



**LESSONS FROM CITRUS PEEL: A BIOMIMETIC
APPROACH FOR PACKAGING DESIGN**

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Master's Thesis

Graduate School
Izmir University of Economics

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ABSTRACT

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Tütüncüođlu, Bilge

Master's Program in Design Studies

Advisor: Prof. Dr. Murat Bengisu

June, 2022

The main purpose of this study was to obtain basic information that can be used in packaging design and provide the highest protection against effects such as water loss, impact, tear and puncture in citrus species in the light of biomimetic principles. It is aimed to use the information obtained from this study to develop new packaging approaches, materials and designs. Another aim of this study was to determine and compare the resistance of citrus species grown in our country as an agricultural product against external factors without additional packaging/coating/processing, and to obtain and compile information that can be used in estimating their shelf life.

Keywords: Biomimicry, Biomimetics, Exocarp, Evolution, Mechanical properties, Bio-inspired structures

ÖZET

NARENCİYE KABUKLARINDAN ALINAN İLHAM: BİOMİMETİK İLKELER İLE AMBALAJ TASARIMINA YAKLAŞIM

Tütüncüođlu, Bilge

Tasarım alıřmaları Yüksek Lisans Programı

Tez Danıřmanı: Prof. Dr. Murat Bengisu

Temmuz, 2022

Bu alıřmanın temel amacı, biyomimetik ilkeler ıřıđında, narenciye türlerinde su kaybı, darbe, yırtılma ve delinme gibi etkilere karřı en yüksek korumayı sađlayan ve ambalaj tasarımında kullanılabilir temel bilgileri elde etmektir. Bu alıřmadan elde edilen bilgilerin yeni ambalaj yaklařımları, malzemeleri ve tasarımları geliřtirmek için kullanılması amalanmaktadır. Bu alıřmanın bir diđer amacı da lkemizde tarım ürünü olarak yetiřtirilen narenciye türlerinin ek paketleme yapılmadan dıř etkenlere karřı direncinin belirlenmesi, karřılařtırılması ve raf ömürlerinin tahmininde kullanılabilir bilgilerin elde edilmesi ve derlenmesidir.

Anahtar Kelimeler: Biyotaklit, Biomimetik, Ekzokarp, Evrim, Mekanik Özellikler, Dođadan İlham Alınan Yapılar

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CHAPTER 1: INTRODUCTION

Plants have colonized virtually every environment while being unable to move from the site of seed germination, plants have evolved an arsenal of solutions that make them suitable for life in the most demanding and extreme conditions (Schäfer et al., 2020). One of their solutions is to shelter their embryos in a protective closed shell structure. Biological shells display a large morphological, biochemical and mechanical diversity across the kingdom of plant species (Huss et al., 2020). Research on layers of fruit shells so far has mostly focused on mechanical properties. Although it is important to the food, fibre and packaging industries, little has been done on the form-structure-function-relationship (Seidel et al., 2013). Therefore, the central question of this thesis is the following: How Can Biomimicry be used as a guide to develop innovative approaches in the packaging industry using citrus exocarps?

1.1 Purpose of this Study

The main purpose of this study was to obtain basic information that can be used in packaging design and provide the highest protection against effects such as water loss, impact, tear and puncture in citrus species in the light of biomimetic principles. For this purpose, it is aimed to compare the physical and mechanical properties of the outer skin (exocarp) of different citrus species, their resistance to water loss and effects such as impact, puncture and tear. The physical and mechanical properties of the outer shells has been associated with parameters such as porosity, density, microstructure and peel thickness.

Another aim of this study was to determine and compare the resistance of citrus species grown in our country as an agricultural product against external factors without additional packaging/coating/processing, and to obtain and compile information that can be used in estimating their shelf life. It is aimed to use the information obtained from this study to develop new packaging approaches, materials and designs.

1.2 Definition of Biomimicry

Biomimicry, as introduced by Janine Benyus (2009) in her book *Biomimicry—Innovation Inspired by Nature*, is focused on merging biological functions form, process and/or system, into various designs, constructing the connection between the

contexts of nature to design (Stevens et al., 2020). As for etymology of biomimicry, the word itself comes from the greek words *bios*, which means life, and *mimesis*, imitation. Biomimicry views nature as a model, mentor, and as a blueprint for further design developments (Benyus, 2009).

There are many terms synonymous with biomimicry, namely biomimesis, biognosis, bioinspiration, biomimetic design, bioanalogous design, biologically-inspired design. Biomimicry emulates natural models, systems, and processes to solved directed problems. Biomimicry is motivated by an understanding of natural selection, a process through which advantageous traits are perpetuated as the best adapted organisms tend to survive and reproduce in greater numbers than those with less effective adaptations. (Shu et al., 2011)



CHAPTER 2: LITERATURE REVIEW

2.1 Principles of Biomimicry

While practicing biomimicry, there are obvious three level of mimicry: the organism level, behavior level and ecosystem level. The first level of biomimicry is the organism level, mimicry of certain organism or a part of them. Compared to organism level, behavior level is more complex since it involves mimicry of behavior of which every organism behaves, involving relations of the organism to its larger context. Lastly ecosystem level biomimicry focuses on a building mimics of the natural process and cycle of the environment as a whole. Because of this, ecosystem level is considered the hardest level. (Benyus, 2009; Pathak, 2019).

In Fig 1. there is an in depth representation of level of biomimicry compared to the previous three levels. Additional to the three levels of biomimicry the vertical, horizontal axis represent five additional sub-levels of biomimicry. The biomimicry designs are categorized as their visual aspects (form), what it is made of (material), how it is constructed (construction), how it is works (process) and capacity of the design (function). According to these levels, biomimicry approaches are completed (Pathak, 2019).

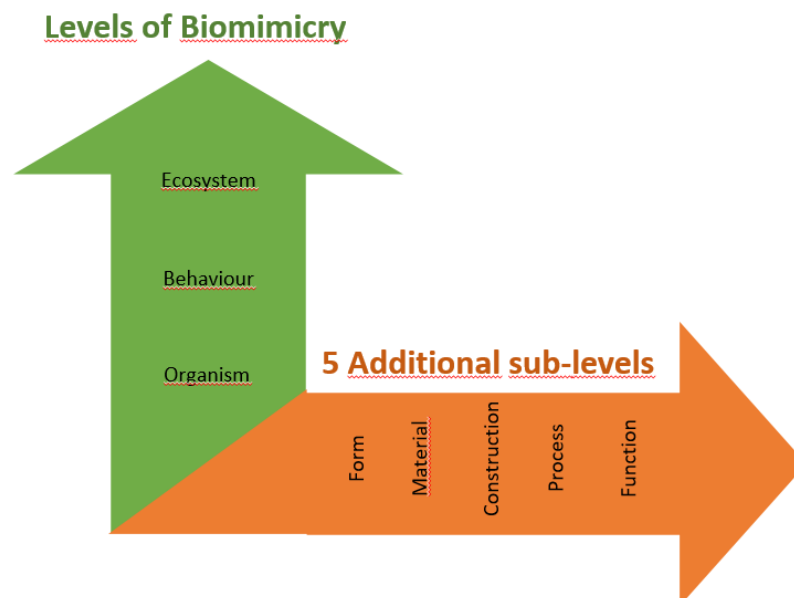


Figure 1. Levels of biomimicry (Source: Pathak, 2019)

Aside from levels of biomimicry, approaches to biomimicry has been categorized as well. Approaches to biomimicry fall into two main categories; top-down approach and bottom-up approach. If a biomimicry approach starts with defining a human need or a problem then searching the ways how other organism or ecosystems solve this, it is defined as top-down approach. If this approach starts by identifying a specific characteristic, behavior or function then applying them to the human design it referred as bottom-up approach (Aziz and El sherif, 2016).

2.2 Notable Examples of Biomimicry

Currently, the study and the extraction of biological key principles have been generally adopted worldwide. Moreover, their translation into design guidelines for a new generation of materials and technological solutions is a driving force in many research fields (Schäfer et al., 2020).

One of the most famous examples of design inspired by biological key principles is the Velcro. It was designed by Georgette Mestral in 1948. He was inspired by the how the hooks of the plant burrs (*Arctium lappa*) stuck in the fur of his dog. The sticking principle of nature was applied to the novel type of zip fastener (Schäfer et al., 2020). Paralell to this, Munari debated that orange is the best packaging available and discussed about the orange from the view point of a designer. Physical and morphological aspects of were compared with the desired aspects of the ideal packaging. Orange indeed has great packaging qualities such as; modular containers with segmentations, attractive color and soft internal lining to protect the product (Munari , 2008.).

