# PECTIN FROM DRAGON FRUIT PEEL (*HYLOCEREUS POLYRHIZUS*) AS POTENTIAL HALAL GELLING AGENT

BY

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# ABSTRACT

Dragon fruit (*Hylocereus polyrhizus*) belongs to the group of *Cactae* from the genus *Hylocereus*. This tropical fruit is known to have multi benefits in terms of its nutritional, medical and commercial values. Dragon fruit is either eaten directly or processed into juice and the peel will be discarded. A massive amount of the waste can be transformed into beneficial materials. For example, the peel has a good ratio of insoluble to soluble dietary fibre and can be considered as a good source of pectin. Pectin is a type of plant-based gelling agent and is one of the key ingredients in food products. Commercial gelling agents currently available on the market come from sources of bovine and porcine. Nevertheless, due to religious constraints and restrictions on safety, the use of this animal-based food quality enhancer has become less common. For these reasons, there is an increasing demand for the replacement of animal-based gelling agents with plant-based agents. This study was conducted to achieve multiple objectives. The first was to determine the best extraction conditions to yield the maximum amount of pectin from dragon fruit peels by using hot acid extraction. The second objective was to determine the antioxidant activity of the extracted pectin using DPPH radical scavenging activity. The final objective was to determine the physicochemical characteristics of dragon fruit peel pectin in terms of Degree of Esterification (DE), DSC, moisture and ash content, water and oil holding capacity, swelling capacity and textural attributes. Distilled water was used as a solvent and hydrochloric acid (HCl) was used to achieve the acidic medium. The experiment was conducted in a beaker that was placed on the hot stirring plate. It was found that the best extraction conditions yield the highest pectin amount (33%) were pH 3.5 and 75 minutes of extraction. Antioxidant activity assessment using DPPH assays shows a decreasing trend of 50% inhibition activity of dragon fruit peel pectin, indicating the presence of an antioxidant activity in the extracted pectin. Dragon fruit peel pectin is categorized under the high methoxyl group as the calculated DE is 52%. FTIR spectra had detected polysaccharides, the functional group of pectin. Analysis using DSC revealed that melting temperature ($T_m$) (122.01$^0$C) and melting enthalpy ($\Delta H_m$) (385.40 mJ) of dragon fruit peel pectin is better in comparison to commercial pectin. The moisture content of the extracted pectin (14.03 ± 1.925) was significantly different compared with the commercial pectin ($p < 0.05$). Ash content of the extracted pectin (8.73 ± 1.218) was significantly different from the commercial pectin ($p<0.05$). For holding capacity of water and oil and swelling capacity, tests have been obtained to verify that the extracted pectin has a high potential to be used as a gelling agent. The differences of textural attributes of the extracted pectin gel were found to be insignificant in terms of hardness, cohesiveness and gumminess as compared to the commercial pectin gel. Overall, the results obtained show that the pectin extracted from dragon fruit peel is comparable to any commercial gelling agent. Consequently, the extracted pectin may be used as another source of gelling agents in food industries.

