engineering proceedings

Proceeding Paper

# Beeswax and Castor Oil to Improve the Moisture Barrier and Tensile Properties of Pectin Based Edible Films for Food Packaging Applications †

**A Dharhshini and Yamini Sudha Sistla**

Department of Chemical Engineering, Shiv Nadar University, Gautam Buddha Nagar-201314, India; email1@email.com
* Correspondence: email2@email.com
† Presented at the 2nd International Electronic Conference on Processes: Process Engineering—Current State and Future Trends (ECP 2023), 17–31 May 2023; Available online: https://ecp2023.sciforum.net/.

**Abstract:** Biopolymer-based edible films and coatings are vital in making the global food-packaging industry more sustainable. These films/coatings protect and extend the shelf life of food by acting as barriers to moisture, oxygen, microorganisms, and Ultra Violet light. Polysaccharides, due to abundant availability from natural plant-based resources and the tendency to form a gel in water, are excellent low-cost choices for packaging films. Additives such as hydrophobic agents, plasticizers, binders, and antimicrobial agents will improve the properties of films and coatings. The present work aims to develop pectin-based packaging film (from 5% *w*/*v* film forming solution) by adding castor oil as a hydrophobic agent, beeswax as a plasticizer, and Clove oil as an antimicrobial agent. Films were developed by using $2^3$ (two-level three-factor) statistical factorial design of experiments. The amount of castor oil (5% & 15%), beeswax (5% & 10%) and clove oil (2% and 4%) are taken as the three factors. The developed films were analyzed for physical, moisture barrier, morphological, thermal, tensile properties and resistance to microbial growth. The results indicated that clove is a good antimicrobial agent. Further, bees wax greatly changed by enhancing the anti-microbial activity, elongation, and moisture barrier properties. Castor oil integration remarkably lowered the moisture and oxygen transmission rates relative to pure pectin films and some other additives reported in the literature. The optimized biofilms had a thickness of ~0.10 ± 0.004 mm, pH = 3, and transparency of ΔE = 9.15 to 25. The elongation at break increased at least four times. The films were thermally stable at 400 °C. The detailed statistical analysis and analysis of various studies indicate that the amount of castor oil ($p < 0.05$), a combined effect of castor oil and beeswax ($p < 0.05$) is significant on barrier properties while the effect of beeswax ($p < 0.05$) is also significant on mechanical properties.

**Keywords:** polysaccharide; pectin; bio-edible films; beeswax; water vapour transmission rate (WVTR); mechanical test

**Citation:** Dharhshini, A.; Sistla, Y.S. Beeswax and Castor Oil to Improve the Moisture Barrier and Tensile Properties of Pectin Based Edible Films for Food Packaging Applications. *Eng. Proc.* **2023**, *5*, x. https://doi.org/10.3390/xxxxx

Published: 17 May 2023



## 1. Introduction

Biodegradable edible films are gaining significant attention world-wide due to increasing awareness of environmental challenges associated with synthetic polymeric films. This is because of the fact that some biopolymers are safe to consume and most of the biopolymers are environmental friendly nature and are biodegradable. Biopolymer films are renewable and with edible functional additives, they degrade much more readily than synthetic films. The biopolymers can be used as active green packaging materials to increase the shelf life of food such as fresh produce and processed. Biopolymer packaging keeps the freshness of food due to green ingredients, and also improves consumer health and safety. The bio-edible films and coatings are usually made from natural polymers like polysaccharides, proteins, and lipids along with other additives. A good film should have good moisture barrier properties, mechanical strength, flexibility, thermal properties, gas barrier properties, etc. The great diversity in the polymeric structures of polysaccharides, proteins, and lipids influences the linkage of polymers, degree of polymerization, etc. these greatly vary the film properties. As a result of which the films are tailored by altering the compositions and film-forming conditions like temperature, relative humidity, pH, etc. Polysaccharides having the gel-forming ability with water include cellulose, carrageenan, chitosan, starch, etc. In general, polysaccharides are hydrophilic and have poor gas barrier properties. However, on adding naturally occurring additives their properties are enhanced. The scope of this research is narrowed down to pectin-based bio-edible films as pectin is abundantly available in the peels of fruits and vegetables and also because of economical advantage. Pectin is an anionic compound having 1- 4-D-galacturonic acid residues. The property of pectin varies depending upon the degree of methyl esterification of uronic acid, based on which they are classified as high methoxy pectin (HMP) or low methoxy pectin (LMP). HMP forms excellent films. Pectin is a long chain of Homogalacturonan (HG), Xylogalacturonan (XGA), and Rhamnogalacturoanan (RGA). However, HG forms 60% of the and the rest by XGA and RGA.