In some cases, biomimicry methods surpass the capabilities of conventional manufacturing, e.g., fabrication in the nano and microregimes. Such example is the spider silk, often referred as one of the strongest biological materials in the world. Spintex Engineering have developed method to produce fiber at room temperature without harsh chemicals with water as a by-product (Fig.2). They inspired by spinning process of the spider, starts with gel-like liquid then turn into into a solid, through 'shear', or physical force within room temperature and without harsh waste products. (Spintex Engineering, 2022; Shu et al., 2011)

Another motivation to develop designs considering the biomimetic is that biomimetic manufacturing methods include the concepts of self-cleaning, self-assembly and self-organization found in biology (Shu et al., 2011). In terms of self-cleaning, lotus plants

were one of the most famous example that researched and studied worldwide. Lotus plant is capable of staying dirt-free in their natural muddy habitat. They can achieve this feature with their specialized wax-coated cuticle that is superhydrophobic and with their unique shape. While wax coating repell the liquids, micro-topography of lotus leaf surface allows liquid droplets to pass by resulting in self-cleaning function (Fig. 2). This aspect has been applied to paints, glass, textiles, and more, reducing the need for chemical detergents and costly labor (Asknature, 2020)



a) Vials of synthetic silk



b) Lotus leaf surface and lotus inspired coating

Figure 2. Applications of biomimicry (Source: Spintex Engineering, 2022; Asknature, 2020)

2.3. Morphological Analyses on Citrus Peel

The mechanical harvesting causes damage to the fruits this results in various unwanted changes in physio-mechanical properties of fruits. Therefore, measurement of post-harvest properties of fruits is essential for adaptation of design of various handling, packaging, storage and transportation systems (Singh and Reddy, 2006).

One of the issue stated by the previous researches is the vast range of variability in oil gland size and shape that was found within citrus peels. This variety could be due to various such as timing of harvest and stage of fruit development. Caputo et al., used Light and scanning electron microscopy (SEM) analyses to observe oil glands of mature fruits and compare these results among three citrus species. Oil glands were compared in size, gland density (number/mm²) and yields of essential oil (Caputo et al., 2020).

Free fall test was conducted on fruits for measuring their impact resistance. One of the prominent example for this test was Pummelo. Their fruit weight up to 6 kg and a maximal height of the fruit bearing trees of 15 meters resulting in high potential energy on the citrus fruit. If their high potential energy causes fruits to macroscopically damaged, their fruits won't survive at the tropical climate of the Southeast Asia. As a solution to this problem, pummelo fruits evolved their structure to dissipate large amounts of energy and to withstand deceleration forces of several kN without being macroscopically damaged. Pummelos were dropped from the height of 10m (n=6), 13.5m (n=2), and 18m (n=2) onto a concrete floor. After this, the pummelos were analyzed macroscopically by eye (Fischer et al., 2010).

Tensile tests were conducted to analyze the behavior of the citrus peel under applied tensile loads measuring their rupture force, tensile strength and modulus of elasticity. In the test of Singh and Reddy, peel pieces were carefully dissected from the equator of five randomly selected samples then cut into peel strips of 15 mm (polar) and 60 mm (equatorial). Rupture force was taken as the maximum peak force required to rupture the peel of the citrus. The test conducted by Putra et al also follows similar principles in tensile testing. In this test, biodegradable plastic films made from dragon fruits were cut length of 50 mm and a width of 10 mm then fixed to a universal tensile test and pulled at a constant speed and a maximum load of 5 kgf. From the results of the tensile test elongation and tensile strength was calculated (Putra et al., 2019; Singh and Reddy, 2006).

Another aspect that tensile test can show is that how much storage span of the fruits effect their skin failure. Masoudi et al., experimented in six batches within six months was conducted and apparent modulus of elasticity, failure stress, failure strain, failure energy, and toughness were tested. Apples from three species were stored next to one another at 4 to 5°C with 65 to 70% with relative humidity. Later, changes between batches were plotted and discussed (Masoudi et al., 2007). Addition to the tensile test, Pitts et al. debate that three point bending as an alternative experimental method to measure tensile elastic modulus and firmness of the fruits. (Pitts, Davis and Cavalieri, 2008).

Fluid-loss experiment is another common test for measurement of fresh fruits. In the experiment of the Purvis, ten whole fruit samples were numbered with a felt-tip pen and weighed at the start of each experiment and weighted daily. For 82 days their Storage room temperatures were maintained at 5°C and 21 °C for the entire time with

relative humidity of the storage rooms ranged from 85 to 95% (Purvis, 1984). In the experiment of Masoudi et al., electric oven was used to dry the specimens. Firstly, randomly selected nine apples from three different species were stored in batches for were weighted using a scale with 0.1 g accuracy. Then they were placed in an aluminum box, and positioned inside an electrical oven for 10 days at 77°C. Their fluid-loss percentage were determined by using the weight of the apple samples before and after drying and the standard wet-basis moisture content equation. (Masoudi et al., 2007)

2.4 Design with Citrus Peel

Citrus fruit peels are usually misspent byproducts in the industry, although they hold valuable potential to be used in many different ways. Underutilization of the fruits peels also represent growing problems, such as microbial spoilage to the environment. Designing and utilization with this uncharted material was limited with factors such as cost of drying, storage and shipment. (Caputo et al., 2020)

Citrus peels generally utilized in the production of drinks, juices, and liqueurs. After their essential oil extracted, the oils were commonly used for cosmetic and food industry (Caputo et al., 2020). After citrus fruits have been utilized in the industry, about 60% of the product is left over as a bio-waste. Fasson Crush Citrus by Favini (Fig 3.), utilizes bio-waste of citrus peel after their essential oil were extracted. It's label contains post-consumer waste (40%) and citrus pulp (15%) (Anon., 2022a). Left-over orange peels could be re-purposed as a packaging as well. A British student at Brunel University London developed a usable food packaging from orange peels (Fig. 3). This packaging consists of biopolymers, vegetable glycerine, orange peel bio-waste and water (Anon., 2022c).

In the citrus industry, sustainability is also an important aspect to consider while designing. Aphytis by Koppert Spain was redesigned for better sustainability. Their packaging is made from 100% biodegradable cardboard and bioplastic PLA. This packaging contained film of natural cotton with a sugar solution and strips of green films have (Fig. 3) parasitic wasp to biocontrol of the number of scale insects used by citrus farmers (Koppert ,2021). Citrus peels were studied in textile industry as a sustainable alternative to the leather and traditional textiles. In Fig. 4 handbag made from citrus peel leather mixture stabilized because of pectin inside the albedo (white part of the citrus peel) since it performs a firming and water-regulating function and

contributes to the stabilization of the citrus peel leather (Luckynelly, 2021). In fig. 4 , orange fiber textile consist of repurposed orange peel bio-waste from juice industry. It still contains essential oil and vitamins of the orange peel due to the nanotechnology. This fiber is available in various color both with options of opaque and matte that can be combined with other yarn types (Orange Fiber, 2018).



a) Aphytis by Koppert



b) Fasso Crush Citrus by Favini



c) Citrus peel packaging

Figure 3. Citrus peel packaging design (Source: Koppert, 2021; Anon., 2022a; Anon., 2022c)



a) Citrus Leather Handbag



b) Orange Fiber Fabric

Figure 4. Orange peel in textile design (Luckynelly, 2021; Orange Fiber, 2018)

CHAPTER 3: METHODOLOGY

3.1 Materials and Methods

In this study, several types of citrus fruits were used such as; oranges, sour oranges, grapefruit, lemon, bitter orange and tangerine harvested from several regions across Turkey (Table1).

Table 1. Types of Citrus Fruit Used in this Study and Region of Harvest

Citrus Fruit	Species of Citrus Fruit	Harvested Region
Oranges	Washington	Mersin, Turkey
	Finike	Alanya, Turkey
Sour Oranges	Seville	Balçova, Turkey
Grapefruit	Star Ruby	Alsancak, Turkey
Lemon	Kütüden	Antalya, Turkey
	Lizbon	Antalya, Turkey
Tangerine	Satsuma	Özdere, Turkey

Different types of citrus fruit peel have been analyzed with different methods (Table 2). Citrus fruits peel (Exocarp) consists of two main parts - epicarp (flavedo) and mesocarp (albedo). Epicarp is the outermost layers of peel, that contains pigments and large follicles of oil glands. Mesocarp is located just below the epicarp, it is white and spongy. (Caputo vd. 2020). Particular attention has been paid to the properties of the peels to favor the fruit/mechanical preservation. (Table 2).

Table 2. Testing Methods and Related Properties

Mechanical or Physical Property of Interest	Selected Method
Energy Absorption	Free Fall Experiments
Tensile strength, strain at rupture	Mini Tensile Test, 3-Point Bending Test
Fluid loss	Fluid-Loss Test
Morphology	Analysing Under Digital Microscope

3.2. Energy Absorption Analysis Methods

Fruits provide many excellent structures for energy absorption in order to protect their integrity (Seidel et al., 2013). Especially the pericarp of fruits evolved in such a way to provide protection for the plant embryo against the sharp teeth of animals or impact forces on the ground (Ha et al., 2020). That's why the relationship between energy absorption and the fruit should be evaluated.

To measure citrus fruit impact strength, free fall experiments were used (Fischer et al., 2010). The fruits were dropped from heights of 6.3 m (n = 6), 10.5 m (n = 2), and 18 m (n = 2) onto a concrete floor. The impact strength was evaluated qualitatively by visual inspection. Only a single piece of fruit was used for each fruit type in the pilot test. In the final test four pieces of fruit were used for each fruit type. The fruits were dropped from a single height of 14.3 m to compare the relative damage that occurred in the fruits. In order to not confuse identify the samples from each other, clearly, sample numbers were written drawn on to their skin. Then photographs of each sample were taken from side and bottom before and after the test (Table 4).