# خلاصة البحث

فاكهة التنين (*Hylocereus polyrhizus*) تنتمي إلى أسرة صبّار من جنس *Hylocereus*. وهذه الفاكهة مشهورة بعدّة فوائد من ناحية التغذية، والفائدة الطبّية، وقيمتها التجارية. فاكهة التنين يمكن أكلها مباشرة أو تحويلها إلى عصير ويتخلّص من القشرة في الحالتين، حيث تنتج كمية كبيرة من النفاية يمكن أن يتم تحويّلها إلى مواد ذات منفعة اقتصادية وبيئية. على سبيل المثال، القشرة فيها نسبة جيّدة من الألياف غير القابلة للذوبان إلى الألياف الغذائية القابلة للذوبان وتُعْتَبَر مادة جيّدة لاستخلاص البكتين. البكتين مادة جلاتينية متبلرة تستخلص من النباتات، وعنصرا أساسيا في المنتجات الغذائية. المادة الجلاتينية التجارية المتوفّرة حالياً في السوق جاءت في معظمها من منتجات حيوانية مثل الأبقار والخنازير. ولذلك، بسبب القيد الديني بالحلال الطيب والسلامة، فإن استخدام محسّن جودة الطعام القائم على الإنتاج الحيوانات أصبح أقلّ انتشارًا. ولهذه الأسباب، هناك طلب متزايد لاستبدال الجلاتين المنتج من الحيوانات بالجلاتين النباتي أو مواد التبلّور كبدائل للجلاتين الحيواني منتجة من النباتات للاستخدام في الأطعمة والأشربة. قامت هذه الدراسة لتحقق عدة أهداف، الهدف الأوّل هو تحديد أفضل وضع للاستخلاص الحمضي ودرجة الحرارة ليؤتي المنتج الأقصى من البكتين من قشرة فاكهة التنين. والهدف الثاني هو تقدير النشاط المضاد للأكسدة لمستخلص البكتين باستخدام الفينيل الراديكالي لعمل كسحي (DPPH). والهدف الأخير هو تحديد الخصائص الفيزيائية والكيميائية لبكتين قشرة فاكهة التنين من حيث درجة الأسترة (DE)، التحليل الحراري باستخدام الماسح الحراري (DSC)، المحتوي المائي، ومحتوى الرماد، القدرة الاستيعابية للماء والزيت، القدرة الانتفاخية، وعوامل القوّام. تم استخدام الماء المقطّر كمحلول مذيب وحمض الهيدروكلوريك (HCl) مستعمل لتحقّق وسطي حمضي معتدل. أجريت هذه التجربة في كأس وُضِع في سخان مع محرك مغناطيسي للخلط. أظهرت النتيجة أنّ الوضع الأمثل والذي يعطي أفضل انتاجية استخلاص من البكتين (33%)، درجة الحموضة 3.5 و75 دقيقة مدة الاستخلاص. تجربة النشاط المضاد للأكسدة باستخدام (DPPH) أثبتت أن لبكتين قشرة فاكهة التنين مضادات أكسدة نشطة وخفضت معدل الأكسدة بنسبة 50%. البكتين من قشرة فاكهة التنين مصنّف تحت أسرة الميثيل العليا كما بحساب معدل الأسترة 52%. اكتشفت أطياف الأشعّة تحت الحمراء أن البكتين يتكون من مجموعات السكرات العديدة (polysaccharides). التحليل باستخدام تقنيّات تحليل الحرارة اكشف عن دراجة الانصهار ($122.01^0C$) ($T_m$) والمحتوى الحراري للانصهار (385.40 mJ) ($\Delta H_m$) وعليه يمكن القول أن بكتين قشرة فاكهة التنين أفضل من البكتين التجاري. وأعرض المحتوى المائي لمستخلص البكتين (14.03 ± 1.925) وهذا يظهر اختلافاً كبيراً مقارنة بالبكتين التجاري ($p < 0.05$). وأظهر محتوى الرمّاد لمستحلص البكتين (8.73 ± 1.218) اختلافا كبيرا من البكتين التجاري ($p<0.05$). أظهرت نتائج تحاليل القدرة الاستيعابية للماء والزيت، والقدرة الانتفاخية، أنّ البكتين المستخلص يمكن استخدامه في الأطعمة للتبلّور. اكتشف الفرق في عوامل القوّام لمستخلص مرطّب البكتين أي أنه غير مناسب بالنسبة إلى الصلابة، والتماسك، والالتصاق بدلاً عن البكتين التجاري للتبلور. مجمل القول، اتّضحت النتيجة بأنّ مواصفات مستخلص البكتين من قشرة فاكهة التنين مماثلة للبكتين التجاري، وعليه فإن مستخلص البكتين يمكن استخدامه كمادة أخرى للتبلور في الصناعات الغذائية.

## APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science (Halal Industry Science)

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*This work is especially dedicated to my beloved parents, Hjh. Siti Khalijah bt Mat Zin and Allahyarham Hj. Muhammad bin Daud. I also dedicated this work to my beloved husband, Muhammad Afiq bin Mohamed, thank you for the patience and unwavering support over the years, for my sisters, in- laws, nephews and nieces for their prayers. With hope Allah Al Mighty shower His blessings on them always.*

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Thank you.

