation of cleanliness and gloss development on 'Red Delicious' apples

The flow chart outlines the processes for the evaluation of the level of cleanliness and gloss attained in this study.

Drying in this study corresponds to the sections of the packing line where (1) the washed apple surfaces are dewatered by means of blowing fans and brushes on a conveyor belt prior to coating, and (2) coatings applied to the apples are dried and solidified through solvent evaporation by circulating heated air. However in Michigan drying is carried out at high RH due to the climatic conditions. Drying as a process is the removal of moisture or humidity, but the high humidity condition used in this study was unable to achieve a dry surface. Therefore the effect or result of the drying process on the packing line is denoted in quotation marks: "dried", "dryness".

3.2. Materials and Methods

3.2.1. Fruit, Detergents and Coatings[1]

'Red Chief Red Delicious' apples harvested in Michigan, in 2006, 2007 and 2008, were DPA drenched and stored under controlled atmosphere (CA) at a commercial packing house. In the later part of the last quarter of each year respectively, the fruit were transported to Michigan State University where they were portioned into storage units with continued storage under CA of 1 ^{o}C, 1.5% O_2 and 3% CO_2.

Commercially available alkaline (Field Clean and Fruit Cleaner 395) and neutral (EpiClean) fruit detergents used in this study were donated by Pace International, LLC. (Yakima, WA), and FMC Technologies, Inc. (Lakeland, FL). These two companies and Cerexagri, Inc. (Monrovia, CA) donated commercially available shellac fruit coatings (Shield-Brite AP-40, Fresh-Cote 214 and Premium Apple Lustr) all of which have different volatile contents. The predominant volatile compositions were isopropanol (≤ 20% to 12%) and morpholine (≤ 5% to 3%). Isopropyl alcohol, for the evaluation of the efficiency of cleaning treatments, was obtained from Sigma-Aldrich Chemical Co. (St. Louis, MO).

[1] *The mention of a product/company is for identification purposes only and does not imply an endorsement by the author.*

3.2.2. Laboratory Packing Line

A pilot packing line was customized by Michigan Orchard Supply (MOS, South Haven, MI) for the purposes of this study, to simulate that of a commercial line. The washing brushes (WB) made from tufted brushes of 0.010" level nylon consisted of four rows; each brush was 18" long and had a 3.5" outside diameter rotating at a speed of 99 RPM. The system was enhanced by attaching a dump tank (DT), detergent and water-wash tanks with a heating system and associated piping. A Norpro instant immersion heater was used to construct the heating system (Everett, WA) which was equipped with blue spirit laboratory thermometers (Fisher Scientific, Waltham, MA) for temperature control. Separate pressure pumps with valve units were also attached for the detergent and water wash tanks. Mounted 28 mm above the WB system were TeeJet 6504 and 8005 nozzles (Spraying Systems Co., Wheaton, IL) for the detergent and rinse water systems respectively. At pressures of 30 and 60 psi, the 6504 nozzle discharged 0.35 and 0.49 GPM of detergent respectively whilst the 8005 nozzle discharged 0.43 and 0.61 GPM of water respectively.

The waxing brushes made from waxlon® consisted of four rows; each brush was 18" long and had a 3.5" outside diameter rotating at a speed of 99 RPM. The unit was placed in an environmental chamber with an accuracy of ± 0.3 ^{o}C and ± 2.5% RH (model SM-32S-SH; Thermotron Industries, Holland, MI) to generate specific temperature and RH, of 15 and 35 ^{o}C, and 60% and 85%, during waxing. The shellac coating was dispensed at 10 psi through an XR-TeeJet 8003VK nozzle (Spraying Systems Co., Wheaton, IL) fitted to a customized 400 ml tube, which was connected to an air pressure system.

The drying section of the waxing line was an environmental chamber (model SM-8-SH; Thermotron Industries, Holland, MI), with convectional heating capability, set to 50 ^{o}C. It equipped with a humidifier controller system with a ± 0.3% RH accuracy to monitor and control selected RH of 25% and 60%.

3.2.3. Cleaning Treatments

The apples were washed and "dried" under different conditions using the pilot packing line. Below in Table 3.1 is the compilation of the factors, and their levels, of the cleaning treatments used. In the DT section, the effects of alkaline and neutral detergents, DT water temperature of 18 and 40 ^{o}C, and fruit dwell time (which is the amount of time the fruit is in contact with the content of the DT) of 30 and 60 s were assessed. In the WB section a constant rinse water temperature of 40 ^{o}C at rinse pressures of 30 and 60 psi were used, with fruit dwell times of 60 and 120 s. Thirty two cleaning treatments per detergent were done and 16 evaluated when no detergent was used, and the factors were studied to evaluate the efficacy of the packing line in cleaning apples prior to waxing.

Table 3.1. Factors (detergent time, temperature dwell time, temperature and spray rinse pressure) on different sections of the washing line affecting the efficacy of the packing line in achieving optimal surface cleanliness on apples

Section of washing line	Factor	Level
DUMP TANK	Detergent	Alkaline
		Neutral
	Temperature	18 ^{o}C
		40 ^{o}C
	Dwell Time	30 s
		60 s
WASHING BRUSHES	Detergent	Alkaline
		Neutral
	Temperature	40 ^{o}C
	Dwell Time	60 s
		120 s
	Rinse Pressure	30 psi
		60 psi

3.2.4. Surface Assessment after the Different Cleaning Treatments

A method to evaluate the level of cleanliness that resulted from subjecting the fruit to different combinations of washing conditions was developed. The method constituted using a solvent extraction system to determine the amount of residue left on the apple surface after each treatment, and correlated to surface cleanliness. This novel system involved rinsing the surface of the cleaned apple fruit (*ca.* 190 – 250 g) with 200 ml of isopropyl alcohol for 2 min (30 s on each axis). The rinse process was repeated three times with new fruit and fresh isopropyl alcohol. The three rinses were combined and the residue from evaporation of the alcohol was weighed as an index of surface cleanliness. This was replicated 8 times, averaging 24 apples per cleaning treatment with 8 three-combined washes for evaluation; more residue extracted indicated a lesser cleaning power of the cleaning treatment used. A total of 80 surface treatments with 1920 apples were used for this part of the study.

3.2.5. Assessment of Surface Temperature, Dryness, and Wax Viscosity Prior to Waxing

Cleaned fruit from section 3.2.3 were "dried" at 50 ^{O}C for either 90 or 300 s at 25% or 60% RH. The temperature of the fruit after emerging from each drying condition was measured using an infrared thermometer gun, with ± 2 ^{O}C accuracy at less than 80% RH (Catalog No. S90202, Fisher Scientific, Hampton, NH), at a distance of *ca.* 30 – 35 in. The average and standard deviation of 6 replicates were reported. Surface moisture, remaining after drying, was quantified by absorbing the moisture on the surface with absorbent #2 filter paper (Whatman Inc., Florham Park, NJ) and recording the difference in weight. This was repeated for 6 fruits; the average and standard deviation were reported.

The rheological profile of the coatings were studied by measuring their viscosity at 1, 2.5, 5, 10, 20, 50 and 100 RPM using a Brookfield viscometer (model 1/2RV TDV-I; Middleboro, MA). Spindle no.1 at a constant depth in a 600 ml beaker was used. The experiments were conducted at room temperature and the flow properties measured for wax at ambient temperature (20 ^{O}C) or heated to 40 ^{O}C; the two temperatures were applied to both the original coating and after solvent evaporation (7% w/w) to a more viscous consistency. Models described by Steffe et al. (2006a) were used to determine the shear rate and stress from which apparent viscosity was calculated using the power law model $\sigma = K \dot{\gamma}^{n}$.

Nine replicates were conducted for each coating, fluid consistency and spindle speed.

3.2.6. The Waxing Process

To study the effect of different factors of the application process such as coating formulation, drying time, temperature and RH with their interactions on the efficiency of the waxing process, cleaned and "dried" fruit were coated with shellac coatings in an environmental chamber (detailed in section 3.2.2) using a combination of the factors listed in Table 3.2.

Measurement of Fruit Gloss

Coated fruit were left overnight to allow the wax to set prior to gloss measurements. Gloss was measured using a non-destructive customized glossmeter. Three apples were measured from each treatment, and three measurements were taken per apple; 130 images were obtained per measurement making a total of 1170 images per treatment. The average gloss and standard error per coating treatment were reported.

Construction of a customized non-destructive gloss device

A prototype glossmeter (Figure 3.2) was successfully designed and built specifically for the purposes of this study to be able to non-destructively measure the gloss on whole fruit. The non-destructive glossmeter was constructed with a Fire-i^{TM} Digital Camera, and two 60 W incandescent light bulbs serving as the light source. A black box housed the device to prevent extraneous light sources from seeping in. The apple fruit automatically rotated at 360^o on a rotary jig fruit holder while 130 images were taken. In operation the glossmeter checks the pixels for the red, green and blue channels. The blue channel was chosen because it gave consistent low and high values when measuring a matte and glossy spheres respectively. The threshold was set at 230 and all blue pixels above the threshold were recorded. The average number of saturated blue pixels for all images was calculated as an index of fruit gloss (Gloss Units) using MATLAB. The device was calibrated against standard glossy and non-glossy black and red spheres before the collection of each data set. Gloss as determined by the glossmeter was correlated to human perception of gloss; a consumer sensory panel using three different levels of gloss, and measuring the gloss of the same fruit on the glossmeter was conducted.

Consumer Sensory Evaluation

A cohort of 50 untrained panelists comprising students, faculty and staff, spanning a wide range of ages, at Michigan State University was recruited. A ranking test with a 3-level scale was used in a single session conducted under white incandescent light at room temperature. Apples were coated using three different waxing conditions a day prior to the evaluation to allow efficient drying and setting of the coating. Random 3-digit codes were assigned to each treatment and presented on trays to the panelists in a random fashion on the evaluation day. The ranking used was as 1 (least glossy); 2 (moderately glossy); and 3 (highly glossy). The gloss on the same fruit evaluated by the panel was measured on the customized glossmeter.

This study has Institutional Review Board (IRB) approval (#X07-469) as exempt.

Table 3.2. Factors of the wax application treatments evaluated for their effects on gloss development

Factor	Level	Description
Wax Viscosity	Original 7%	Volatile content (w/w) evaporated
Waxer Temperature	15 ^{o}C 35 ^{o}C	Temperature in wax application chamber, simulating extreme conditions in packing house
Waxer Relative Humidity	60% 85%	Humidity in the wax application chamber, typical of environmental conditions
[1]Drying Time	90 s 300 s	50 ^{o}C when the fruit come out of the waxer
[1]Relative Humidity in the Dryer	25% 60%	Conditions in the drying oven for both washed and waxed fruit

[1]The drying time and relative humidity in the dryer represent the different levels of surface dryness and temperature

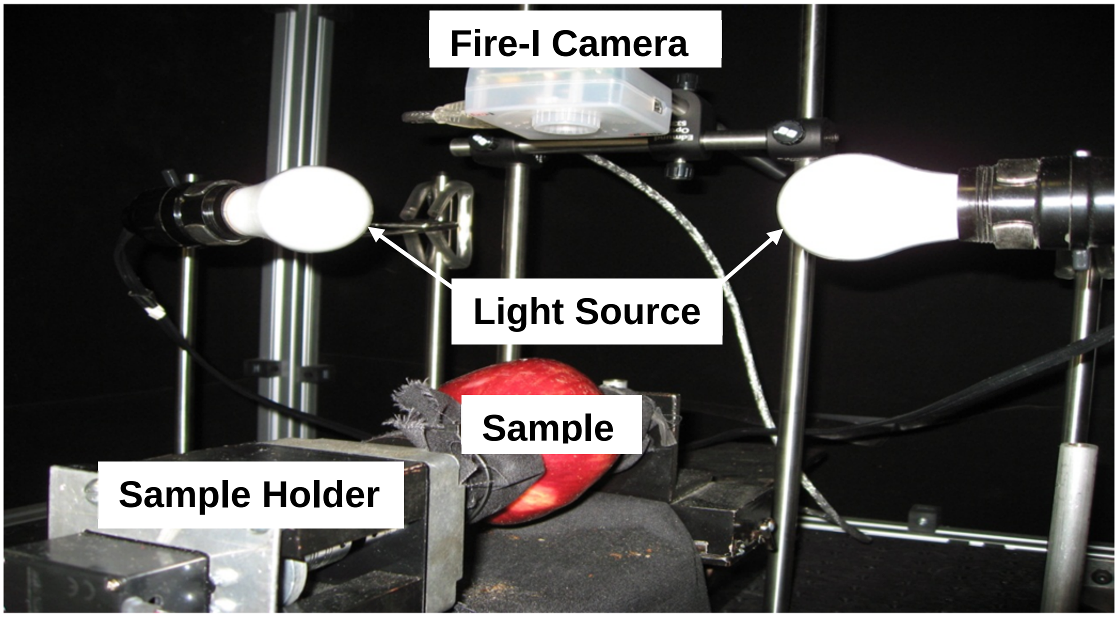


Figure 3.2. Customized glossmeter for non-destructive apple gloss measurement

3.2.7. Statistical Analyses

While a full factorial design was used to assess the effect of washing treatment on surface cleanliness, a fractional factorial design was used to assess the effect of coating conditions on the development of gloss. The designs provided an 84% and 100% power respectively for identifying significant differences in the treatments used, and also allowed the evaluation of the main effects with their 2-way interactions.

The collected data was analyzed using the Statistical Analysis System (SAS Version 9.1) software (SAS Institute, Cary, NC). Analysis of variance (ANOVA) at confidence interval of 95% ($\alpha = 0.05$) was evaluated and Tukey's multiple comparison test for the means was used to determine significant differences in the detergent type and the levels of the other factors used. Multiple comparison adjustment for the means using Bonferroni's correction was used to analyze the effect of the waxing conditions on the quality attribute of gloss. The Spearman rank correlation was used to find the correlation between the human perception of gloss and the glossmeter measurements.

3.3. Results and Discussion

3.3.1. The effect of surface treatments on cleanliness

Critical points in the cleaning section of the packing line were identified in order to minimize their variability, and to optimize the packing line to improve surface cleanliness and ensuing gloss. A simulation of existing washing treatments used in the industry was employed; several controllable factors such as the type of detergent, water temperature, rinse pressure, and dwell time were identified to contribute to the surface cleanliness of apples. The fruit detergents were used at concentrations according to the manufacturers' specifications. A detergent was applied to a section of the washing line at a given time such that when it was applied in the DT, only water was applied on the WB and vice versa. The control was no detergent in either section of the washing line.

Effects of the dump tank temperature and detergents

A DT temperature of 40 ^{o}C resulted in significantly lower residue recovered from the fruit surface, indicative of a greater cleaning power compared to 18 ^{o}C (Figure 3.3). The higher temperature was better able to remove the natural wax and the residual fungicides present on the fruit, recovering 0.037 ± 7.1E-04 g in comparison to 0.042 ± 6.8E-04 g recovered using 18 ^{o}C in the DT. Hot water systems not only produce cleaner fruit, but have been shown with various heat treatments by many studies to control insects and diseases in produce as well as maintaining and enhancing quality (Smith et al., 2000; Fallik et al., 2001; Spadaro et al., 2004; Bai et al., 2006; Wang et al., 2006). It is essential that these heat treatments are closely monitored to prevent heat injury to the fruit.

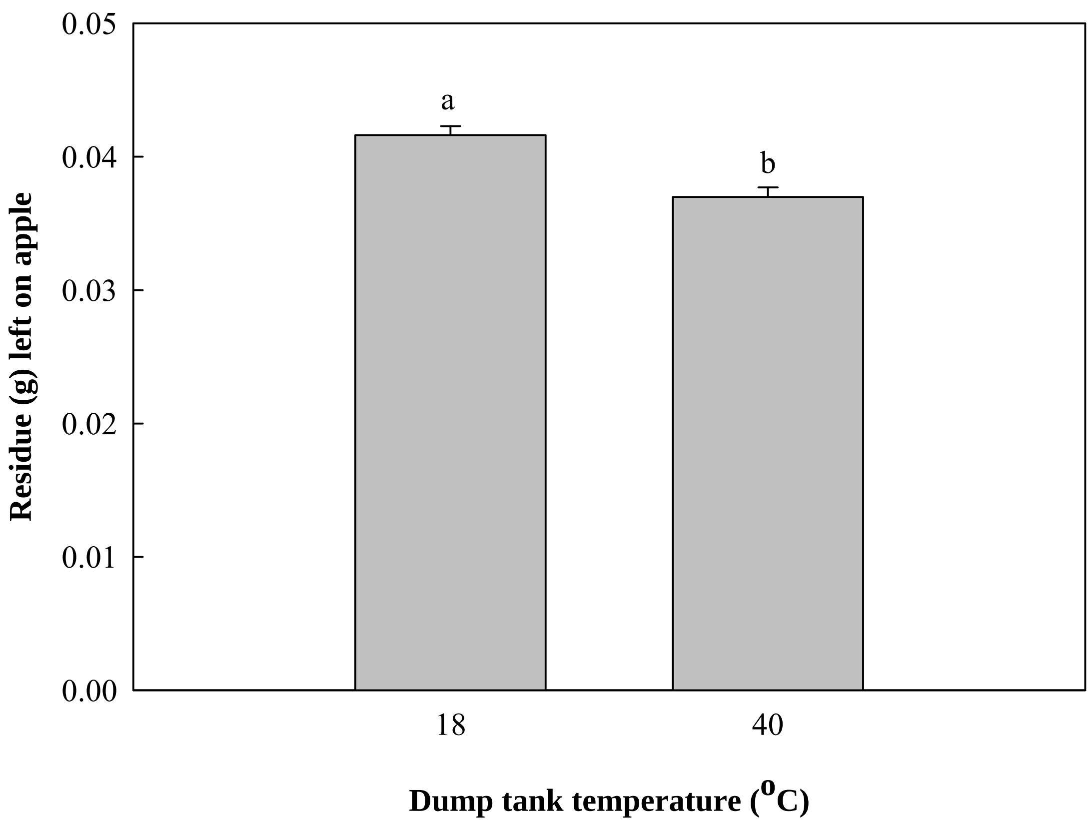


Figure 3.3. Effect of 18 and 40 ^{o}C dump tank temperature on apple surface cleanliness
Bar columns with the same letter are not significantly different (means ± SE; n = 672; p ≥ 0.05)

There was no significant difference in the cleaning power of the alkaline detergent and when no detergent (i.e. only water) was used (Figure 3.4), irrespective of where the detergent was applied, either in the DT or on WB. The rubbing action of manually cleaning apples accounts for the observed similar cleaning power of systems with no detergent to that with detergent (Kenney et al., 2002); in this study this would be attributed to the scrubbing action of the brushes. Overall the use of fruit detergents is reported by Alvindia et al. (2007); Hansen et al. (2006) and Kenney et al. (2002) to impact treatment efficacy.

The alkaline detergent produced significantly cleaner apples than the neutral detergent. Differences in their formulation could be the reason for the significant difference observed. The alkaline detergent used in this study contained sodium hydroxide and/or potassium hydroxide and/or their phosphate salts, together with anionic, cationic and non-ionic surfactants. In the formulation of the neutral detergent however, only an anionic component was listed as a major ingredient. Surfactants act to reduce surface tension of the solution which is required for adsorption of the surfactants at the interface. Interactions across an interface are stronger for molecules of the same nature and ions of opposite charges (Rosen et al., 2002). Thus the alkaline detergent having different charged ends is better able to attract, remove and disperse oppositely charged molecules of the residue, compared to the neutral detergent which is limited to the removal of predominantly the positively charged residues. Also, when neutral detergents are used in an immersion cleaning system, as in the DT, the dirt rises to the surface and is re-deposited on the part of the substrate which is pulled out through the top layer of dirt (Moore, 2003). This could be due to the absence or low concentration of dispersants such as silicates in the neutral formulation. The alkaline detergent by having a caustic base has a strong dissolving power by penetrating the residue and facilitating its separation from the fruit (Moore, 2003). By virtue of possessing different charged surfactants, the alkaline detergent also had the advantage of synergy. Synergy of a surfactant mixture of different charge types is an important method to enhance performance of detergents (Rosen et al., 2002).

Effects of the dwell time in the dump tank and on the brushes, and rinse pressure

No significant differences were recorded for the level of cleanliness that resulted from subjecting the fruit to dwell times of either 30 or 60 s in the DT. The 60 s dwell time produced a residue recovery of 0.039 ± 7.1E-04 g whilst that of 30 s produced a recovery of 0.040 ± 7.0E-04 g. Walker et al. (1999) reported that increasing dwell time from 10 to 23 s in a pressure wash for citrus produced a significant change in the level of cleanliness by decreasing the number of pests by 6.1-fold. The 13 s increase in dwell time which produced a significant change in the level of cleanliness could be attributed to the peel morphology being different from that of an apple, and also the differences in the variables studied.

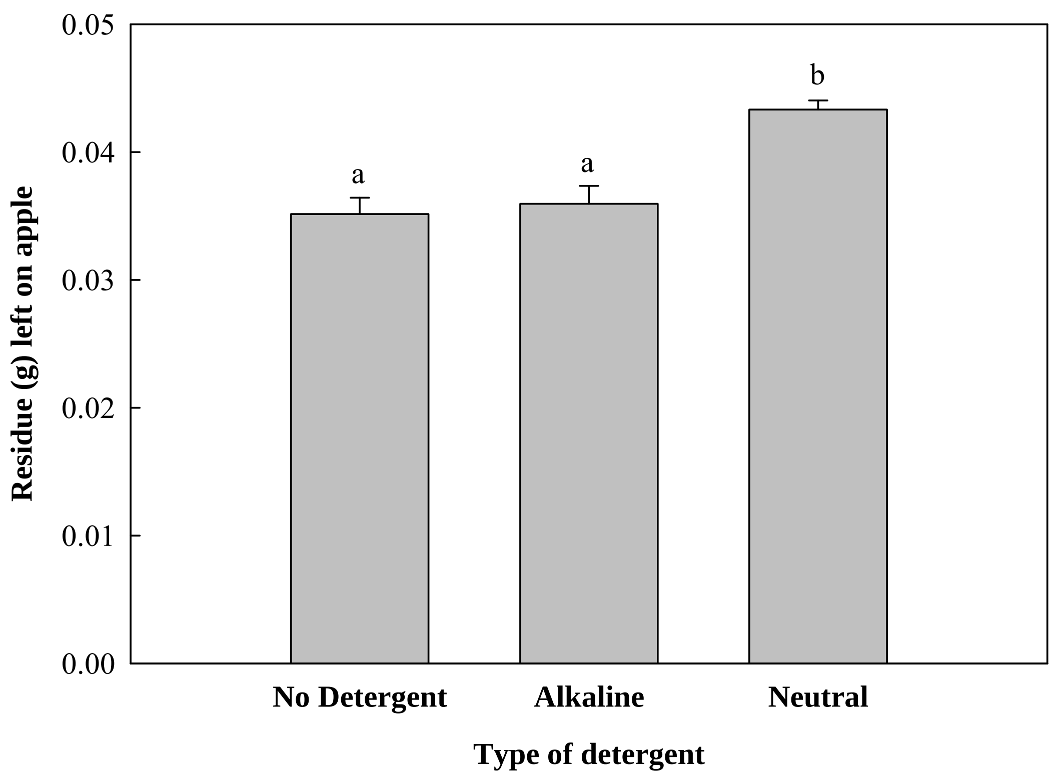


Figure 3.4. Effects of no detergent vs. using an alkaline vs. neutral detergent on the surface cleanliness of apples

Bar columns with the same letter are not significantly different (means ± SE; $n = 672$; $p \geq 0.05$)

Fruit surface cleanliness resulting from the dwell times of 1 and 2 min on the WB were also observed not to be significantly different. Similar amounts of residue, 0.039 ± 6.9E-04 g and 0.039 ± 7.2E-04 g respectively were recovered. Thus increment in the dwell times in both the DT and on the WB were observed not to be significantly different in achieving a cleaner surface.

Also, the power of the rinse pressure used had no significant effect on cleanliness. The 30 psi rinse recovered a residue of 0.039 ± 7.1E-04 g, whilst that of 60 psi rinse recovered 0.040 ± 6.9E-04 g. High pressures washes have been recorded to enhance the cleaning efficiency of systems used for produce. According to Neven et al. (2006) the use of high pressure proved effective in cleaning (removal of moth and eggs) than hot water sprays and dips. Walker et al. (1999) reported that high pressure significantly cleaned the surface of citrus fruit. The observational dissidence in this study and Walker's was probably because of the differences in the pressure levels, 30 and 60 psi rather than 325 and 150 psi since such high pressures would have damaged the apple.

Interaction of temperature, detergent, dwell time and pressure on the level of cleanliness

Out of the ten possible two way interactions, the significant pairs were detergent*temperature of DT, detergent*dwell time on WB, temperature of DT*dwell time on WB, and dwell time in DT*rinse pressure. It is interesting to note that neither detergent nor DT temperature were significant in a two way interaction with either the dwell time in DT or the rinse pressure, while the interaction between dwell time in DT and rinse pressure was significant.

Graphs of the interactions demonstrate the effect of one factor on the other. In the detergent*DT temperature interaction (Figure 3.5A), increasing the DT temperature not only resulted in a cleaner surface but its effect was improved with the use of the alkaline detergent. At

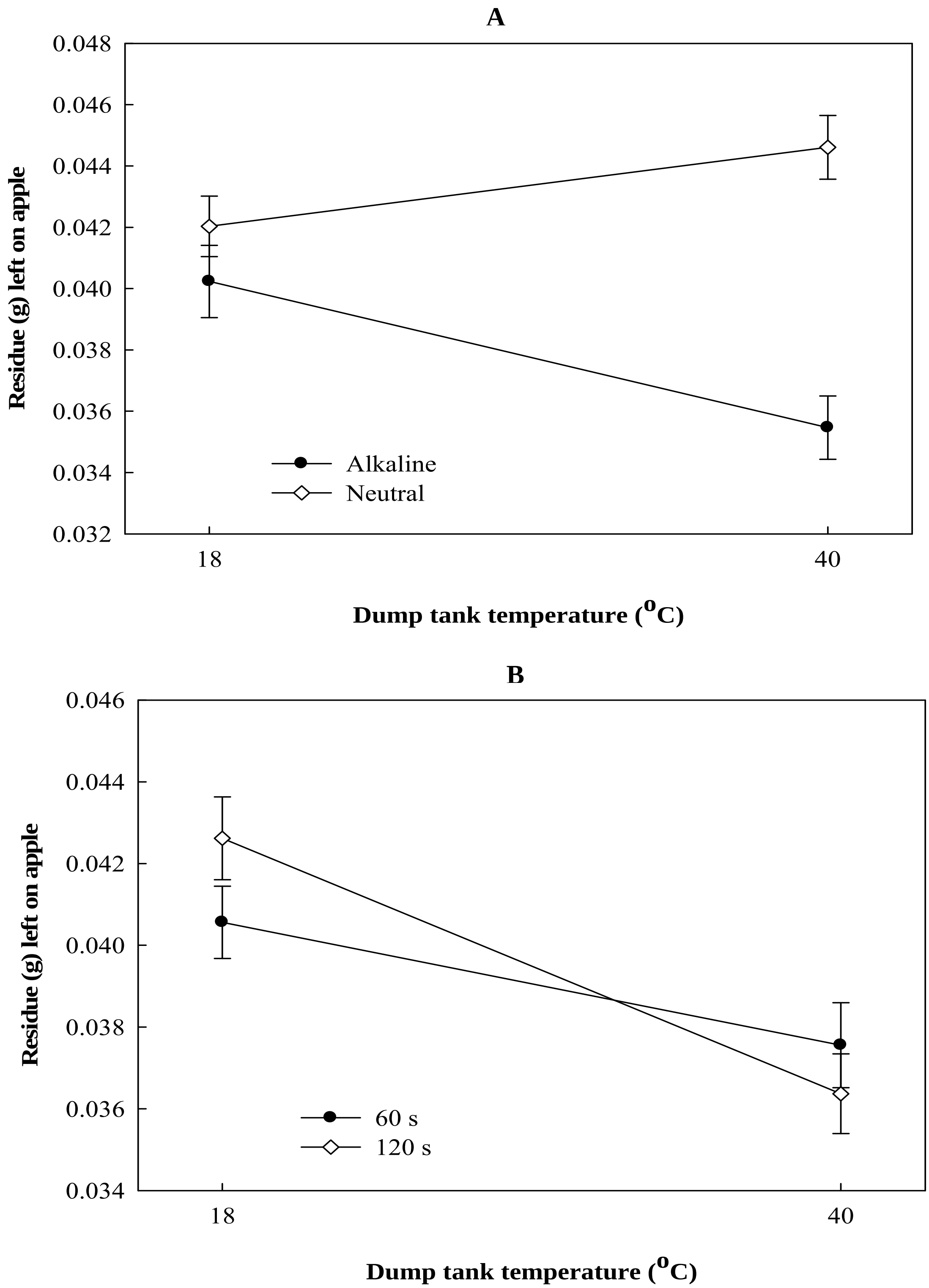


Figure 3.5. Two way interactions of 18 and 40 °C dump tank temperature with (A) alkaline and neutral detergents and (B) 60 and 120 seconds dwell time on the washing brushes on the surface cleanliness of apples (means ± SE; n = 512 for A, 672 for B; $p \leq 0.05$)

18 ^{o}C there was no significant difference in the level of cleanliness regardless of the detergent type, but at 40 ^{o}C there was a significant difference with the alkaline producing a significantly cleaner surface. Temperature greatly influences cleaning efficiency as an increase in temperature increases the activity of the cleaning agents (Chateau et al., 2004) by enhancing the formation of micelles with the dissolution of residue and its ultimate removal and dispersion at the interface (Rosen et al., 2002). Unlike the synergistic influence of the surfactants with temperature in the alkaline detergent to produce a cleaner surface, the neutral detergent recorded no significant difference in the residue recovered at both temperatures.