3.3. Measurement of Mechanical Properties of Wet and Dry Peels.

Using the ASTM D638M method to determine the tensile strength (MPa) and elongation at break (ϵ ,%), specimens were cut into rectangular strips (2 cm x 8 cm) and the initial grip separation was set at 60 mm and the test speed at 0.1 mm/s. Five measurements were taken from each sample. The tensile strength of the samples were calculated by dividing the maximum load by the cross-sectional area of the sample, and the elongation at break (ϵ ,%) was calculated by the Eq. 1 of this thesis (Churchill et al., 1980).

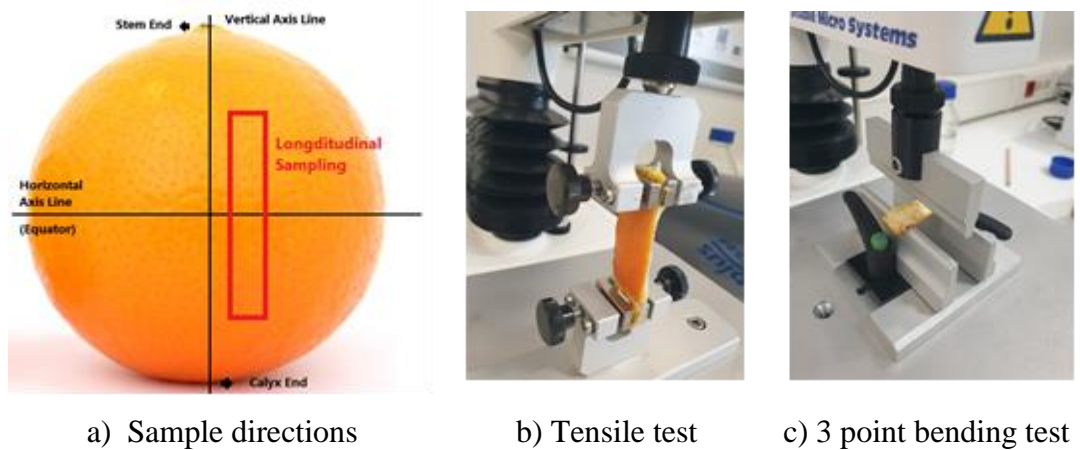


Figure 5. Specimen and Tests

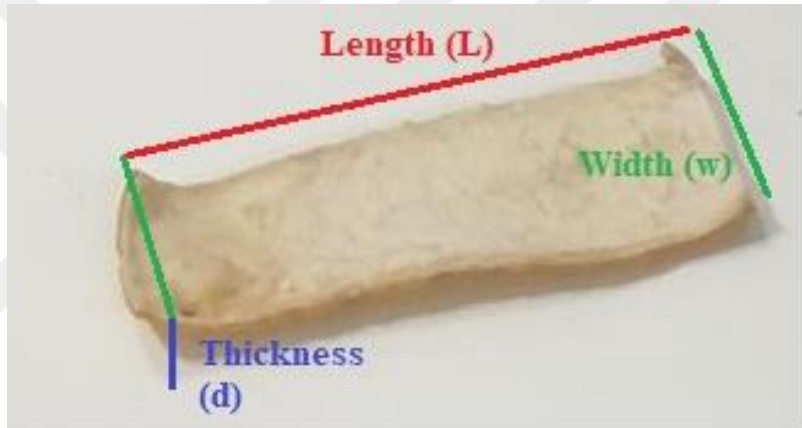


Figure 6. Sample dimensions

3.4. Fluid-Loss Measurement Methods

Transpiration (fluid loss) and respiration are major factors determining physiological deterioration (Purvis, 1984). Therefore, peel contribution over fruit physiological deterioration was investigated.

To compare the ability of citrus peels to prevent fluid loss, after washing and cleaning, five samples of each fruit type were kept in the laboratory at random locations for up to 6 weeks without any coating or treatment, and their weights were measured every week (Galed et al., 2004). A ranking was made in terms of the protectiveness of different fruit skins, taking into account the weight losses, peel thickness, density, and porosity obtained from these measurements.

3.5. Morphological Analysis Methods

Morphological features of citrus peels were examined with an optical microscope. For optical microscope analysis, very thin sections obtained from the epicarp and mesocarp layers of citrus peels and their morphology and porosity were determined under the light microscope. In addition, the surfaces of the shells were examined directly by a USB digital microscope.



CHAPTER 4: RESULTS AND DISCUSSION

4.1 Evaluation of Energy Absorption Analysis

Our free fall tests were conducted with five whole citrus fruits from the same drop height as explained in Chapter 3. This way the citrus types can be compared within each other. Fruits were visually analyzed at a macroscopic scale by eye, evaluated comparatively by their crack sizes and numbers. After the test, potential energy was calculated (Table 3), crack sizes were demonstrated and relative damage of the whole fruit was displayed (Table 4) (Fischer et al., 2010).

Table 3. Free Fall Test Results

Grapefruit	Weight (kg)	Peel Thickness (mm)	Potential Energy (J)
1st Sample	0.273	5,2	38,2
	Damage: Four Cracks(\cong 1 mm) + Slightly Deformed (Maintaining Sphere Shape)		
2nd Sample	0.232	6,3	32,5
	Damage: Three Large Cracks (\cong 8 mm) + Slightly Deformed (Maintaining Sphere Shape)		
3rd Sample	0.231	7,7	32,4
	Damage: Four Cracks (\cong 6mm)+ Slightly Deformed (Maintaining Sphere Shape)		
4th Sample	0.263	5,5	36,5
	Damage: Two Large Cracks (\cong 70 mm)+ Slightly Deformed (Maintaining Sphere Shape)		
5th Sample	0.247	5,7	34,8
	Damage: Four Cracks (\cong 2 mm)+ Deformed (Crooked Shape)		

Table 3. (continued) Free Fall Test Results

Lemon	Weight (kg)	Peel Thickness (mm)	Potential Energy (J)
1st Sample	0.117	4,66	16,3
	Damage: Two Cracks ($\cong 4$ mm) + Slightly Deformed (Maintaining Sphere Shape)		
2nd Sample	0.117	4,33	16,3
	Damage: Two Cracks ($\cong 5$ mm) + Slightly Deformed (Maintaining Sphere Shape)		
3rd Sample	0.104	7	14,5
	Damage: Two Cracks ($\cong 8$ mm)+ Slightly Deformed (Maintaining Sphere Shape)		
4th Sample	0.043	4,5	6,0
	Damage: Two Cracks ($\cong 4$ mm)+ Slightly Deformed (Maintaining Sphere Shape)		
5th Sample	0.188	7	26,3
	Damage: Two Cracks ($\cong 8$ mm)+ Slightly Deformed (Maintaining Sphere Shape)		
Orange	Weight (kg)	Peel Thickness (mm)	Potential Energy (J)
1st Sample	0.257	6,83	35,9
	Damage: Four Large Cracks ($\cong 5$ mm) + Heavily Deformed (detached piece/pieces)		
2nd Sample	0.277	7,73	38,7
	Damage: No Cracks + Heavily Deformed (detached piece/pieces)		
3rd Sample	0.240	5,16	33,6
	Damage: No Cracks + Heavily Deformed (detached piece/pieces)		
4th Sample	0.236	7,83	33,0
	Damage: Two Large Cracks ($\cong 6$ mm) + Heavily Deformed (detached piece/pieces)		
5th Sample	0.244	4,16	35,1
	Damage Four Cracks ($\cong 4$ mm) + Heavily Deformed (detached piece/pieces)		

Table 3. (continued) Free Fall Test Results

Sour Orange	Weight (kg)	Peel Thickness (mm)	Potential Energy (J)
1st Sample	0.163	5	22,8
	Damage: Two Cracks (≈ 4 mm) + Slightly Deformed (Maintaining Sphere Shape)		
2nd Sample	0.173	3,3	24,1
	Damage: Two Cracks (≈ 5 mm) + Slightly Deformed (Maintaining Sphere Shape)		
3rd Sample	0.155	6	21,7
	Damage: Three Cracks (≈ 5 mm) + Slightly Deformed (Maintaining Sphere Shape)		
4th Sample	0.146	5,3	20,4
	Damage: Two Cracks (≈ 9 mm) + Slightly Deformed (Maintaining Sphere Shape)		
5th Sample	0.063	4,6	8,83
	Damage: One Crack (≈ 5 mm)+ Slightly Deformed		
Sour Orange	Weight (kg)	Peel Thickness (mm)	Potential Energy (J)
1st Sample	0.163	5	22,8
	Damage: Two Cracks (≈ 4 mm) + Slightly Deformed (Maintaining Sphere Shape)		
2nd Sample	0.173	3,3	24,1
	Damage: Two Cracks (≈ 5 mm) + Slightly Deformed (Maintaining Sphere Shape)		
3rd Sample	0.155	6	21,7
	Damage: Three Cracks (≈ 5 mm) + Slightly Deformed (Maintaining Sphere Shape)		
4th Sample	0.146	5,3	20,4
	Damage: Two Cracks (≈ 9 mm) + Slightly Deformed (Maintaining Sphere Shape)		
5th Sample	0.063	4,6	8,83
	Damage: One Crack (≈ 5 mm)+ Slightly Deformed		