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| | |
|---|---|
| DE | Degree of Esterification |
| DFPP | Dragon fruit peel pectin |
| CP | Commercial pectin |
| WHC | Water Holding Capacity |
| OHC | Oil Holding Capacity |
| SC | Swelling Capacity |
| FTIR | Fourier Transform Infra- Red |
| DSC | Different Scanning Calorimetry |
| S.D | Standard Deviation |

# CHAPTER ONE

# INTRODUCTION

## 1.1 BACKGROUND OF THE STUDY

Dragon fruit (*Hylocereus polyrhizus*) or pitaya is a tropical fruit which belongs to the *Cactae* family from the genus *Hylocereus*. The plant is tolerable to drought, heat and poor soil (Hua et al., 2016) *H. polyrhizus* is native to Central America and is becoming popular in Malaysia, Thailand, Vietnam, Philippine, and China. It has been widely cultivated in fertilized lands and grows abundantly in warm, and humid environment. In Malaysia, *H. polyrhizus* has gained popularity due to its sweet, refreshing fruit taste. The tiny black seeds embedded in the flesh, the edible part of the fruit are crunchy and has provided a unique taste. Dragon fruits are red, purplish colored and the fruits have green scales, creating an exotic, scaly appearance which contributes to its name. Apart from its taste and features, *H. polyrhizus* fruits have high concentrations with essential nutrients including antioxidants, vitamins and minerals. It is also rich vitamin C and contain anti- aging properties that can be used to treat photo aging (Leong, Show, Lim, Ooi, & Ling, 2017). Besides vitamin C, it contains A and B vitamins, protein, polyunsaturated fatty acid (PUFA), minerals including calcium, iron, potassium and sodium (Sonawane, 2017). *H. polyrhizus* is eaten raw or made as juice. In this light, the peels of the fruit are mostly discarded, creating considerable wastes (Fadzillah et al., 2019). Studies have suggested that the peel might contain a rich amount of pectin (Rahmati, Abdullah, Momeny, & Kang, 2015). Due to its high nutrient content, extracting pectin from *H. polyrhizus* peels could be a good idea to make the waste useful.

Meanwhile, the use of pectin, especially in the food industry, has benefited consumers and suppliers alike. This plant-based material has distinctive characteristics as a gelling agent, a stabilizer as well as a thickening agent. Studies have reported that the global demand of pectin has exceeded 40, 000 tons per annum and is increasing by about 5% in the main market sectors (Pagliaro, Ciriminna, Fidalgo, & Delisi, 2016). In 2015, the average price of pectin exceeded 15 USD per kilogram and the market was close to reach USD 1 billion (Idrovo et al., 2019). According to the analysis reported in 2017, the global market size for pectin was estimated at USD 964.1 million and is growing at Compound Annual Growth Rate (CAGR) of 7.1 % in 2025 (Cooper, 2017). The application in the food industry is caused by the consumers' demand for a gelatin alternative. The distinctive characteristics of pectin as a gelling agent, a stabilizer and a thickening agent resembles gelatin (Saha & Bhattacharya, 2014) and thus, researchers are giving attention for its use as a substitute to gelatin (Abang Zaidel, Md Rashid, Hamidon, Md Salleh, & Mohd Kassim, 2017; Begum, Yusof, Aziz, & Uddin, 2017; Jaswir et al., 2016).

Pectin and gelatin are categorized under the same group of hydrocolloids gelling agent. In food productions, hydrocolloids gelling agent has been used in jellies, desserts, yoghurts and candies to provide thickening without stiffness through the formation of gels. The addition of gelling agents helps to increase the consistency in the texture, taste and mouth feel of food products. In this regard, gelatin is the most common gelling agent available in the market as it is sourced from the skins and bones of animals such as cows and pigs, which are readily available and cheaper compared to other sources (Karim & Bhat, 2008; Nik Aisyah, Huda, Azhar, & Fazilah, 2014). In addition, the ability of gelatin to form a gel, bind water, stabilize, and improve the texture of food products made it among the most important ingredients in food manufacturing. In this light, gelatin has been extensively used in  popular food products include desserts, dairy products, meat, bakery, wine and juice to improve their quality (Hanani, 2016). Apart from the food industry, gelatin is widely used in the biomedical and pharmaceutical fields as a gelling agent, such as in the preparation of three- dimensional tissue regeneration and soft and hard capsule, respectively (Karim & Bhat, 2008).

Despite the extensive application of gelatin in food production, its usage has several shortcomings linked to religious and health concerns as some types of gelatin are sourced from pigs, which cannot be consumed by Muslims. The prohibition of consuming certain foods, including pork, is mentioned in the Holy Quran, chapter 2, verse 173:

> "He hath only forbidden you dead meat, and blood, and the flesh of swine, and that on which any other name hath been invoked besides that of Allah. But if one is forced by necessity, without wilful disobedience, nor transgressing due limits, then is he guiltless. For Allah is Oft-forgiving Most Merciful."

