

HIGH WATER VAPOR PERMEABILITY ETHYLENE-VINYL ACETATE COPOLYMER  
COMPOSITE FOR FRESH HOLY BASIL (*Ocimum sanctum* L.) PACKAGING



A Thesis Submitted in Partial Fulfillment of the Requirements  
for the Degree of Master of Engineering in Chemical Engineering

Department of Chemical Engineering

FACULTY OF ENGINEERING

Chulalongkorn University

Academic Year 2021

Copyright of Chulalongkorn University

ฟิล์มบรรจุภัณฑ์ที่มีอัตราการส่งผ่านไอน้ำสูงจากโวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิต สำหรับ  
บรรจุกะเพราสด



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิศวกรรมศาสตรมหาบัณฑิต  
สาขาวิชาวิศวกรรมเคมี ภาควิชาวิศวกรรมเคมี  
คณะวิศวกรรมศาสตร์ จุฬาลงกรณ์มหาวิทยาลัย  
ปีการศึกษา 2564  
ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

Thesis Title HIGH WATER VAPOR PERMEABILITY ETHYLENE-VINYL  
ACETATE COPOLYMER COMPOSITE FOR FRESH HOLY  
BASIL (*Ocimum sanctum* L.) PACKAGING

By Mr. Nuttinan Boonao

Field of Study Chemical Engineering

Thesis Advisor Professor ANONGNAT SOMWANGTHANAROJ, Ph.D.

Thesis Co Advisor Associate Professor KANITHA TANANUWONG, Ph.D.

---

Accepted by the FACULTY OF ENGINEERING, Chulalongkorn University in  
Partial Fulfillment of the Requirement for the Master of Engineering

..... Dean of the FACULTY OF  
ENGINEERING  
(Professor SUPOT TEACHAVORASINSKUN, D.Eng.)

THESIS COMMITTEE

..... Chairman  
(Associate Professor THARATHON MONGKHONSI, Ph.D.)

..... Thesis Advisor  
(Professor ANONGNAT SOMWANGTHANAROJ, Ph.D.)

..... Thesis Co-Advisor  
(Associate Professor KANITHA TANANUWONG, Ph.D.)

..... Examiner  
(Chutimon Satirapipathkul, Ph.D.)

..... External Examiner  
(Assistant Professor Wanchai Lerdwijitjarud, Ph.D.)

นัททินันท์ บุญเนาวิ : फिल्मบรรจุภัณฑ์ที่มีอัตราการส่งผ่านไอน้ำสูงจากไวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิต สำหรับบรรจุกะเพราสด. ( HIGH WATER VAPOR PERMEABILITY ETHYLENE-VINYL ACETATE COPOLYMER COMPOSITE FOR FRESH HOLY BASIL (*Ocimum sanctum* L.) PACKAGING) อ.ที่ปรึกษาหลัก : ศ. ดร.อนงค์นาฏ สมหวังธนโรจน์, อ.ที่ปรึกษาร่วม : รศ. ดร. ชนิษฐา ธนานุวงศ์