Table 4. Images of samples after the test










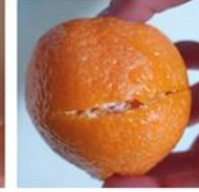








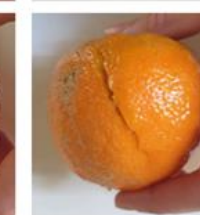































Mandarin (Top row: Top view Bottom Row: Side view)				
1 st Sample	2 nd Sample	3 rd Sample	4 th Sample	5 th Sample
				
				
Sour Orange (Top row: Top view Bottom Row: Side view)				
1 st Sample	2 nd Sample	3 rd Sample	4 th Sample	5 th Sample
				
				
Orange (Top row: Top view Bottom Row: Side view)				
1 st Sample	2 nd Sample	3 rd Sample	4 th Sample	5 th Sample
				
				

Table 4. (continued) Images of samples after the test

Lemon (Top row: Top view Bottom Row: Side view)				
1 st Sample	2 nd Sample	3 rd Sample	4 th Sample	5 th Sample
				
				
Grapefruit (Top row: Top view Bottom Row: Side view)				
1 st Sample	2 nd Sample	3 rd Sample	4 th Sample	5 th Sample
				
				

These drop tests caused different outer damages depending on the fruit type. Based on table 4, it is hard to clearly determine which exocarp is more efficient in absorbing the impact. In terms of maintaining shape and having less cracks, sour orange was more efficient. When exocarp thickness is considered, lemon is more successful since its peel is thinner than that of sour orange.

Interestingly, being damage absorbant is inversely proportioned to the potential energy of the fruit. Schäfer et.al. debated that pomelo fruit were adapted to squander potential energy resulting from falling from a height of up to 15 meters and the heavy weight of the fruit (up to 6 kg) without being macroscopically damaged (Schäfer et al., 2020). In our tests, the fruits with larger potential energy (orange and grapefruit) were relatively lesser protective compared to the other fruits (sour orange and lemon).

Two oranges exploded into two pieces around pedicel region (sap of the fruit), exposing the pulp. Other three remained in a single piece but their pup was damaged. Both oranges had their juice seep and small pieces were scattered to the concrete. Based on this data, oranges have lower damage absorbent qualities.

Lemon and grapefruit fractured twice from fruit's equator. Their juices were spilled and the whole fruit was deformed slightly. But lemons maintained their shape compared to the grapefruit and had less cracks. This could be explained by their different potential energy and exocarp structure difference. Although they have approximate exocarp thickness, the grapefruits have more than double amount of potential energy than lemons.

4.2 Evaluation of Tensile Strength and Strain at Rupture

In the methodology, it was mentioned that our plan was to cut samples into rectangular strips (2 cm x 8 cm). Unfortunately, small citrus species such as lemon and mandarin doesn't have enough space for this size. Samples were cut to the size of 2 cm x 6 cm instead of 2 cm x 8 cm. There was +/- 3 mm tolerance in the initial sliced samples.

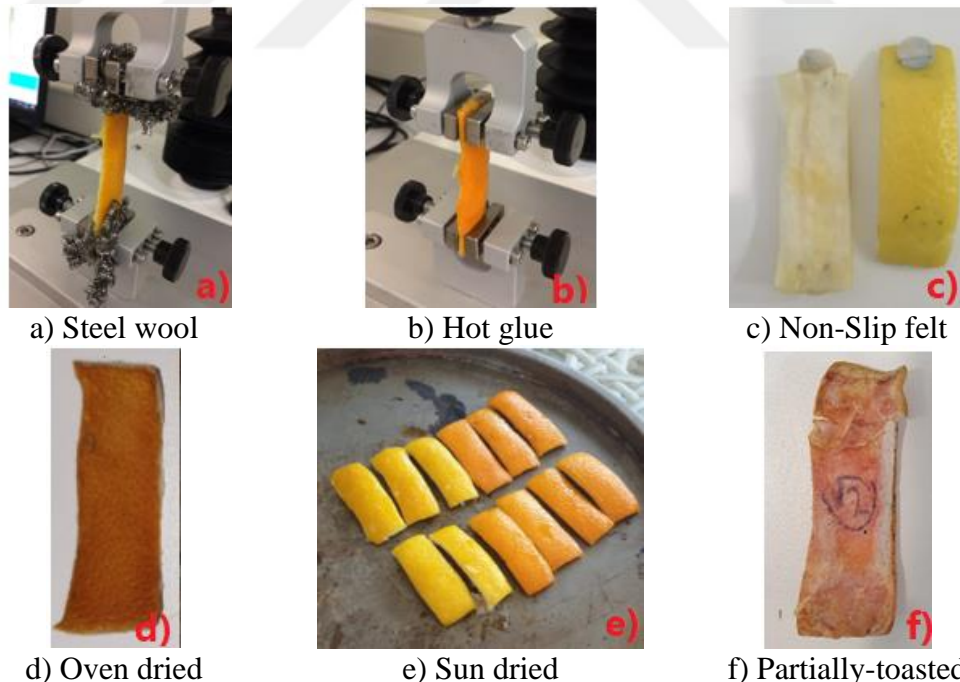


Figure 7. Methods used for preparing samples

A problem faced in mini tensile tests, was that the issue of the samples was slipping past the tensile grips while testing. Unfortunately, there weren't any solutions in the literature about this issue. For overcoming this, six methods were tested (Fig. 7). The

first two methods (a,b) were eliminated due to mini tensile grip width limitation. Method c was not suitable since it teared the surface during testing. Method d was rejected because the samples deformed during drying. Method e was applied to the three-point bending. Since wet samples can't be applied to this test, method e was selected due to least crooked drying process. Method f (drying the grip ends) was the most successful so it was applied to the mini tensile samples.

4.2.1. Mini Tensile Tests

In this test, the focus was on observing the behavior of the citrus peels under applied tensile loads. Parameter coefficients such as brittleness and flexibility was obtained with a texture analyzer (Brookfield Texture Analyzer, AMETEK, USA). Citrus fruits were peeled carefully dissected perpendicularly to the equator with a caliper and lancet (ASTM, 1983), (ASTM, 1993).

$$e = \frac{(L_{max} - L_0)}{L_0} \quad (1)$$

$$\sigma = \frac{F}{A} \quad (2)$$

Using the ASTM D638M method to determine the tensile strength (MPa) and elongation at break (ϵ ,%), specimens were cut into rectangular strips (2 cm x 6 cm) and the test speed was adjusted to 0.1 mm/s. Ten samples were selected from each citrus fruit . Tensile strength σ (P) was calculated by dividing the peak rupture force (N) to the cross-sectional area (A=thickness x width, m²) of the initial specimen (Singh and Reddy, 2006) The change in elongation was measured from variant such as L_{max} (peak rupture length) and L_0 (starting length).

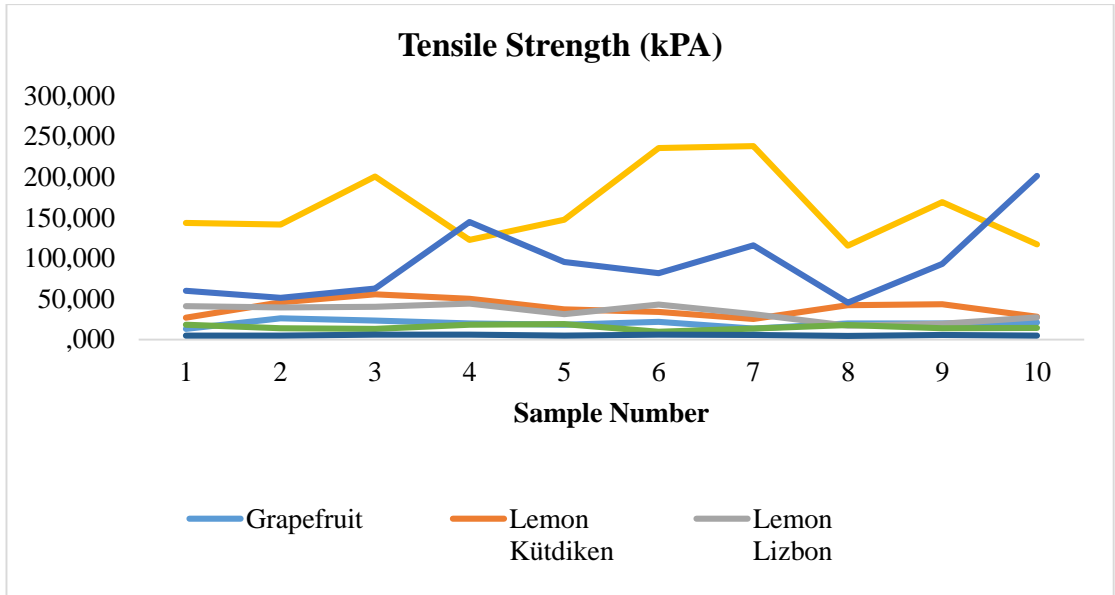


Figure 8. Tensile strength results of different citrus fruit peels

Table 5. Tensile strength of different citrus fruit peels (KPa)