## 2. Materials and Methods

The chemical grade pectin powder was brought from Loba Chemie Pvt. Ltd. in Mumbai, India. The molecular weight varies from 30,000 and 100,000. The methoxyl concentration in pectin was 6–10%, and the DE (degree of esterification) was 63–66%. Bees wax and castor oil were procured from local vendor. Other chemicals such as sodium hydroxide buffer tablets were procured rom Sigma Aldrich. Double distilled water is made in our lab.

### *2.1. Film Development*

The experiments proceed with the preparation of 5% weight/volume (*w*/*v*) pectin-based control films, where 5 g of pectin powder is agitated in 100 mL distilled water. Lab scale magnetic stirrer was used to agitate and homogenize the film forming base solution at a temperature of 45 °C for 30 min and at 500 rpm stirrer speed. During the last 10 min of this process, 0.5 g of emulsifier was added to help the immiscible components combine. The solution was cooled to ambient temperature and the power of hydrogen (pH) was checked, it was around 2.8 which was because of the acidic nature of pectin. It was adjusted around 3 using a diluted sodium hydroxide solution. Further, the solution was mixed in the magnetic stirrer along with which a melted mixture of beeswax, castor oil, and clove oil of appropriate composition was added. The solution is further homogenized using a homogenizer (IKA T25 ULTRA-TURRAX) at speed of 4000–6000 rpm. To achieve proper mixing of the small particles the solution is ultrasonicated for 45 min. 40 mL of perfectly homogenized solution is poured into lab scale petri dish of 100 mm diameter by spreading it uniformly. These are then kept in the humidity chamber (NECSTAR NEC-HTC-150) to dry the films at a controlled temperature and relative humidity (40%–50%). The dried films are peeled and stored in a desiccator containing silica gel.

### *2.2. Characterization*

#### 2.2.1. Moisture Content and Water Solubility

To analyze the moisture content bound in the film, a 4 cm × 4 cm film was kept in a hot air oven for 3 h at a temperature of 110 ± 5 °C. The percent weight loss against the initial weight of the film was calculated as $((W_{initial} - W_{final})/W_{initial}) \times 100$, where $W_{initial}$ weight of the film and $W_{final}$ is the weight of the dry film. To confirm the results, the analysis was done by taking at least three samples from the same film.

Similarly, to analyze the water solubility of a film, a 3 cm × 3 cm was taken and soaked in 15 mL of water for 30 min. The time taken for the film to completely dissolve was noted.

2.2.2. Thickness

The thickness of the film developed by spreading 40 mL of film solution on a 100 mm diameter petri dish was measured. To measure the thickness of this film, a micrometer screw gauge with a least count of 0.001 mm was used. The thickness at five random positions on the 100 mm diameter film was measured and the average was reported as the film thickness.