ผลไม้และผักสดยังคงหายใจทำให้เกิดไอน้ำจำนวนมากภายในบรรจุภัณฑ์ส่งผลให้เกิดการเจริญเติบโตของเชื้อราและแบคทีเรียก่อโรคและส่งผลกระทบต่อลักษณะภายนอก เพื่อแก้ปัญหานี้จะใช้ฟิล์มบรรจุภัณฑ์ที่มีการซึมผ่านของไอน้ำและแก๊สออกซิเจนที่เหมาะสมกับอัตราการหายใจและอัตราการคายน้ำของผลิตภัณฑ์ผักและผลไม้สด ในงานวิจัยนี้ได้ทำการผลิตฟิล์มบรรจุภัณฑ์ไวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิตโดยหลอมผสมเอทิลีนโคพอลิเมอร์กับสารเติมแต่ง 2 ชนิดคือไดอะตอมไมต์และซอร์บิแทนโมโนสเตียเรตเพื่อทำการปรับความสามารถในการซึมผ่านของก๊าซ ซึ่งรวมถึงอัตราการส่งผ่านไอน้ำ (WVTR) และอัตราการส่งผ่านออกซิเจน (OTR) ให้อยู่ในช่วง 73.88 - 87.60 g/m<sup>2</sup>·day และ 3,662.47 - 11,342.80 cc/m<sup>2</sup>·day ตามลำดับ ฟิล์มบรรจุภัณฑ์มีความต้านทานแรงดึงในช่วง 15.94 - 24.77 MPa และมุมสัมผัสน้ำในช่วง 9.0° - 87.7° เพื่อทดสอบประสิทธิภาพของฟิล์มบรรจุภัณฑ์ที่พัฒนาขึ้นในงานวิจัยนี้ กะเพราสดถูกบรรจุในถุงบรรจุภัณฑ์ที่ทำจากฟิล์มบรรจุภัณฑ์ไวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิตสูตร (EVADSM) ที่มีอัตราการซึมผ่านไอน้ำและแก๊สออกซิเจนที่เหมาะสมในการยืดอายุกะเพราสดและฟิล์มบรรจุภัณฑ์พอลิพรอพิลีนที่มีรูพรุนในเชิงพาณิชย์ ถุงที่ทดสอบทั้งสองถูกเก็บไว้ที่ 10±0.5 °C เป็นเวลา 3 สัปดาห์ จากผลการทดลองพบว่ากะเพราสดที่บรรจุในฟิล์มบรรจุภัณฑ์ไวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิตนั้นมีการสูญเสียน้ำหนักน้อยกว่าและมีปริมาณน้ำหนักของกะเพราที่เสื่อมสภาพน้อยกว่าในช่วงเวลาการสุ่มตัวอย่างเดียวกัน และที่อุณหภูมิ 10 °C ฟิล์มบรรจุภัณฑ์พอลิพรอพิลีนที่มีรูพรุนในเชิงพาณิชย์สามารถยืดอายุกะเพราสดให้อยู่ในสภาพที่ยอมรับได้เป็นเวลา 6 วัน ในขณะที่ฟิล์มบรรจุภัณฑ์ไวนิลอะซิเตทเอทิลีนโคพอลิเมอร์คอมพอสิตสูตร (EVADSM) สามารถยืดอายุกะเพราสดให้อยู่ในสภาพที่ยอมรับได้เป็นเวลา 14 วัน

สาขาวิชา วิศวกรรมเคมี

ปีการศึกษา 2564

ลายมือชื่อนิสิต .....

ลายมือชื่อ อ.ที่ปรึกษาหลัก .....

ลายมือชื่อ อ.ที่ปรึกษาร่วม .....

# # 6370148221 : MAJOR CHEMICAL ENGINEERING

KEYWORD:

Nuttinan Boonao : HIGH WATER VAPOR PERMEABILITY ETHYLENE-VINYL ACETATE COPOLYMER COMPOSITE FOR FRESH HOLY BASIL (*Ocimum sanctum* L.) PACKAGING.  
 Advisor: Prof. ANONGNAT SOMWANGTHANAROJ, Ph.D. Co-advisor: Assoc. Prof. KANITHA TANANUWONG, Ph.D.

Fresh fruits and vegetables still respire, which causes large amount of water vapor inside the package. This results in the growth of fungal and bacterial pathogens and defecation in the external appearance. To solve this problem, the packaging film with an appropriate value of gas permeability that matches the respiration rate of fresh fruits and vegetable products will be used. In this study, we produced EVA copolymer composite packaging films by melt blending EVA copolymer with diatomite and sorbitan monostearate additives and adjusted gas permeability which includes water vapor transmission rate (WVTR) and oxygen transmission rate (OTR) to be in the range of 73.88 - 87.60 g/m<sup>2</sup>·day, and 3,662.47 - 11,342.80 cc/m<sup>2</sup>·day, respectively. The packaging films had tensile strength in the range of 15.94 - 24.77 MPa and water contact angle in the range of 9.0° - 87.7°. To test the performance of the packaging films, fresh holy basil was packed in a pouch made from selected EVA copolymer composite packaging films (EVADSM) that had a suitable rate of permeation of water vapor and oxygen gas to extend the shelf life of fresh holy basil or commercial perforated polypropylene film. The pouches were kept at 10±0.5 °C for 3 weeks. The packaging test results show that, at similar storage time, fresh holy basil packed in a EVA copolymer composite pouch had lower weight loss and weight of deteriorated basil compared with those kept in a commercial perforated polypropylene pouch. At storage temperature of 10 °C, the commercial perforated polypropylene can prolong the shelf life of holy basil at a satisfactory quality for 6 days and the selected EVA copolymer composite pouch (EVADSM) can prolong shelf life of holy basil at a satisfactory quality for 14 days

Field of Study: Chemical Engineering

Student's Signature .....