	Maximum	Minimum	Average
Grapefruit	26,2	13,2	19,8
Lemon Kütdiken	56,0	26,0	39,1
Lemon Lizbon	44,5	17,2	33,6
Mandarin	238,4	116,0	163,5
Orange Finike	201,4	45,5	95,3
Orange Washington	19,1	9,7	15,3
Sour Orange	6,2	4,7	5,4

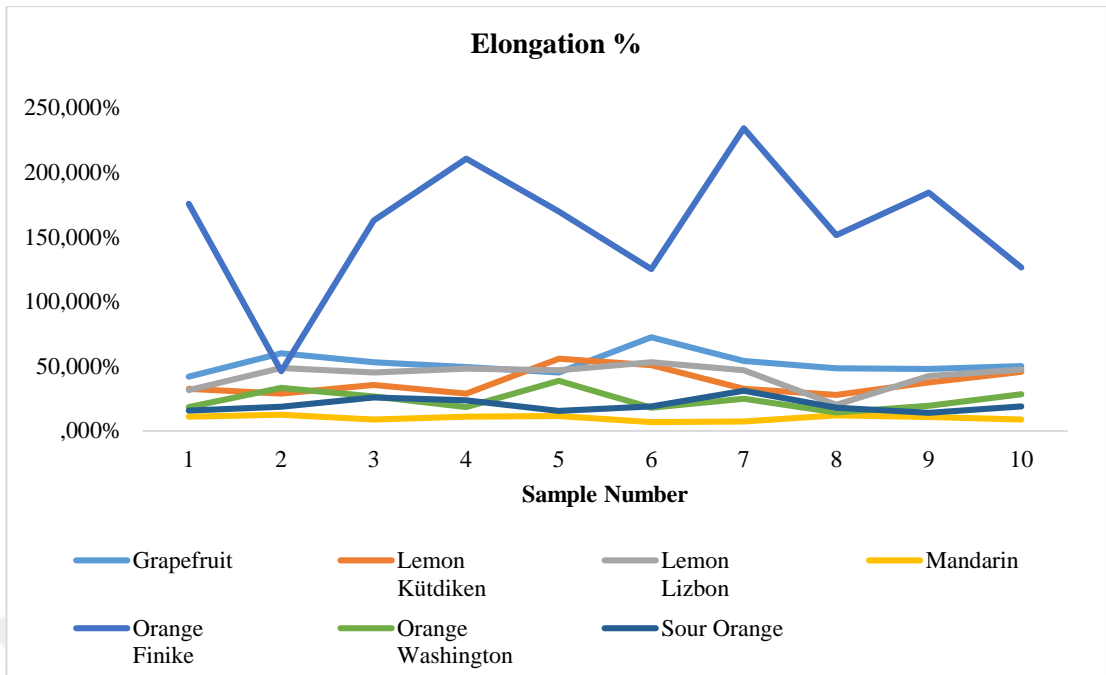


Figure 9. Elongation of different citrus peels

Elongation is the change in the length of the sample due to applies force. The extension until break gives us the elasticity of the material. The higher the extension (%) value the more elastic the material, the lower the extension value the more fragile the material (Putra et al., 2019).

The results of the measurement of the elongation can be seen in Figure 9. From Table 6, we can see that the samples have demonstrate a percent elongation ranging from 234, 2 % to 6, 8 %. As you can be seen, there is a wide spectrum of results between species of fruits.

Putra Endo et al. debated that measurement results of the percentage of elongation are inversely proportional to the tensile tests. We could see the same statement in our measurements. The sharp decreases, increases and large variations in elasticity could be interpreted as decreasing intermolecular bond distance and structure of fruit. The reduction of the intermolecular force between the chains causes the chain movement to be freer so that flexibility increases (more elastic) (Putra et al., 2019). By structure of fruit it could be interpreted as uneven distribution of peel tissue cell and gaps (Juxia, et al., 2015)

Table 6. Elongation analysis table

	Maximum	Minimum	Average
Grapefruit	72,5%	41,8%	52,2%
Lemon Kütüden	55,8%	27,9%	37,6%
Lemon Lizbon	53,0%	20,2%	43,0%
Mandarin	12,4%	6,8%	10,0%
Orange Finike	234,2%	46,2%	158,7%
Orange Washington	38,6%	14,2%	24,0%
Sour Orange	31,1%	13,9%	20,0%

$$\sigma_{fl} = \frac{M(\frac{d}{2})}{I} \quad (3)$$

$$M = (\frac{F}{2})\alpha \quad (4)$$

$$M = (\frac{L}{2})(\frac{F}{2}) \quad (5)$$

$$I = \frac{(wd^3)}{12} \quad (6)$$

4.2.2. Three Point Bending Tests

In three-point bending M is the moment and I is the moment of inertia. For three-point bending, where L is length between the two contact points, w is width, and the d is the thickness of the sample (Bengisu, 2001) (Fig. 6). Fruits must be sturdy enough to resist damage during packaging, storage, and transportation to the market. To measure the efficiency and performance of the peel as a packaging, three-point bending tests were executed. For this test, moment (Nm), moment of inertia (m⁴) and three-point bending strength (MPa) were calculated and plotted as a graphic.

In general, biological materials exhibit common characteristics such as low compressive elastic modulus, a large compressive failure strain, and they exhibit plastic failure in compression (Pitts et al., 2008). These characteristic could be monitored in following data charts as well.

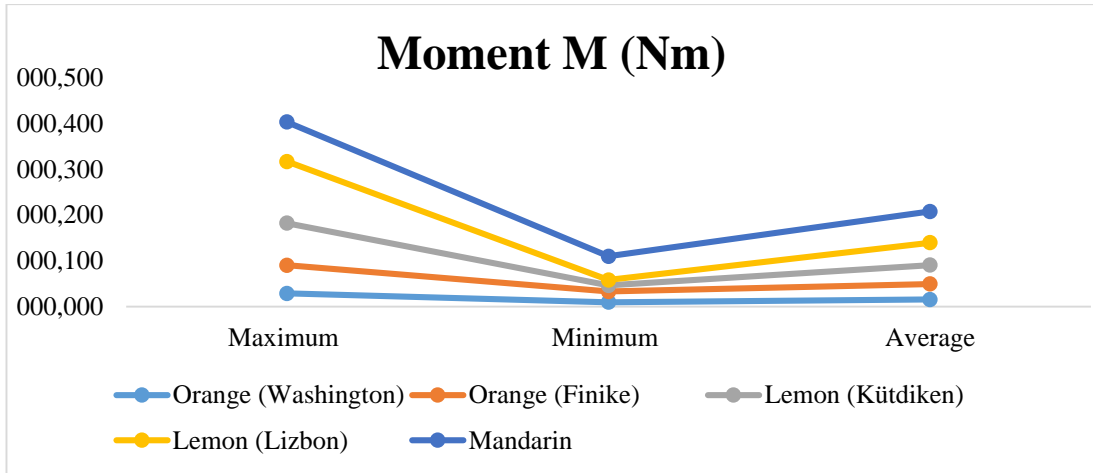


Figure 10. Moment N (Nm) analysis results

Table 7. Moment M (Nm) analysis table

	Maximum	Minimum	Average
Orange Washington	0,02926	0,00998	0,01598
Orange Finike	0,06133	0,02339	0,03371
Lemon Küttdiken	0,09188	0,01309	0,04119
Lemon Lizbon	0,13435	0,01209	0,04888
Mandarin	0,08620	0,05154	0,06816

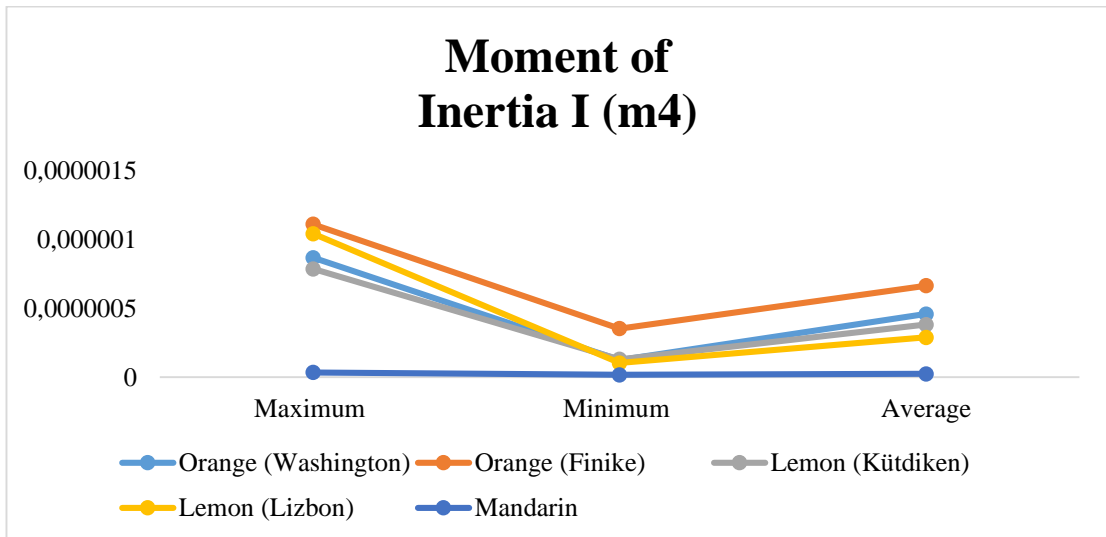


Figure 11. Moment of Inertia I (m4) analysis results

Table 8. Moment of Inertia I (m4) analysis table

	Maximum	Minimum	Average
Orange Washington	8,66166E-07	1,24097E-07	4,58474E-07
Orange Finike	1,10963E-06	3,53427E-07	6,63275E-07
Lemon Kütdiken	7,85122E-07	1,3058E-07	3,81779E-07
Lemon Lizbon	1,04071E-06	1,03571E-07	2,89304E-07
Mandarin	3,53717E-08	1,74361E-08	2,43536E-08

The higher the Flexural Modulus, the stiffer the material; the lower the Flexural Modulus, the more flexible it is. It could be stated that three-point bending strength (MPa) is directly proportional to the moment but in directly proportional to the moment of inertia.