For the interaction of DT temperature*dwell time on WB (Figure 3.5B) increasing the temperature together with increasing the dwell time on the WB produced a cleaner fruit. An 18 ^{o}C wash with a dwell time of 60 s resulted in cleaner fruit, while the 120 s dwell time produced cleaner fruit at 40 ^{o}C. Since the higher temperature of 40 ^{o}C was more efficient at removing extraneous variables on the surface of the fruit, an increase in exposure time will facilitate the cleaning process and produce a cleaner surface. Considering the dwell time in DT*rinse pressure interaction (Appendix B), both 30 and 60 psi rinse pressures at 30 s in the DT produced the same level of cleanliness. At 60 s in the DT, the 30 psi recovered the lower amount of residue. Since the action of the rinse pressure is on both the fruit and brushes, the reason that the 60 psi rinse produced the less cleaner fruit could be due its more powerful force dislodging residue that had been removed, from the fruit, by the brushes back onto the fruit. This would be more pronounced with exposure time and could account for the higher residue recovery from fruit rinsed at 60 psi for 60 s. In the interaction of detergent*dwell time on WB (Appendix B), it was observed that 60 s on the washing brushes was what produced the cleaner fruit with the alkaline detergent whereas in the case of the neutral detergent it was the 120 s dwell time on the brushes. At 60 s with the alkaline detergent, most of the extraneous variables had been removed such that an increase in dwell time did not contribute significantly to surface cleanliness. But with the neutral detergent, which is less aggressive, an increase in exposure time was required to observe its effectiveness.

3.3.2. The effect of dewatering on surface dryness

Dewatering and drying are necessary after cleaning to complete the surface treatment phase required for waxing. Surface dryness and temperature are critical to achieving optimal wax application in terms of a uniform and even coverage, and humidity plays a vital role in the dryness of the fruit surface. Dew point, the temperature at which condensation occurs at a specific vapor pressure, is an important factor to consider in order to make provision for possible condensation on the surface of the apples. Moisture will condense on the fruit when the vapor pressure at a set temperature exceeds the maximum for that temperature (Wills et al., 2007). To avoid condensation, it is therefore critical to keep the temperature above the dew point by maintaining a warm fruit surface, and if possible controlling the humidity of the processing environment. For this study, fruit were dewatered and "dried" at 50 ^{o}C in 25 or 60% RH which have dew point temperatures of 24.56 and 40.07 ^{o}C respectively. In either condition, the fruit were "dried" for 90 or 300 s. The purpose of varying the humidity and drying time was to simulate different climatic conditions and lengths of the dewatering tunnel respectively. The control was fruit that had just emerged from the washing brushes and had not been "dried". Prior to entering the dryer, the surface temperature of the fruit was above the dew point for the 25% condition but below that for 60% RH. Upon exiting the dryer the surface temperature was still below the dew point for the 60% RH conditions (Figure 3.6).

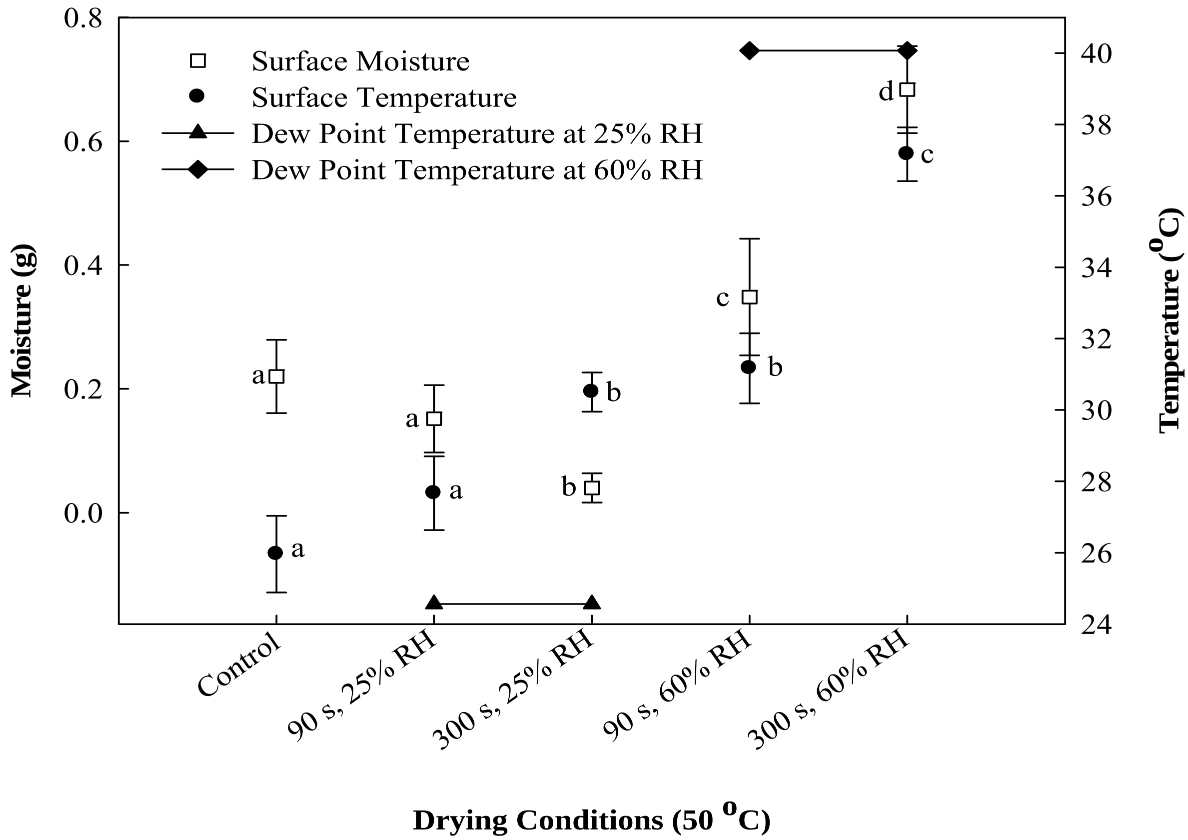


Figure 3.6. Surface moisture (g) and temperature (^{o}C) of cleaned apples "dried" at 50 ^{o}C for 90 and 300 seconds in RH conditions of 25% and 60%
Data symbols with different letters are significantly different (mean ± SD; n = 30; p ≤ 0.05)

The average fruit surface temperature was 25.95 ^{o}C, upon exiting the washing section of the line and prior to entering the dryer. After drying in the oven in 25% RH, the surface temperature increased from 27.67 to 30.50 ^{o}C for the drying duration of 90 and 300 s respectively. There was an increase from 31.17 to 37.17 ^{o}C in the 60% RH drying conditions (Figure 3.6). It is obvious that the longer duration and more humid conditions produced the warmest fruit surface. As a result of the surface temperature of fruit from the 60% RH drying conditions being below the dew point, there was significantly more condensation of moisture on the fruit surface compared to those from the 25% RH treatment. There was an observed minimal decrease of 31.06% in surface moisture at 25% RH, with a maximum increase of 210.61% at 60% RH (Figure 3.6). Once the fruit have gone through the dewatering and drying phase, they are conditioned for waxing, but the presence of moisture would prevent an even and intact coating disposition of the wax.

3.3.3. Changes in apparent viscosity of the coatings with solvent concentration and temperature

The rheological characteristic of the wax is important as it influences the outcome of the application process. Temperature plays a critical role in the rheological properties of fluids (Steffe et al., 2006b) just as changes in formulation. Viscosity, a controllable factor, is particularly important since flow properties influence the spreadability. It is related to spreadability such that a high viscosity will reduce flow properties and therefore affect the distribution and coverage on the fruit surface.

Assessment of the rheological profile of the coatings revealed them to exhibit time-independent non-Newtonian behavior (data not shown). The slope of shear stress against rate using the power law model $\sigma = K \dot{\gamma}^{n}$ was calculated as the apparent viscosity of the coatings (Steffe et al., 2006b). The apparent viscosity decreased with an increase in temperature from 20 or 40 ^{O}C, which resulted in a fluid with a more rapid/increased flow (data not shown). Studies on the effect of temperature on the rheological properties of solid shellac reported a sharp decrease in melt-viscosity with temperature (Goswami et al., 2003). This shows that solid shellac and solution of shellac have the same behavior pattern with increasing temperature. According to

Goswami et al. (2003), the decrease in viscosity with increasing temperature is due to changes in the conformation of the shellac molecules as a result of decreased molecular chain entanglement.

It was hypothesized that viscosity would increase as the solvent matrix evaporates, and an increase of 4.6 – 11.4% in apparent viscosity was observed when 7% (w/w) of the solvent was evaporated. If the solvent is acting as a plasticizer or diluent then the increase could be explained by an increase in entanglement and/or decrease in flexibility of the molecular chains of the coating. Figure 3.7 shows the changes in shear stress and apparent viscosity with increasing volatile vaporization. During processing, there are chances of volatile vaporization with improper handling and storage of the coating, which could lead to an increase in viscosity.

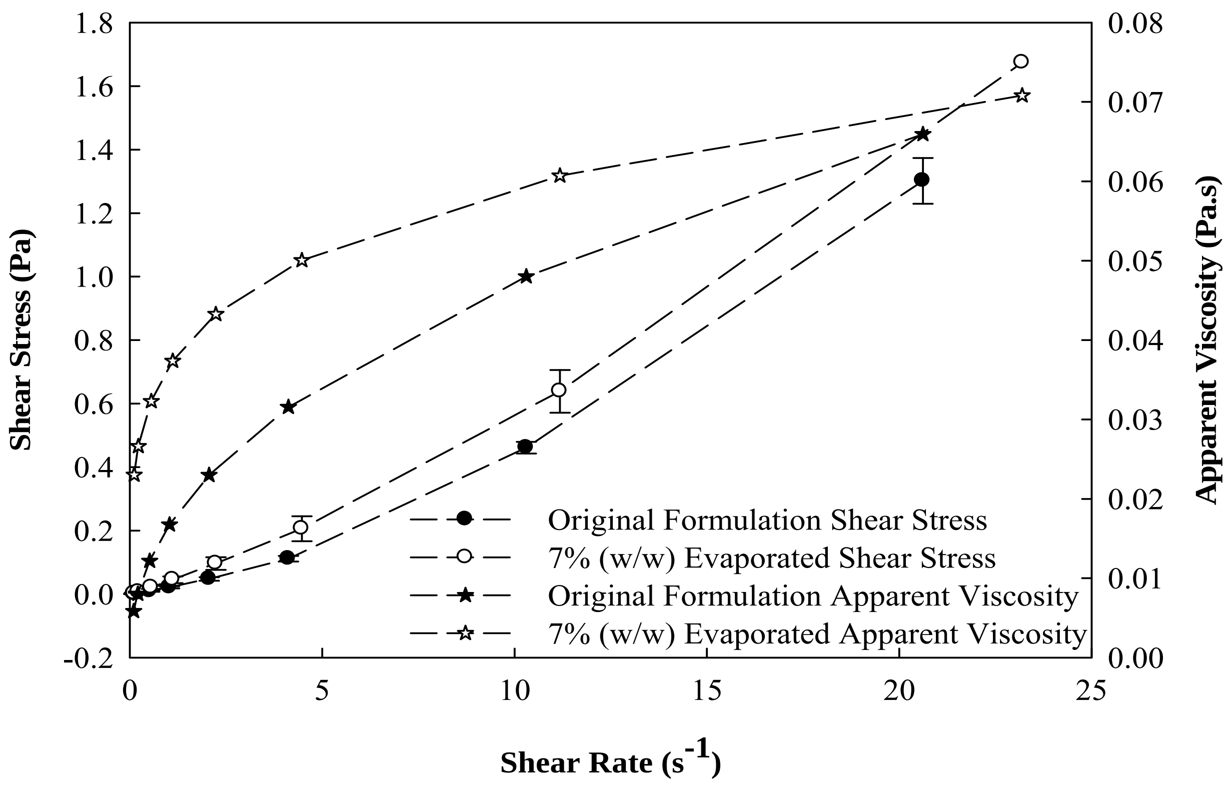


Figure 3.7. Apparent viscosity and shear stress of shellac fruit coating at 20 ^{o}C with varied shear rates (mean ± SD)

Changes in viscosity, with accompanied changes in flow behavior, will affect the uniformity and thickness of the coating layer and hence the gloss and barrier properties, which can influence quality such as changes in weight and firmness. Information garnered in this section provided insight into the flow characteristics and behavior of the coatings in the waxing system, hence the effect of viscosity in the wax application process on gloss. There was however the importance of a valid means to measure ensuing gloss to effectively study the influence of the wax application variables on it.

3.3.4. Correlation of the customized glossmeter to human perception of gloss

The effects of wax application variables namely viscosity, the temperature and RH during the waxing and drying process, as well as surface dryness on the development of gloss were studied. Gloss determined by the gloss device had to be correlated to gloss perception by humans. This was necessary to identify and develop a scale based on the human ability to perceive differences in gloss, and it involved several sensory evaluations with modifications to the gloss device. The first correlation study involved fifty untrained panelists who evaluated and rated the gloss on apples as "slightly", "moderately" and "highly" on a 15-point scale using standards marked as "non-glossy" and "glossy" to understand the aesthetic scale. The gloss on the fruit evaluated by the panel was measured on a customized glossmeter, which was a modified Ocean Optic Spectrometer mounted over an inverted cup in a black box. The panel could not consistently distinguish between the "moderately" and "highly" gloss fruit but the glossmeter was able to distinguish between the different intensities of gloss though with high variability. This led to the redesign and the development of a new device to improve precision using a Fire-i™ 1394 Firewire camera over a rotary jig fruit holder described in the methodology section.

Fifteen of the 50 panelists who were able to successfully distinguish and identify the sequence of the degree of gloss from the first study were selected to evaluate another set of fruit to establish a correlation. They were presented with 3 test sets of 4 apples each, and gloss ranks were determined by both the sensory panel and the customized glossmeter. Spearman rank correlation coefficients were computed between the gloss ranks produced by the sensory panel and that obtained from the glossmeter measurements. Rank correlation coefficients were computed for the overall dataset, for each panelist and for each test set of apples evaluated. There was evidence for a significant association between the ranking yielded by the glossmeter and the sensory panel ($P < 0.0001$). The rank correlation was estimated to be equal to 0.38. From a validation standpoint, it was concluded that the estimated rank correlation between the sensory panel and the glossmeter was fairly low. Subsequent analyses showed that the low correlation may be due to the test sets and the experimental design model used. Running an Analysis of Covariance (ANCOVA) showed that approximately 88% of the variation in glossmeter readings was explained by a model that included the effects of sample set and the average assessment of a sensory panel. This led to the design of a new consumer evaluation test recruiting another 50 panelists and described in the methodology section 3.2.6.

The Spearman rank correlation coefficient between the ranking of the new data set yielded by the glossmeter and the sensory panel was significantly different ($P = 0.0024$) but with a fairly low rank correlation of 0.247. The frequency table (Table 3.3) indicated a 45.6% (sum of the diagonal percentages: 22.45 + 14.29 + 8.84) agreement between the panel ranking and the glossmeter measurement; there was a mismatch of 54.4% (sum of the off-diagonal percentages) between the sensory panel and the glossmeter. The chi-square test for independence indicated a significant association between the panel ranking and the glossmeter measurement ($P<0.0001$).

Table 3.3. Frequency table of panel ranking by glossmeter measurement

	Glossmeter Measurement*			
Panel Ranking*	1	2	3	**Total** Frequency Percent
1	33	5	10	48
	22.45	3.40	6.80	32.65
2	4	21	26	51
	2.72	14.29	17.69	34.69
3	12	23	13	48
	8.16	15.65	8.84	32.65
Total				
Frequency	49	49	49	147
Percent	33.33	33.33	33.33	100.00

* 1 = least glossy, 2 = moderately glossy, and 3 = highly glossy

Glossmeter rankings of 2 (moderately glossy) and 3 (highly glossy) were not consistently distinguishable by the panel. Therefore rankings 2 and 3 were collapsed into one category and the correlation association re-analyzed. The rank correlation coefficient between the ranking yielded by the glossmeter and the sensory panel was significantly different ($P = 0.0024$) but this time with a rank correlation of 0.523. Identifying the gloss levels imperceptible to the human senses enabled the setting of the glossmeter scale within that of human perception. A frequency table (Table 3.4) indicated complete agreement between the panel ranking and the glossmeter ranking for 78.9% of the test samples; there was a mismatch of 21.1%. The chi-square test for independence indicated a significant association between the panel ranking and the glossmeter ranking (P<0.0001).

Table 3.4. Frequency table of panel ranking by glossmeter measurement after merging the moderately and highly glossy fruit into one category

	Glossmeter Measurement*		
Panel Ranking*	A	B	**Total** Frequency Percent
A	33 22.45	15 10.20	48 32.65
B	16 10.88	83 56.46	99 67.35
Total Frequency Percent	 49 33.33	 98 66.67	 147 100.00

* A = least glossy, B = moderately glossy + highly glossy

A Spearman correlation coefficient of 0.773 was estimated between the glossmeter rankings and its measurements. Given that the glossmeter rankings were based on customized gloss measurements, this correlation may be considered as an upper limit to compare agreement between the panel and the glossmeter. When the gloss on a coated fruit was compared to that of an uncoated fruit, there was a 100% agreement between the panel and glossmeter rankings.

The prototype gloss device therefore provided an efficient and accurate means to non-destructively measure gloss from different waxing conditions.

3.3.5. The effects of wax application process conditions on gloss prior to refrigerated storage

The performance of the coating system is an interaction not only with the type of fruit but also with the wax properties (Cisneros-Zevallos et al., 2005). The effect of changes in apparent viscosity of the coating, as a result of varying wax temperature, was studied on gloss. Though there were significant differences in the apparent viscosities with varying the wax temperature, preliminary studies showed no significant differences in the gloss that developed when the wax temperature was either at 20 or 40 ^{o}C. This was attributed to heat transfer during the spray application which may have rendered the wax temperature the same, and hence similar viscosity, by the time it made contact with the apple peel. However significant differences in gloss were observed with varying the viscosity of the wax at the same temperature. Thus for the remainder of the study only the wax viscosity was evaluated, excluding its temperature.

Prior to wax application cleaned fruit were “dried” in 25 or 60% RH at 50 ^{o}C condition, which is commonly found in northeastern packing houses. “Dried” fruit from the 60% RH treatment had condensation as a result of the surface temperature being below that of the dew point (Figure 3.6), and thus entered the wax applicator wet. This meant a dilution of the applied coating. The conditions in the wax applicator used in this study were a temperature and RH of 15 or 35 ^{o}C, and 60 or 85% respectively. No visual differences in the wax distribution were observed when the wax was applied in 15 or 35 ^{o}C conditions. However there was a difference observed between the two temperatures when the humidity was set to 85%. Fruit waxed in the 35 ^{o}C, 85% RH exited the wax applicator with a thin coverage of coating which appeared to be diluted; the surface temperatures prior to entering the wax applicator under the stated conditions were below that of the dew point. Drying coated fruit at 50 ^{o}C in 60% RH visibly washed off the coating as a result of condensation, and the wash-off was more pronounced in the longer duration. The drying conditions before and after applying wax influenced surface temperature and moisture, thereby dictating the final gloss on the fruit by way of dew point temperature and condensation.

A relationship between how dry the surface of the apples needs to be, and what surface temperature has to be attained to give the best gloss was determined. As the humidity and duration in the dryer increased, and the moisture on the surface of the fruit increased as a result, there was a decrease in the gloss that developed. The effect of surface temperature and moisture on gloss could therefore be said to be influenced by the dew point temperature such that if the surface temperature was below the dew point, condensation prior to and after coating application diluted the applied coating and decreased the gloss that developed.

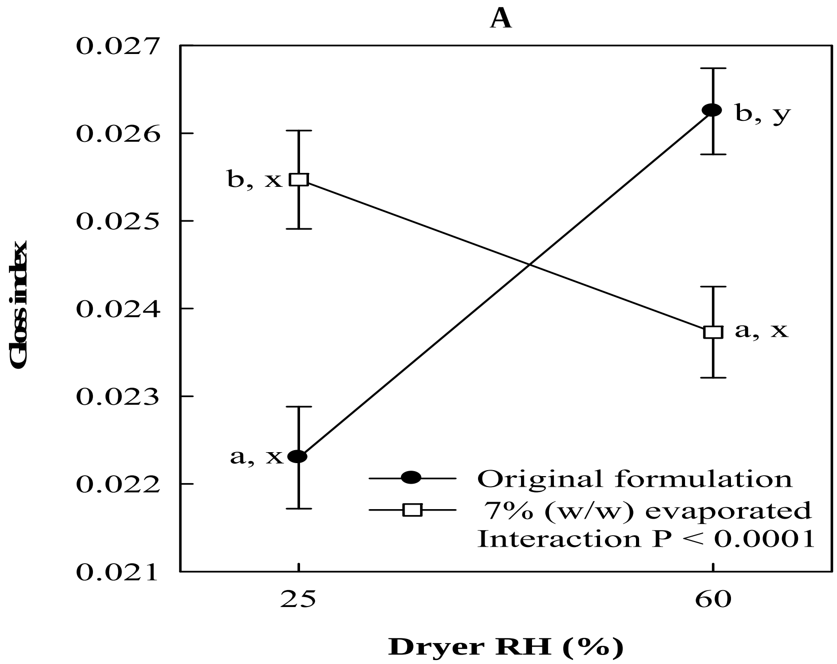
Only the humidity in the drying oven played a significant role in the gloss that evolved. This was obviously due to dilution and removal of the coating in the presence of condensation. However, the effect of the factors evaluated on each other resulted in significant effects on the resulting gloss. Significant interaction effects were identified between wax viscosity*RH in drying oven, temperature in wax applicator*drying time, RH in wax applicator*drying time and RH in wax applicator*RH in drying oven on the gloss on apples.

An increase in RH from 25% to 60% while drying fruit coated with the original formulation (0.051 – 0.062 Pa.s) resulted in a significant difference in the gloss whereas for the higher viscous coating (0.057 – 0.066 Pa.s) no significant differences were recorded (Figure 3.8A). It was observed that the more viscous wax did not spread readily and evenly on the fruit, and was therefore unable to produce a uniform coating coverage as a result of the evaporation of solvents which concentrates the solid content thus producing a system with high viscosity and reduced spreadability. The difficulty in the utilization of viscous formulation is also reported by Rojas-Argudo et al. (2009) in a study on locust bean gum-based edible coatings. Under the 25% RH condition in the dryer, the higher viscous coating produced a significantly higher gloss, and at 60% RH the higher gloss was produced by the original formulation. The author hypothesize that drying in 25% RH (without the incident of condensation) provided conditions that did not interfere with flow behavior. Therefore sections of the fruit peel that were coated had relatively more wax (compared to that of the less viscous coating) setting in those areas. Preliminary studies with wax volume showed that coating with a greater amount of wax result in higher gloss; hence though fewer areas were visually observed to be covered with wax from the use of the more viscous coating, the greater amount of wax in the covered areas translated into higher gloss. In the case of the original formulation, the author hypothesize that the 60% RH influenced spreadability and thus an enhanced uniform coverage translating into greater gloss.

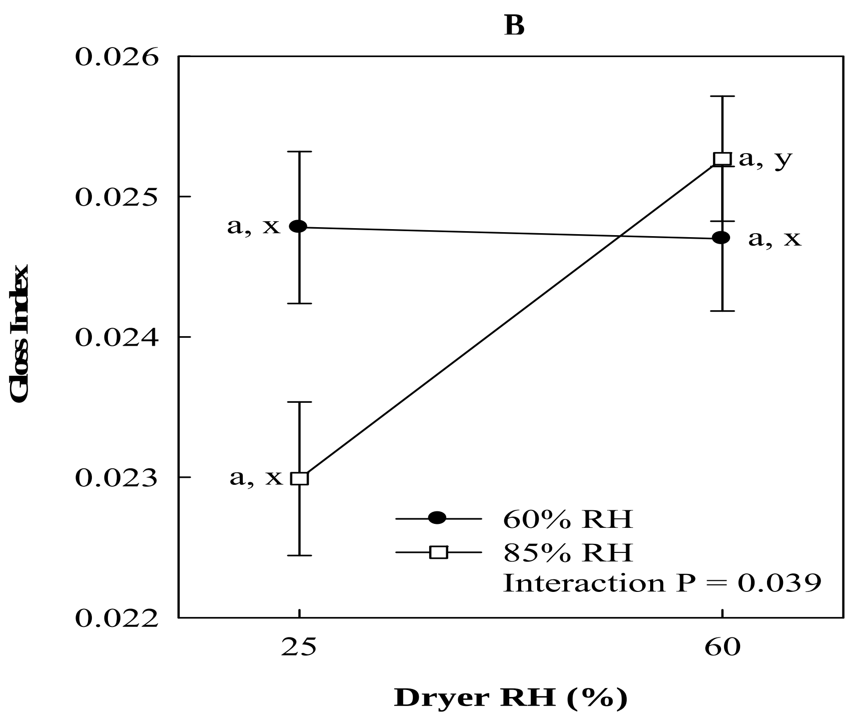
An interesting interaction of the higher RH (85%) in the wax applicator with the higher RH (60%) in the drying oven producing the higher gloss was observed (Figure 3.8B). In the 25% drying oven RH condition, fruit coated in the 60% wax applicator RH developed more gloss whereas it was those coated in 85% wax applicator RH that had the higher gloss in the 60% drying oven RH. Under the wax applicator humidity of 60% no significant differences were observed in the gloss level when "dried" in either 25 or 60% drying oven RH, but at the 85% wax applicator RH the fruit "dried" in 60% RH developed a significantly higher gloss compared to those "dried" in 25%. The moisture content in the higher humidity conditions of both the wax applicator and drying oven environments could have possibly acted as a plasticizer freeing the molecular chains and therefore increasing flow properties with the results of a coating coverage of greater surface area, leading to the recorded higher gloss. Gloss increased with an increase in drying time but it significantly decreased when the RH in the wax applicator was increased from 60% to 85% (Figure 3.8C). The longer duration in the "drying" oven made it possible for an effective curing of the coating for better gloss, and the lower gloss at 85% wax applicator RH could be explained by the effect of dew point and dilution of applied wax with condensation. The gloss level that evolved from the lower wax applicator temperature of 15 ^{O}C decreased with increasing drying time. For 35 ^{O}C wax applicator temperature, the gloss increased with increased duration in the drying oven (Figure 3.8D). The observed trends were however not significantly different. How the microstructure of the fruit peel during the wax application process impacts gloss levels is discussed in Quist et al. (2011).