This ayah clearly states that Muslims are forbidden from eating all portions of the pig, its offspring, and other animals not killed in accordance with Islamic traditions. In accordance with Muslim, the Jewish community must also has to abstain from eating pork, while Hindus are also prohibited from eating beef and cow products and some practiced vegetarianism (Karim & Bhat, 2009).

In addition to the religious restrictions on the use of pork and meat products, swine flu infections and disease outbreaks  such as Creutzfeldte Jacob Disease caused by Bovine Spongiform Encephalopathy (BSE)  prion in the 1980s have increased consumer awareness of the source of gelatin used in their foods (Ali, Joe M., & Ismail, 2015; Mohd Dali, Sulaiman, A. Samad, Ismail, & Alwi, 2007).

Scientists have studied extensively the alternatives for mammalian gelatins (porcine and bovine) following these events (Elgadir, Mirghani, & Adam, 2013; Jaswir et al., 2016; Karim & Bhat, 2008, 2009; Mariod & Adam, 2013; Nik Aisyah et al., 2014). One of the potential alternatives for mammalian gelatins, for example, is fish gelatin. Nevertheless, the use of fish gelatin is limited because of the lack of available and inexpensive raw materials, the different quality of the jelly produced and the risk of allergic reactions (Karim & Bhat, 2009; Nik Aisyah et al., 2014). Other than fish gelatin, it is also possible to replace existing mammalian gelatins with chicken skin gelatin. In this way, due to the insufficient sources of resources, the industrial use of poultry gelatin often faces the same challenge (Ali et al., 2015; Mhd Sarbon, Badii, & Howell, 2013). Research have also indicated alternatives to plant-based gelatin (Adzahan et al., 2014; Karim & Bhat, 2009; Lugovska & Sidor, 2015; Milani & Maleki, 2012), and the use of pectin as a replacement has been given attention by researchers in recent years (Abang Zaidel et al., 2017; Fadzillah et al., 2019; Jaswir et al., 2016). The data pectin from tropical fruits like dragon fruits, on the other hand, is still insufficient. (Abang Zaidel et al., 2017; Adzahan et al., 2014; Izalin, Kharidah, Jamilah, & Noranizan, 2016; Naderi et al., 2012; Tang, Wong, & Woo, 2011). This research was therefore intended to provide a deeper understanding on physicochemical properties of pectin extracted from dragon fruit peel that can be used in future applications as a gelling agent and a prospective replacement for gelatin.

## 1.2 STATEMENT OF THE PROBLEM

Hydrocolloid gelling agent is an important ingredient in food productions as it changes the rheology and improve the quality of food products. At present, gelatin, which is derived from pig and cattle body parts, is the most commonly used hydrocolloid gelling agents (Hanani, 2016; Mariod & Adam, 2013). The use of gelatin raises concern in certain religious community as Muslim and Jewish are not allowed to eat pork and its derivatives. The halal status of the cattle is also questionable and the emergence of bovine spongiform encephalopathy (BSE) epidemic in European countries in the 1980s brought medical concerns over the safety of products that use bovine gelatin (Karim & Bhat, 2008). For these reasons, there are many studies that tried to find alternative for gelatin, particularly from plant sources (Jaswir et al., 2016; Nur Farhana, Amin, Sadeq Hasan, & Shuhaimi, 2017).

Dragon fruits are usually eaten raw or mixed in a drink, leaving their peels in waste. It is proposed that pectin can be extracted from the peel and used as a gelling agent to reduce waste and convert it into something useful. Therefore, as a possible gelling agent, this research was conducted to determine the properties of pectin extracted from dragon fruit peel. The results of this study will be useful, especially in the food industry, because plant- based materials are free from religious issues and have no health risks.

## 1.3 SIGNIFICANCE OF THE STUDY

Common gelling agent available in the market is gelatin. However, the consumption of gelatin raises many issues especially from religious and health conscious, as mentioned previously. Thus, both consumers and researchers are finding the alternative from other sources, including plant- based. Pectin is another type of gelling agent with high possibility to be gelatin alternative. The goal of this analysis is to extract the pectin from dragon fruit peel as a new source of gelling agent. In addition, this research is expected to turn the waste (peel) into something valuable (pectin). Thus, this study can be used to propose a new source of gelling agent that is free from any religious and health issues.