At Fig. 10. and Fig 11, where we could see Moment of Inertia and Moment values, stiffness of peel indicates higher moment need to tear. So it could be stated that for better packaging or peel performance, moment of inertia should be lower, moment and three-point bending strength should be higher. According to this reasoning, out of five species, highest to lowest packaging performances were; mandarin, lemon (Lizbon), lemon (Kütdiken), orange (Washington), orange (Finike). Also this ranking is indirectly proportional to the peel thickness.

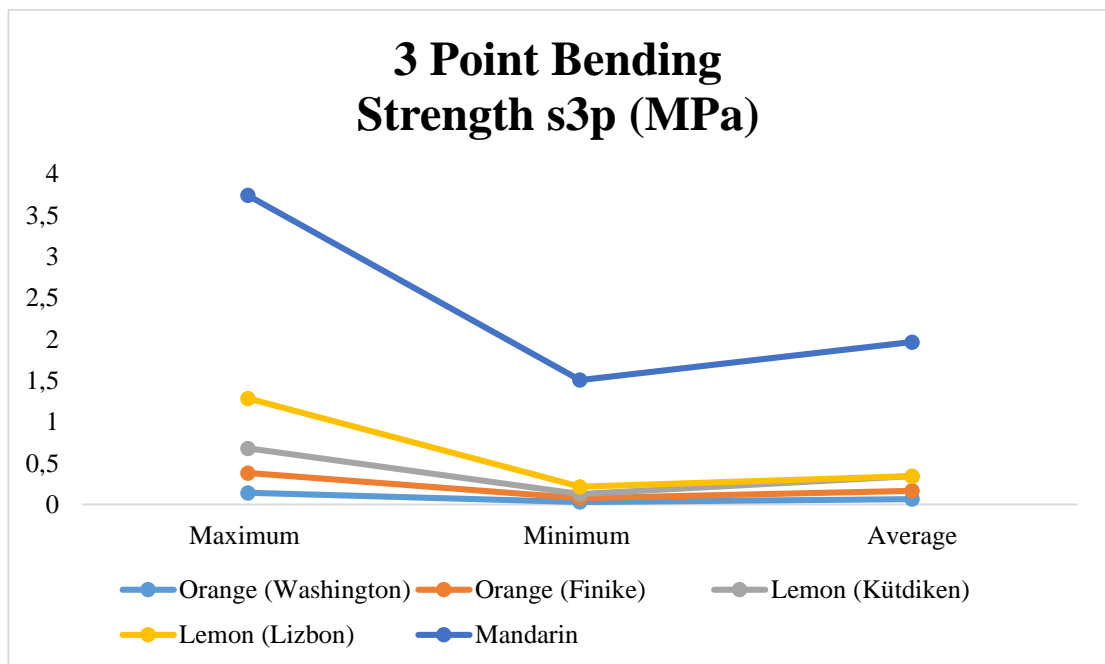


Figure 12. 3 Point Bending Strength s3p (MPa) analysis results

Table 9. 3 Point Bending Strength s3p (MPa) analysis table

	Maximum	Minimum	Average
Orange Washington	0,139	0,0306	0,0647
Orange Finike	0,241	0,046	0,1
Lemon Kütüden	0,297	0,048	0,117
Lemon Lizbon	0,604	0,090	2,893
Mandarin	2,458	1,291	1,623

4.3 Evaluation of Fluid-Loss Measurement Methods

Fluid loss from citrus fruits measured as a whole fruit, peel and fruit to determine the efficiency of the fruit peel as a packaging. This way, it was possible to measure the effect of peel on protecting the fruit itself. According to Fig. 13, citrus fruit sections were named and divided for measurement (Aruoma et al., 2012).

Weight loss was determined by selecting five samples from selected citrus species then weighting them. Fruits were measured by weighing (± 0.1 g) each fruit daily for 3 weeks. Relative weight loss was evaluated as a percentage of the starting weight. These samples were kept at different locations in a well ventilated, constant-temperature room between 22-25oC (Hagenmaier and Baker, 1995).

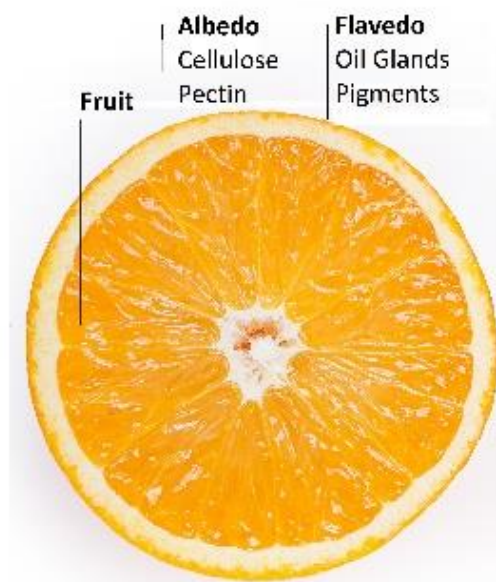

















Figure 13. Citrus fruit cross section

Table 10. Fruits before and after fluid-loss test

	Fruit and peel (Before)	Whole fruit (Before)	Whole fruit and peel (Before)
Lemon			
Grapefruit			
Orange			
Mandarin			
Sour Orange			

Washed, uncoated samples were divided into peel and fruit or left as whole. As seen in Table 10, specimen photos were gathered to spot visual differences between day 0th day and day 28th of the experiments. Average data from five samples from each species determined and shown in graphic form (Figs.14 and 15). This way we could spot the relation between peel, fruit and whole fruit more clearly. Peels shrank in an uneven way, resulting in asymmetric shapes. Both whole fruit and fruit itself shrank in more uniform manner.

Color change is another aspect to measure since possible color changes would influence the organoleptic properties (being of a substance that stimulate the sense

organs) of dried citrus peel samples and would limit their potential applications (Manjarres-Pinzon et al., 2013). Although there are color differences between the peels before and after drying tests, mandarin fruit has the most distinct difference. This aspect of mandarin would not be suitable since brighter and lasting colors on packaging is favored. In this aspect oranges and sour oranges would be a better choice since they have less color change compared to the other specimens.

Larger citrus fruits were expected to have lower rates of moisture loss compared to the smaller citrus fruits. It is due to higher surface area to volume ratio to the later one. Also it was expected protruding areas, such as neck of the fruit, to lose weight faster than the smoother surfaces. According to the Lufu et al. (2020), the rates of weight loss should be, from low to high, grapefruit, sour orange, orange, lemon, mandarin. But the outcome in Fig. 14, is somewhat different: orange, grapefruit, sourorange, lemon, mandarin. Although oranges used in this study (Average 0th day weight: Orange 190,0 gr. Grapefruit: 266,4 gr. Sour orange: 194,1 gr.) are smaller than grapefruits and sour oranges, they have lower rates of moisture loss. Lower rates of moisture loss is a critical feature in fresh fruit and vegetable packaging.

The liquid loss graphic in Fig. 15 is also important since citrus peels represent approximately 30-40 g/100g of the fresh fruit weight and could be used to develop value-added product (Manjarres-Pinzon et al., 2013). Movement of the peel graphic in Fig. 15 aligns with the previous statement since graphic lines comes halt in %70-72 at 3th day. In the same graphic, fruit liquid loss rate of lemon and sour orange is stated. Comparing these data to the whole fruit data of the same species could indicate the performance of the citrus fruit as a packaging. At day 28th, sour oranges inner fruit liquid loss (without the peel) is at -64,4% and whole citrus fruit (with the peel) is at -31,43%. As for Lisbon lemon, inner fruit weight loss is at -41,6% and the whole fruit weight loss is at -36,9%. Comparing the difference of weight loss, it can be concluded that sour orange peel has better moisture preserving quality between the two. However, overall, it can be seen that the best performance is displayed by oranges (Fig. 14). On average, oranges lost only 27% of their weight due to loss of moisture in 28 days while the closest to them were sour oranges with 31% weight loss and the worst performance was observed in lemons with 37% weight loss.