Figure 3.8. Interactions of varied coating conditions of viscosity, temperature, RH, and time on gloss development (estimate ± SE; n = 414; Bonferroni's adjustment $p \leq 0.05$) prior to regular atmosphere refrigerated storage

Figure 3.8 (cont'd)

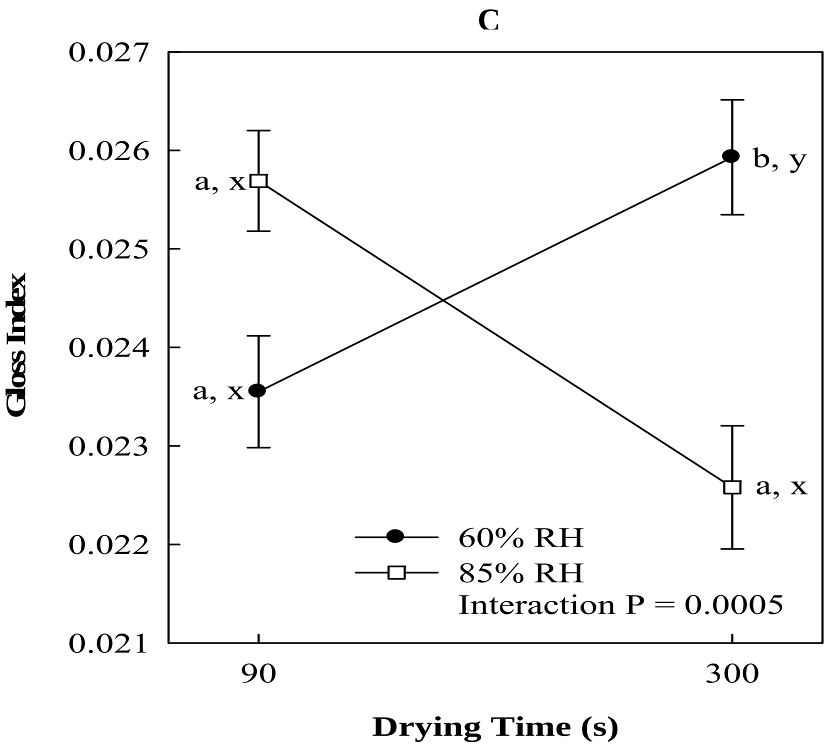


(a,b) Comparing Viscosity within each Dryer RH: Vertically comparison
(x,y) Comparing Dryer RH within each Viscosity: Horizontally comparison along each line

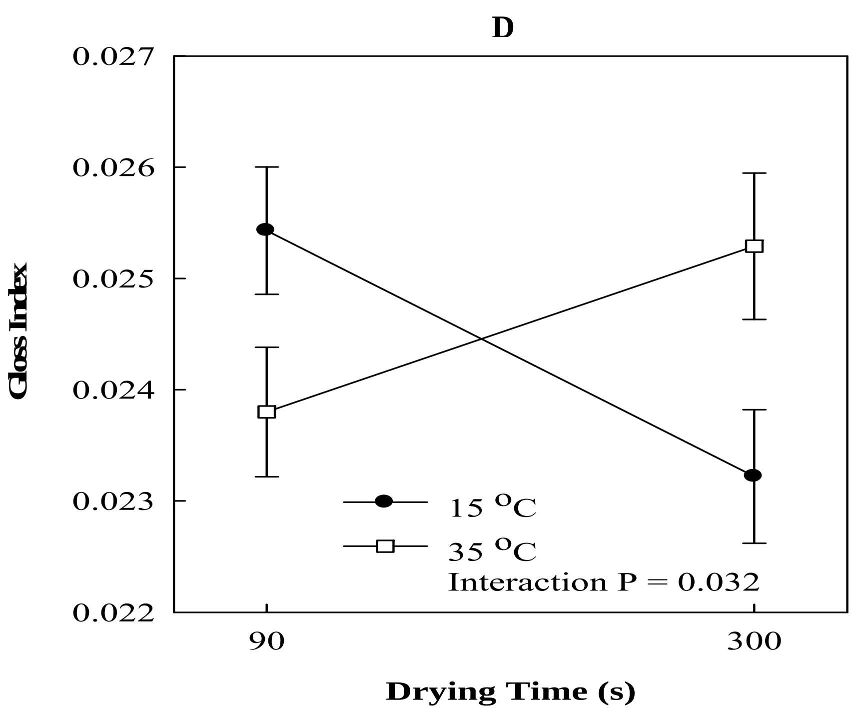


(a,b) Comparing Waxer RH within each Dryer RH: Vertically comparison
(x,y) Comparing Dryer RH within each Waxer RH: Horizontally comparison along each line

Figure 3.8 (cont'd)



(a,b) Comparing Waxer RH within each Drying Time: Vertically comparison
(x,y) Comparing Drying Time within each Waxer RH: Horizontally comparison along each line



3.4. Conclusion

Based on the factors used in this study a 40 ^{o}C water-alkaline fruit detergent solution in the DT for at least 60 s produced a significantly cleaner fruit surface. Rinse conditions of at least 30 psi with 40 ^{o}C water for at least 60 s in combination with the above contributed to ensuring a clean fruit surface. If alkaline fruit detergent is used on the washing brushes, then fruit should be rinsed thoroughly on exiting the brushes with the same rinse conditions as when exiting the dump tank.

This study successfully met the challenge of developing a non-destructive gloss device for the evaluation of gloss development on apples using a combination of different simulated packing line coating application processes. High gloss values were recorded on fruit that attained surface temperature above that of the dew point. Therefore we recommend dry and warm fruit surface for a good gloss. In addition, the utilization of coatings that exhibited good flow properties and provided more coverage on the fruit surface resulted in a good coating finish. These findings can be extrapolated to other apple varieties and regional packing houses.

Gloss could therefore be used as an indicator of the optimization of the packing line, and a non-destructive glossmeter will not only complement but facilitate the assessment and consequently the development of an optimal packing process.

3.5. Acknowledgements

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CHAPTER 4

The Effect of Wax Application and Storage on the Gloss and Quality of 'Red Delicious' Apples (*Malus domestica*)[1]

[1]To be submitted to the Journal of Agricultural and Food Chemistry

4.1. Introduction

Coating apples is used to improve appearance and quality; appearance through the impartation of gloss and quality through the reduction of water loss, delayed ripening and senescence (*1, 2*). The efficacy of the coating system is influenced by more than the type of fruit and coating. It is also dictated by the coating thickness and permeability, surface coverage, and environmental conditions such as temperature and humidity (*2, 3*). Critical to gloss development are environmental conditions and other wax application factors such as drying time and wax formulation which affect the thickness and uniformity of the coating layer. The coating layer reinforces the barrier properties of the peel influencing respiration and transpiration, thereby controlling weight loss and firmness retention during storage. It is therefore important to investigate how environmental conditions affect the waxing process and thereby influence quality which is key to the successful commercialization of produce. A recent study by Quist *et al.* (*4*) expounds on how these conditions affect the initial gloss quality but further studies are needed to learn how gloss and other quality attributes, weight and firmness, are affected by storage.

For red colored apples, the intensity and characteristics of the red skin promotes the perception of quality (*5*). Surface quality, characterized by gloss, is one measure of the quality of an object, and gloss is one of the most important properties considered when evaluating visual properties. Surface gloss is crucial to human perception of quality, and gloss measurements are routine in assessing quality. Gloss is therefore used to describe the appearance of a material and is basically the interaction of light with the surface as a function of specular reflectance (*6, 7*). Specular gloss is an important factor considered in the estimation of the smoothness or roughness of surfaces. A smooth surface appears glossy whereas a rough surface has a matt appearance as a result of reduced intensity of the specular reflection. Gloss therefore describes a surface's ability to reflect light in the specular direction. It's the superposition of the topography of the surface, the angle of the incident light and the refractive index of the surface that affect gloss, and hence influence the surface appearance (*6*). Unlike certain commodities, surface gloss adds value to apples (*8, 9*) by increasing both its consumer appeal and market value (*10*); especially for 'Red Delicious' apples which naturally have a low gloss compared to other varieties, and according to Bai *et al.* (*1*) is the reason that led the apple industry to apply shiny coatings to 'Red Delicious'.

However, the complete reliance on appearance to judge quality sacrifices critical attributes like firmness, color and aroma. Gloss, weight and firmness preservation are the quality attributes of interest in this study; these are important parameters in judging the quality and freshness of produce (*7*). Some studies have investigated and used surface gloss as a potential indicator to determine the quality of eggplants (*7, 11*) and bananas (*12*) but none so far is explicitly documented for apples. None to the author's knowledge has also investigated the potential effect of coating conditions on quality during storage of apples.

The objective of this research was to study the effect of gloss on the changes in weight and firmness during storage, as well as how these are impacted by waxing conditions. It also investigated the effect of the peel surface roughness and coating thickness on gloss intensity. The findings from this study enabled the establishment of a relationship between the quality attributes of gloss, weight and firmness per unit time with waxing conditions.

Figure 4.1 details the work plan for this study.

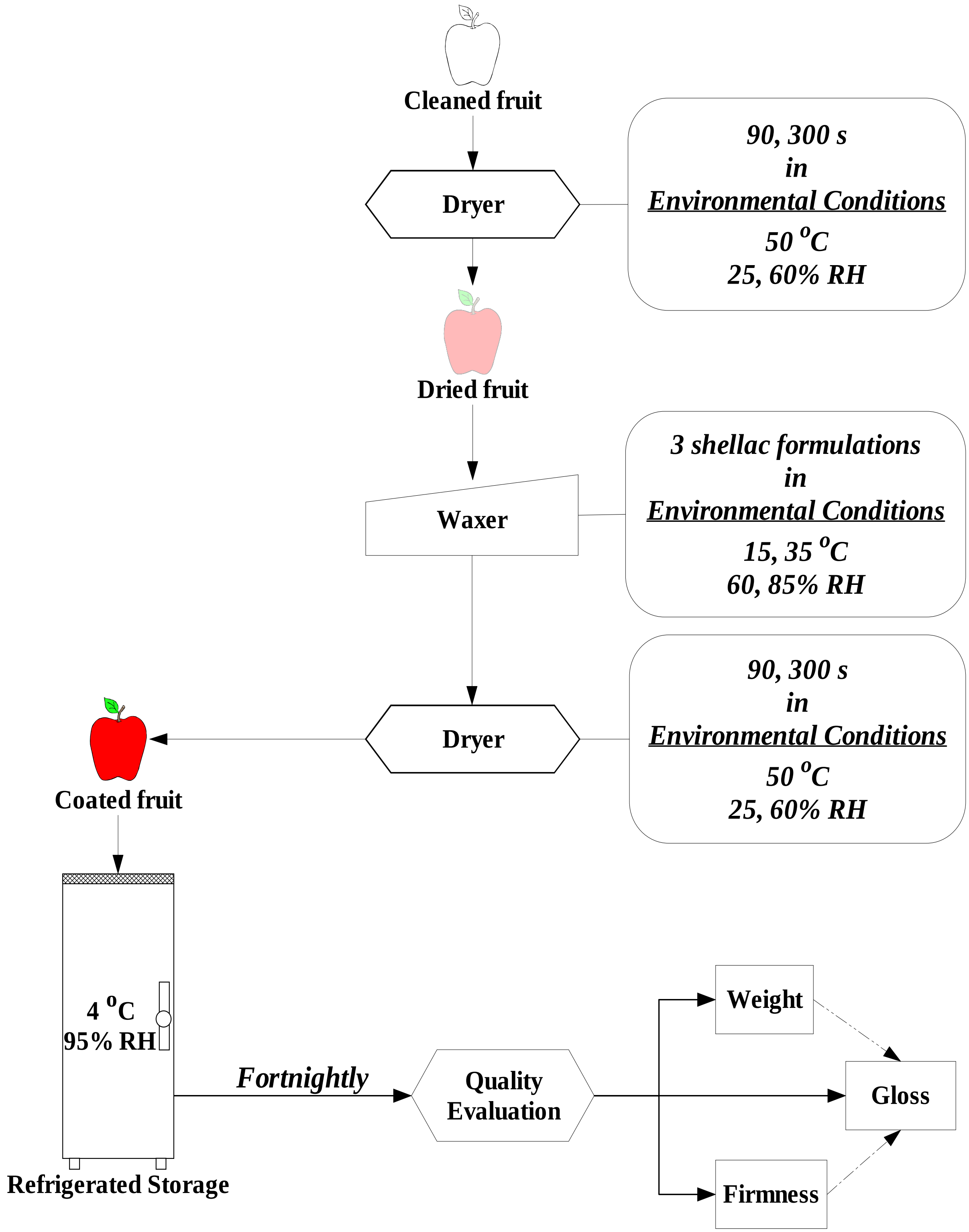


Figure 4.1. Flow diagram of the work plan

Drying in this study corresponds to the sections of the packing line where (1) the washed apple surfaces are dewatered by means of blowing fans and brushes on a conveyor belt prior to coating, and (2) coatings applied to the apples are dried and solidified through solvent evaporation by circulating heated air. However in Michigan drying is carried out at high RH due to the climatic conditions. Drying as a process is the removal of moisture or humidity, but the high humidity condition used in this study was unable to achieve a dry surface. Therefore the effect or result of the drying process on the packing line is denoted in quotation marks: "dried", "dryness".

4.2. Materials and Methods

4.2.1. The Packing Line and Wax Application Process[1]

'Red Chief Red Delicious' apples harvested in 2007 and 2008 were stored under controlled atmosphere (CA) at a commercial packing facility in Sparta, MI. In the later part of the last quarter of each year respectively, the fruit were transported to Michigan State University where they were portioned into storage units with continued storage under CA of 1 ^{o}C, 1.5% O_2 and 3% CO_2. Commercially available shellac fruit coatings (Shield-Brite AP-40, Fresh-Cote 214 and Premium Apple Lustr) with different volatile contents, isopropanol (≤ 20% to 12%) and morpholine (≤ 5% to 3%), were donated by Pace International, LLC. (Yakima, WA); FMC Technologies, Inc. (Lakeland, FL); and Cerexagri, Inc. (Monrovia, CA).

The set-up of the packing line used in this study, consisting of a washer and waxer component and an environmental chamber, is described in detail in Quist *et al.* (*4*). Cleaned and "dried" fruit were coated with shellac coatings of different spreadability in an environmental chamber of 15 or 35 ^{o}C and 60 or 85 % RH. A drying oven set at 50 ^{o}C was used to dry the coated fruit for 90 or 300s under 25 or 65 %. The temperature and RH were carefully monitored to facilitate the study of the effect of diverse environmental and process conditions on wax application and the resultant fruit quality as detailed in Quist *et al.* (*4*). These varied conditions for the remainder of the study will be referred to as treatments.

[1]*The mention of a product/company is for identification purposes only and does not imply an endorsement by the author.*

4.2.2. Microscopy Assessment of Fruit Peel Surface

To evaluate the surface of the coated and uncoated apples, different microscopy techniques, detailed below, were used. For a controlled investigation on the effect of fruit surface microstructure on gloss, varying amounts (0.5, 1.0 and 2.0 ml) of shellac coating were hand coated on uniformly sized fruits. These amounts in terms of gloss levels were designated low, medium and high respectively as preliminary studies showed the gloss value to increase with the amount of coating.

4.2.2.1. Atomic Force Microscope (AFM), and Scanning Electron Microscope (SEM)

Surface roughness of the apple peel was measured using a Nanoscope IV Multimode AFM (Veeco, Santa Barbara, CA). Exposed fruit flesh was dipped in paraffin wax to coat the tissue (and thus prevent water loss and shrinkage which affect the surface structure). The surface morphology was analyzed in contact mode using an NP20 probe obtained from Veeco. The scanning parameters were optimized at scan rates of about 3 Hz and integral and proportional gains of about 1 and 2 kHz respectively. The mean roughness, R_a, arithmetic average of deviations from the center plane, of four sub-samples were reported.

Cross sectional samples were freeze dried in an EMS750X Freeze Drier (Electron Microscopy Science; Hatfield, PA) prior to analysis on the SEM. The freeze dried samples were then mounted on aluminum stubs using high vacuum carbon tabs (SPI Supplies, West Chester, PA), and coated with Osmium (≈ 10 nm thickness) in an NEOC-AN osmium coater (Meiwa Shoji Co. Ltd, Osaka, Japan). Four samples per treatment were micrographed in a scanning electron microscope JEOL JSM - 6400V (Japan Electron Optics Laboratories, Tokyo, Japan) with a lanthanum hexaboride electron emitter, using analySIS Pro software (Version 3.2; Olympus Soft Imaging Solution Co., Munster, Germany), at an accelerating voltage of 10 kV.

4.2.3. Assessment of Fruit Quality

Fruit were stored in a temperature- and humidity-controlled chamber, at 4 ^{o}C and 95% RH, for two months, and quality assessments performed fortnightly. The quest to be able to non-destructively measure the gloss on the whole fruit over the duration of the study led to the development of a non-destructive prototype gloss device. Gloss was measured using the customized device described elsewhere (*4*); the same fruits were evaluated for the entire study, and the gloss average and standard error per storage time were reported.

Fruit firmness of the stored apples was measured using a texture analyzer (TA-XT2i, Texture Technologies Corp., Scarsdale, NY) with an 11 mm diameter probe. Three skin discs 20 mm equidistant from each other were removed from the fruit and the probe driven into the exposed flesh to a 9 mm depth. Three fruits per treatment per storage time were evaluated, and the average of 3 force values required to penetrate the preset depth was used as an index for fruit firmness per fruit. The average and standard error of the 3 replicates were reported.

Five randomly selected apples from each treatment were weighed from the start to the end of the study on an electronic laboratory precision balance with 0.01g sensitivity (Adventurer ARC120; Ohaus, Pine Brook, NJ). The results were expressed as percent weight loss of the initial weight, using the average and standard error.

4.2.4. Statistical Analyses

A fractional factorial design was used in studying the effect of environmental conditions on the efficiency of the waxing process; the design provided a 100% power for identifying differences. Data were analyzed using the Statistical Analysis System (SAS Version 8, SAS Institute, Cary, NC). Analysis of variance (ANOVA) at confidence interval of 95% ($\alpha = 0.05$) and multiple comparison adjustment for the means using Bonferroni's correction was used to analyze the effect of the waxing conditions on the quality attribute of gloss, weight loss and firmness; the main effects and their 2-way interactions were assessed.

4.3. Results and Discussion

4.3.1. The relationship between gloss, peel rugosity and coating thickness

To investigate the relationship between the appearance of the fruit in terms of its glossiness (or lack thereof) and the apple peel surface structure, a study measuring the surface roughness by AFM was conducted. It was observed, as expected, that the R_a values decreased (from 31.2 to 0.4 nm) as the amount of coating applied to a uniform surface area increased. This was a clear indication that a layer of coating led to a corresponding decrease in surface roughness. The shellac was able to fill in the valleys as a result of increasing lateral layer growth on the surface as more coating was applied (*13*). There was a considerable decrease in the surface roughness of the low gloss fruit (0.6 ± 0.1 nm) compared to the control (31.2 ± 2.4 nm), but as the coating was increased to the medium gloss fruit, the surface roughness increased slightly to 1.0 nm, changing the trend (Figure 4.2a, b and c); this according to Salvadori *et al.* (*13*) is the stage where the "tops of the hills grow faster than the valleys are filled". Other studies on membrane casts (*14*) have reported a similar trend of decrease in surface roughness with thickness growth to a certain limit after which it begins to increase.

Based on the observations of the surface roughness with the amount of coating, and preliminary studies that increasing the amount of coating increased the gloss index, investigations to study the relationship between the amount of coating, coating thickness and gloss were carried out. Microstructural analyses of the apple coated surface using the SEM showed the influence of the coating thickness on gloss. The amount of coating used did not only have a direct impact on the gloss level alone but also on the thickness, as expected, such that as the thickness of the coating increased the gloss index also increased (Table 4.1).

Table 4.1. Comparison of gloss index to coating thickness and surface roughness of unwaxed, and low, medium and high gloss apples

Gloss Level	Gloss Index	Thickness (nm)	Roughness, Ra (nm)
Control (unwaxed)	0*	-	31.2 ± 2.4
Low	4.1E-5 ± 2.1E-5	2242.6 ± 784.5	0.6 ± 0.1
Medium	2.5E-4 ± 0.5E-4	4663.9 ± 1264.3	1.0 ± 0.0
High	5.2E-4 ± 0.9E-4	8562.5 ± 911.5	0.4 ± 0.0

* The glossmeter scale was adjusted to zero for the control

It could therefore be inferred that as the surface got smoother with a corresponding increase in the coating thickness (Figures 4.2 and 4.3) the gloss level also increased (Figure 4.4).

Figure 4.2. AFM images of the apple surface with different amounts of coating
(A) Control, (B) 0.5 ml (low gloss), (C) 1.0 ml (medium gloss), and (D) 2.0 ml (high gloss) of coating
x 1.000 µm/div, z 20.000 µm/div except z 200.000µ/div for A.

Figure 4.2 (Cont'd)

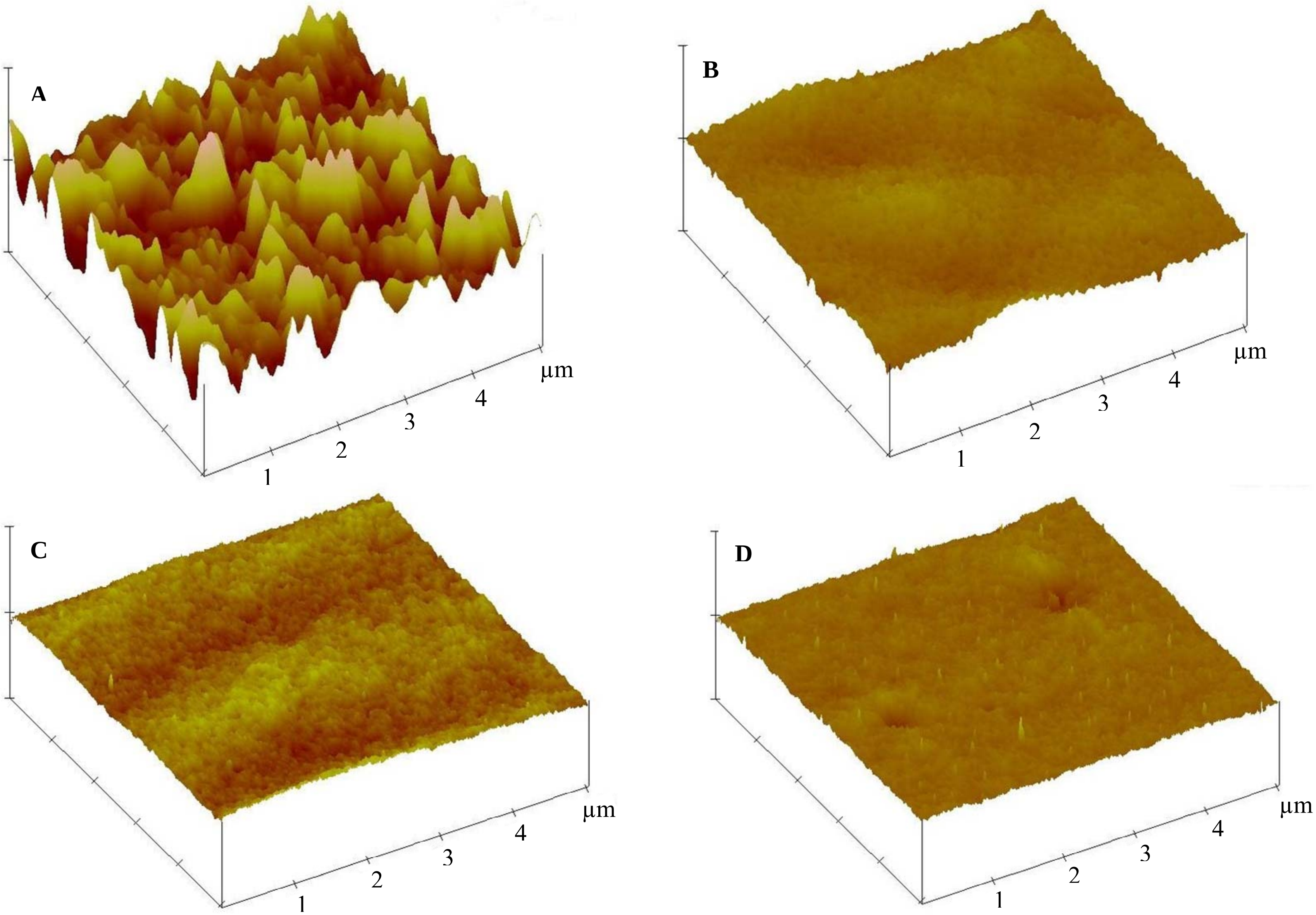
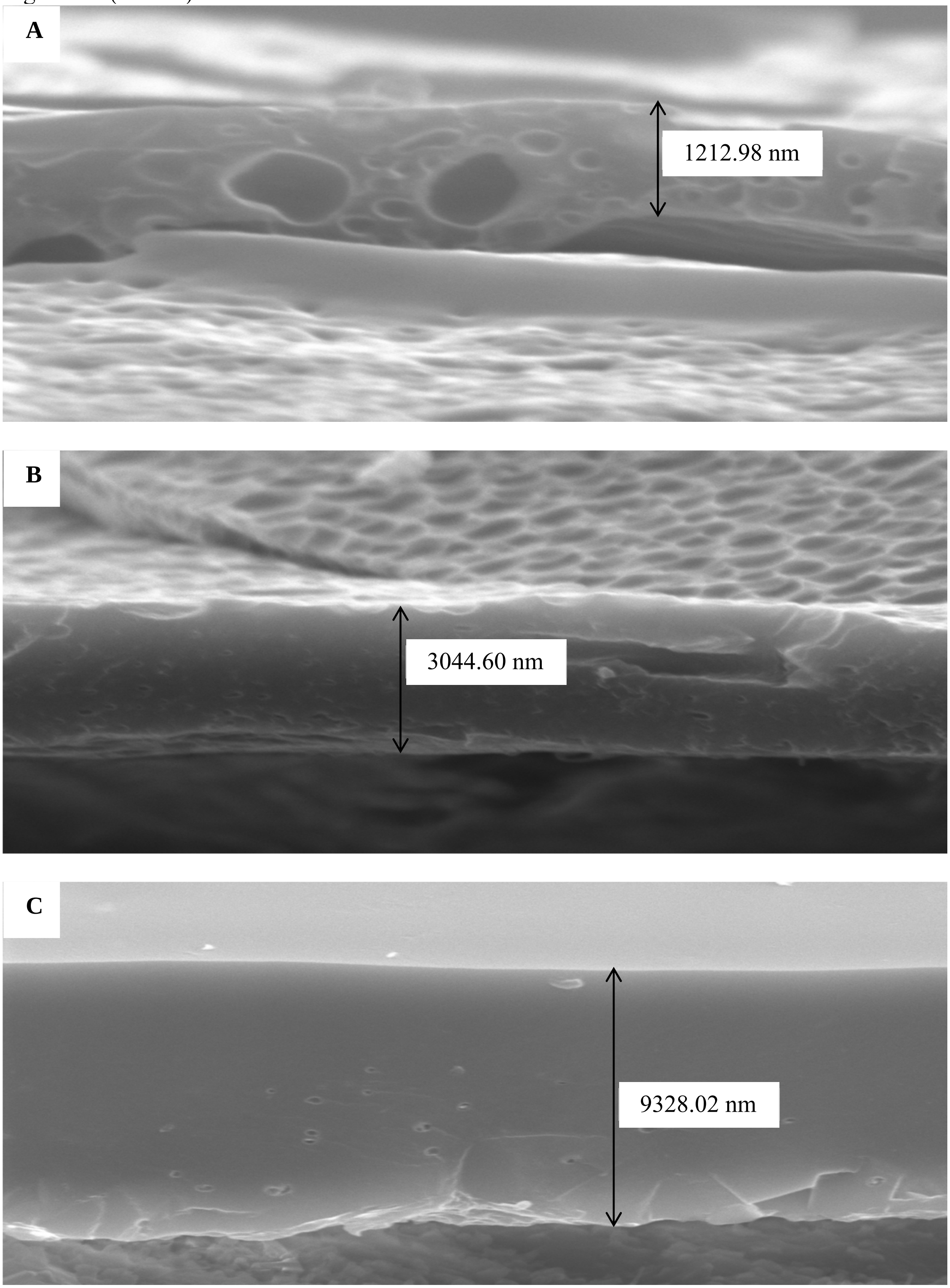


Figure 4.3. Scanning Electron Microscope micrographs of the apple surface with different amounts of coating

(A) 0.5 ml, (B) 1.0 ml, and (C) 2.0 ml of coating classified as low, medium and high gloss respectively

Figure 4.3 (Cont'd)



The nature of a rough surface induces light scattering which causes the irradiance distribution to be shorter and wider as the surface roughness increases. As a result not only is the intensity of the specular reflection weak but also all of the specularly reflected light may not be collected by the photodetector leading to a decreased gloss value as surface roughness increases (*6*). Thus as the valleys are filled with the evolution of a smoother surface, reflection is optimized.