## 1.4 RESEARCH OBJECTIVES

### 1.4.1 General Objective

To study the physicochemical properties of pectin from dragon fruit (*H. polyrhizus*) peel as a new potential halal gelling agent.

### 1.4.2 Specific Objectives

1. To determine the highest percentage of pectin yield that can be produced from different exposure of time and pH value
2. To determine antioxidant activity of DFPP (dragon fruit peel pectin)
3. To characterize the physical and chemical properties of pectin from DFPP and associate these properties with commercial pectin (CP).

## 1.5 RESEARCH QUESTIONS

1. What are the best condition of extraction to produce the highest number pectin?
2. How to determine if the DFPP possesses antioxidant activity?
3. What are the physicochemical characteristics of DFPP? What is the possibility of making DFPP as new source of commercial pectin?

## 1.6 RESEARCH HYPOTHESES

1. Pectin can be extracted at the highest amount with the correct conditions of extractions.
2. Antioxidant activity of DPP is confirmed by DPPH testing.
3. DFPP can be a new source of pectin proven by its physicochemical characteristics.

# CHAPTER TWO

# LITERATURE REVIEW

## 2.1 DRAGON FRUIT (*H. polyrhizus sp.*)

Dragon fruit, known in Malay as *buah naga*, is a tropical fruit belonging to the (*Cactaceae*) family. It is also known as pitaya and in Southeast Asia it is prevalent. Across countries such as Vietnam, Indonesia, Thailand, China and Australia, the dragon fruit plant is widely cultivated. Vietnam is currently the top exporter of dragon fruits, with dragon fruits contributing to around 55% of the country's export fruit turnover. In Malaysia, the extensive cultivation of dragon fruit plants first started in 1999 when the plants were planted in Johor, Setiawan in Perak and Kuala Pilah in Negeri Sembilan in several regions. The cultivation of dragon fruit has expanded extensively where, in 2006, the total production of this fruit was 2,534.2 tons of 927.4 ha of land (Wan. M & Cheah, 2008), and in 2013 the total the cultivation area was 1, 641 Ha and a total of 10, 961 tons of dragon fruits (Hoe, 2017).

A unique plant is the dragon fruit plant itself. This epiphyte plant grows in the hot and humid environment of the peat land. With a thickness of 10- 12 cm, it has dark green stems and can reach up to 10 m or more. The stems clamber and abundantly branch out (Centerchem, 2012). Typically, a matured plant can produce a blooming cereal bloom for just one night.  With countless tiny black seeds, the inner part of the flower is red- violet and the outer part is greenish yellow with purple dyes. Subsequently, the *H. polyrhizus* plant produces fruits with a mouth- watering light sweet taste highly sought after by fruit lovers, particularly in the Asian community. Dragon fruits can ripen in a year for up to six cycles, and the process is propagated by seed or stem cuttings in commercial cultivation. The price of dragon fruits sold on the market depends on its quality, which is determined by various means. Dragon fruit of the best quality can be seen as shown in Figure 2.1(a). It seems to be pretty mature and young. The skin appears to be red in color with more greenish scales and clear from any physical defects and injuries. Whereas the commercially acceptable quality of dragon fruit is shown in figure 2.1(b). The skin is red in color, with few green colors on the scales, and the presence of black spots reveals minor flaws. The fruit is, however free from physical damages and therefore safe to eat.

(a)

(b)

Figure 2.1: The best quality of (a) dragon fruit and (b) the acceptable quality of dragon fruit by the market

Meanwhile, the maturity indexes of dragon fruits are shown in Figure 2.2. The first index is the immature fruit condition which is flower blooming on the $19^{th}$ – $23^{rd}$ days. The skin color and scale is green at this point, indicating that it is not ready for harvest.

The fruit is in the mature stage during second index ($24^{th}$ – $26^{th}$ days). Red spots on the fruits are found, but the fruit scales are still colored green. This is the moment when the fruit is ready to be harvested especially for remote shipping and sea export in particular. The fruit is almost ripe at the third index ($27^{th}$ – $29^{th}$ flower flowering) and the skin is completely red. It means that the fruit is now ready for harvesting by air and sea for remote distribution and shipping. In the fourth maturity index ($30^{th}$ to $33^{rd}$ days of flower bloom) the fruit is fully mature. It is bright red in color and the scales is slightly green. At this point, fruits are only suitable for express air transportation. Eventually, after the flowering $34^{th}$ days, the fruit is in the fifth maturity table. The color of the skin becomes dark red and the color of the scale is yellow. Fruits are considered to be too mature at this point and these fruits are only suitable for the local market to be sold.