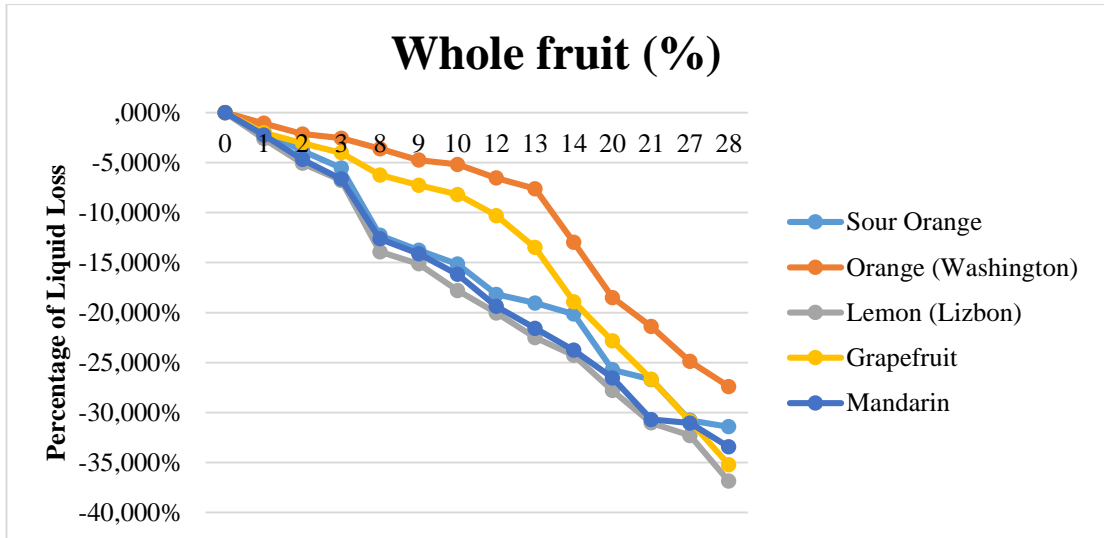


Figure 14. Whole fruit liquid loss

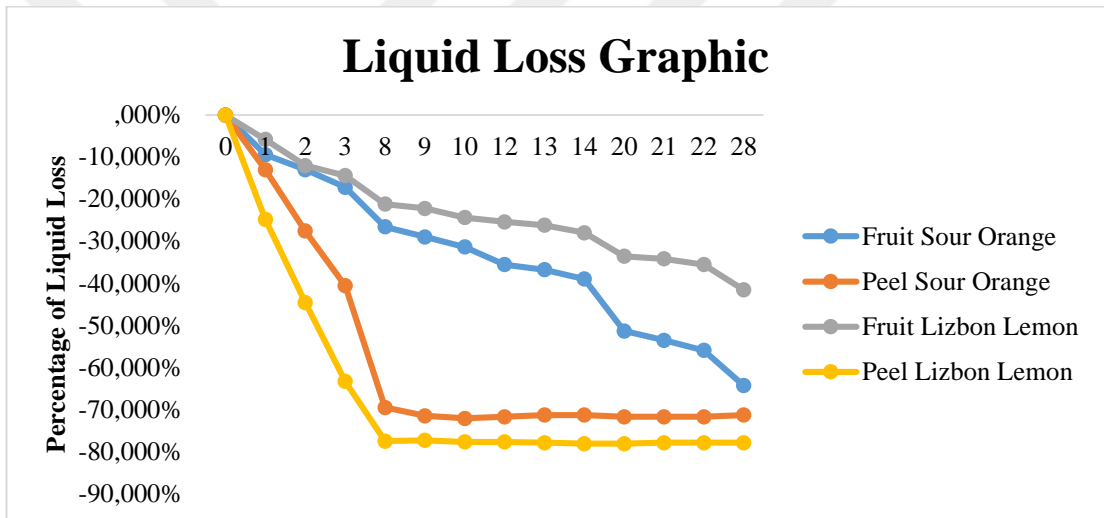


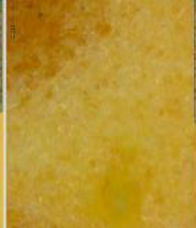



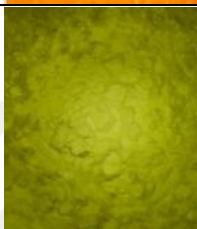
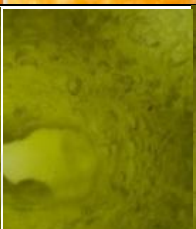
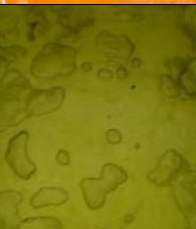


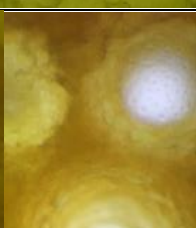
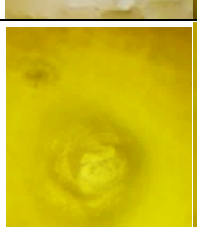


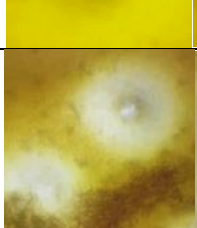

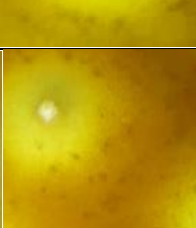


Figure 15. Fruit and peel liquid loss

4.4 Evaluation of Morphology under Digital Microscope

In this test, the main goal was to evaluate the structure of the peel visually. Lemon (Lizbon), sour oranges and oranges (Valencia) were used as samples. We used AM7025X and AM4113T5 model optical microscope. Sample peel were divided into their flavedo and albedo layers and photos were taken at 10X magnification.

Table 11. Albedo and flavedo analysis results (Bar = 0,5mm)

Orange Albedo					
Orange Flavedo					
Sour Orange Albedo					
Sour Orange Flavedo					
Lemon Albedo					
Lemon Flavedo					

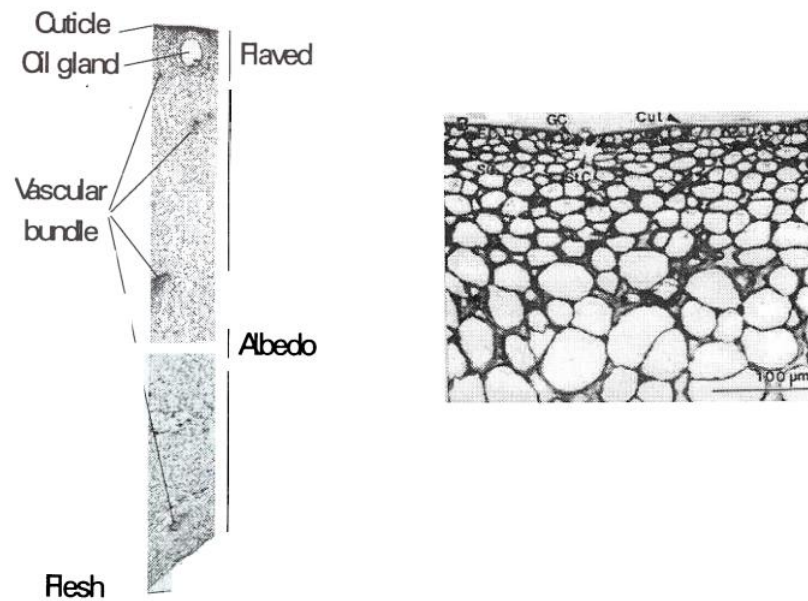


Figure 16. Anatomy of white grapefruit (left, bar equals 0.5mm) and close up of flavedo (right) (Source: Albrigo, 1986)

Citrus peels consist of two main components: the albedo and the flavedo (fig. 9), (fig. 12). The albedo primarily consists of vascular bundles and cells. These cells of the albedo play an important role in providing water to peel and the fruit. The flavedo section is rich in pigments and contains oil glands which is covered by cuticle. The positioning of the flavedo between environment and the fruit, affects peel performance more than the thickness of the albedo. It is due the cuticle, which is the outer most layer of the flavedo. The cuticle enables controlled access between the environment and fruit. It is a layer that plays a primary role for protecting the fruit from the environment (such as insects and microbes) and for monitoring vital gas exchanges (Albrigo, 1986; Petracek, 2002).

Albedo and flavedo structural differences can be spotted rather easily in table 11. At first glance, three particular feature stands out: coloring, oil glands and porous structure. Flavedo has much more vibrant colors compared to the albedo. Also in flavedo, oil glands could be spotted in the structure, especially in lemon. Albedo possess a courser structure due to vascular bundles. It is especially prominent in sour orange.

CHAPTER 5: CONCLUSION

Up to this point, citrus species were analyzed and compared to each other in terms of performance as a potential packaging. The analysis mainly consists of the mechanical and morphological aspects of the citrus fruits. Some important conclusions follow.

Tensile test results are summarized in Figure 17. The graphic seems to display an inverse relationship between peel thickness and tensile strength. This result indicates that a thinner material can be used in some cases, especially for smaller volumes and weights.