Academic Year: 2021

Advisor's Signature .....

Co-advisor's Signature .....

## ACKNOWLEDGEMENTS

Firstly, I would like to express my sincere thanks to my advisor, Professor Anongnat Somwangthanoj, and my co-advisor, Associate Professor Kanitha Tananuwong for their valuable advice and the continuous support thought out of my Master Degree study and this research

Apart from my advisor and co-advisor, I would be thankful to Assoc. Prof. Tharathon mongkhonsi, as the chairman, Dr. Chutimon Satirapipathkul and Assistant Professor Wanchai Lerdwijitjarud, as the members of the thesis committee, who provided kindly suggestions for this research.

I also would like to express my sincere appreciation to PDI trading Co., Ltd for storage information and fresh holy basil supported in this study.

In addition, I am grateful to everyone in the Polymer Engineering Research Laboratory, Department of Chemical Engineering, Chulalongkorn University, for discussion, friendly encouragement and given comments on this research. Furthermore, I would like to thank my laboratory colleagues. Miss Nappaphan Kunanusont and Miss Apinya Wongthanoj for her suggestion, assistance, discussion and friendly encouragement in solving problems.

Finally, I most gratefully acknowledge my family for all their support both financially and mentally throughout the period of this research.

Nuttinan Boonao

## TABLE OF CONTENTS

	Page
.....	iii
ABSTRACT (THAI).....	iii
.....	iv
ABSTRACT (ENGLISH).....	iv
ACKNOWLEDGEMENTS .....	v
TABLE OF CONTENTS.....	vi
LIST OF TABLES.....	ix
LIST OF FIGURES .....	x
CHAPTER I INTRODUCTION.....	1
1.1 General introduction.....	1
1.2 Objectives of the research .....	2
1.3 Scopes of the research .....	2
CHAPTER II THEORY AND LITERATURE REVIEWS.....	4
2.1 Main metabolic process in Postharvest fruit and vegetables .....	4
2.1.1 Respiration.....	4
2.1.2 Transpiration .....	4
2.1.2.1 Intrinsic factors .....	5
2.1.2.2 Extrinsic factors .....	5
2.2 Relative humidity in fresh produce packaging .....	7
2.2.1 Effect of water condensation inside fresh produce packaging.....	9

2.3 Fresh produce packaging .....	11
2.3.1 Commercial fresh produce packaging .....	11
2.3.1.1 Basil packaging .....	12
2.3.2 Ethylene-vinyl acetate copolymer composite films packaging.....	14
2.3.2.1 <i>Ethylene-vinyl acetate (EVA)</i> .....	14
2.3.2.2 Diatomaceous earth (Moisture absorber).....	17
2.3.2.3 Sorbitan monostearate (Antifogging agent).....	20
.....	24
CHAPTER III.....	25
METHODOLOGY.....	25
3.1 Materials.....	25
3.2 Film preparation.....	25
3.3 Properties of the copolymer composite films.....	27
3.3.1 Analysis of water vapor permeability.....	27
3.3.2 Analysis of oxygen permeability.....	27
3.3.3 Mechanical analysis.....	27
3.3.4 Contact angle analysis.....	28
3.3.5 Cold fog analysis.....	28
3.3.6 Opacity analysis.....	28
3.4 Application of the copolymer composite films on the storage of holy basil....	28
3.4.1 Weight loss of holy basil during storage .....	29
3.4.2 Appearance of holy basil during storage .....	29
3.4.3 Deteriorated proportion of holy basil during storage .....	29
3.4.4 Temperature and relative humidity during storage.....	29



3.5 Methodology .....	29
CHAPTER IV .....	31
RESULTS AND DISCUSSION.....	31
4.1.1 Gas barrier properties.....	31
4.1.2 Water contact angle properties .....	33
4.1.3 Cold fogging properties.....	35
4.1.4 Mechanical properties .....	36
4.1.5 Opacity properties .....	40
4.2 Application of the copolymer composite films on the storage of holy basil....	41
4.2.1 Temperature and relative humidity during storage.....	43
4.2.2 Weight loss of holy basil during storage .....	44
4.2.3 Weight of deteriorated holy basil during storage.....	45
4.2.4 Appearances .....	46
CHAPTER V .....	48
CONCLUSIONS .....	48
REFERENCES .....	49
VITA.....	52