2.2.3. Optical Property or Transparency

The transparency analysis of the film was done using a spectrometer (CHN SPEC & CS-580 A). The calibration was done by keeping the film over a white surface and on which the spectrometer gave the color analysis value. Film color was measured by lightness/luminosity (L*), chromaticity (a*), and chromaticity (b*) on the color scale. L* values give the range from black (zero) to white (100), a* ranges from green (negative) to red (positive), and b* ranges from blue (negative) to yellow (positive). The analysis is repeated at 5 different areas of the film and the average was considered.

$$\Delta E = \sqrt{\left(L^{*} - L\right)^{2} + \left(a^{*} - a\right)^{2} + \left(b^{*} - b\right)^{2}}$$

The standard values are L* = 86.75, a* = 0.93, and b* = −1.03, Total transparency or color difference parameter is ΔE.

2.2.4. WVTR

The water vapour transmission rate is analyzed due to the fact that less moisture from the food should be transmitted out so that desirable moisture is present in the food to keep up the freshness of the food. It is usually measured in $g/m^2/day$. The analysis was done by using the facility at Northern India Textile Research Association, Ghaziabad, India, and Sree Chitra Tirunal Institute For Medical Sciences & Technology, Thiruvananthapuram, India. It was done according to the ASTM standard ASTM E96/E96M-05 (water method), maintained at a temperature of 32 ± 2 C °Cand a relative humidity of 50 ± 2%, with air velocity set between 0.02–0.3 m/s.

2.2.5. Water Contact Angle and Mechanical properties

The water contact angle (WCA) was done using the sessile drop method using a device that measures the contact angle between the film and the water droplet as video data (Data Physics OCA 15 Plus, Germany). The analysis was done at Sree Chitra Trunal Institute For Medical Sciences & Technology, Thiruvananthapuram, Kerala, India. Mechanical properties of the films were studied at Sree Chitra Trunal Institute For Medical Sciences & Technology, Thiruvananthapuram, Kerala, India. The study was performed based on the ASTM D882 method. The analysis was done on a film of 10mm width × 150 mm length over a crosshead speed of 10 mm/min.

2.2.6. FTIR

FTIR analysis of the films was done with a Fourier transform infrared spectrophotometer. The spectrum of each sample of the film was done in the range of 4000–500 $cm^{-1}$ with a resolution of 4 $cm^{-1}$. The results obtained from the FTIR analysis provided insights on the structural properties of the film.

## 3. Results and Discussions

All the pectin films with additives such as castor oil, bees wax and clove oil were developed as per the $2^3$ (two-level, three-factor) statistical design of experiments. The details of the experiments are given in Table 1. The films were flexible and easy to get peeled off from the petri dish. All the films were developed from 5 % *w/v* (5 g of pectin per 100 mL of water) concentration film forming solution. The other process parameters such as pH (=3) and drying conditions such as temperature (40 °C) and relative humidity (60%) were kept constant based on our previous studies. The films were kept free from atmospheric moisture and other contaminations by storing them in a vacuum desiccator.

**Table 1.** Details of low and high levels of functional additives.

| Run No | Castor Oil (% *w/w* of Pectin) | Bees Wax (% *w/w* of Pectin) | Clove Oil (% *w/w* of Pectin) |
|---|---|---|---|
| 1 | 5 (low) | 5 (low) | 2 (low) |
| 2 | 15 (high) | 5 | 2 |
| 3 | 5 | 10 (high) | 2 |
| 4 | 15 | 10 | 2 |
| 5 | 5 | 5 | 4 (high) |
| 6 | 15 | 5 | 4 |
| 7 | 5 | 10 | 4 |
| 8 | 15 | 10 | 4 |

### 3.1. Water Solubility

All the films were observed to be completely water soluble. When a 5 cm × 5 cm film was dipped in a 20 mL of water, within 20 min, almost all the film got dissolved in water. However, a minute amount of bees wax was visible floating on the surface of the water based on the amount of wax added. This is because of the hydrophobic nature of bees wax.

### 3.2. Thickness

The bees wax, castor oil, and clove oil integrated pectin films developed were observed to be thin. This can be confirmed from Figure 1 that the thickness of the films was in the range of 0.12 ± 0.004–0.15 ± 0.004 mm.