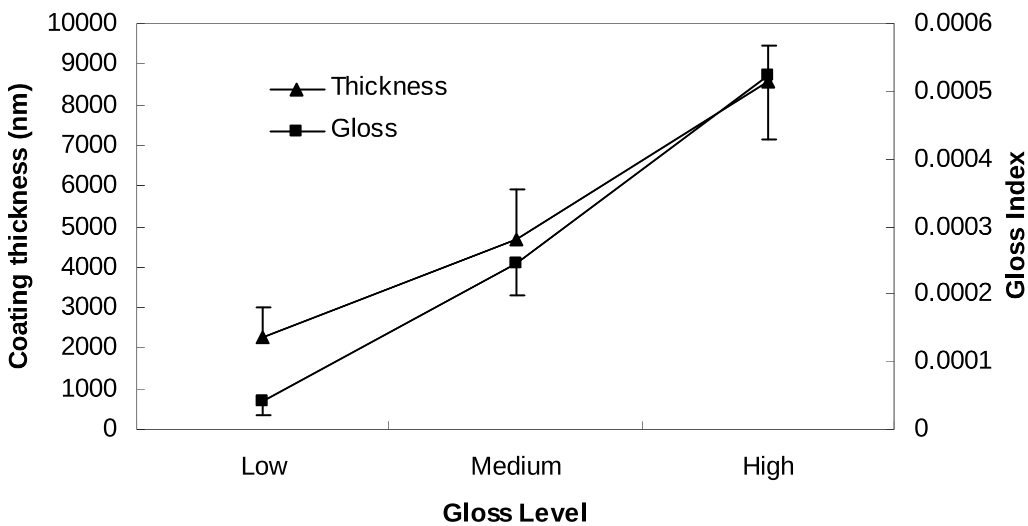


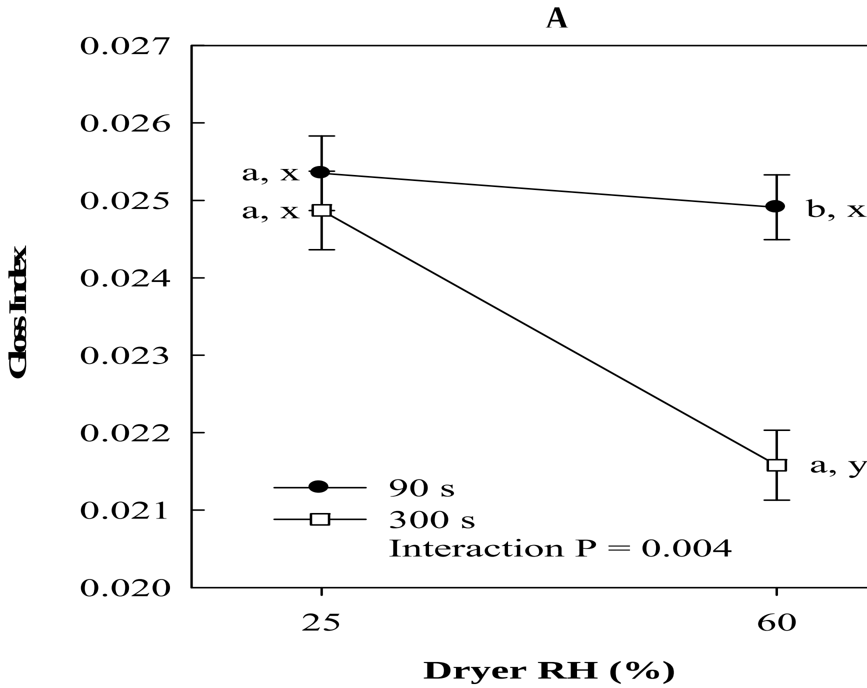
Figure 4.4. Relationship between coating thickness and gloss at low, medium and high gloss levels

4.3.2. The effects of waxing conditions and storage, at 4 ^{o}C in 95% RH, on gloss

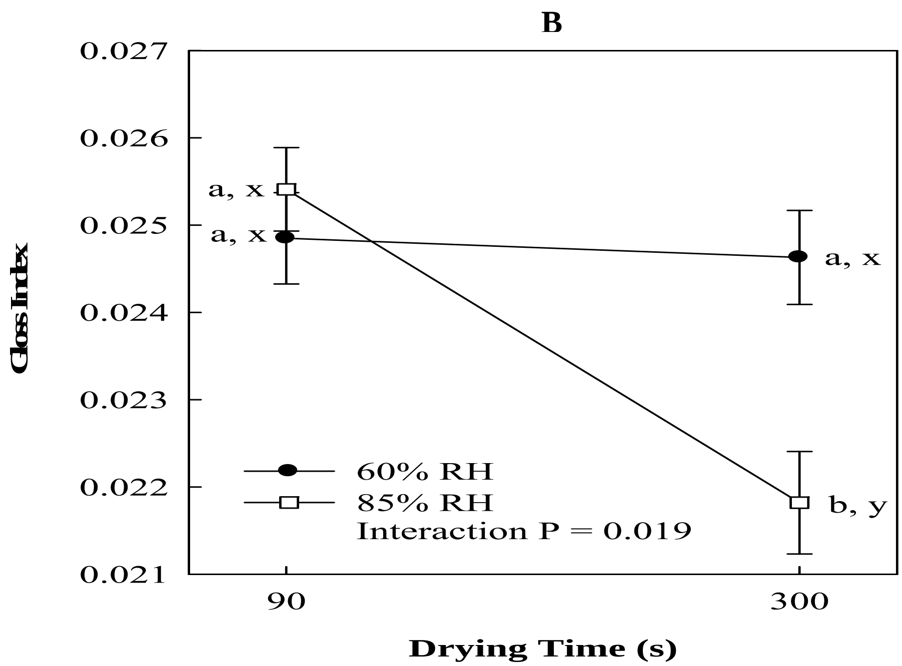
On storing the coated fruit at 4 ^{O}C in 95% RH, gloss was observed to decrease over time which is consistent with studies on bananas (*Musa acuminate*) (*12*), eggplants (*Solanum melongena*) (*11*), apples (*Malus domestica*) (*15*) and mandarins (*Citrus clementina* Hort. ex Tan. × *Citrus tangerine* Hort. ex Tan.) (*16*) among other commodities which also recorded the decline in peel gloss with storage. According to Jha et al. (*11*), gloss levels decline with storage as a result of the decrease in the quantity and structure of surface waxes as observed with bananas and eggplants. Damage of epidermal cells during storage contributes to an increase in surface roughness which translates as decreasing gloss with storage (*7, 11*).

The surface gloss during storage was significantly affected by the RH conditions in the wax applicator, drying time and RH in the drying oven, and obviously the time in storage. There was evidence of an interaction between each of the significant factors and storage time, indicating that the effect of each of these factors on gloss on apples varied across storage times. The effect of the RH in the drying oven during storage, independent of the other coating process factors, showed the fruit "dried" under the 25% RH to have a higher gloss than those under the 60% RH. This observation was similar to that reported by Quist *et al.* (*4*) 24 hours after coating, prior to refrigerated storage. Fruit "dried" under 25% RH achieved a surface temperature of 30.50 ^{O}C, which was above the dew point of 24.56 ^{O}C, while those from 60 % RH treatment with a surface temperature of 37.17 ^{O}C was below the dew point of 40.07 ^{O}C (*4*). The latter therefore had condensation and entered the wax applicator wet, as a result of the surface temperature being below that of the dew point. This meant a dilution of the applied coating and the reason for the lower gloss (*4*). A diluted coating will translate into a thinner layer on the apple, which has been shown in the SEM studies (figures 4.3 and 4.4) to have decreased gloss.

There was an interaction between the RH in the drying oven*drying time (Figure 4.5A) and RH in the wax applicator*drying time (Figure 4.5B). The effect of RH in the drying oven on gloss on apples depended on drying time such that 90 s of drying produced a significantly higher gloss compared to 300 s under 60% RH (Figure 4.5A). The significantly lower gloss at 300 s was because the relatively longer exposure aggravated the effect of condensation due to the surface



(a, b) - Comparing Drying Time within each Dryer RH: Vertically comparison
(x, y) - Comparing Dryer RH within each Drying Time: Horizontally comparison along each line



(a, b) - Comparing Waxer RH within each Drying Time: Vertically comparison
(x, y) - Comparing Drying Time within each Waxer RH: Horizontally comparison along each line

Figure 4.5. Interactions of coating conditions on gloss with storage at 4 ^{o}C in 95% RH (mean ± SE; n = 2031; Bonferroni's adjustment $p \leq 0.05$)

temperature being below the dew point. As a result there was pronounced removal of the applied coating (*4*) with the effect of increased surface roughness and a thinner coating layer, which culminate to reduced gloss. Under the 25% RH in the drying oven it was also the 90 s that developed the higher gloss but it was not significantly different from that developed at 300 s (Figure 4.5A) since the environment was already relatively dry and a prolonged drying did not influence the setting of the applied coating.

The RH in the wax applicator * drying time interaction is an indication that the moisture conditions in the applicator on the apple surface gloss depended on drying time. Fruit coated under 60% RH developed a significantly higher gloss compared to those coated under 85% RH when “dried” for 300 s. At 90 s of drying however, the fruit coated under 85% RH were those with the higher gloss but the gloss level was not significantly different from those coated under 60% RH (Figure 4.5B). Once again the effect of condensation with the consequence of wax removal and dilution, which sum up into the effect of a thin coating on a rough surface, is the reason for the significantly lower gloss observed under the 85% RH in the applicator with prolonged duration in the drying oven.

4.3.3. Quality characteristics of apples during storage at 4 ^{o}C in 95% RH

There was a direct relationship between decreasing attributes of gloss, weight and firmness (Figure 4.6). Numerous studies over the years have recorded significant decreases in weight loss with coating (*17-19*). According to Yaman *et al.* (*17*) the primary mechanism for moisture loss is the establishment of a vapor pressure gradient at different locations on the produce which causes vapor-phase diffusion. The permeability of the coating as well as its

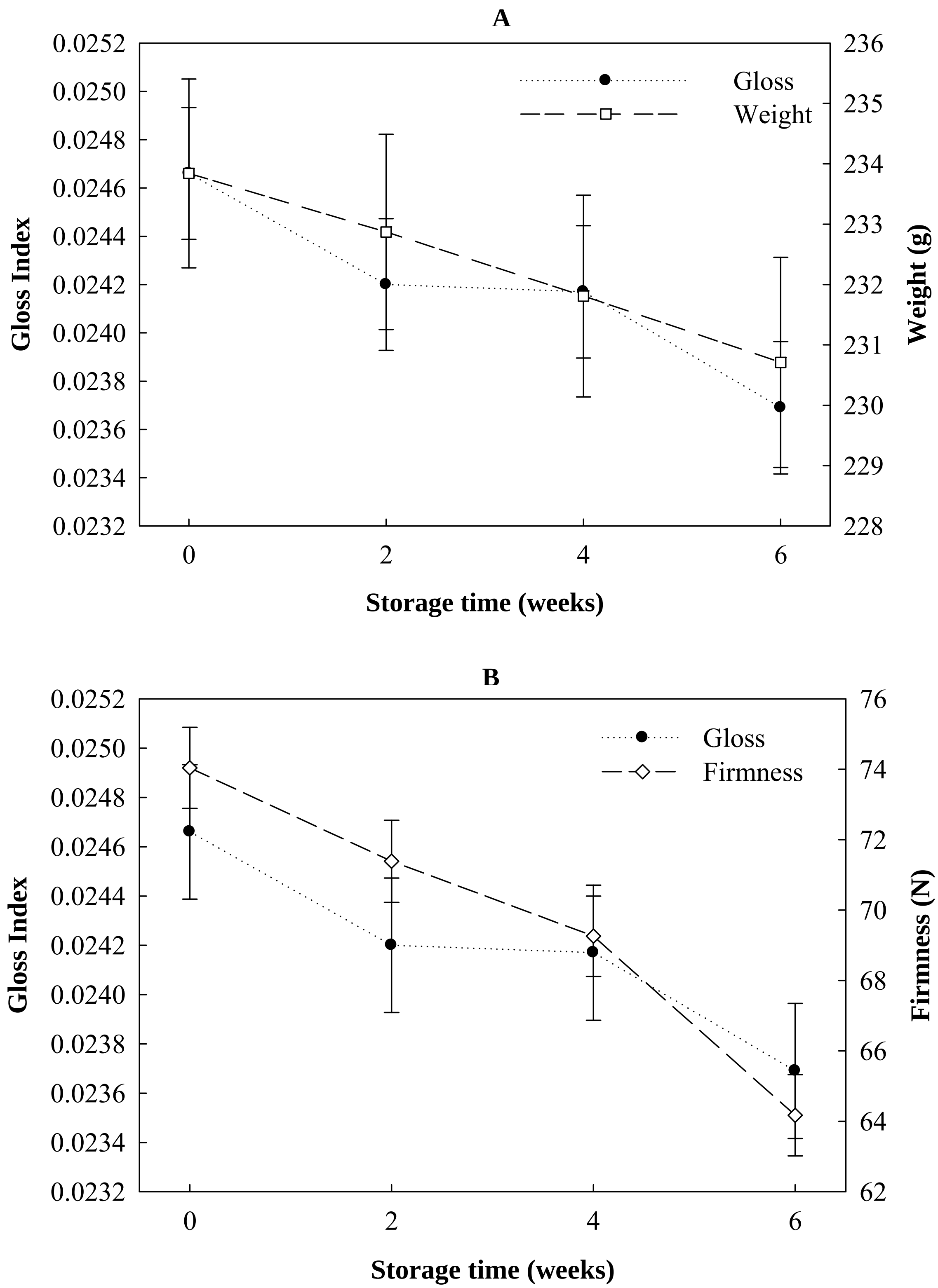


Figure 4.6. Relationship between gloss and (A) weight, and (B) firmness of fruit in 6 weeks of storage at 4 ^{O}C in 95% RH (mean ± SE)

thickness is important in determining how much moisture is lost; permeability is directly proportional to thickness. The temperature and RH also play a critical role as they affect the vapor pressure difference between the atmosphere and the fruit (*3, 17, 20*), hence the rate of transpiration with effects on weight loss and firmness preservation. However it is the nature of the surface of a commodity that has a major influence on the rate of evaporation of moisture (*3*). A commodity with larger pores and lenticels in the peel will have a greater surface area for moisture loss. Respiration also causes weight reduction in fruit as a result of the loss of a carbon atom from the fruit in each respiration cycle when CO_2 is produced and released, into the atmosphere, from an absorbed O_2 (*17, 20*). The treatments which exhibited longer gloss retention with storage were those that retained firmness with a minimal weight loss. Since weight loss in fruit is mostly a result of moisture loss, this observation can therefore be attributed to the fact that the coating treatments with greater gloss retention, by means of a thicker coating layer (Figure 4.4), maintained barrier properties that minimized the loss of moisture and hence controlled weight loss. This finding is similar to those reported by Rojas-Argudo *et al.* (*16*) in a study with mandarins and Jha *et al. (7, 11)* in a study with eggplants where a linear correlation between the decrease in gloss and weight during storage were observed.

The weight of the fruit decreased with time. Overall the percentage weight loss decreased by waxing the fruit, shown in figure 4.7. The coated fruit lost 1.79% in weight at the end of 8 weeks of storage compared to 5.11% in the uncoated control. During the first month of storage, fruit that were treated to lower humidity in the drying oven exhibited slightly more controlled weight loss. At 6 weeks of storage it was the fruit “dried” in the higher humidity conditions that showed a slightly more controlled loss in weight (Figure 4.7), bringing them to par by the second month of storage. The coating was effective in reducing weight loss such that the percentage weight loss of coated fruit at 8 weeks was similar to the weight loss of uncoated fruit at week 2. Coating decreased the weight loss by almost three-fold.

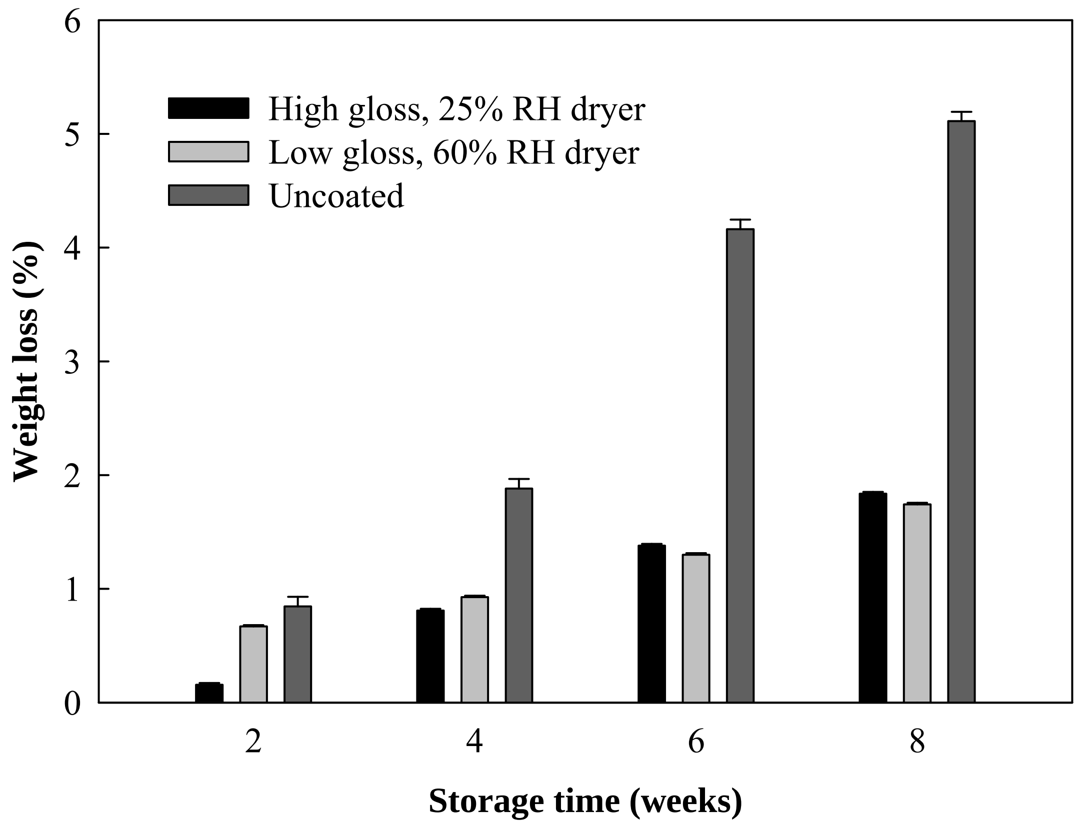


Figure 4.7. Weight loss in apples during storage (mean ± SE) at 4 ^{o}C in 95% RH for 8 weeks

The wax formulation, and RH conditions in the wax applicator had significant effects on the change in weight during storage. The higher viscose formulation (0.057 – 0.066 Pa.s) recorded a more pronounced loss in weight at the onset of storage but had evened out with that of the lesser viscose formulation (0.051 – 0.062 Pa.s) by the end of the study. The more viscose coating had reduced flow properties and spreadability. Visual observations showed a patchy and non-uniform coating coverage (*4*). With more surface area not uniformly covered, the apples coated with the more viscose formulation had a compromised barrier to moisture with more weight loss. However, during the prolonged storage in the high RH (94% in 4 ^{o}C), the coating integrity was reduced (*2*). The effectiveness of the less viscose coating barrier during storage is hypothesized to have been reduced to that of the more viscose formulation. The latter by having relatively more wax (thicker setting) in the areas covered (*4*) may not have been compromised at the same rate. This could be the reason for the similar weight loss for both formulations by the 8th week. Also, the deposit of a thicker layer supposedly protects the fruit from the rigors of temperature and humidity during application and storage. This effect is the function of an increase in the solid content of the coating which better covers the pores and fills in the cracks on the fruit peel (*16*). Coverage of the pores and filling in of the cracks will result in a decrease in surface roughness per unit area (Figure 4.2) which will enhance barrier to moisture loss, resulting in reduced weight loss.

Retention of firmness is another benefit derived from application of coatings to apples. Firmness decreased with storage for both uncoated and coated fruit, with the coated lot retaining firmness longer to almost three-fold that of the uncoated at the end of storage (Figure 4.8). Just as observed in the investigation on weight loss, loss of firmness was accompanied by loss in surface gloss (Figure 4.6B). Ward *et al.* (*12*) in a study on bananas, also found gloss to decrease with the ripening of the fruit. Loss of firmness is a result of fruit ripening (*21*) and retention of firmness is attributed to delayed degradation of insoluble protopectins to the more soluble pectic acid and pectin (*17*). The activity of pectinesterase and polygalactronase increase during ripening with an increase in the deploymerization of pectic substance (*18, 21, 22*) but during storage, the low levels of O_2 and high levels of CO_2 derived from coatings (*23*) impeded the activities of these enzymes resulting in the retention of firmness (*17*). Coating fruit introduces a barrier that inhibits water loss resulting in the maintenance of tugor pressure and the cell wall and middle lamella, all of which diminish with ripening (*18, 22, 24*). The type of coating used however plays a significant role in the maintenance of firmness. Shellac coated apples among other types of coatings used on apples were reported to have the least loss of flesh firmness (*15*).

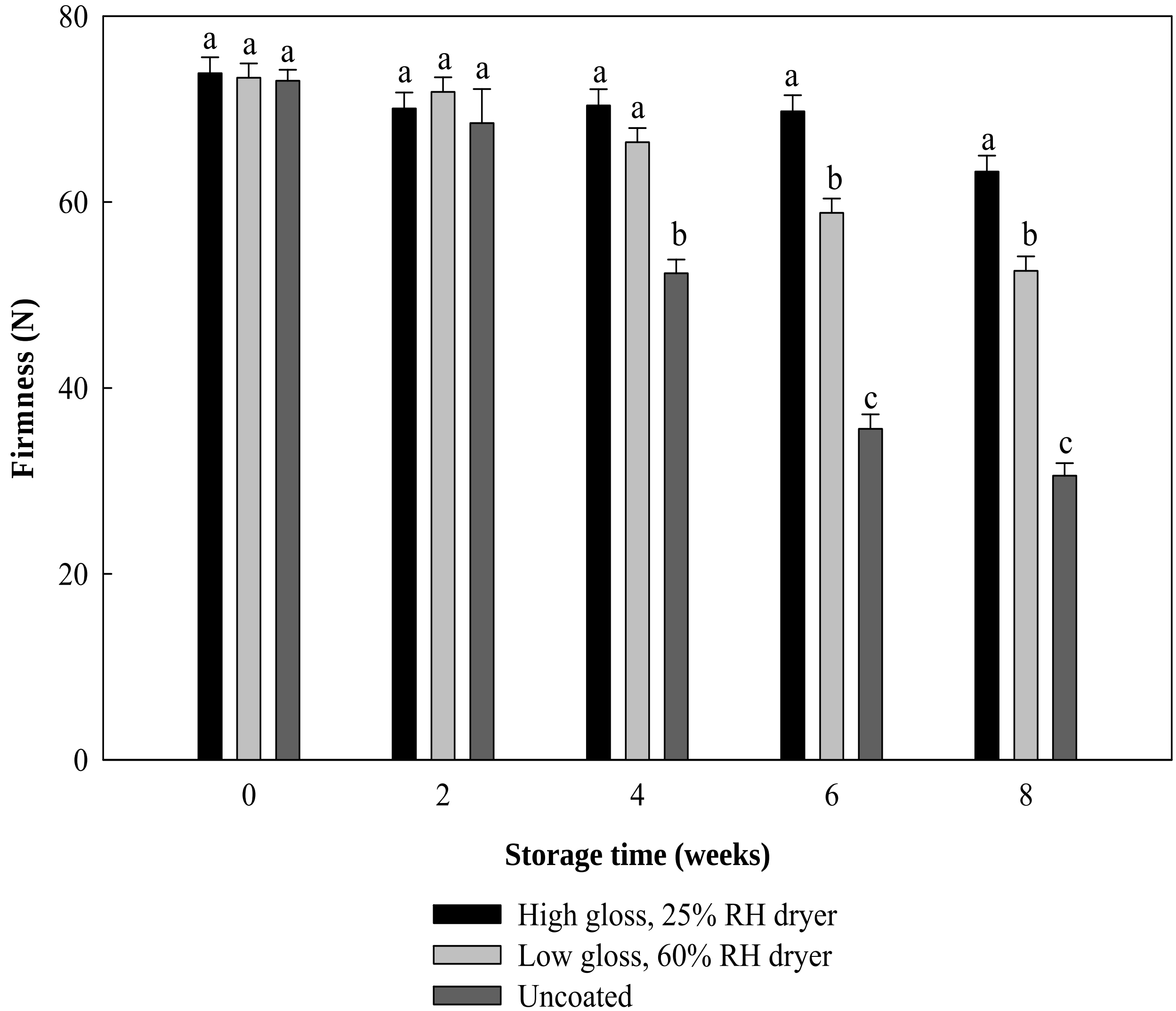


Figure 4.8. Loss in fruit firmness with storage at 4 ^{o}C in 95% RH for 8 weeks
Grouped columns with the same letter are not significantly different (mean ± SE; n = 684; p ≥ 0.05)

In a study on mangoes, a climacteric fruit exhibiting rapid ripening after harvest just like apples, the firmness of coated fruit was found to be significantly different from that of the uncoated (*25*). The researchers found that coated fruit required 7.0 – 5.3 N to compress a 2 mm distance compared to the significantly different 2.8 N need to compress the same distance for uncoated fruit. Yaman *et al.* (*17*) in a study on cherries (*Prunus avium*) found that fruit stored at cold temperature, 0 ^{o}C, had higher firmness than that stored at ambient temperature, 30 ^{o}C. The effect of cold temperature storage on the retention of firmness was reinforced by Meng *et al.* (*26*) study on peaches (*Prunus persica* L. cv. Jiubao) where they found firmness to decrease rapidly at higher storage temperature; fruit stored at 10 ^{o}C softened rapidly with a significant decrease in firmness compared to 5 ^{o}C storage with an effective maintenance of firmness. There is a significant increase in oxygen requirement for respiration at higher storage temperatures. As temperature increases there is an increase in metabolism accompanied by an increase in the respiratory demand and the utilization of stored carbohydrates that lead to loss of firmness (*27*).

Only the RH factors (wax applicator and drying oven) had significant effect on firmness. The effect of coating and RH in the dryer on firmness shown in figure 4.8 revealed that applying coating under low humidity conditions in the dryer slowed down the loss of firmness. The observation of the latter could be the effect of the comparatively more intact and even coating layer derived as a result of the "drier" environment. This reinforced the peel barrier properties which reduced the rate of water loss and culminated in an effective maintenance of firmness. High RH (94% – 98% in 1 ^{o}C), however, in prolonged storage reduces coating integrity with a loss in barrier properties (*2*) which causes an increase in the loss of weight and flesh firmness in fruit which many not have had pronounced differences at the beginning of storage (*16*). This could be the reason for the significant loss in firmness at week 6 of the fruit “dried” under 60% RH (Figure 4.8). Also if the fruit temperature is not near that of the air temperature a high RH will not prevent moisture loss (*3*), as the fruit will have to lose moisture till the vapor pressure around it is in equilibrium with that of the atmosphere.

Clearly, reduced gloss decay contributed to maintaining weight and preserving firmness during storage. A study on bananas (*12*) report an increase in peel roughness and a decrease in gloss with ripening. They attributed the increase in the roughness of the peel to the damage of epidermal cells which is partly the effect of moisture loss resulting in a shriveled surface. Since peel rugosity is directly proportional to the coating thickness, a rougher surface would have a thinner coating layer with reduced barrier properties which promotes more rapid water loss and respiratory exchange; the result of which is pronounced weight and firmness loss. It could also be hypothesized that at high RH the top layer of the coating is dissolved leaving the compromised valleys filled with moisture. The presence of moisture in the valleys would facilitate further dissolution of the remaining coating resulting in reduced barrier properties. The accompanying lower gloss levels, with the loss in weight and firmness, would be the result of the increasing optical scattering as a result of increasing surface roughness.

4.4. Conclusion

Fruit with higher gloss retention recorded the lowest weight loss and reduction in firmness which indicated the effect of coating and its barrier properties on enhancing and maintaining quality respectively. The gloss level and barrier properties are highly dependent on the nature of the fruit surface, and the thickness and uniformity of the coating layer. The smoother surface and thicker layer recorded the highest gloss value and in turn exhibited better quality retention. The gloss, weight and firmness decreased during storage, as the apples ripened, and this could be attributed to increasing surface roughness with a loss in coating thickness and barrier properties respectively. Monitoring RH in the dryer during the coating process is critical as it was observed to have significant effects on the loss in firmness. Low humidity settings in the drying oven would be recommended as its effect slowed down the loss of firmness during storage. The effects of the environmental conditions in the wax applicator, however, on fruit quality during storage were varied, showing neither a systematic decrease nor increase.

The influence of surface roughness and coating thickness on gloss can be successfully studied using the AFM and SEM and used as an indicator of fruit quality. Just as knowledge of the surface roughness and coating thickness can potentially help predict the quality by means of gloss levels, knowledge of the initial gloss at the onset of storage can also be potentially used as an index to predict the quality, via weight and firmness, at any given time during storage.

4.5. Acknowledgements

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CHAPTER 5

The Effect of Edible Coating Application Process and Storage Temperature on the Respiration and Quality of 'Red Delicious' Apples (*Malus domestica*), and the Transmission of Gases through the Coating[1]

[1]To be submitted to the Journal of Agricultural and Food Chemistry

5.1. Introduction

Edible coatings provide a barrier to respiratory gases and water vapor which slow down respiration, transpiration and related physiological responses and decay. However edible coating interaction with temperature and relative humidity is crucial to the maintenance of quality. The application of edible coatings also imparts gloss, which together with the barrier properties are highly dependent on the thickness and uniformity of the coating layer such that thicker and smoother coatings exhibit higher gloss and enhanced barriers (*1*).

By enrobing the fruit, edible coatings serves as a package providing the function of a packaging material such as modified atmosphere (MA) through changes in the permeability of its skin and its internal atmosphere. A MA relies on the modification of gases through the film by providing a low O_2 and a high CO_2 environment with the benefit of decreased respiratory rate, delayed compositional changes and slowed physiological deterioration. These benefits cumulate into slowing down ripening with its associated changes (*2*). In view of the internal atmosphere of fruit being different from that of their external atmosphere, fruit are said to be MA packages for their tissues (*3*). According to Banks *et al.* (4), it is the modification of the fruit's internal composition that achieves all MA effects. The internal composition is mainly affected by respiration and diffusion. While respiration tends to reduce the internal O_2 partial pressure, gas diffusion exerts the opposite effect. The change in the atmospheric composition is dictated by the permeability of gases through the coating, and the respiration rate and gas diffusion through the produce. The ability to reduce respiratory activity under MA conditions is a benefit only if the levels of O_2 and CO_2 are within that tolerated by the commodity of interest (*2*). Physiological benefits of MA accrue from a decline in processes linked to respiration and ethylene synthesis and action (4).

Disorders associated with low O_2 and high CO_2, as a result of the modification of the internal atmosphere, have been linked to edible coatings. Oxygen concentration dropping too low and/or CO_2 concentration rising too high will induce anaerobic respiration due to decreased O_2 uptake and its reduced concentration in the fruit's internal atmosphere (2, 5, 6). Gaseous exchange occurs through the pores and cuticle of the fruit peel. The application of surface coatings increase the resistance to gaseous exchange by covering the cuticle and pores of the skin which culminates in decreases in the transmission rates of gases between the internal and external atmospheres. Thus surface coatings, through these effects, have the tendency to modify the composition of the internal atmosphere, suppress the respiration rate and reduce transpiration in fruits (4).

Respiration, dependent on O_2 concentration and temperature, generally decreases in low levels of O_2, and increases with high temperature (7, 8). There is a two to three-fold increase in biological activity with every 10 ^{o}C rise in temperature within the temperature range encountered in the commercial chain (9, 10). Yearsley *et al.* (8) found the respiration rate of both 'Cox's Orange Pipping' and 'Braeburn' to increase with temperature increases from 0 – 24 ^{o}C. Poor temperature control can accelerate deterioration with the induction of anaerobic respiration (*2*). For normal respiration, O_2 and CO_2 must enter and escape respectively through the peel of the commodity (11) with the permeability of the peel influencing the transfer of respiratory gases. Permeability is influenced by temperature, relative humidity and air movement around the film package (*2*). Temperature dependence of film permeability and fruit respiration is determined by the film type and the physiology of the commodity respectively (7). The resistance of O_2 uptake by many produce is at the skin, and Beaudry *et al.* (7) discovered that the rate of O_2 consumption by the commodity increased more rapidly than the skin permeance to O_2 as temperature increased from 0 - 25 ^{o}C. Consequently O_2 levels in both the external and internal atmosphere decrease while that of CO_2 rises due to continued respiration (8).