## LIST OF TABLES

	Pages
<b>Table 2.1</b> The approximate moisture content of some typical food and vegetable [14].....	8
<b>Table 2.2</b> The characteristics of plastic films used in this study [15].....	10
<b>Table 2.3</b> Water vapor transmission rate of commercially plastic used in fresh produce packaging [2].....	12
<b>Table 2.4</b> Characteristics of plastic films used in Kotepong et al. (2015)'s study [10].....	13
<b>Table 2.5</b> Summary resulted of packaging test [10].....	13
<b>Table 2.6</b> Effect of EVA content on gas barrier properties (OTR and WVTR) of LDPE.	16
<b>Table 2.7</b> Mean contact angle (MCA) of EVA (layer A) with 3% nonionic surfactant and pure LDPE (layer B) with 1% nonionic surfactant [26].....	23
<b>Table 2.8</b> Molecular weight of all non-ionic surfactants [26].....	23
<b>Table 3.1</b> Chemical composition of diatomaceous earth.....	25
<b>Table 3.2</b> Weight ratio of binary blend of EVA / Diatomite.....	26
<b>Table 3.3</b> Weight ratio of binary blend of EVA / SM.....	26
<b>Table 3.4</b> Weight ratio of tertiary blend of EVA / SM/ Diatomite.....	26
<b>Table 4.1</b> Gas barrier properties of all EVA copolymer composite films.....	32
<b>Table 4.2</b> The contact angle of all EVA copolymer composite films.....	33
<b>Table 4.3</b> The opacity properties of all EVA copolymer composite films.....	40
<b>Table 4.4</b> The summary properties of EVA copolymer composite films.....	41

## LIST OF FIGURES

	Pages
<b>Figure2.1</b> Effect of temperatures (5, 10, 15, 20 and 25 °C) on respiration rates of basil .....	6
<b>Figure2.2</b> Effect of temperatures (5, 10, 15, 20 and 25 °C) on respiration rates of sweet basil [10] .....	6
<b>Figure2.3</b> Modified atmosphere packaging application [11] .....	7
<b>Figure2.4</b> Moisture condensation mechanism in fresh produce packaging [3] .....	8
<b>Figure2.5</b> %Fungal growth (a) and %weight loss (b) of strawberry packed in NVS package and OPP package [9] .....	9
<b>Figure2.6</b> Marketability percentage of after 28 days of green chillies packaging in all packaging material [15] .....	11
<b>Figure2.7</b> Chemical Repeating unit of EVA [18] .....	15
<b>Figure2.8</b> Effect of diatomite concentration (0-8%) on oxygen transmission rate of LDPE polymer [21] .....	18
<b>Figure2.9</b> Effect of diatomite concentration (0-8%) on water vapor transmission rate of LDPE polymer [21] .....	18
<b>Figure2.10</b> Effect of diatomite concentration (0, 3, 5, 7 and 9%) on carbon dioxide transmission rate o EVA polymer [22] .....	19
<b>Figure2.11</b> SEM images of EVA-diatomite polymer composite film at 0, 3, 5, 7 and 9% respectively (a-e) [22] .....	20
<b>Figure2.12</b> Chemical structure of sorbitan monostearate (SM) (C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> ) [23] .....	21
<b>Figure2.13</b> Antifogging agent mechanism in polymer matrix [24] .....	21
<b>Figure2.14</b> Water contact angle of solid surface from superhydrophilic to superhydrophobic [25] .....	22