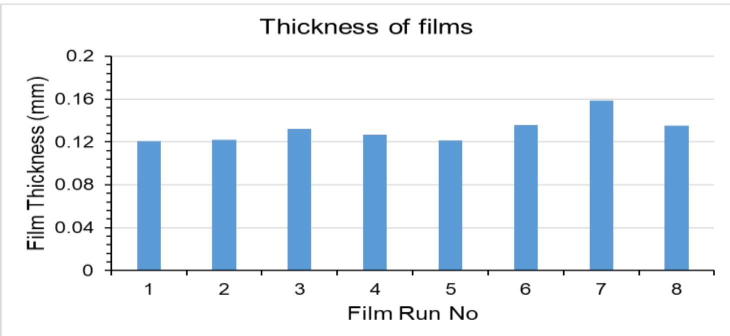


**Figure 1.** The thickness of films (run number as per Table 1).

### 3.3. Transparency

All the films were transparent enough when compared with a transparent plastic sheet. The transparency parameter ΔE of the films was observed to be in the range of 15 ± 2–20 ± 2. The ΔE values less than 50 usually referred to as transparent films. Therefore, the bees wax, castor oil and clove oil integrated pectin films developed in the present work are transparent.

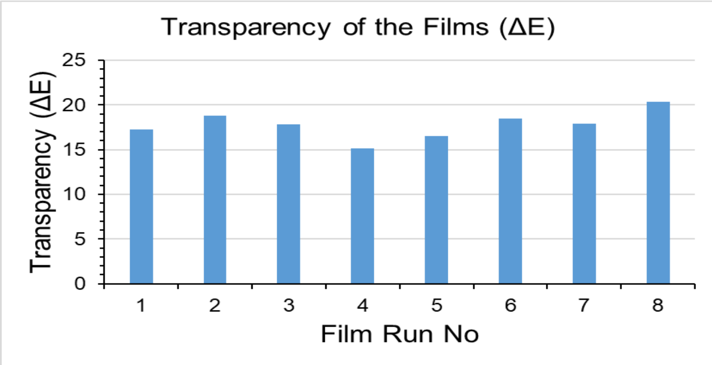


**Figure 2.** Transparency of the films (run number as per Table 1).

### *3.4. WVTR*

The moisture transmission to and from the food material will cause drying and fungal growth. Therefore, the packaging film must possess good moisture barrier properties. The moisture barrier property of the films was studied in terms of water vapor transmission rate (WVTR). The WVTR of the bees wax, castor oil and clove oil integrated pectin films developed in the present work is in the range of 1017.12 g/m²/day–1739.73 g/m²/day. The WVTR of control pectin is 1815.70 g/m²/day. A comparison of the WVTR values of the eight composite films and the control pectin film is shown in Figure 3. This shows that the hydrophobic nature of bees wax, castor oil and clove oil has reduced the WVTR to a significant level. It was observed that the Run No 8 which has higher percentage of bees wax, castor oil and clove oil has shown lowest WVTR. This means that the highest moisture barrier property is exhibited by Run No. 8.

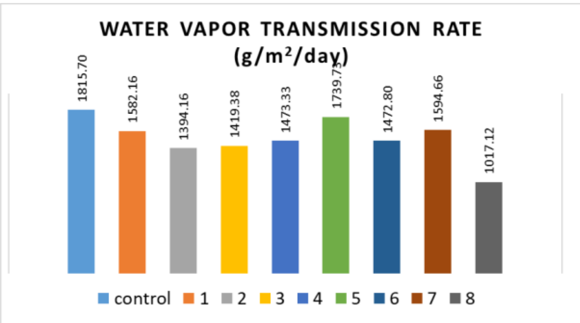


**Figure 3.** Water vapor transmission rate of the films (run number as per Table 1).

### *3.5. Water Contact Angle and Mechanical Properties*

The water contact angle of the films was observed to be in the range of 75–85 ± 5° after 12 sec of sessile drop. This is considerably higher than the control pectin film for which water contact angle is 62°. The tensile strength of the film was in the range of 8–12 ± 3 MPa and elongation at break of 15% at a strain rate of 10 mm/min. At similar strain rates, the control pectin film has tensile strength of 15 MPa and elongation at break of 3%. This indicates that the additives bees wax, castor oil and clove oil have improved the mechanical proeprties.