Fruit of the same strain have shown variations in their internal gas concentrations (11); these variations are the results of differences in skin permeance, respiration rates, and in the fruit surface area to mass ratio (3, 12). But the temperature and the physiological stage of the commodity in relation to their climacteric will determine the magnitude of the differences (8, 12). In general the external atmosphere is more influenced by temperature than the internal. This is because the internal atmosphere is more dependent on the intercellular spaces and/or cell matrix, and the composition of the external atmosphere, rather than the skin permeance (8) which is dependent on surrounding environmental conditions. The skin permeance is also influenced by the coating integrity and its thickness. A uniform coating allows the diffusion of gases to the same degree on all areas of the peel whereas inconsistencies in the coating may introduce different gas diffusion properties in different sections of the commodity's peel. A thick coating layer will provide a greater barrier to the entry and exit of respiratory gases which translates to decreased normal respiration, and the opposite occurs for a thin coating layer.

The objective of this study was therefore to investigate the effect of barrier properties through coating application, and storage temperature on the respiration of 'Red Delicious' apples. It also aimed at establishing a relationship between gloss and the barrier properties and hence the effect of gloss on changes in respiration. The study, outlined in Figure 5.1, determined how temperature alters the respiration rate of both coated and uncoated ‘Red Delicious’. The transmission of respiratory gases through the coating were carried out at 2 levels of coating thickness and 3 levels of temperature to determine how these factors affect the diffusion of CO_2 and O_2 through the coating layer. The findings of this study enabled the evaluation of coating performance with storage, and the relationship between coating, permeation of respiratory gases, respiration rates, and weight loss, and how they affect and are affected by changes in gloss intensity.

Drying in this study corresponds to the sections of the packing line where (1) the washed apple surfaces are dewatered by means of blowing fans and brushes on a conveyor belt prior to coating, and (2) coatings applied to the apples are dried and solidified through solvent evaporation by circulating heated air. However in Michigan drying is carried out at high RH due to the climatic conditions. Drying as a process is the removal of moisture or humidity, but the high humidity condition used in this study was unable to achieve a dry surface. Therefore the effect or result of the drying process on the packing line is denoted in quotation marks: “dried”, “dryness”.

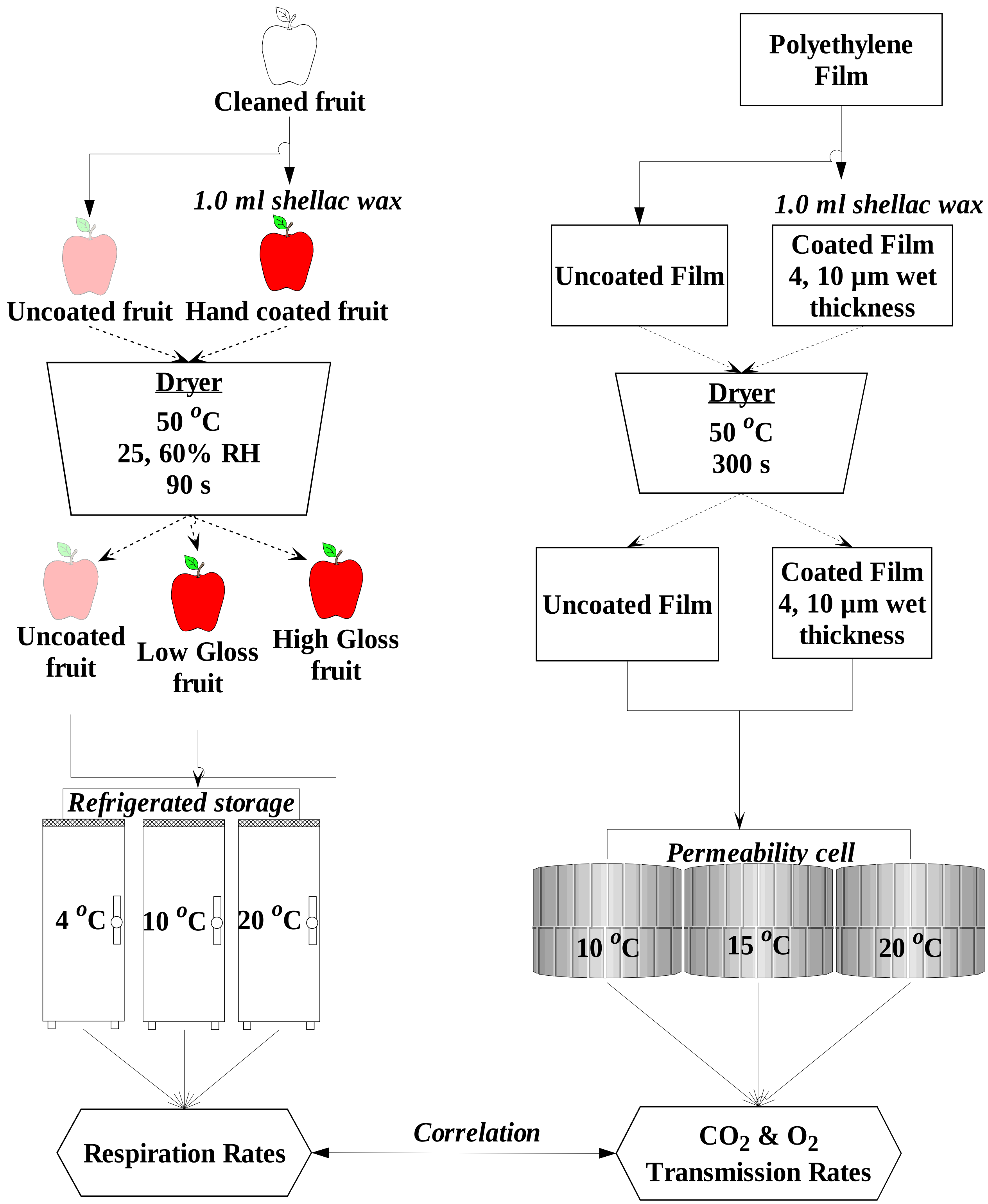

Figure 5.1. Flow diagram of the work plan

5.2. Materials and Methods

5.2.1. Surface Treatment and Coating of fruit[1]

'Red Chief Red Delicious' apples harvested in 2008, stored under controlled atmosphere (CA) at a commercial facility in Belding, MI, were transported to Michigan State University in May 2009 where they were portioned into storage units with continued storage under CA of 1 ^{o}C, 1.5% O_2 and 3% CO_2. Fruit from CA were equilibrated to room temperature overnight prior to surface treatment in preparation for coating and subsequent wax application. The surface treatment section of the packing line, consisting of a heated dump tank and heated pressure rinse, is described in Quist *et al.* (13). The fruit were cleaned, using commercially available alkaline detergent donated by Pace International LLC. (Yakima, WA), employing the optimum identified surface treatment described in Quist *et al.* (13). Using the method of Bai *et al.* (6) with modifications, 1.0 ml of commercially available shellac fruit coating, donated by Cerexagri, Inc. (Monrovia, CA), was evenly spread on the fruit by hand. The fruit were "dried" at 50 ^{o}C in 25% or 60% RH for 90 s prior to a month's storage. The control was uncoated fruit subjected to the same procedure.

[1]*The mention of a product/company is for identification purposes only and does not imply an endorsement by the author.*

5.2.2. Microscopy of Fruit Peel Surface - Scanning Electron Microscope (SEM), Atomic Force Microscope (AFM), and Digital Optical Microscope (DOM)

Shellac coating of varied amounts (0.5, 1.0 and 2.0 ml) were hand coated on uniformly sized apples, for a controlled investigation on the effect of coating thickness on gloss. Cross sectional samples were freeze dried and micrographed in a scanning electron microscope JEOL

JSM - 6400V (Japan Electron Optics Laboratories, Tokyo, Japan) with a lanthanum hexaboride electron emitter, using analySIS Pro software (Version 3.2; Olympus Soft Imaging Solution Co., Munster, Germany) as previously described in Quist *et al.* (*1*). Four samples per treatment were evaluated (*1*). A Keyence digital microscope (Keyence Corp., Woodcliff Lake, NJ), at 1000x magnification, was used to capture 3D images of the surface of each treatment for an evaluation of the surface structure.

5.2.3. Analysis of Respiration Rate and Fruit Quality

The flow-through system was used in determining the respiration rates. Coated and uncoated fruit were stored at 4, 10 and 20 ^{o}C in 1 L capacity glass jars and analyzed for respiratory gases for a month, using the experimental setup described by Beaudry (14). Two experimental units were setup in the refrigerated chambers. One set was used to study the changes in gloss and weight at the start and end of the respiration study, week 1 and 4, which will be referred to as 'Study I' in the results and discussion section. The second set, referred to as 'Study II', subjected to the same treatment and storage conditions, was used for weekly respiratory measurements. The jar lids were fitted with ports for atmospheric gas inlet and outlet to allow the attainment of steady state. A glass sampling septum was attached to the fruit, in the second experimental setup, using silicone sealant; this allowed sampling of internal gases. Three jars, each containing a weighed fruit, per coating treatment and experimental setup were placed in each refrigerated chamber.

Gas samples (100 µl) were withdrawn from the headspace and glass septum (external and internal) weekly with a 0.5 ml insulin plastic syringe and analyzed for CO_2 using an infrared gas analyzer (ADC-225-MK3, The Analytical Development Co. Ltd., Hoddesdon, England). The gas sample was injected into the gas analyzer which uses N_2 as the carrier gas at a flow rate of 200 ml/min, and connected to a chart recorder. Accumulated CO_2 from the headspace and core of the fruit were calculated relative to a standard (Matheson Gas Products Inc., Montgomeryville, PA) containing 4.85% CO_2 and 1.95% O_2. The respiration rate was modeled after (9):

$$R_{CO_2} = \frac{\left(y_{CO_2}^{out} - y_{CO_2}^{in}\right) \times F}{100 \times M}$$

Where R_{CO_2} = Respiration rate, CO_2 produced (mlCO_2 kg^{-1} h^{-1})

y = Volumetric concentration, %v/v

F = Flow rate (ml h^{-1})

M = Mass of the fruit (kg)

Two determinations were made per jar, and the average of three jar replicates was reported.

Methods described in Quist *et al.* (1, 13) using a laboratory precision balance and a non-destructive prototype glossmeter are what were used to measure weight and gloss respectively.

5.2.4. Evaluation of CO_2 and O_2 Transmission Rates

To determine the coating barrier, a polyethylene (PE) film of thickness of 50 µm was used as the carrier. One ml of fruit coating was uniformly deposited on the film to a wet thickness of 4 and 10 µm using a K-hand coater bar no. 0 (white) and US5 (grey) respectively (RK Print Coat Instruments Ltd., Litlington, UK). The film was dried at 50 ^oC for 5 min, and thickness measured at three positions around the film and in the center using a digital micrometer (Model CD-6"BS, Mitutoyo Corp., Japan) to verify the differences in the coating thickness. The

CO_2 and O_2 transmission rates (CO_2TR and O_2TR) through the coated and uncoated PE were evaluated at 10, 15 and 20 ^oC using ASTM D1434-82 (15), and the activation energy determined. The CO_2TR and O_2TR were measured using the Permatran-C 4/41 (Mocon, Minneapolis, MN) and the 8001 Oxygen Permeation Analyzer (Illinois Instruments, Inc., Johnsburg, IL) respectively. The tested film areas were 3.24×10^{-4} m^2 for CO_2TR and 5.0×10^{-3} m^2 for O_2TR. Uncoated film was used as control, and the average of four replicates was reported.

5.2.5. Statistical Analyses

A full factorial design was used to assess the effect of coating, peel transmission properties and storage temperature on respiration rates. Data was analyzed using the Statistical Analysis System (SAS Version 8) software (SAS Institute, Cary, NC). Analysis of variance (ANOVA) and Tukey's multiple comparison test for the means were used to determine significant differences in respiration rates, gloss and weight changes at the different temperatures which enabled a correlation to peel permeation.

5.3. Results and Discussion

An efficient application of fruit coating is dependent on the surface preparation. The surface preparation in this study was achieved by simulating a commercial line but the application of coating was conducted in a controlled environment to achieve a predetermined level of gloss. This allowed the investigation of the respiratory behavior of low vs. high gloss apples. Two experimental setups were used in this study. Setup one to evaluate changes in respiration, weight and gloss at the start and end of the study, to minimize the influence of human tampering with the storage jars. Setup two was solely for respiratory and internal CO_2 measurements. Both setups were stored at 4 ^oC, to simulate storage conditions prior to marketing, and at both 10 and 20 ^oC to simulate marketing conditions. Information on the transmission of gases, from the transmission rate studies, will provide additional insight into the changes in gloss, weight and respiratory behavior of the apples under marketing conditions.

5.3.1. The effect of coating thickness and gloss levels on respiration rate and quality

Previous studies on coating conditions and gloss showed that apples coated and "dried" in a humid environment produced lower gloss fruit. This was attributed to the effect of the interaction of attained surface temperature and dew point temperature with a pronounced effect of condensation which resulted in the dilution of the applied wax (13). Reduction in the amount of applied coating on the fruit also translated into a thinner coating layer with increased surface rugosity, all of which impacted gloss (*1*). Thus the study was started with the prior knowledge that the apples processed in the 25% RH would have a higher gloss and thicker coating layer than those in 60% RH which would have a lower gloss with thinner coating. The two levels of gloss will be referred to as low and high gloss respectively for the remainder of the study.

Study I

The first experimental setup focused on changes in respiration, gloss and weight. The gloss was significantly affected by the drying treatment (50 ^oC in 25% or 60% RH for 90 s) given to the apples and the duration of storage (1 and 4 weeks). Gloss was higher for the fruit "dried" under the 25% RH and both gloss levels were observed to decrease over time (Figure 5.2), confirming the observation of previous studies where the fruit were waxed in a conventional packing line (1, 13). Previous studies on gloss and coating thickness using the SEM, showed a thicker layer with a corresponding increase in gloss as the amount of applied coating was increased (*1*). A smooth surface appears glossy and light scattering is reduced by the smoothening of a rough surface. This results in a stronger and less dispersed specular reflection (16) and is the reason for the observed higher gloss when a higher amount of coating and smoother surface is present. The remarkable evolution of a thinner surface with decreasing amounts of coating applied, observed with SEM images (*1*) together with digital images of the surface of coated fruit (Figure 5.3), could account for the decreasing gloss intensity with storage. This observation infers that with storage, there was progressive thinning and evolution of a decline in the smoothness of the finish which resulted in the observed decreasing gloss with time.

With regards to the respiration rate and weight of the apples, it was also the drying treatment together with the storage temperature that showed significant effects. The rate of respiration was lower, just as the loss in weight was slower in the higher gloss fruit (Figure 5.4). Figure 5.5 and SEM micrographs from previous studies (*1*) show the progressive increase in coating thickness and gloss with the application of more coating. The higher gloss fruit by virtue of a thicker coating therefore reduced the rate of transpiration and respiratory exchange as a result of an enhanced barrier to gases, translating into reduced respiration rates and weight loss. Park *et al.* (17) in a study on sucrose polyester coatings on apples also observed the thicker coatings to have reduced respiration rates and delayed changes in quality as a result of higher gas barriers. Banks *et al.* (4) explain the resistance of the skin to the diffusion of gases to be dependent on what proportion of the pores are blocked by the coating and also the extent to which the coating adheres to the fruit surface especially along the edges of the pores.

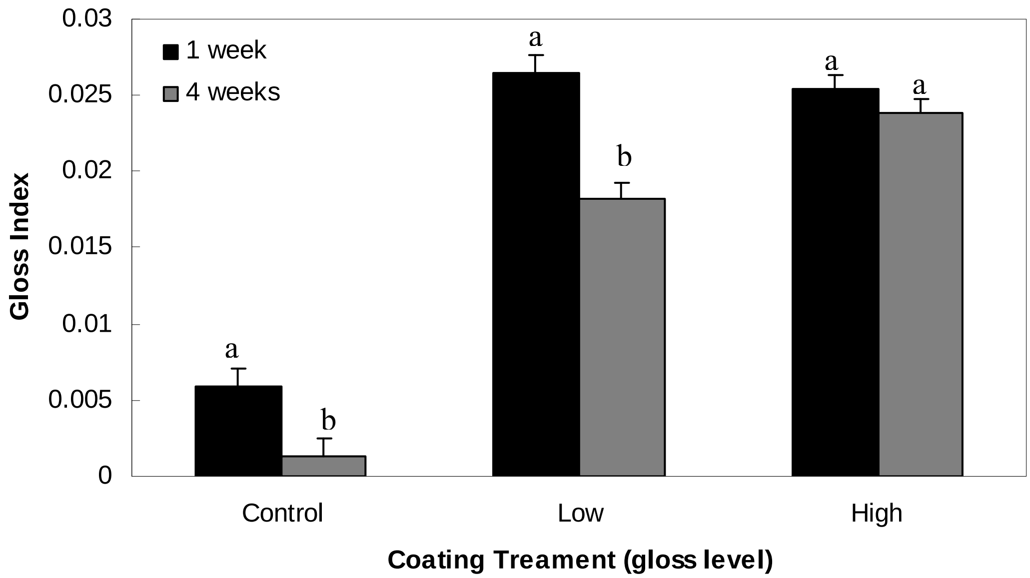

Figure 5.2. Changes in gloss of unwaxed, low and high glossed apples at 1 and 4 weeks of storage
Gloss level group columns with the same letter are not significantly different (mean ± SE; n = 72; $p \geq 0.05$)

Microstructural analyses of the apple coated surface using the SEM showed air spaces in the coating layer of the low gloss fruit. As more coating was applied, with a corresponding increase in gloss, the air bubbles decreased in size and progressed to a dense and thicker coating in the high gloss fruit (*1*). A loosely adhering coating offers the opportunity for gaseous exchange in the space between the fruit and the coating, whereas the opposite applies in the situation of a tightly adhering coating (4). Thus the air spaces in the low gloss fruit allowed more

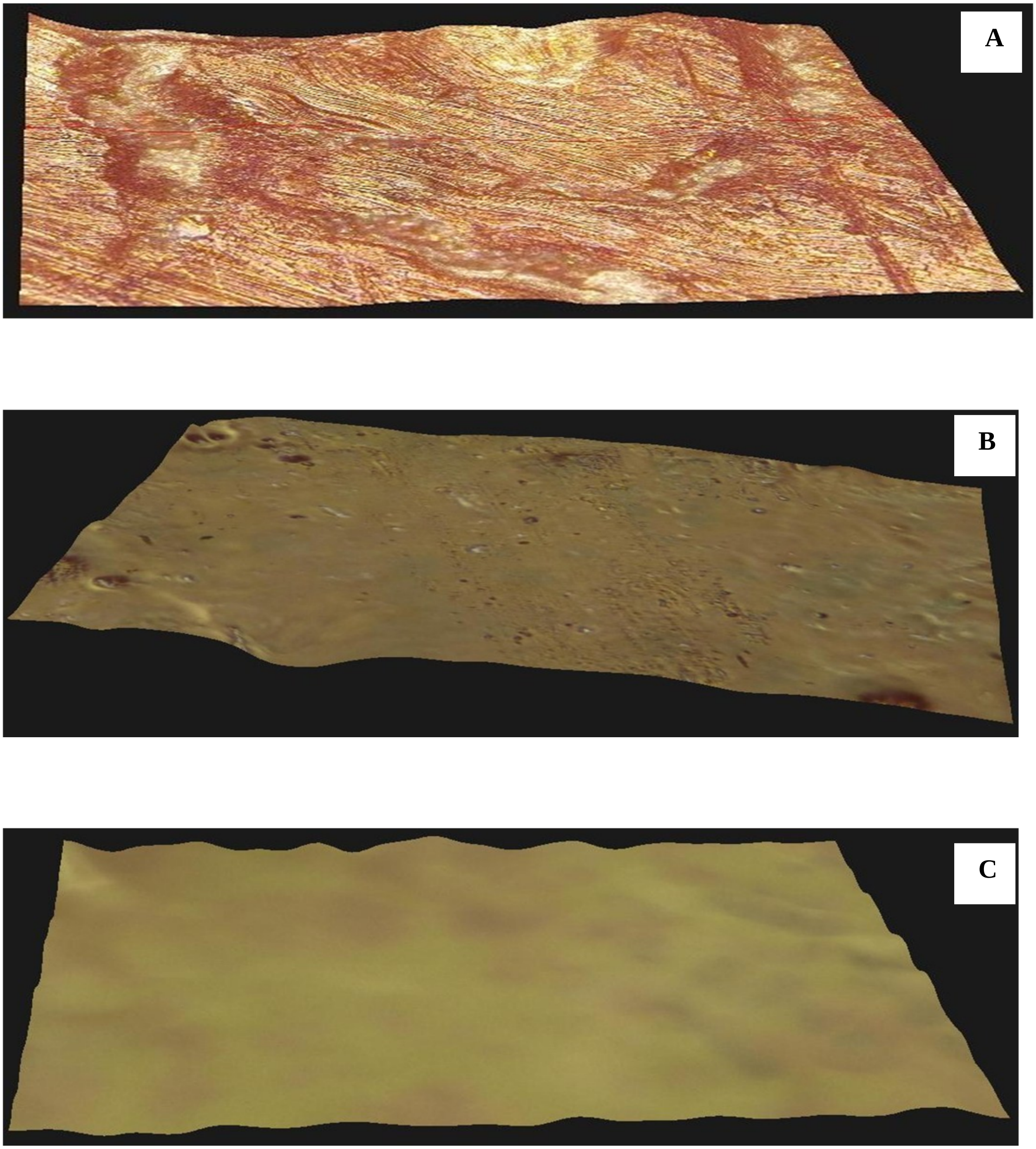

Figure 5.3. Digital optical images of the apple surface with different amounts of coating (A) Control, (B) 0.5 ml, low gloss, and (C) 2.0 ml, high gloss, of coating

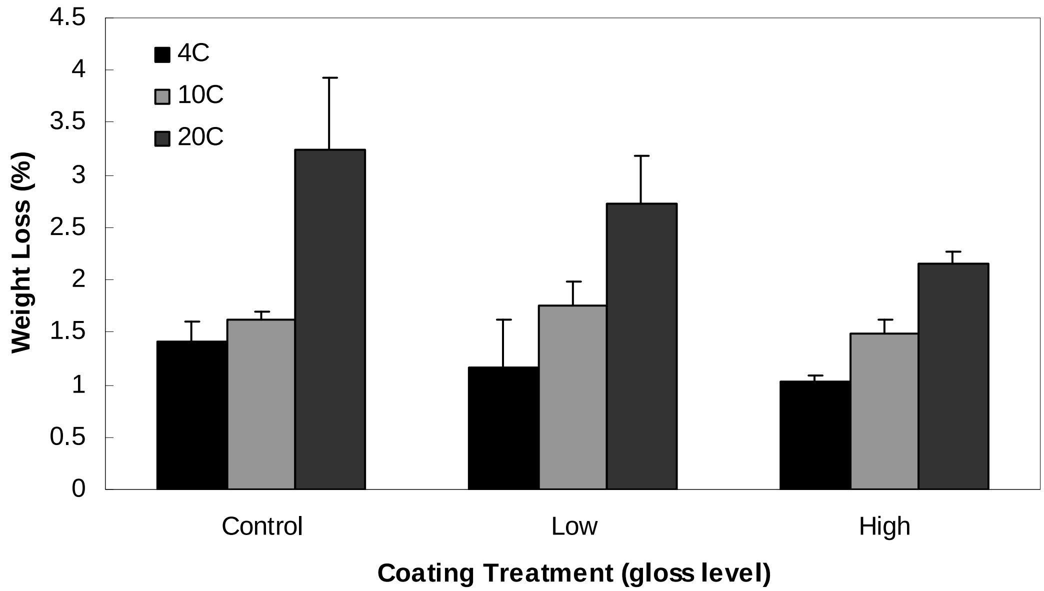

Figure 5.4. Weight loss in unwaxed, low and high gloss apples stored at 4, 10 and 20 ^{O}C (mean ± SE)

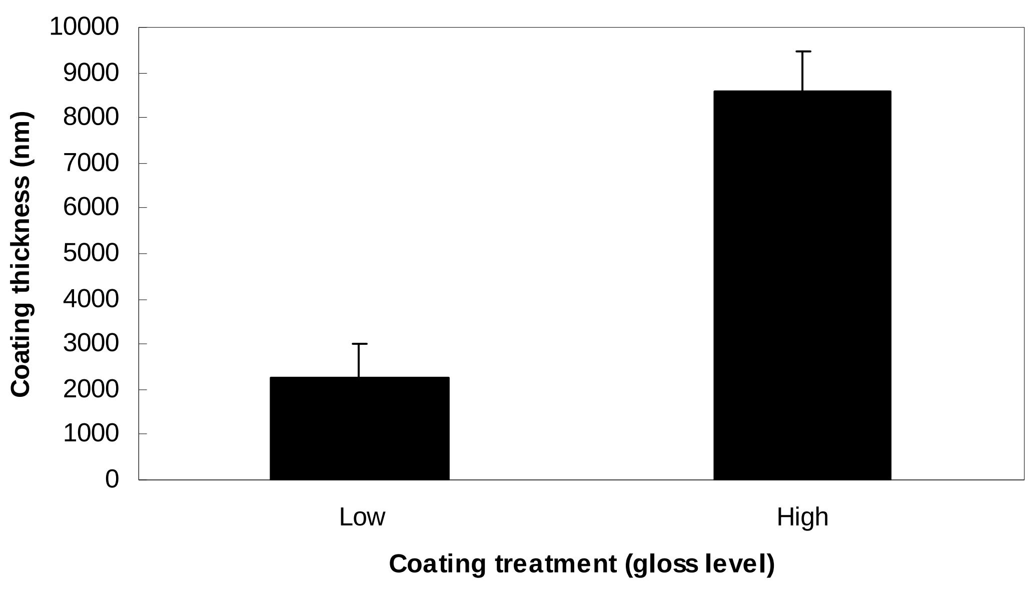

Figure 5.5. Thickness of shellac coating on low and high gloss apples (mean ± SD)
Data exported from SEM studies in Quist *et al.* (*1*)

gaseous exchange and give a reason for the lower respiration rate observed in the high gloss fruit with smaller and fewer air spaces in a denser and thicker coating layer.

Increase in the temperature of the refrigerated chamber resulted in increases in the rate of respiration and weight loss. Temperature is identified as a critical extrinsic factor influencing respiration; low temperatures are generally known to reduce biochemical reaction rates and vice versa (9). The association of weight loss with higher respiration rates could be due to the utilization of stored carbohydrates in the fruit tissues for respiration (8) thereby decreasing the amount of tissue composition which translates to weight loss. Increasing temperatures are associated with ripening, while ripening is associated with decreasing gloss and increasing peel roughness (18). Since the peel gloss is directly proportional to the coating thickness, low gloss fruit would have thinner coatings. This would translate into reduced barrier properties which promotes more rapid water loss and respiratory exchange resulting in a pronounced weight loss (*1*), as observed in the fruit stored at the higher refrigerator temperatures.

Study II

In the second experimental setup, all three factors – storage temperature (4, 10 and 20 ^{o}C), time (1, 2, 3 and 4 weeks), and gloss treatment (thin and thick coated) - under study significantly affected both the respiration rates and internal CO_2 concentrations in the fruit. The reason the duration of storage was significant in this setup and not the first could be due to variability in individual fruit response to the coating treatment and/or due to the opening of the jars for internal CO_2 sampling. In the case of the latter, this would result in infiltration of the surrounding's respiratory gases which would offset the conditions in the jar weekly.

Increases in storage temperature were once again shown to cause an increase in respiration rates (Table 5.1) as well as internal CO_2 concentrations (Figure 5.6). These observations are consistent with the findings of increasing respiration rates of apples as storage temperature was increased from 0 – 24 ^{o}C (8). The drying treatments, resulting in different levels of gloss, applied to the fruit were not shown to significantly affect respiration and this could again be due to variability in individual fruit. The variability in the fruit response to a coating treatment is due to inherent variability in the fruit skin resistance to gas diffusion and hence respiration rate. The variation in the proportion of blocked pores, by coating, also accounts for differing fruit response to coating treatments (4). There were observed differences, though not significantly different, in the uncoated and coated fruit (Table 5.1). The uncoated fruit recorded higher rates of respiration due to the lack of an enhanced barrier allowing a faster and unimpeded exchange of respiratory gases. Also as coating was applied, the valleys which may serve as a source of entry and exit for respiratory exchange were filled up, as observed from the optical images (Figure 5.3), and therefore reduced the gaseous exchange rate with a corresponding decrease in respiration rates. Overall, though differences were observed in the respiration rates of the coated and uncoated apple, the fact that the differences were not significantly different implies that the exchange of respiratory gases occurs predominately through the pores and not the cuticle, confirming the findings of Banks and his team (4).