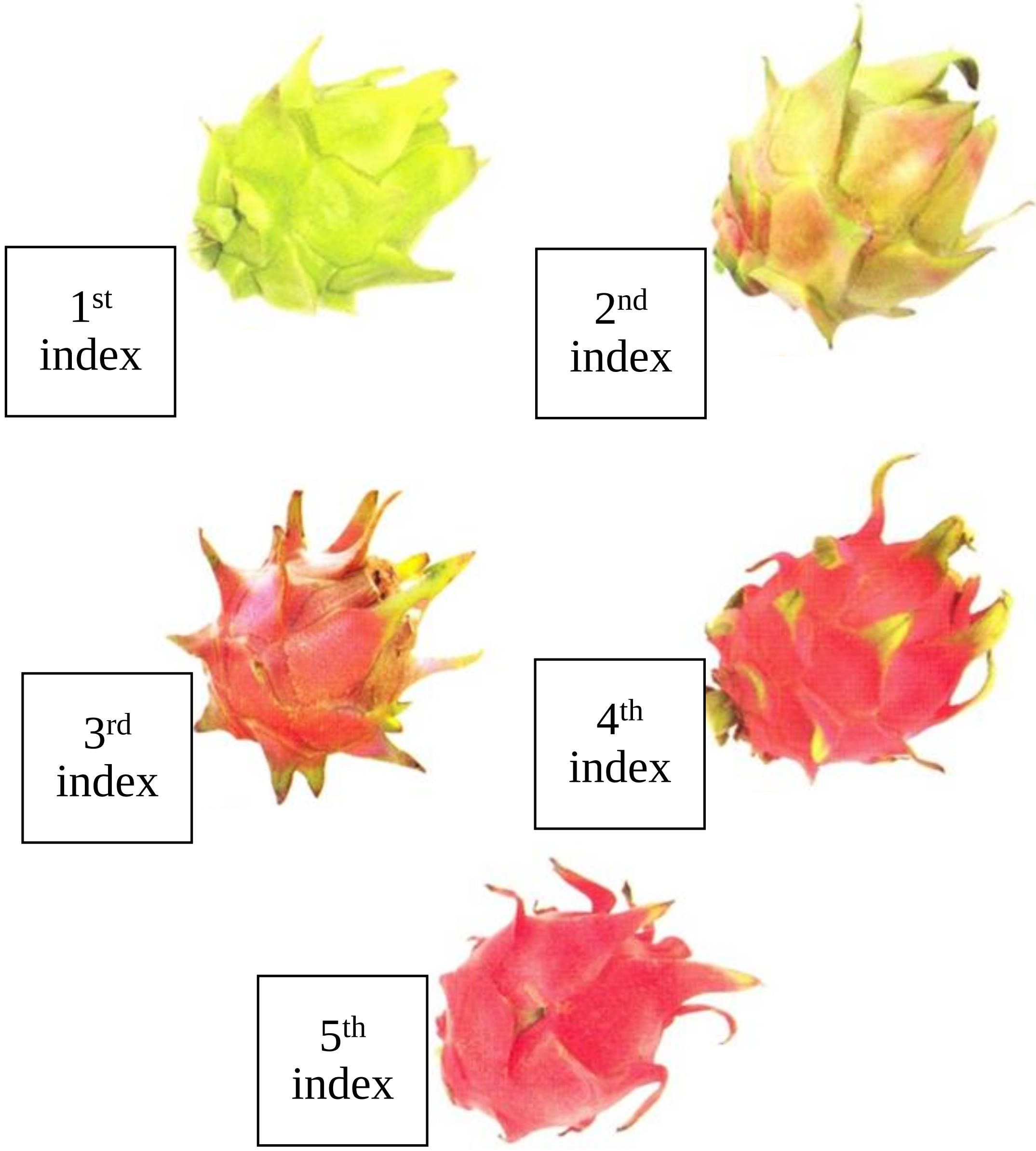


Figure 2.2: Maturity index of dragon fruits