### *3.6. FTIR*

The fourier transform infrared spectroscopy (FTIR) of the four film samples is shown in Figure 4. The Figure 4 clearly shows that all the films are of the same nature with only a slight difference in the intensity of transmittance. Figure 4 also shows that no chemical transformations happened during the film forming process and solvent evaporation.

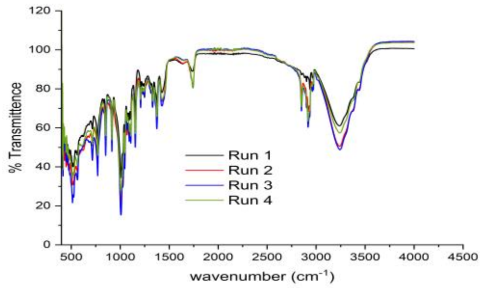


**Figure 4.** FTIR of the films (run number as per Table 1).

## 4. Conclusions

The present work reports pectin biopolymer based films for food packaging applications. Bees wax, castor oil and clove oil were used as hydrophobic and plasticizer additives. The films developed were thin, transparent and easily soluble in water. Further, the films showed excellent moisture barrier properties compared to control pectin films. The films were also found to be biodegradable. The results suggest that pectin-based films with bees wax, castor oil and clove oil have the potential to be used as edible packaging materials with enhanced mechanical, barrier, and antimicrobial properties. This research could contribute to sustainable food packaging practices with biodegradable films.

**Acknowledgments:** The WVTR analysis was made possible with the support of the Sree Tirunal Institute Chitra for Medical Sciences & Technology, Trivandrum, Kerala, India and the Northern India Textile Research Association (NITRA),Ghaziabad, India.

## References

1. Mehraj, S.; Sistla, Y.S. Optimization of process conditions for the development of pectin and glycerol based edible films: Statistical design of experiments. *Electron. J. Biotechnol.* **2022**, *55*, 27–39. https://doi.org/10.1016/j.ejbt.2021.11.004.
2. Dhall, R.K. Advances in Edible Coatings for Fresh Fruits and Vegetables: A Review. *Crit. Rev. Food Sci. Nutr.* **2013**, *53*, 435–450. http://dx.doi.org/10.1080/10408398.2010.541568.
3. Cortes-Rodríguez, M.; Villegas-Yepez, C.; Gonzalez, J.H.G.; Rodríguez, P.E.; Ortega-Toro, R. Development and evaluation of edible films based on cassava starch, whey protein, and bees wax. *Heliyon* **2020**, *6*, E04884. https://doi.org/10.1016/j.heliyon.2020.e04884.
4. Food Science and Quality Management ISSN 2224-6088 (Paper) ISSN 2225-0557 (Online) Vol 3, 2012. Available online: www.iiste.org (accessed on).
5. Mehraj, S.; Pandey, G.; Garg, M.; Santra, B.; Grewal, H.S.; Kanjilal, A.; Sistla, Y.S. Castor Oil and Cocoa Butter to Improve the Moisture Barrier and Tensile Properties of Pectin Films. *J. Polym. Environ.* **2023**, *31*, 312–326. https://doi.org/10.1007/s10924-022-02581-4.
6. de Moraes Crizel, T.; de Oliveira Rios, A.; Alves, V.D.; Bandarra, N.; Moldão-Martins, M.; Flôres, S.H. Biodegradable Films Based on Gelatin and Papaya Peel Microparticles with Antioxidant Properties. *Food Bioprocess Technol.* **2018**, *11*, 536–550 https://doi.org/10.1007/s11947-017-2030-0.