The coated fruit recorded the higher concentrations of internal CO_2. At 4 ^{o}C storage, the internal CO_2 concentration among the coated samples were not significantly different as a result of the low physiological activity at low temperatures but as the storage temperature increased with an increase in respiration and metabolism, significant differences were progressively

Table 5.1. Respiration rate* of apples with varied coating thickness stored at 4, 10 and 20 ^{o}C for 4 weeks

Storage Time (Week)	Storage Temperature (^{o}C)	Respiration ($mgCO_2kg^{-1}h^{-1}$) Coating Treatment (gloss level) Control	Low	High
1	4	$0.47^{a,\sim}$	$0.03^{a,\sim}$	$0.30^{a,\sim}$
	10	$1.55^{b,\sim}$	$1.42^{b,\sim}$	$1.39^{b,\sim}$
	20	$2.67^{c,\sim}$	$2.13^{b,\sim,\wedge}$	$2.13^{c,\wedge}$
2	4	$0.16^{a,\sim}$	$-0.04^{a,\sim}$	$0.19^{a,\sim}$
	10	$1.51^{b,\sim}$	$1.36^{b,\sim}$	$1.28^{b,\sim}$
	20	$2.60^{c,\sim}$	$2.10^{b,\sim}$	$2.18^{c,\sim}$
3	4	$0.35^{a,\sim}$	$0.06^{a,\sim}$	$0.27^{a,\sim}$
	10	$1.48^{b,\sim}$	$1.40^{b,\sim}$	$1.33^{b,\sim}$
	20	$2.74^{c,\sim}$	$2.73^{c,\sim}$	$2.68^{c,\sim}$
4	4	$0.33^{a,\sim}$	$0.22^{a,\sim}$	$0.32^{a,\sim}$
	10	$1.62^{b,\sim}$	$1.55^{b,\sim}$	$1.43^{b,\sim}$
	20	$2.89^{c,\sim}$	$2.66^{c,\sim}$	$2.92^{c,\sim}$

*Residual for the respiration rate was not normal, so the data was log transformed

Numbers in the same category column, specific to a storage time with the same letter; and numbers in the same row, specific to a storage temperature and time, with the same symbol are not significantly different ($\alpha = 0.05$)

identified between the different levels of gloss. Though the coated fruit produced less CO_2 by virtue of the lower respiration rate, the presence of the coating, which enhances the peel barrier, reduced the rate at which gases escaped from the interior of the fruit. Hence, a more concentrated amount over time and higher in the high gloss fruit with a more dense and thicker layer of coating (Figure 5.6).

At the end of the study, the fruit were cut open and no CO_2 damage was observed.

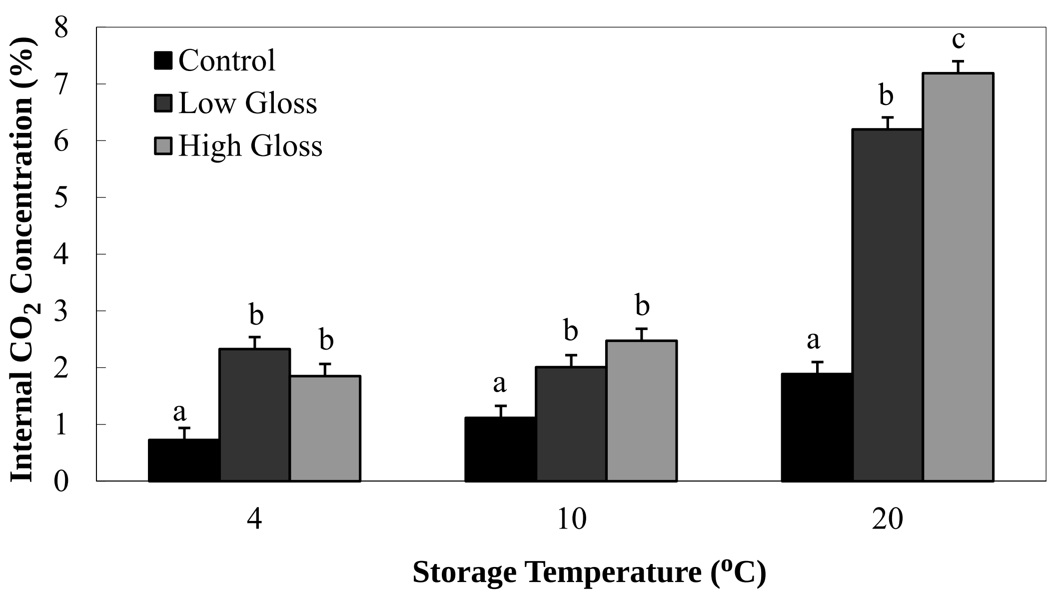

Figure 5.6. Internal CO_2 of coated apples stored at 4, 10 and 20 ^{o}C for 4 weeks
Group columns with the same letter are not significantly different (mean ± SE; n = 144; $p \geq 0.05$)

5.3.2. Respiratory gas transmission properties of the wax coating

Respiration is not only O_2 dependent but also influenced by peel permeation (11); the more available O_2 is, the higher the respiration rate. The coating on an apple dictates how O_2 and

CO_2 diffuse in and out of the fruit respectively. Understanding the permeation characteristics of the coating will provide an explanation for the influence of the fruit peel on respiration. For explicit analyses of the influence of the fruit coating and its thickness on the exchange of respiratory gases, shellac coating of 4 and 10 µm wet thicknesses were bar-coated onto a 50 µm PE film and dried. The dried films recorded thicknesses of 53.98 µm and 60.33 µm respectively. Both the CO_2TR and O_2TR through shellac, increased with increasing temperature. The CO_2TR was higher than that of O_2 for all testing parameters, thickness and temperature (data not shown).

At 10 ^{o}C, CO_2TR decreased from 667.1 ± 132 to 301.5 ± 20.3 mg/m^2.h for the 4 and 10 µm coated thickness respectively, compared to 123.5 ± 11.1 to 56.2 ± 1.2 mg/m^2.h for the O_2TR; a similar trend was observed for the other temperatures (Figure 5.7). The relatively low barrier of the coating to CO_2 was ideal for the release of CO_2, produced during respiration; such release prevented anaerobic respiration and the breakdown of the internal tissue (or formation of byproducts). The O_2TR indicated how much O_2 each coating thickness and consequently gloss level allowed to permeate its peel for respiration. Based on the transmission rate data, the high gloss fruit by having a thicker coating should have had a decreased gaseous exchange and a corresponding significant lower respiration rate. In the same vein, the low gloss apple should have had a higher respiration rate, and a rate significantly higher than that of the latter should have been observed for the uncoated. This once again goes to prove that the exchange of respiratory gases occurs mainly through the pores.

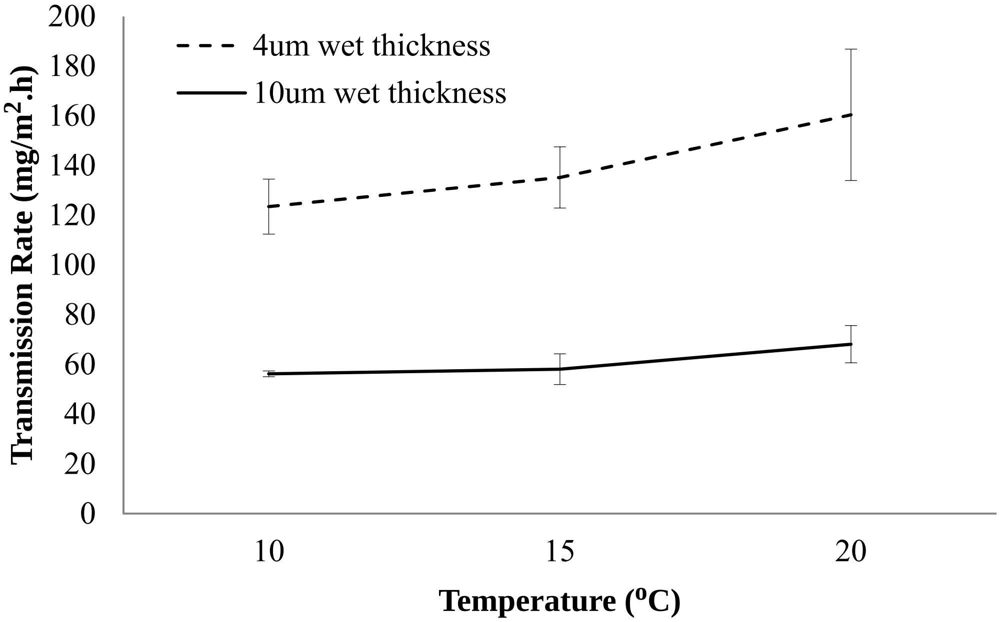

Figure 5.7. O_2 transmission rate through shellac coating of 4 and 10 μm wet thicknesses at 10, 15 and 20 ^{o}C (mean ± SD)

Temperature increases also resulted in increases in the transmission rate, as expected, in both of the shellac thicknesses evaluated (Figure 5.7). As the temperature was increased, the chains of the shellac polymer gained more energy which resulted in an increase in their segmental mobility. The mobility of the chains expanded the intercellular spaces allowing a greater amount of gases to be transmitted. However, as the thickness increased a thicker barrier over the intercellular spaces was created which was why the 10 μm coating recorded less significant transmission rates than the 4 μm (Figure 5.7). The observation of the effect of temperature on changes in respiration rates (Figure 5.4 and Table 5.1) is concurrent with the observation in the changes in O_2TR with temperature (Figure 5.7); the lower storage temperature recorded lower respiration rates as well as reduced gaseous transmission rates. A reduction in O_2 to less than 10% (by volume) is what is required to achieve a reduction in respiration.

However this reduction is temperature dependent such that when temperature is lowered the oxygen requirement is reduced (7, 9, 19). Hence as more O_2 permeates the peel with temperature increases, the respiration rate is higher with the availability of more O_2, and a response in the increased production of CO_2.

The activation energy, E_a, of O_2 transmission rate were 17.5 ± 5.2 and 17.4 ± 2.8 kJ/mol for the 4 and 10 μm coated thickness respectively, and 38.6 ± 6.9 and 33.3± 5.7 kJ/mol respectively for the CO_2 transmission rate. The E_a was greater for CO_2, compared to that of O_2, indicating that the CO_2TR underwent the most change with temperature increases which is evident from the data (Figure 5.7).

5.4. Conclusion

Reduced gloss occurred when coated apples were “dried” under high humidity (25% compared to 60% RH). Also gloss was observed to decrease over time, as a result of changes in peel roughness (18), and confirmed earlier studies (1, 13). Coating the apples reduced weight loss. This was more pronounced in the higher gloss fruit, with an enhanced thicker barrier to minimize the loss of moisture. Higher storage temperatures increased the O_2 demand for respiration (7) which was evident in the increased O_2TR with higher temperatures and increased respiration rates by means of CO_2 production. Respiration rates were observed to be lower for coated fruit and much lower for the higher gloss fruit. Therefore the reinforced barrier reduced the permeation of respiratory gases.

The gloss intensity, as influenced by the thickness of the coating layer and the roughness of the apple surface, could be potentially used as an indicator of the permeation of respiratory gases. Therefore gloss levels could be used as a potential index to predict respiration rates and changes in weight during storage.

5.5. Acknowledgements

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CHAPTER 6

SUMMARY, CONCLUSIONS & RECOMMENDATIONS FOR FUTURE STUDIES

Commercial cleaning practices were evaluated in an attempt to identify cleaning factors that produced significantly cleaner apple surfaces, as a preparatory step in conditioning the fruits for further studies and analyses. A 40 ^{o}C water-alkaline fruit detergent solution in the DT for at least 60 s, together with a 40 ^{o}C – 30 psi rinse for at least 60 s produced significantly cleaner fruit surfaces. The section for the utilization of the alkaline fruit detergent can be either the dump tank or the washing brushes. In the case where the detergent is used on the washing brushes, the fruits should be rinsed thoroughly with the same rinse conditions as when exiting the dump tank.

Simulating a combination of commercial packing line coating application processes, different gloss intensities evolved that were successfully measured on a customized non-destructive glossmeter. Higher gloss intensities were observed not only with apples that were “dried” under low humidity but with those that obtained a surface temperature above the dew point, and were “dry” and warm prior to the application of the fruit coating. The less viscose coatings with good flow properties were also observed to give a better coverage on the fruits, and recorded higher gloss values.

Throughout the 8weeks storage study, the fruits with the higher gloss values recorded the lowest reduction in firmness and weight loss as a result of the function of the barrier properties of the coating. The uniformity and thickness of the coatings were also observed to influence the gloss and quality attributes. The smoother and thicker coatings recorded higher gloss values with greater firmness and weight retention. Respiration rates were also observed to be lower for coated fruits and much lower for the higher glossed fruit, as a result of the reinforced barrier of the thicker (higher gloss) coating minimizing the permeation of respiratory gases.

As the apples continued to ripen during storage, the gloss, weight and firmness were observed to decrease as expected. These changes were accompanied by decreases in coating thickness and increases in the surface roughness, as evaluated with the SEM and AFM respectively. Storage in high temperature environments increased the respiration rates, by means of CO_2 production, since the demand for O_2 increases with temperature. The changes in gloss with its associated changes in weight, firmness and respiration, were attributed to the changes in peel coating roughness and thickness which culminated a corresponding decline in the coating's barrier properties.

Knowledge of the initial gloss at the onset of storage can be potentially used as an index to predict the fruit quality, via weight, firmness and respiration, at any given time during storage. In that vein, gloss could therefore be used as an indicator of the optimization of the packing line and processes. Complementing the packing line with a non-destructive glossmeter will facilitate the development and assessment of an optimal packing process. Information on the surface roughness and coating thickness, using the AFM and SEM respectively, can also be potentially used as an indicator of fruit quality by means of gloss levels and its association with respiratory gases transmission rates, and changes in the quality attributes of interest in this study. Thus while visual perception is key to judging the quality of produce, scientifically the microstructure of the surface can also be used as a tool to judge and predict quality.

Recommendations for improving the packing line based on the findings from the pilot and scale-up studies are outlined in the report submitted to the Michigan Apple Committee (Appendix A). A good suggestion for future studies would be to expand the study on the environmental conditions used to evaluate the wax application process and ensuing gloss. This would involve testing different wax formulations to identify which gives the highest gloss intensity for specific sets of temperature and relative humidity. In doing so, a range of environmental conditions that influence gloss to similar but insignificant extents could be identified, allowing the formulation of wax coatings for specific range of conditions. It would be very good for apple packing houses to have coatings formulated for use in high RH regions such as that of Michigan and New York. This way, packing houses can easily change the waxes on their packing lines as the seasons change with their accompanying changes in temperature and relative humidity. A study on the turbidity of the water in the dump tank and how it affects gloss should be conducted. This would make possible the determination of the turbidity at which the dump tank water interferes with the deposition of the coating and therefore with the development of gloss. The acquisition of such knowledge will facilitate the establishment of a point at which the dump tank water has to be changed during processing.

It would also be ideal to incorporate a glossmeter as a non-destructive testing unit in the packing line. This would minimize errors and losses as fruits will not have to be moved from one unit to the other before evaluating the efficiency of the system in producing the desired gloss. It would ensure efficiency of the system by possibly shutting down the line automatically or issuing a sound signal when the gloss value is below the set point. This would provide an on-going evaluation on the line and therefore allow adjustments of the line to ensure acceptable gloss intensities whenever gloss is detected to be lower than the set value.

Since packing houses handle different varieties of apples, it is highly recommended that the findings from this study be tested on apples other than ‘Red Delicious’, to verify the adaptation to all apples. This will ensure consistency and universality in the implementations of the findings from this study. Therefore packing lines could be enhanced to pack different varieties of apples on the same settings and adjustments.

APPENDICES

APPENDIX A

Copy of the Final Report for the Michigan Apple Committee

FINAL REPORT

for

“Evaluation and Improvement of the Appearance of MI ‘Red Delicious’ Apples”

RECOMMENDATIONS FOR ACHIEVING A GOOD APPLE GLOSS

Submitted to

The Michigan Apple Committee

February 2010

Start Date: Fall 2006

Completion Date: Spring 2010

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Conducted by: E. Enyonam Quist

Testing Facility: School of Packaging

Michigan State University

East Lansing, MI 48824

1. BACKGROUND

Pilot packing line components were set up at the School of Packaging, Michigan State University, to test and evaluate critical points on the packing line that significantly contribute to achieving a good waxing job. The different factors on the line that were tested, and their outcomes, were reported in the interim report 'Guidelines for Achieving a Good Apple Gloss' (June 1, 2009). This included the effect of (1) having a heated water system on the packing line, (2) maintaining a warm fruit surface temperature taking into consideration the environmental temperature and relative humidity (RH), as well as that of (3) using a coating with higher solid content specially formulated for humid environments. A scale-up was conducted on a commercial packing line to test and verify the findings from the pilot line reported in the interim report.

2. OBJECTIVE

The objective of the project was to evaluate and improve the apple packing line, and packing line operations, to achieve the desirable fruit finish by:

1. Identifying the parameters on the washing section of the line that will ensure efficient cleaning of the fruit surface in preparation for further processing
2. Evaluating different wax application treatments on apple gloss while maintaining environmental conditions of temperature and RH common to Michigan
3. Studying gloss decay with storage and correlating such decay to quality attributes of firmness and weight loss
4. Evaluating different commercially available wax formulations that could provide better apple gloss when applied under high humidity environments

5. Scaling-up: Verify the finding from the laboratory pilot trials by running trials on a commercial line and evaluate:
    a. The extent to which gloss improves when using a heated water system on the packing line
    b. A new wax formulation with high solid content that provided a high gloss on Michigan apples when applied at high RH during the pilot trial
    c. The effect of dew point on gloss development

3. SIGNIFICANCE

The significance of this study is that:

- The packing process specific to Michigan will be modified, based on observations in the pilot plant, ensuring the delivery of appealing apples
- Waxes used by the Michigan industry are those recommended by manufacturers for packinghouses without taking the humidity of the environment into consideration, thus the observation of lower gloss compared to higher gloss from regions with lower humidity conditions. This critical observation will foster communication with formulators to develop new coatings that is adapted for the Michigan climate
- The recommended changes require the contribution of both packers and wax formulators to achieve the desired goal
- Reports from MAC indicate that the investment to provide the consumer with appealing fruits may substantially increase the annual revenue from the sale of fresh ‘Red Delicious’

4. SUMMARY OF THE PILOT FINDINGS

4.1. Fruit cleaning

The type of detergent and temperature of the dump tank were the factors with significant effects on the cleanliness achieved after washing. The use of alkaline detergent proved to do a better job than the neutral detergent. There was no significant difference where the detergent was applied, be it in either the dump tank or on the washing brushes. The use of 40^{O}C in the dump tank resulted in lower residue recovery from the fruit surface indicative of a better cleaning power compared to 18^{O}C (Figure A.1). The power of the rinse pressures (30 or 60 psi) used in this study was not significant in achieving a cleaner fruit surface. The dwell times in both the dump tank (30 or 60 seconds) and on the washing brushes (60 or 120 seconds) also did not play a significant role in achieving a cleaner surface.

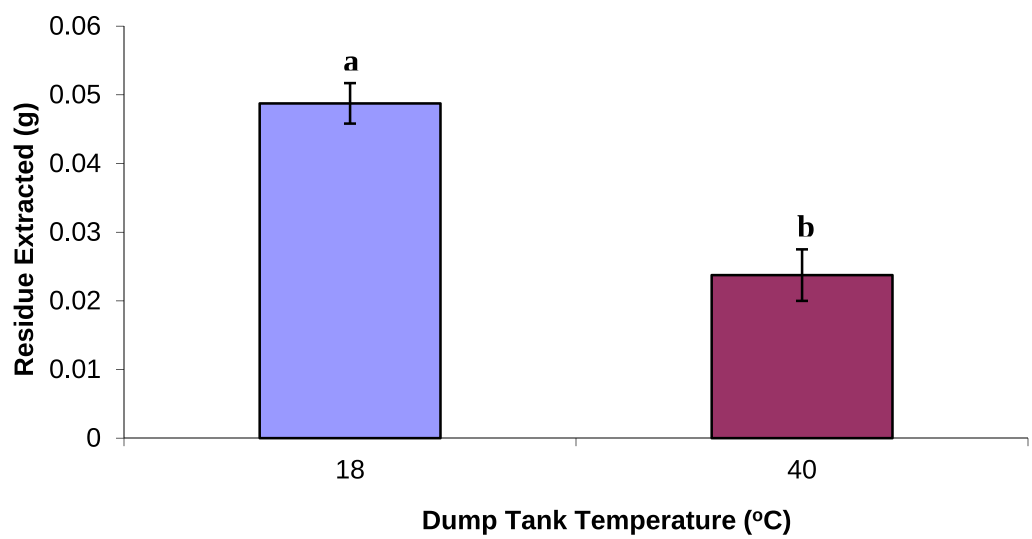

Figure A.1. Effect of dump tank temperature on surface cleanliness (per 3 apples)
Bar columns with the same letter are not significantly different (means ± SD; n = 672; $p \geq 0.05$)

4.2. Wax application and drying

The RH in the drying oven significantly affected the gloss that evolved, with the 25% RH producing the higher gloss fruit (Figure A.2). There was also an effect of interactions of factors used in the waxing process that resulted in significant gloss level differences; these included the wax viscosity and drying time.

The longer drying time (300 compared to 90 seconds) produced drier fruit surfaces, as expected, at the lower RH of 25% compared to 60% when applied at 50^{O}C. For example when comparing fruits that had not been dewatered to fruits emerging from dewatering (just before waxing), there was a decrease of 31% and an increase of 210% in surface moisture in the 25% and 60% RH drying environments respectively. This will have a significant effect on the wax coating thickness and uniformity on the entire fruit and eventually affect the gloss. Drier apples will have a uniform thick coating and consequently a higher gloss. Apples with a wet surface when the wax is applied will not have a uniform coating and patches of wax will be observed throughout the apple surface. As a consequence the gloss will be very low.

4.3. Wax formulation

A preliminary study with high solid content waxes, while keeping all other ingredients the same, performed better when applied in a high humidity environment and gave higher gloss apples during our initial pilot trials. We refer to these waxes as 'new' formulation for the remainder of this report. A subset of the data obtained during our initial pilot trial is shown in figure A.3. It is, however, important to keep in mind that the interaction with other packing line factors, such as drying time (that relates to line speed and length of drying tunnel), temperature and RH in the drying tunnel, impact the solidification of the wax on the surface of the apple which during the pilot trial were very easy to control and maintain. The solidification of the wax in the surface is critical for gloss development.

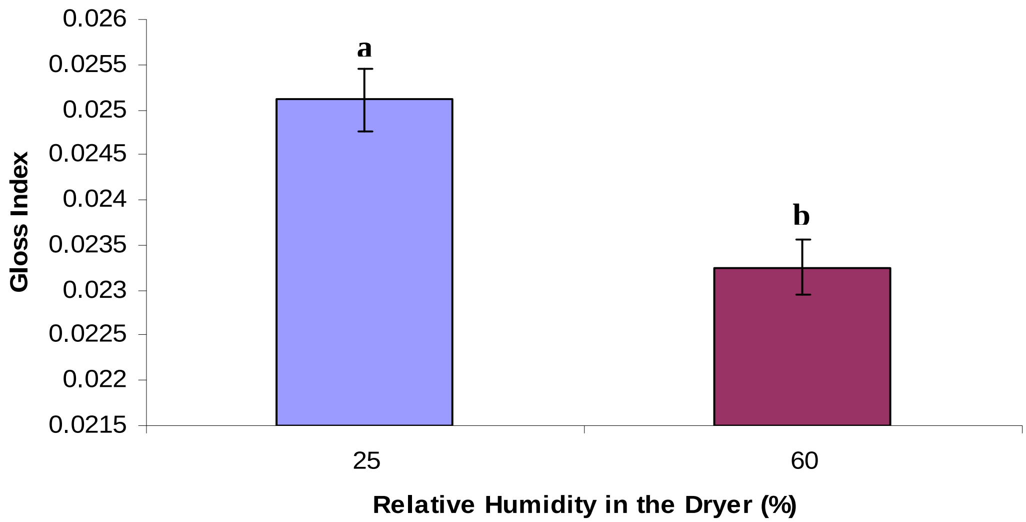

Figure A.2. Effect of humidity in the drying oven on gloss level of coated fruits
Bar columns with the same letter are not significantly different (means ± SD; n = 2031; $p \geq 0.05$)

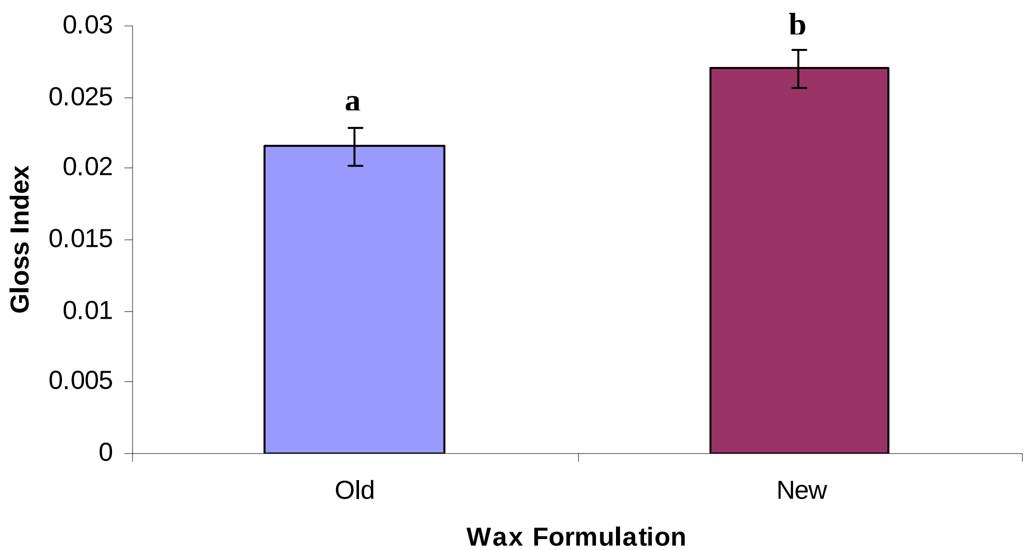

Figure A.3. Effect of coating formulation on gloss level of fruits dried in 60% RH for 90sec during the pilot trial
Bar columns with the same letter are not significantly different (means ± SD; n = 18; $p \geq 0.05$)

5. SCALE-UP: TESTING ON THE COMMERCIAL LINE

Both the ‘old’ (current formula) wax and the ‘new’ formulation were tested on the day of the scale-up with MI ‘Red Delicious’ fruits. The fruits were tested using both heated and cold dump tanks. The cold dump tank was simulated using a plastic bucket filled with unheated water from the dump tank on the packing line. Due to challenges posed by the amount of wax and using a hand sprayer, the team resorted to dipping fruits into a tub of shellac coating and transferring coated fruits to the waxing brush bed by hand. This application was used for both the ‘old’ and ‘new’ formulations to ensure efficient comparison of the gloss that developed from the two formulations. The brush bed was cleaned between formulations by running fruits until it seemed to be dry and clean.

5.1. FINDINGS/RESULTS FROM THE COMMERCIAL LINE

5.1.1. Temperature of the water system and surface temperature of the apples

Based on the findings in the pilot study, it was recommended to maintain a 40^{O}C (104F) temperature in the dump tank together with a 40^{O}C (104F) rinse. These conditions on fruits, equilibrated to room temperature prior to entering the packing line, with dwell times of at least one minute in both the dump tank and on the washing brushes ensured a surface temperature of 24 - 28^{O}C (75.82 – 82.4F) entering the waxing component. The dwell time, temperature, and RH, in the dewatering section contributed to the surface temperature of the apples before they entered the wax applicator.

However, during the scale-up, there was a challenge with testing the theory of having a heated water system on the packing line as the rinse water was cold. Our preliminary testing on the line showed the temperature on the fruits to drop from 30^{O}C (86F), upon exiting the dump tank, to 18^{O}C (64.4F) upon exiting the cold rinse on the washing brushes. Since the spreadability of applied coatings is highly influenced by the surface temperature and moisture content on the fruit in addition to the dew point temperature, a decision was made to bypass the washing brush bed. Thus fruits were held in mesh bags for 4 minutes in the dump tank and delivered to the dewatering brush bed. The surface temperature of the apples transitioning through the packing line is shown in figure A.4. The dwell time on the dewatering brushes was an average of 0.5 minutes.