On the other hand, Figure 18 indicates that as the average fruit weight increases, the peel thickness increases roughly. This may be a strategy developed by evolution in order to protect the fruit from damage when it falls to the ground. Figure 19 seems to support this view. As seen in this figure, as the weight of the fruit is increased, the potential energy increases during the fall. This translates into a higher energy possible to damage the fruit. This damage is partly counteracted by a thicker peel. A simple lesson derived from these results would be that a thicker package thickness would be beneficial to protect heavier ingredients, which is usually applied by designers, in many cases instinctively. Combining the lessons from the two relationships, it could be suggested that thinner packaging materials would be sufficient for smaller volumes and weights while thicker protective materials should be preferred especially if the individual volume is relatively large. The peel thickness is a good indicator for a packaging material with similar tensile strength. For example, a designer could safely assume that a packaging thickness of about 1 mm is sufficient for a material with a tensile strength of 100-200 KPa if the product has similar features and size to an average mandarine. However, if the product is as large and heavy as a grapefruit, the peel thickness should be increased to 7-8 mm to protect it from damage related to static forces such as compression caused by other products transported in the same container, and dynamic forces such as vibration and impact during transportation.

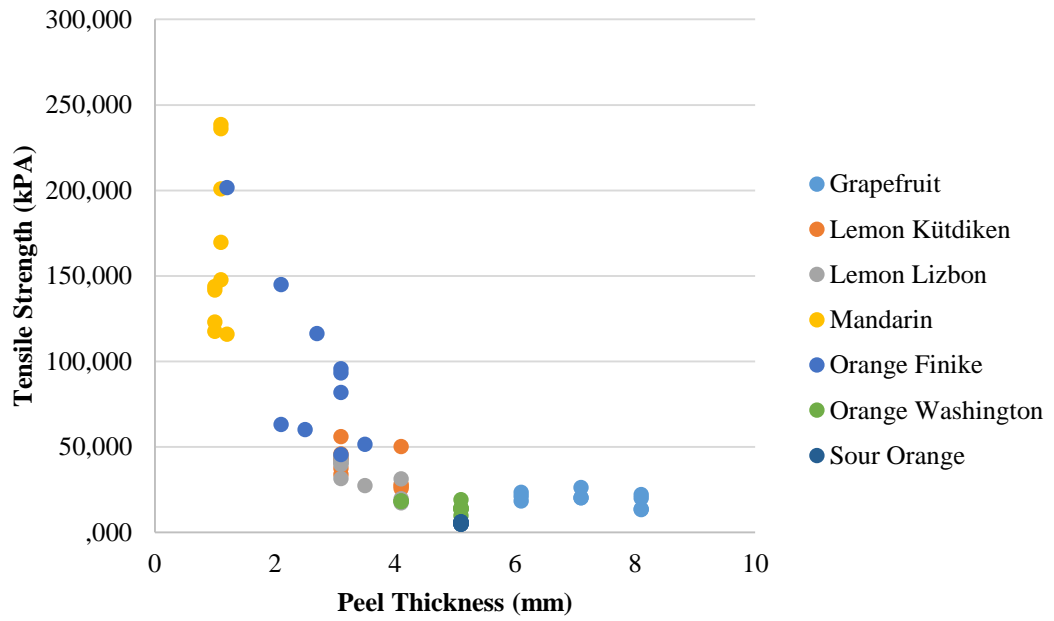


Figure 16. Relation of tensile strength and peel thickness

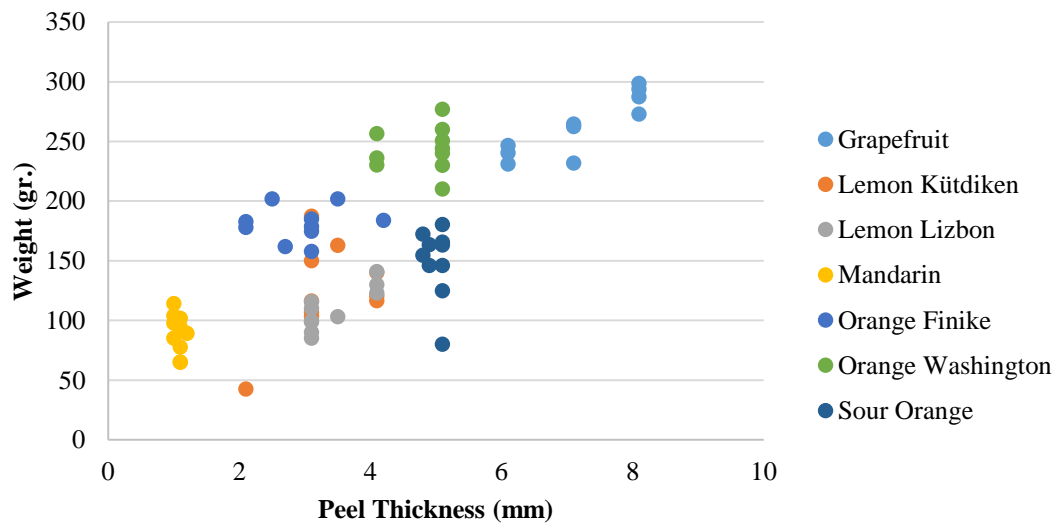


Figure 17. Relation of weight and peel thickness

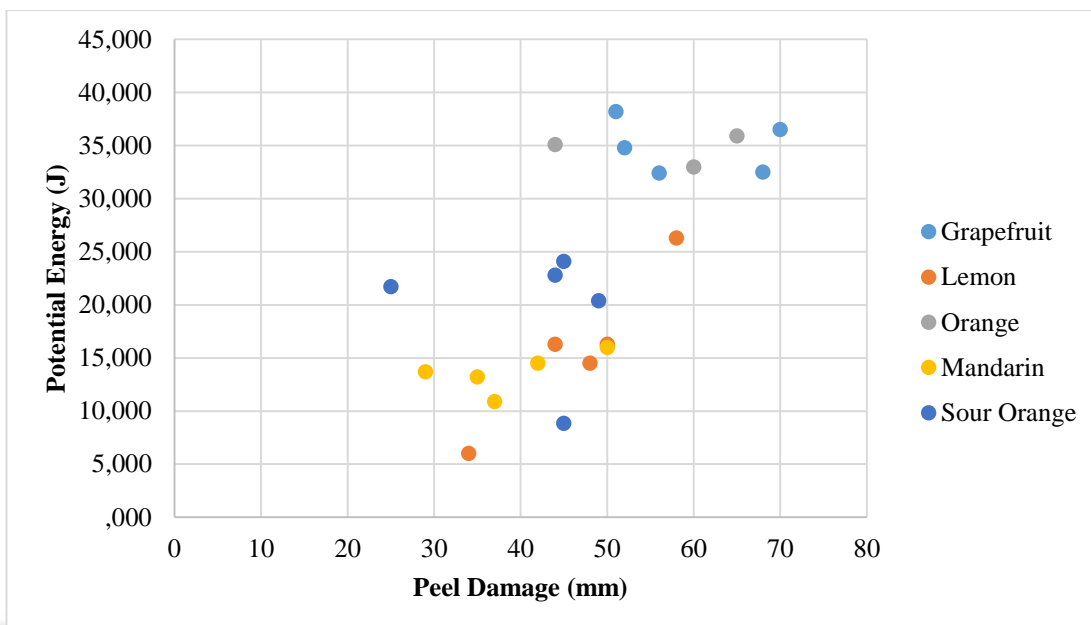


Figure 18. Relation of potential energy and peel damage

Fluid loss test results are summarized in Figure 14 and 15. As suggested in Chapter 4, among the five species studied, Washington oranges showed the best performance in terms of resistance to moisture loss, i.e. drying. However, this result does not consider the peel thickness. When we compare the following data (Table 12) it can be inferred that actually mandarin peel has the best performance in terms of protection against fluid loss since it is the thinnest and the total fluid loss is comparable to other types of citrus with much thicker peel.

Table 12. Comparison of Average Peel Thickness and Moisture Loss in 28 Days

Citrus Type	Average Peel Thickness (mm)	Moisture Loss (28 days, %)
Mandarin	1	33
Lemon (Lizbon)	3,5	37
Orange (Washington)	4,5	27
Sour Orange	5	31
Grapefruit	7	35

Since citrus peels contain the natural antioxidants and antimicrobial agents, one of the rising trend is to incorporate citrus peel into a sustainable, biodegradable PLA-based active food packaging (Fadzil and Othman,2021). Also, citrus peels have high pectin content and this could be beneficial for utilizing citrus peels as polymers films.

Cellulose reinforced pectin citrus peel films have higher tensile strength compared to the unreinforced citrus peel films. (Bátori, 2017).

Based on the present results and information based on the literature, a new packaging material can be suggested that is prepared from dried and powderized citrus peel, binder (agar agar or glycerin) and water. Mandarin peel would be a suggested due to its higher tensile strength. Such a film could be used to protect fresh fruit and vegetables, meat, cheese and similar products that contain water. It is suggested that citrus peel films could be applied as an inner layer of the corrugated tray. This layer would protect the corrugated cardboard from humidity that is caused by the fruits and vegetables.

Ongoing research presented fungus as an alternative material to the corrugated cardboard with collaboration with major brands. According to research and fungus start-up companies, fungus has two main advantages over corrugated cardboard; it generates less solid waste and it takes less time to grow the needed raw material (Anon, 2022b). These advantages could be applied to the citrus peel as an alternative material to corrugated cardboard. There could be development of a material similar to corrugated cardboard from citrus peel. Further research is needed to develop these ideas.

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