<b>Figure 2.15</b> Effect of 3 and 5% all non-ionic surfactant on mean contact angle of layer A (EVA 15% of vinyl acetate groups) [26] .....	24
<b>Figure 3.1</b> Methodology of this research	30
<b>Figure 4.1</b> Effect of diatomite and SM additives on oxygen and water vapor transmission rate of all EVA copolymer composite films .....	32
<b>Figure 4.2</b> Effect of diatomite and SM additives on water contact angle of EVA copolymer composite films .....	34
<b>Figure 4.3</b> Effect of diatomite and SM additives on cold fogging properties of EVA copolymer composite films .....	35
<b>Figure 4.4</b> Direction for measuring mechanical properties including MD and TD directions .....	36
<b>Figure 4.5</b> Effect of diatomite and SM fillers on tensile strength of EVA.....	37
<b>Figure 4.6</b> Stress-strain curve of all blends in both (a) MD and (b) TD .....	38
<b>Figure 4.7</b> Effect of diatomite and SM fillers on elongation at break of EVA .....	39
<b>Figure 4.8</b> Example of basil 80 g packed in test packaging A (EVADSM) and B (commercial perforated PP). .....	42
<b>Figure 4.9</b> Temperature and %Relative humidity in all test packaging at different storage time .....	43
<b>Figure 4.10</b> Weight loss of holy basil in all test packaging at different storage time ..	44
<b>Figure 4.11</b> Weight of deteriorated (%) of holy basil in all test packaging at different storage time .....	45
<b>Figure 4.12</b> Apperences of holy basil in all test packaging pouches at different storage times .....	47

# CHAPTER I

## INTRODUCTION

### 1.1 General introduction

Fresh fruit and vegetable products suffer high losses in quality during the supply chain from producers to the consumer because metabolic activity (respiration and transpiration) still remains after harvest [1]. One way to minimize post-harvest food loss is to use packaging. Modified atmosphere packaging technique (MAP) is widely used to slow down this problem for a long time. Gases ( $O_2$ , and  $CO_2$ ) around the products are properly managed inside the packaging [2]. However, most polymeric films used in MAP have a low water vapor transmission rate compared to moisture loss from fresh fruit and vegetable products with high water activity. This results in excess moisture condensed inside packaging, which leads to the growth of fungal bacterial pathogens, loss of visibility, and defects in the external appearance, such as rotting [3]. Temperature change during storage, distribution and marketing can also exacerbate condensation problems [4].

Holy basil (*Ocimum sanctum* L.) is one of high perishable fresh culinary herbs. It is an ingredient in a variety of Thai dishes and its exporting is increasing in demand every year. Temperature and relative humidity are important factors that affect the shelf life of holy basil. Low storage temperature of holy basil can cause chilling injury symptom and decrease its shelf life rapidly [5]. Many research studies about temperature and MAP material for extending shelf life of holy basil. The temperature at 10 °C and MAP polyethylene was shown to extend shelf life of holy basil for 9 days [6]. The LDPE bags could also be used to delay the changes in biochemical parameters of holy basil under 5 °C storage [7].

In this research, a kind of effectively controlled in-packed moisture condensation copolymer composite packaging films were obtained from ethylene-vinyl acetate (EVA) as the polymer matrix with diatomite and sorbitan monostearate (SM) as the additives. The effect of these additives on gas permeability, mechanical

properties, cold fogging, water contact angle and opacity properties of ethylene-vinyl acetate copolymer films were investigated. Moreover, the packaging tests of selected copolymer composite packaging films on basil storage such as weight loss, appearances, weight of deteriorated basil and, temperature and humidity change during storage were also investigated.

## 1.2 Objectives of the research

1.2.1 To investigate the effects of diatomite and SM on ethylene-vinyl acetate copolymer films' gas barrier (OTR and WVTR), water contact angle, cold fogging, mechanical and opacity properties.

1.2.2 To develop an effectively control in-pack moisture condensation packaging by ethylene-vinyl acetate copolymer composite film for fresh holy basil (*Ocimum sanctum* L.) packaging.

## 1.3 Scopes of the research

### 1.3.1 Part I

1.3.1.1 EVA copolymer composite films were prepared by binary blend of diatomite/EVA, binary blend of SM/EVA, and tertiary blend of diatomite /SM/EVA.

1.3.1.2 The concentration of diatomite in diatomite/EVA blend was varied at 0, 3, and 5%wt respectively whereas the concentration of SM in SM/EVA blend was fixed at 3%wt and the concentration of diatomaceous earth and SM in diatomaceous earth/SM/EVA blend was fixed at 3 phr.

1.3.1.3 Gas barrier, mechanical, cold fogging water contact angle and opacity properties of all prepared EVA copolymer composite films were studied.