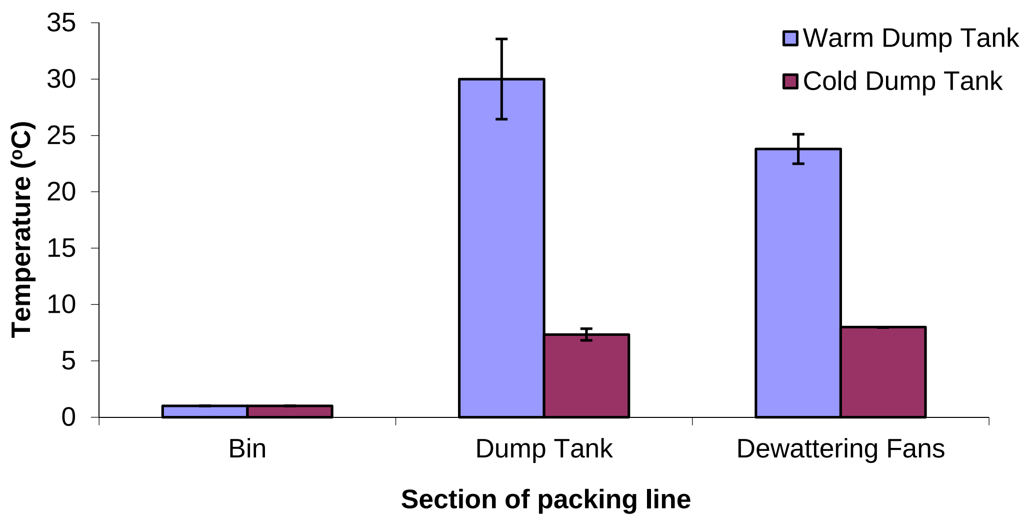

Figure A.4. Surface temperature of apples transitioning through the packing line (means ± SD; n = 24)

5.1.2. Surface Moisture of the apples

The ambient (15^{o}C, 70% RH) and the dewatering (18^{o}C, 61% RH) environmental conditions had dew point temperatures of 9.6 and 11^{o}C respectively. The surface temperature of the fruits treated to the warm dump tank was well above the dew point temperature while that of those treated to the cold was below it (figure A.4). As a result of condensation on the fruits treated to the cold dump tank, higher surface moisture content was recorded (Figure A.5). The higher surface moisture also impacted gloss development as the presence of moisture on the fruit in the wax applicator would dilute the wax applied as will be shown in the next section.

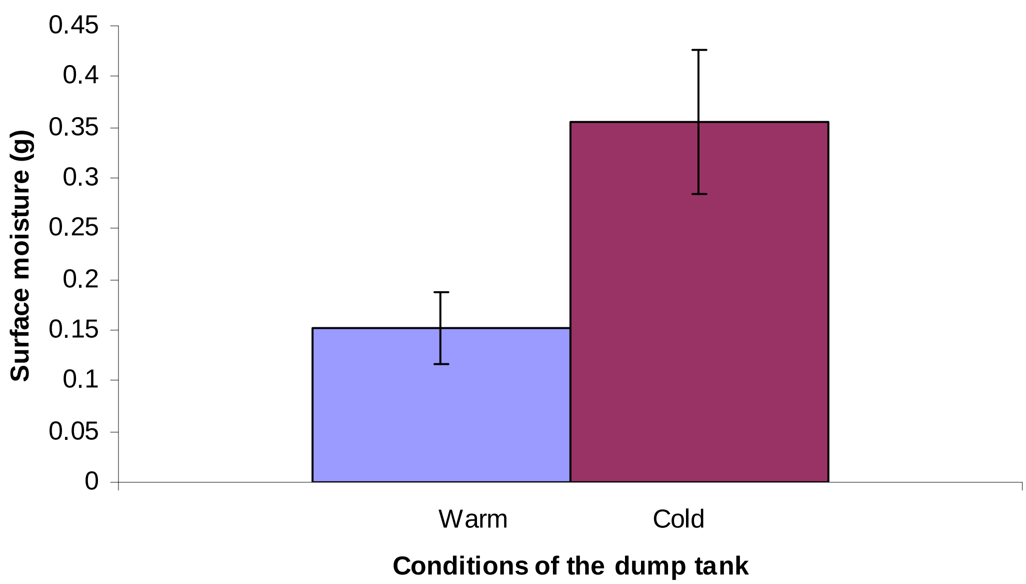

Figure A.5. Surface moisture on apples, treated in either a warm or cold dump tank under 15^{o}C and 70% RH, just prior to entering the wax applicator (means ± SD; n = 12)

5.1.3. Impact of wax formulation and processing on gloss

Fruits treated to the warm dump tank, waxed with either the 'old' or 'new' shellac formulation recorded significantly higher gloss values than those treated to the cold dump tank (Figure A.6). This observation could be attributed to the surface temperature of the warmer fruits being above the dew point temperature, and thus minimal surface moisture to significantly dilute the applied wax.

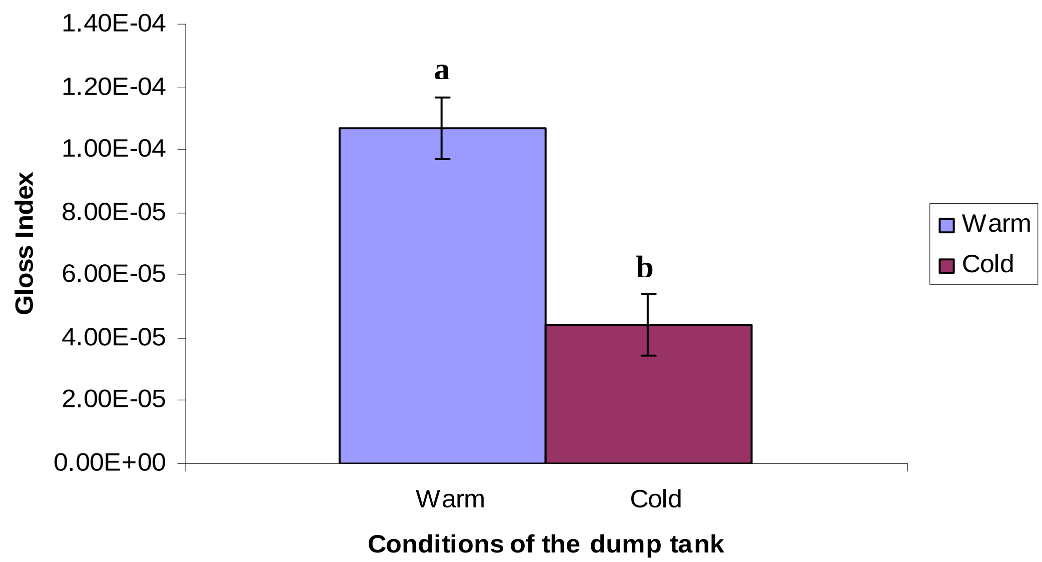

Figure A.6. Comparing gloss on apples treated in either a warm or cold dump tank, and waxed with the 'old' formulation

Bar columns with the same letter are not significantly different (means ± SE; $n = 31$; $p \geq 0.05$)

In the warm dump tank however, just as in the cold, it was the 'old' formulation that recorded the higher gloss values as shown in figure A.7. The observations on the commercial line contradict that of the pilot. This could be attributed to better control of the experiments and factors, such as drying time and temperature, in the latter compared to the former where minimal adjustments were made on the line during the scale-up. A scale-up will have more variability than a controlled pilot trial. The 'new' formulation with higher solid content may require more time to solidify compared to the current formulation in which the coating seemed to be completely dry and less sticky with the evaporation of the volatile solvents.

Although the low performance of the 'new' wax was unexpected it is possible that factors that produced a positive outcome in the pilot trials do not perform in the same manner on the commercial line, which is the reason why it is critical to run a scale-up to validate the results obtained in the pilot trials.

As a result of the scale-up we are confident that a close control of the dump tank temperature and apple surface temperature and moisture content will have a positive impact on the gloss development on a commercial line since these were validated.

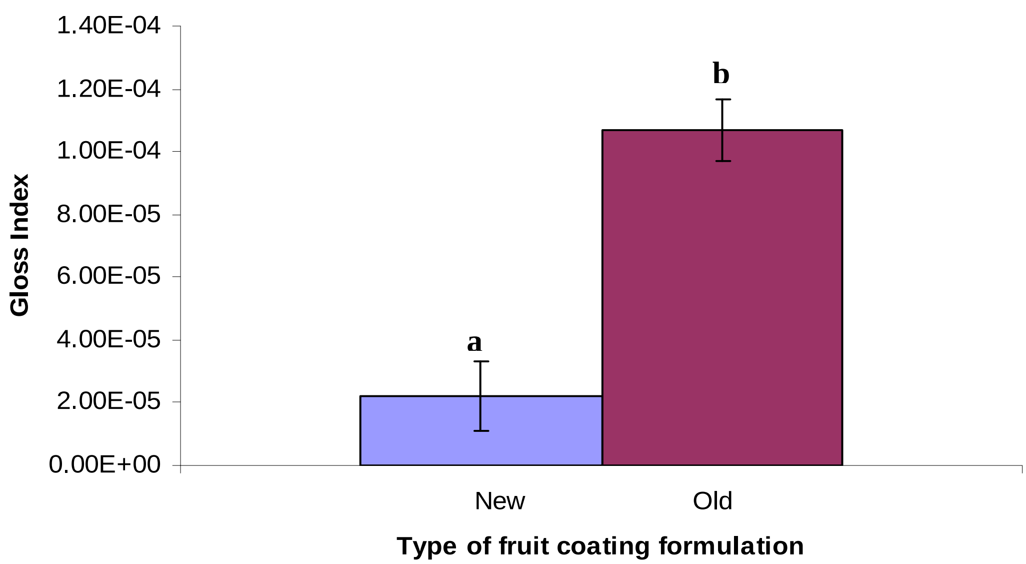

Figure A.7. Comparing gloss on apples treated in a warm dump tank and waxed with either the 'old' or 'new' formulation

Bar columns in the same group with the same letter are not significantly different (means ± SE; $n = 31$; $p \geq 0.05$)

6. RECOMMENDATIONS

These recommendations outlined below are based on both the findings in the pilot and commercial scale packing lines:

1. To ensure efficient cleaning of apples and a warm surface before being introduced into the wax applicator, it is recommended to use at least 40^{o}C water in the dump tank together with at least 30psi, 40^{o}C rinse water. Though the heating systems for both the dump tank and washing brushes may be equipped with a digital regulator-display, we recommend the use of a stick thermometer on a regular basis to check that the systems are heating efficiently. The key is to have an efficient heating system to enable attaining fruit surface temperature above the dew point.
2. We recommend that the surface moisture and temperature of the fruits be critically monitored as they transition through the packing line. Fruit surface temperature above the dew point will prevent condensation of moisture and ensure a good gloss. Knowledge of the dew point temperature (Table A.1) will enable appropriate adjustment of the temperature of the water system and dwell times in the dump tank and/or washing brushes to maintain surface temperatures above the dew point. The surface temperature of the apples can be monitored using a hand held infra-red thermometer gun and surface moisture can be measured by absorbing with an absorbent paper.
    a. The temperature and dwell time in the drying tunnel may also need to be adjusted based on the environmental conditions and surface temperature the fruits attain prior to being dried. These adjustments will be specific to each packing line as a result of the different sizes and lengths of individual lines. A monitoring system is suggested as a means to either control the speed of fruit through the drying zone and/or sound a

warning based on the dew point falling near or below expected fruit surface temperatures.

b. Surface moisture was 0.1 – 0.2g for an average sized apple (*ca*. 250-300g) with surface temperature above the dew point. Therefore we recommend that the surface moisture on fruit with surface temperature above the dew point does not exceed 0.2g to ensure efficient application and drying of the applied coating.

3. We recommend the use of a coating that has a minimum Zahn viscosity of ~45 Zahn seconds when using a 0.078" orifice Zahn cup. Using a viscosity below that is likely to drip off the fruit while that above it may not provide an efficient and uniform spread on the fruit, both of which impact gloss. Since the initial assumption of changing the solid content to provide a higher gloss has not shown consistent results we highly recommend more work to be done on coating formulations by the companies to improve formulations suited for Michigan.

4. The effect of the type of detergent on surface cleanliness was not assessed during the scale-up so as not to interfere with the main objective of the scale-up. Therefore this recommendation is based solely on observations during the pilot:-

   We recommend the use of an alkaline detergent in either the dump tank or on the washing brushes. In the case where the detergent is used on the washing brushes, it is recommended to thoroughly rinse the fruits with the rinse conditions mentioned in "Recommendation 1" above. We also recommend the use of a sanitizing agent such as chlorine or chlorine dioxide (as is practiced in WA) in the dump tank to eliminate bacteria and kill spores on the fruit surface. This can be coupled with the alkaline detergent in either the dump tank or on the washing brushes, together with another sanitizer on the washing brushes prior to rinsing the fruits.

Table A.1. Temperature, Relative Humidity and Dew Point Chart

Temperature (F)	Relative Humidity / Dew Point Temperature (F)												
	40%	45%	50%	55%	60%	65%	70%	75%	80%	85%	90%	95%	100%
78	51.75	54.96	57.87	60.54	63.00	65.29	67.43	69.43	71.33	73.12	74.83	76.45	78.00
79	52.64	55.87	58.79	61.47	63.94	66.23	68.38	70.40	72.30	74.10	75.81	77.44	79.00
80	53.53	56.77	59.71	62.39	64.88	67.18	69.34	71.36	73.27	75.08	76.80	78.44	80.00
81	54.43	57.68	60.62	63.32	65.81	68.13	70.29	72.33	74.24	76.06	77.79	79.43	81.00
82	55.32	58.58	61.54	64.25	66.75	69.08	71.25	73.29	75.22	77.04	78.77	80.42	82.00
83	56.21	59.49	62.46	65.18	67.69	70.02	72.20	74.25	76.19	78.02	79.76	81.42	83.00
84	57.10	60.39	63.37	66.10	68.62	70.97	73.16	75.22	77.16	79.00	80.75	82.41	84.00
85	57.99	61.29	64.29	67.03	69.56	71.91	74.12	76.18	78.13	79.98	81.73	83.40	85.00
86	58.88	62.20	65.20	67.96	70.50	72.86	75.07	77.15	79.10	80.96	82.72	84.40	86.00
87	59.77	63.10	66.12	68.88	71.43	73.81	76.03	78.11	80.07	81.94	83.71	85.39	87.00
88	60.67	64.01	67.03	69.81	72.37	74.75	76.98	79.07	81.05	82.92	84.69	86.38	88.00
89	61.56	64.91	67.95	70.73	73.31	75.70	77.93	80.04	82.02	83.89	85.68	87.38	89.00
90	62.45	65.81	68.86	71.66	74.24	76.64	78.89	81.00	82.99	84.87	86.66	88.37	90.00
91	63.34	66.71	69.78	72.59	75.18	77.59	79.84	81.96	83.96	85.85	87.65	89.36	91.00
92	64.22	67.62	70.69	73.51	76.12	78.54	80.80	82.92	84.93	86.83	88.64	90.36	92.00
93	65.11	68.52	71.61	74.44	77.05	79.48	81.75	83.89	85.90	87.81	89.62	91.35	93.00
94	66.00	69.42	72.52	75.36	77.99	80.43	82.71	84.85	86.87	88.79	90.61	92.34	94.00
95	66.89	70.32	73.44	76.29	78.92	81.37	83.66	85.81	87.84	89.77	91.59	93.34	95.00

Table A.1 (cont'd)

96	67.78	71.22	74.35	77.21	79.86	82.32	84.62	86.78	88.81	90.75	92.58	94.33	96.00
97	68.67	72.13	75.26	78.14	80.79	83.26	85.57	87.74	89.79	91.72	93.57	95.32	97.00
98	69.56	73.03	76.18	79.06	81.73	84.21	86.52	88.70	90.76	92.70	94.55	96.32	98.00
99	70.44	73.93	77.09	79.99	82.66	85.15	87.48	89.66	91.73	93.68	95.54	97.31	99.00
100	71.33	74.83	78.00	80.91	83.60	86.09	88.43	90.63	92.70	94.66	96.52	98.30	100.00
101	72.22	75.73	78.92	81.83	84.53	87.04	89.38	91.59	93.67	95.64	97.51	99.30	101.00
102	73.11	76.63	79.83	82.76	85.47	87.98	90.34	92.55	94.64	96.62	98.50	100.29	102.00
103	73.99	77.53	80.74	83.68	86.40	88.93	91.29	93.51	95.61	97.60	99.48	101.28	103.00
104	74.88	78.43	81.65	84.61	87.33	89.87	92.24	94.47	96.58	98.57	100.47	102.27	104.00

7. REQUESTED COMPLEMENTARY STUDY: GLOSS DECAY WITH TIME

During a progress report session on the pilot findings, the Michigan Apple Committee requested a study to evaluate gloss decay over time. The research team complemented the decay study with quality attributes of weight and firmness, and also correlated that to gloss thickness using scanning electron microscopic techniques.

The weight of the fruits decreased with time and coating the fruits decreased the weight loss by almost 3-fold (Figure A.8); the coated fruits lost 1.79% in weight at the end of the 8 weeks storage compared to 5.11% in the uncoated. Firmness also decreased across time (Figure A.9), and in both evaluations of weight and firmness it was observed that the higher gloss fruits were able to maintain quality longer.

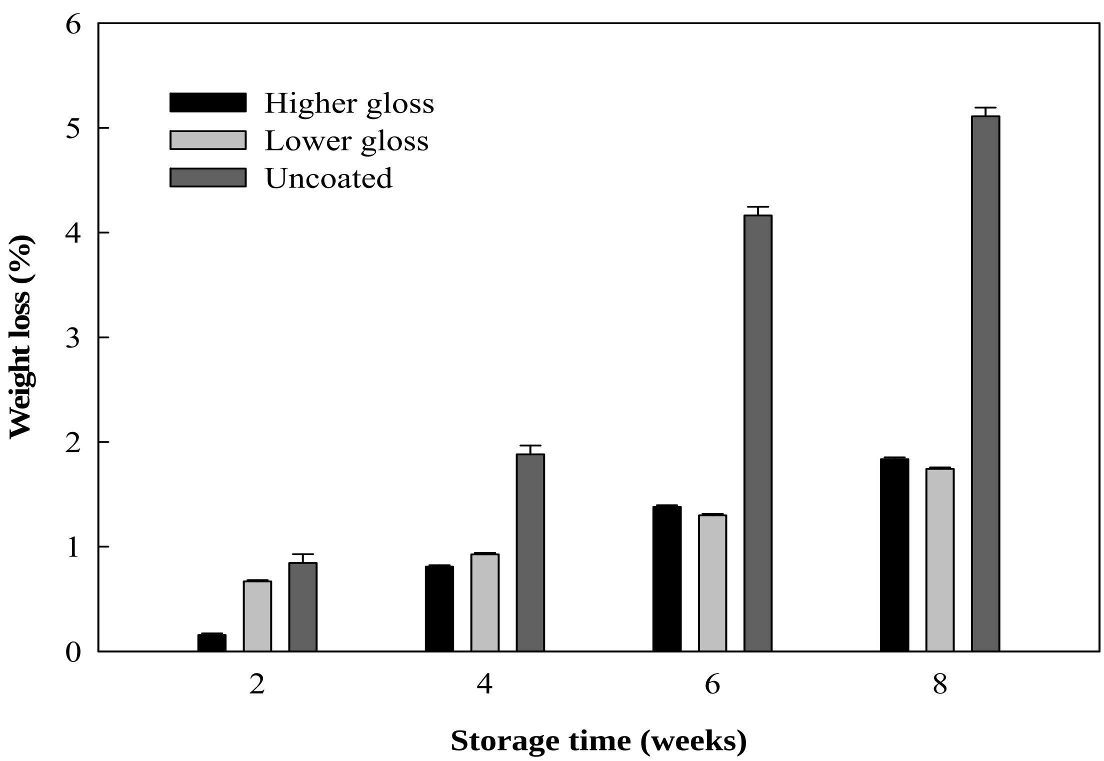

Figure A.8. Weight loss in apples during storage at 4^{o}C, 95% RH (estimate ± SE)

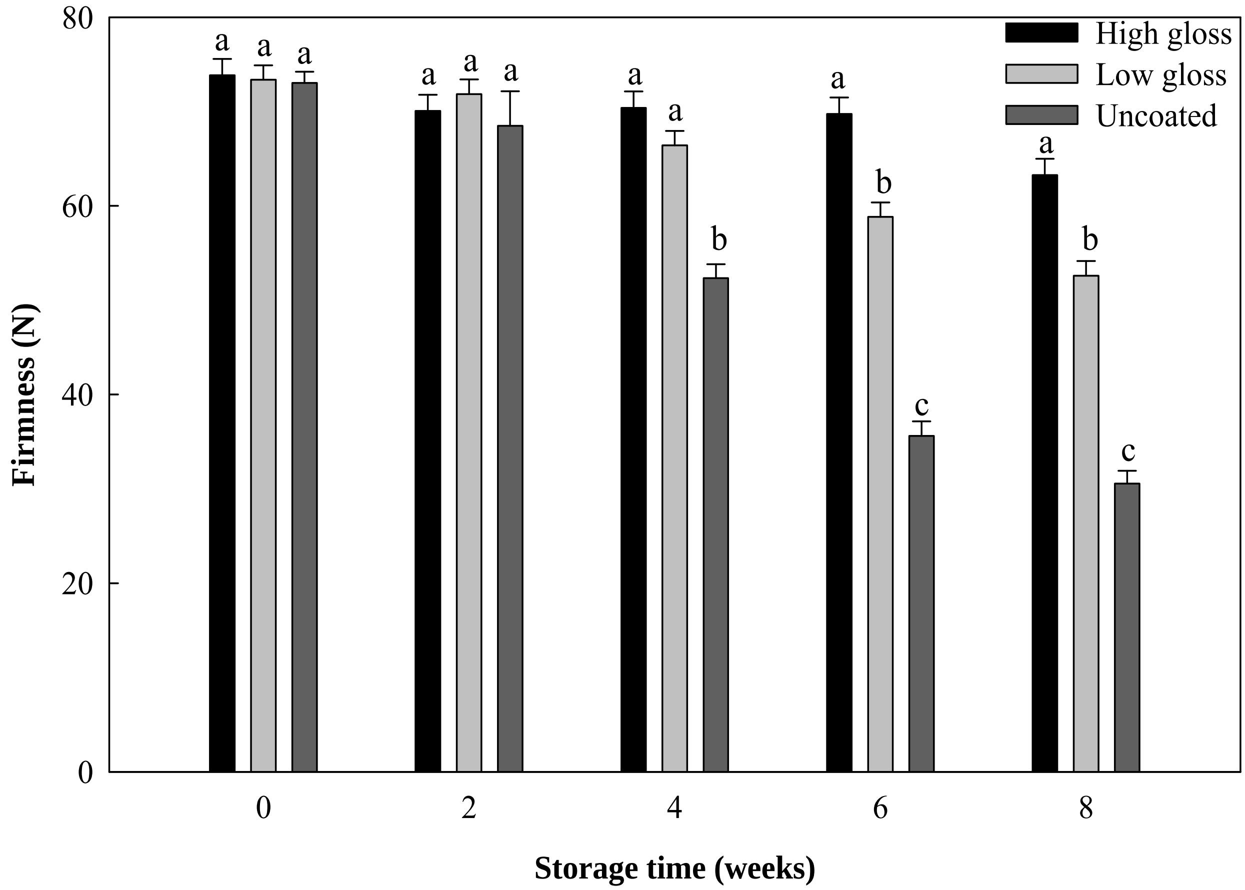

Figure A.9. Loss in fruit firmness with storage at 4^{o}C, 95% RH
Grouped columns with the same letter are not significantly different (estimate ± SE; n = 684; p ≥ 0.05)

Gloss, just like weight and firmness also decreased over time. A similar trend of decay is observed in all three (Figure A.10); the fruits with longer gloss retention during storage were those that retained firmness with minimal weight loss.

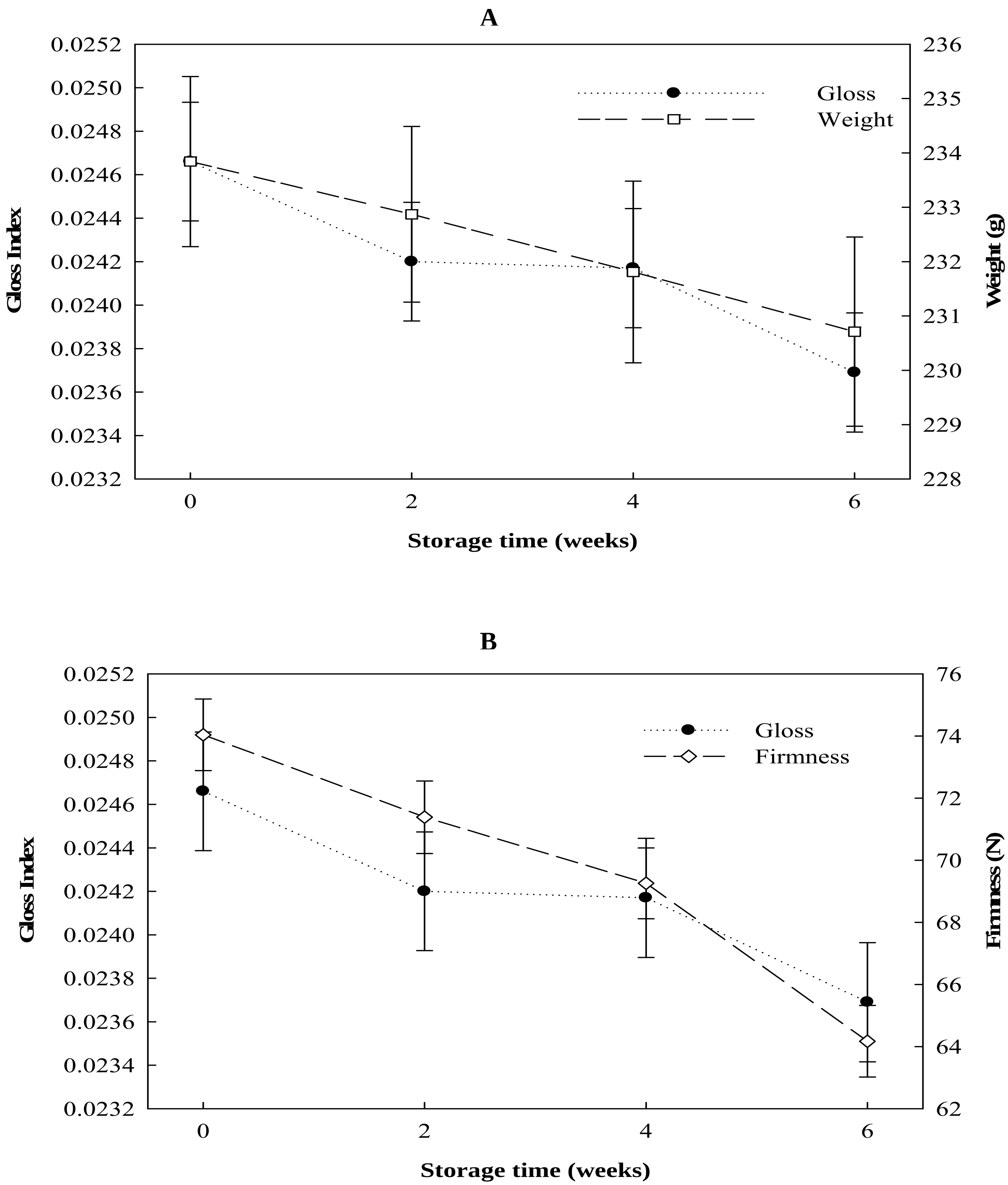

Figure A.10. Relationship between gloss and (A) weight, and (B) firmness of fruits during storage at 4^{0}C, 95% RH (estimate ± SE)

Studies on the microstructure of the coating showed that the thickness of the coating translates to gloss (Figure A.11). Since the higher gloss fruits had thicker coatings, it could be said that the barrier properties were enhanced which is the reason for the slower deterioration of quality in the higher gloss fruits.

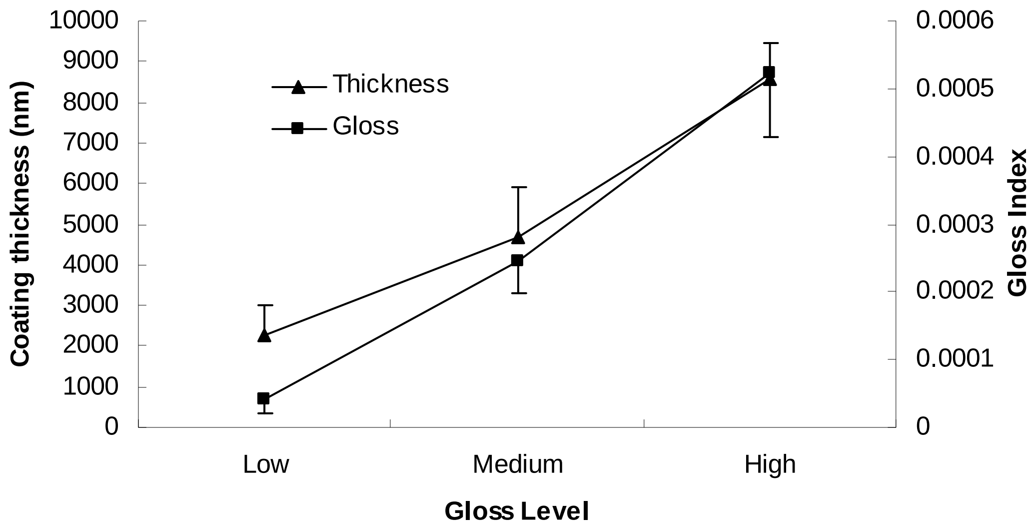

Figure A.11. Relationship between coating thickness and gloss (mean ± SD)

APPENDICES

Appendix A – Method to measure water temperature

- Use a thermometer stick at predetermined intervals in various sections of the dump tank and rinse spray.
- Immerse the thermometer into the body of water (for the rinse spray, collect a sample in a beaker) and wait till the dial (or in the case of a digital stick, the display) is stable.
- Use the average of about three measurements each for the dump tank and rinse spray.

Appendix B – Method to measure surface temperature

Measure the fruit surface temperature just prior to entering the wax applicator.

- Use an infrared thermometer gun with ± 2^{o}C accuracy at less than 80% RH (e.g. Catalog No. S90202, Fisher Scientific, Hampton, NH). (Note: be sure of the IR thermometer cone/area of incidence/measurement as this varies with distance from apple. Longer distances from the apple surface may result in measuring an area larger than the apple.)
- Measure no fewer than 6 fruit surface temperatures for each batch. Use the average and standard deviation and compare to the dew point temperature chart.

Appendix C – Method to measure surface moisture

Measure the surface moisture of fruits prior to entering the wax applicator.

- Quantify the amount of moisture on the apple surface by absorbing the moisture with absorbent #2 filter paper (e.g. from Whatman Inc., Florham Park, NJ)
- Weigh the absorbent paper on a laboratory precision balance with at least a 0.01g sensitivity (e.g. from Adventurer ARC120; Ohaus, Pine Brook, NJ). Record the difference in weight of

the paper before and after use as the amount of surface moisture. (See Recommendation 2b under Section 6).

- Record the surface moisture of 6 fruits, and use the average and standard deviation.

Appendix D – Method to measure wax viscosity

Measure the viscosity of the fruit coating being used, intermittently throughout the day, with a 0.078” orifice Zahn cup (e.g. from Weschler Instruments, Cleveland, OH) and a timer.

- Since temperature affects viscosity, we recommend that the temperature of the coating should be measured (using a thermometer stick) prior to each viscosity measurement.
- Insert the Zahn cup vertically into the coating.
- Start the timer when the top of the Zahn cup emerges from the surface of the coating.
- Stop the timer when the steady flow of the wax through the orifice ceases.
- Record the time and repeat three times.

APPENDIX B

The Remainder of the Interactions of Packing Line Factors on the Level of Cleanliness

The figure below is complementary to the discussion in Chapter 3, section 3.3.1, on page 88 - 89.

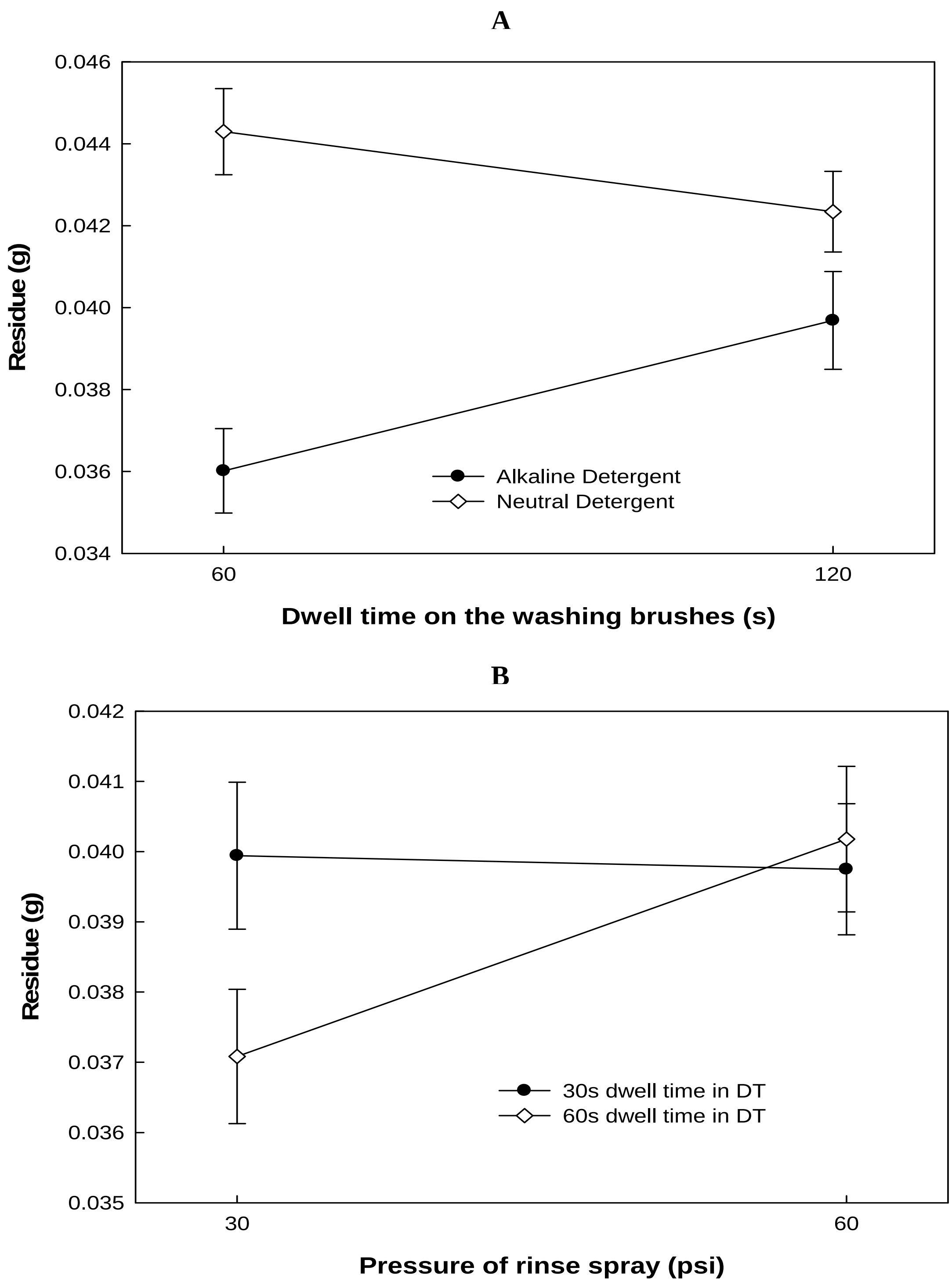

Figure B1. Two way interactions of (A) 60 and 120 seconds dwell time on the washing brushes with alkaline and neutral detergents and (B) pressure of rinse spray with 30 and 60 seconds dwell time in the dump tank on the surface cleanliness of apples (means ± SE; n = 512 for A, 672 for B; $p \leq 0.05$)

APPENDIX C

Consumer Panel Consent Form

School of Packaging, Michigan State University

Consumer Panel Consent Form

Evaluation of Fresh Apples

Dear Participant:

Michigan State University researchers are working to improve the quality of **fresh apples** and asking that panelists participate in a study to evaluate the resulting gloss on the apple after different treatment conditions. The treatment conditions include cleaning the apples in different fruit cleaners, varying the time, water temperatures and pressures after which the apples are waxed with shellac (FDA approved food grade wax) under different environmental conditions. Each evaluation will take about 10 minutes or less after you receive your samples. You will be given a coupon or food treat worth $2 or less as an appreciation for your participation and completion of the questionnaires.

Though none is anticipated, if you have a problem **upon touching the samples**, notify the on-site sensory evaluation coordinator and/or principle investigator immediately. You will be released from participating in this study. Please note if you are injured as a result of your participation in this research project, Michigan State University will assist you in obtaining emergency care, if necessary, for your research related injuries. If you have insurance for medical care, your insurance carrier will be billed in the ordinary manner. As with any medical insurance, any costs that are not covered or in excess of whatever are paid by your insurance, including deductibles, will be your responsibility. Financial compensation for lost wages; disability, pain or discomfort is not available. This does not mean that you are giving up any legal rights you may have. Your response is confidential and we will protect your confidentiality to the full extent of the law.

School of Packaging, Michigan State University

Participation in this study is voluntary. You may refuse to **grade any of the apples** without penalty, and your decision to refuse participation or discontinue participation during this study will be honored promptly and unconditionally.

If you have any questions during your reading this consent form, or during or after your participation, please do not hesitate to contact the on-site sensory evaluation leader and/or the principle investigator, Dr. Maria Rubino, via phone at 517-355-0172, or regular mail at 130 Packaging Building, East Lansing, MI 48824. She can also be reached via email at mariar@msu.edu for any inquiry you might have related to your participation in the study. In case you have questions or concerns about your role and rights as a research participant, please feel free to contact Dr. Peter Vasilenko, Ph.D., Director of Human Research Protections, by phone: (517) 355-2180, fax: (517) 432-4503, email: irb@msu.edu or regular mail: 202 Olds Hall, East Lansing, MI 48824-1047.

I voluntarily agree to participate in the study.

SIGNED ____ DATE______________

APPENDIX D

Questionnaire for the Sensory Evaluation of the Gloss on Apples - Consumer Panel Rating

School of Packaging, Michigan State University

Evaluation of Fresh Apples

Questionnaire for Sensory Evaluation

Name: ____________________ **Email Address:** _______________ **Date:** ________

Type of Sample: Red Delicious Apples

Characteristic Studied: Gloss on apples from different packaging conditions

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Instructions:

1. Receive the 5 samples. Two samples are marked as "Non glossy" and "Glossy" while others are marked with a 3-digit numeric code.
2. Use the "Non Glossy" and "Glossy" samples to understand the aesthetic attribute that we are asking you to identify. "Non-glossy" represents 2 and "Glossy" represents 13 according to the key provided in instruction # 5.
3. Once you understand the aesthetic attribute that we want you to analyze, go ahead and note the sample codes for the coded samples in the next section according to its position on the tray.
4. Visually analyze the samples from left to right and note the intensity of the gloss on the fruits.
5. Rank the samples according to the following key for degree of glossiness, by drawing a line through the corresponding grid.

0 – 3	Non glossy	4 – 7	Slightly glossy
8 - 11	Moderately glossy	12 - 15	Highly glossy

School of Packaging, Michigan State University

Evaluation of Fresh Apples

Code: ________

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Non-glossy Highly Glossy

Code: ________

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Non-glossy Highly Glossy

Code: ________

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

Non-glossy Highly Glossy

Comments:

APPENDIX E

Selected Panel Consent Form

School of Packaging, Michigan State University

Selected Panel Consent Form

Evaluation of Fresh Apples

Dear Participant:

Before you decide to sign this consent form and continue to participate in this study, please read this document carefully for the information related to the study, ingredients, packaging material and procedures used in the study. Potential risks and benefits from your study, assurance of your privacy and your rights as a human subject in our study are also listed.

If you have any questions during your reading this consent form, or during or after your participation, please do not hesitate to contact the on-site sensory evaluation leader and/or the principle investigator, Dr. Maria Rubino, via phone at 517-355-0172, or regular mail at 130 Packaging Building, East Lansing, MI 48824. She can also be reached via email at mariar@msu.edu for any inquiry you might have related to your participation in the study. In case you have questions or concerns about your role and rights as a research participant, please feel free to contact Dr. Peter Vasilenko, Ph.D., Director of Human Research Protections, by phone: (517) 355-2180, fax: (517) 432-4503, email: irb@msu.edu or regular mail: 202 Olds Hall, East Lansing, MI 48824-1047.

PLEASE NOTE THAT UPON YOUR SIGNING THIS CONSENT FORM, YOU VOLUNTARILY AGREE TO PARTICIPATE IN THIS STUDY. YOUR SIGNATURES INDICATE YOU HAVE READ ALL THE INFORMATION PROVIDED IN THIS CONSENT FORM AND THAT YOU HAVE HAD AN ADEQUATE OPPORTUNITY TO DISCUSS THIS STUDY WITH THE PRINCIPLE INVESTIGATOR AND HAVE HAD ALL YOUR QUESTIONS ANSWERED TO YOUR SATISFACTION. A COPY OF THIS CONSENT

School of Packaging, Michigan State University

Selected Panel Consent Form

Evaluation of Fresh Apples

FORM WITH YOUR SIGNATURE FOR YOUR RECORDS CAN BE PROVIDED UPON YOUR REQUEST.

I voluntarily agree to participate in the study.

SIGNED ____ DATE______________

School of Packaging, Michigan State University

Selected Panel Consent Form

Evaluation of Fresh Apples

Invitation to Participate: You are invited to participate in the study that assesses the effect of different packing and environmental conditions on gloss development on fresh apples.

Purpose of the study: We are investigating the effect of different processing conditions on the resultant **gloss on apples**. This study would help establish the best processing conditions to obtain the best sheen on apples and also correlate human perception of gloss to a customized glossmeter.

Procedure of the study: Each panelist would be served red delicious apples waxed with shellac under different environmental conditions and would be asked to rank the sample after **visual examination**. Each sample would be coded with a unique 3-digit code. We are asking that panelists participate in this study that will include approximately 5 sensory evaluation sessions. Evaluations should last about 20 minutes or less.

Sample Preparation: All the materials used in this study are approved by the FDA for contact with food. The apples will be cleaned with either alkaline or neutral fruit cleaners for different lengths of time under varied water temperatures and pressures. The cleaned fruits will then be waxed with shellac under different environmental temperatures and relative humidity to develop different levels of sheen.

Potential Risks: Since all the materials used to prepare and wax the apples are FDA approved food grade, these samples pose no adverse health risk, provided the subject has not been identified as being susceptible to an allergic reaction to apples or surfactants or waxes upon contact. Though none is anticipated, if you have a problem **upon touching the samples**, notify the on-site sensory evaluation coordinator and/or principle investigator immediately. You will be released from participating in this study. Please note if you are injured as a result of your participation in this research project, Michigan State University will assist you in obtaining emergency care, if necessary, for your research related injuries. If you have insurance for medical care, your insurance carrier will be billed in the ordinary manner. As with any medical insurance, any costs that are not covered or in excess of whatever are paid by your insurance, including deductibles, will be your responsibility. Financial compensation for lost wages; disability, pain or discomfort is not available. This does not mean that you are giving up any legal rights you may have. Your response is confidential and we will protect your confidentiality to the full extent of the law.

Expected Benefits: This study will enable the researchers understand gloss development under different packing conditions, and will help validate and correlate readings on a customized glossmeter to human perception of gloss.

Assurance of confidentiality: Any information obtained in connection with this study that could be identified with you will be kept confidential by ensuring that all consent forms and response sheets are securely stored. All data collected and analyzed will be reported in an aggregate format that will not permit associating subjects with specific responses or findings. **Your privacy will be protected to the maximum extent allowable by law.**

School of Packaging, Michigan State University

<u>Selected Panel Consent Form</u>

Evaluation of Fresh Apples

Withdrawal from the study: Participation in this study is voluntary. You may refuse to grade any of the apples without penalty, and your decision to refuse participation or discontinue participation during this study will be honored promptly and unconditionally.

APPENDIX F

Questionnaire for the Sensory Evaluation of the Gloss on Apples -

Selected Panel Ranking

School of Packaging, Michigan State University

Evaluation of Fresh Apples

Questionnaire for Sensory Evaluation

Name: ________________________ **Date:** _______________

Type of Sample: Red Delicious Apples

Characteristic Studied: Gloss on apples from different packaging conditions

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Instructions:

1. Receive the samples and note each sample code in the next section according to its position on the tray.
2. Visually analyze the samples from left to right and note the intensity of the gloss on the fruits.
3. Write "1" in the box of the sample which you deem to have the least surface gloss. Write "2" for the next, "3" for the one next to that and "4" for the sample with the highest surface gloss.
4. If two samples appear the same, then give them the rank that is average of the ranks. For example if samples contending for rank # 2 and 3 seem similar, assign both of them rank= 2.5.

Code:	________	__________	__________	_________
Rating:	☐	☐	☐	☐

APPENDIX G

Questionnaire for the Sensory Evaluation of the Gloss on Apples -

Consumer Panel Ranking

School of Packaging, Michigan State University

Evaluation of Fresh Apples

Questionnaire for Sensory Evaluation

Name: ________________________ **Email Address: _______________________** **Date: ______**

Type of Sample: Red Delicious Apples

Characteristic Studied: Gloss on apples from different packaging conditions

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Instructions:

1. Receive the samples and note each sample's code in the next section according to its position on the tray.
2. Visually analyze the samples from left to right and note the intensity of the gloss (surface sheen) on the fruits.
3. Rank the samples, relative to each other, according to the following key for degree of glossiness.

1	Least glossy	2	Moderately glossy	3	Most glossy

Code: ________ __________ __________

Rating: ☐ ☐ ☐

Comments:

APPENDIX H

Rheological Profile of Shellac Coatings Used in this Research

The table and figures in this appendix complement the discussion in Chapter 3, section 3.3.3.

Table H.1. Apparent behavior of fruit shellac coatings (original formulation and that of 7% weight vaporized) at 20 and 40 ^{o}C

Coating	Formulation	Temperature (^{o}C)	Consistency Coefficient, K	Flow Behavior Index, n
Co. A	Original	20	0.0165	1.4578
		40	0.0084	1.5975
	7% (w/w) evaporated	20	0.0365	1.2107
		40	0.0142	1.4793
Co. B	Original	20	0.0104	1.5789
		40	0.0053	1.8636
	7% (w/w) evaporated	20	0.0138	1.4878
		40	0.0086	1.6302
Co. C	Original	20	0.0188	1.3687
		40	0.0103	1.5234
	7% (w/w) evaporated	20	0.0246	1.3796
		40	0.0128	1.5097

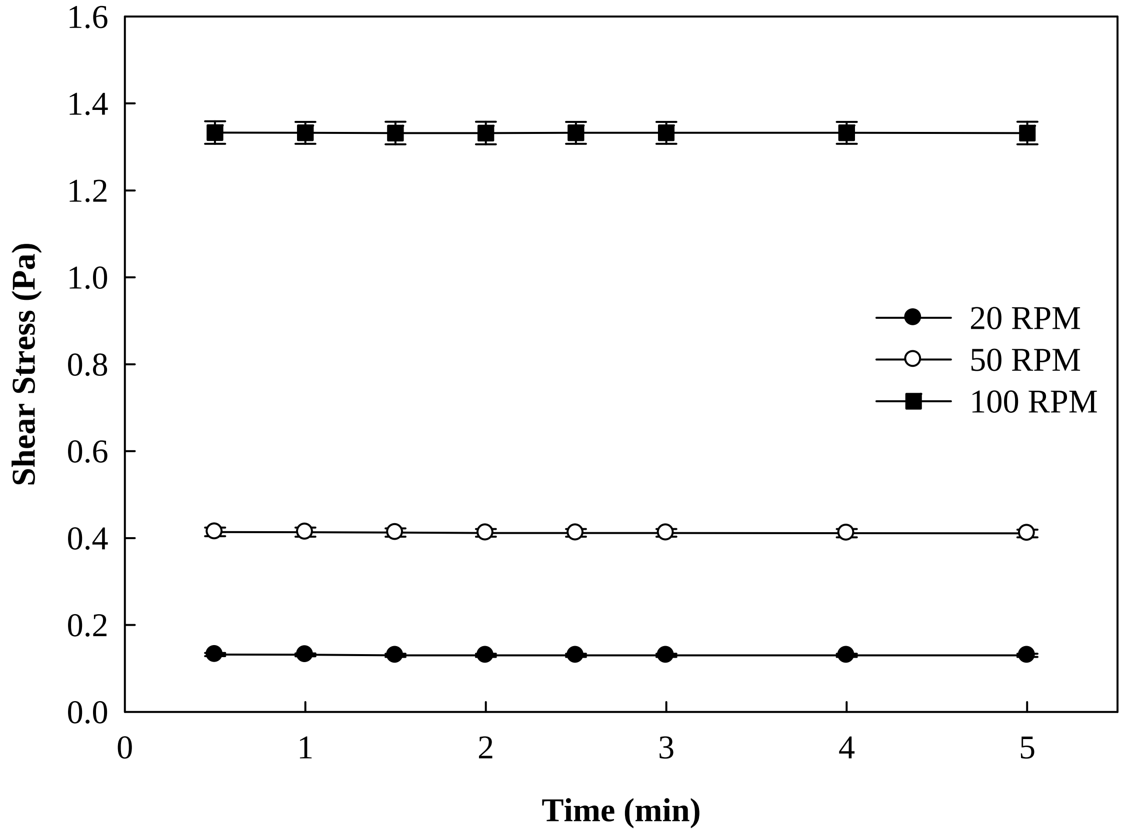


Figure H.1. Time-independent behavior of fruit shellac coating at 20, 50 and 10 RPM (mean ± SD)

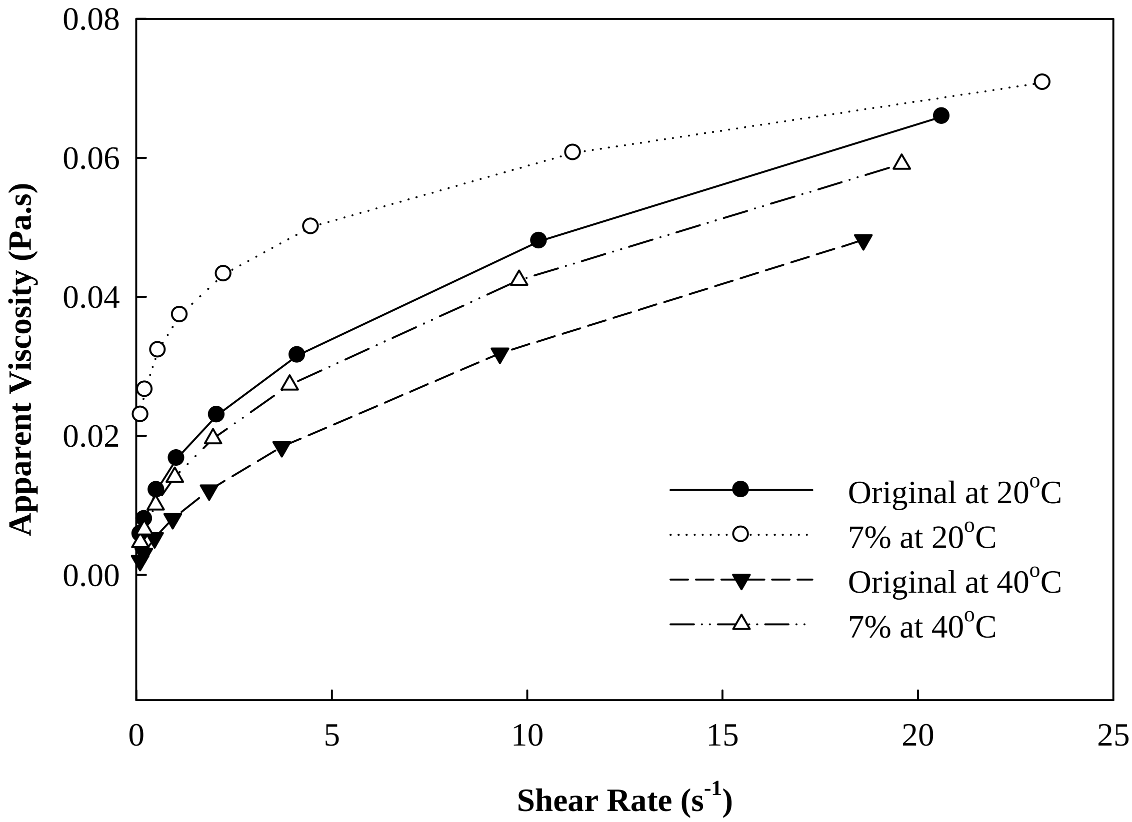


Figure H.2. Apparent viscosity of fruit shellac coating (original formulation and that of 7% weight vaporized) at 20 and 40 ^{o}C with varied shear rates

APPENDIX I

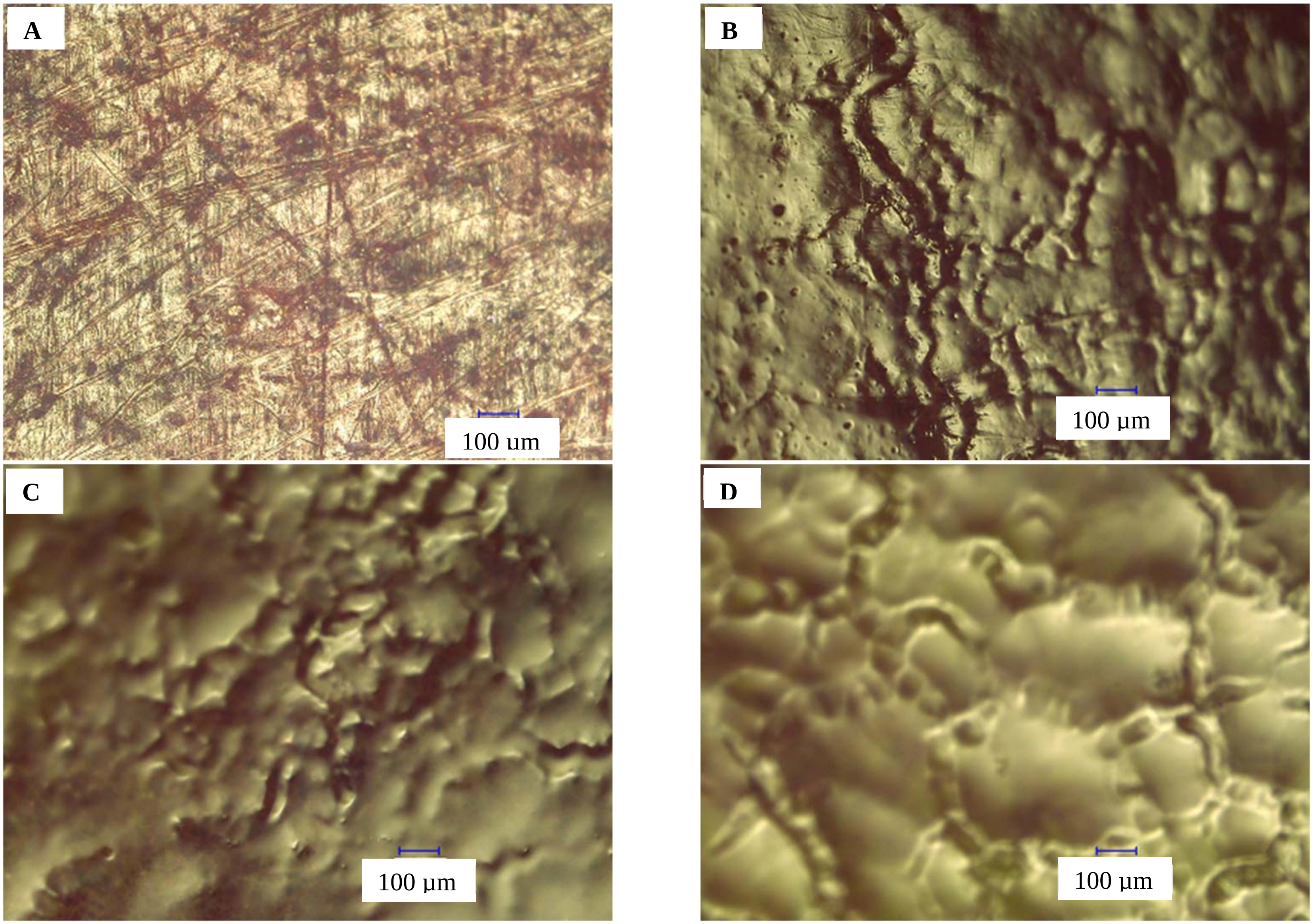
Digital Microscopic Images of Uncoated and Coated Apple Surface

This figure is complementary to the result and discussion sections in Chapter 4 (section 4.3) and Chapter 5 (section 5.3).

Figure I.1. Digital microscopic images of the apple surface with different amounts of coating at 200x magnification

(A) Control; (B) 0.5 ml, low gloss; (C) 1.0 ml, medium gloss; and (D) 2.0 ml, high gloss

Figure I.1 (Cont'd)



APPENDIX J

CO_2 Transmission Rate through Shellac of Varied Thicknesses

The figure below is complementary to the discussion in section 5.3.2 of Chapter 5.

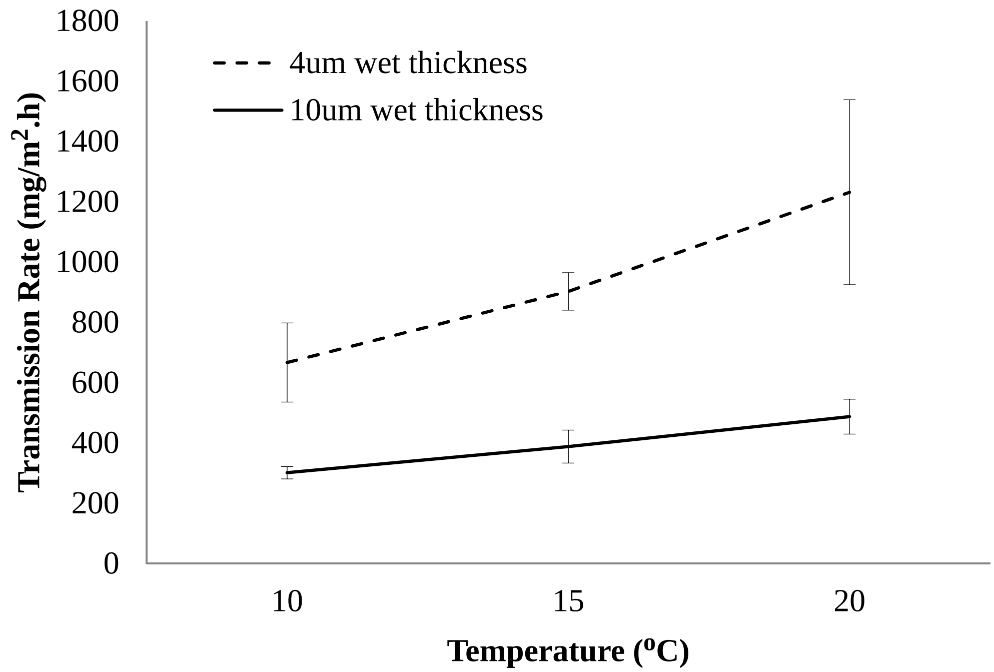


Figure J.1. CO_2 transmission rate through shellac coating of 4 and 10 µm wet thicknesses at 10, 15 and 20 ^oC (mean ± SD)