### 1.3.2 Part II

1.3.2.1 Basil was packed in selected EVA copolymer composite film and commercial polypropylene film from factory

1.3.2.2 The packaging test including %weight loss, appearances, %weight of deteriorated basil, temperature and humidity change during storage were investigated



## CHAPTER II

### THEORY AND LITERATURE REVIEWS

#### 2.1 Main metabolic process in Postharvest fruit and vegetables

##### 2.1.1 Respiration

Respiration is one major metabolic process that happens in post-harvest fruit and vegetables. This process uses oxygen in the atmosphere to change nutrients correcting from photosynthesis such as protein, lipid, carbohydrate to water, heat, and energy. But an atmosphere that has too low oxygen can cause anaerobic respiration that effecting the odor and sensory quality of fruit and vegetables. If fruit and vegetables in post-harvest have high respiration rates, their shelf-life is reduced rapidly [8]. The equations that represent aerobic respiration and anaerobic respiration were shown in **Equation 2.1** and **Equation 2.2** respectively.



##### 2.1.2 Transpiration

Fruit and vegetable products are always released water through stomia to the surrounding air to reduce heat inside. The driving force of this process is the concentration difference between moisture concentration in a product's surface and water vapor pressure in the surrounding air. Typically, moisture content inside the product is higher than outside so water is released from inside to outside air [8]. There are two types of factors that impact transpiration rate: intrinsic and extrinsic factors.



### 2.1.2.1 Intrinsic factors

Products which have high surface area lead to high transpiration rate when compared with products which have low surface area. For example, leafy green vegetables and cauliflowers have a higher transpiration rate than oranges and tomatoes. Structural characteristic is also an important factor. Water loss is reduced in products with tissue resistance, such as a waxy surface coating, than in products without these structures [4] [8].

### 2.1.2.2 Extrinsic factors

Temperature and relative humidity are important extrinsic factors that can affect transpiration rate of produce. In terms of temperature, the rate of transpiration increases as the storage temperature increases. On the other hand, the rate of transpiration increases as the storage relative humidity decreases. Air movement and atmospheric pressure, in addition to temperature and relative humidity, can alter the rate of transpiration of produce [9] [10] [4] [8] .

Thus, when researching the storage shelf life of postharvest fruits and vegetables. It is critical to keep relative humidity and storage temperature under appropriate control.

Kotepong et al. [10] studied the effect of temperature on the transpiration rate of fruits and vegetables. The effect of temperature on the transpiration rate of basil and sweet basil was shown in **Figure 2.1** and **Figure 2.2** respectively. For basil, transpiration rate proportionally increased when increasing temperature storage. For sweet basil, transpiration rates at 5, 10 and 15 °C were not significantly different but when increasing temperature to 20 and 25 °C transpiration rate increased.

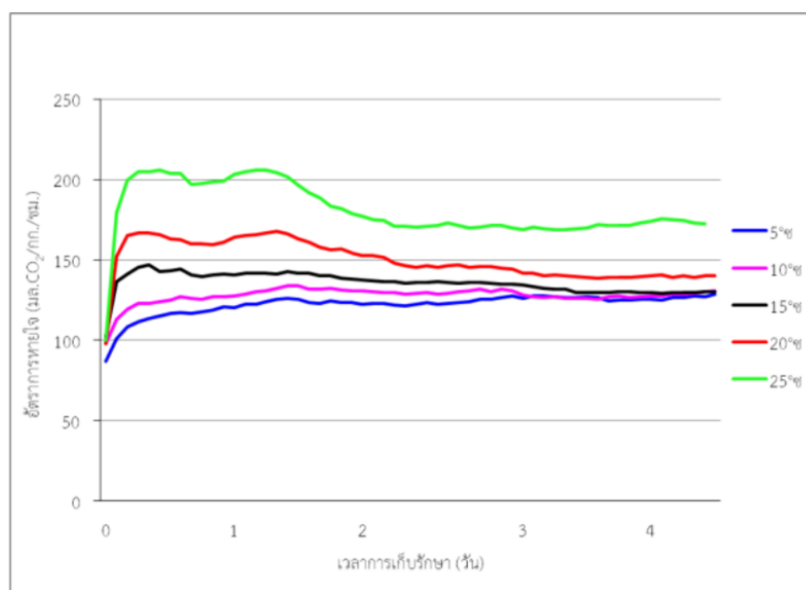


Figure 2.1 Effect of temperatures (5, 10, 15, 20 and 25 °C) on respiration rates of basil [10]

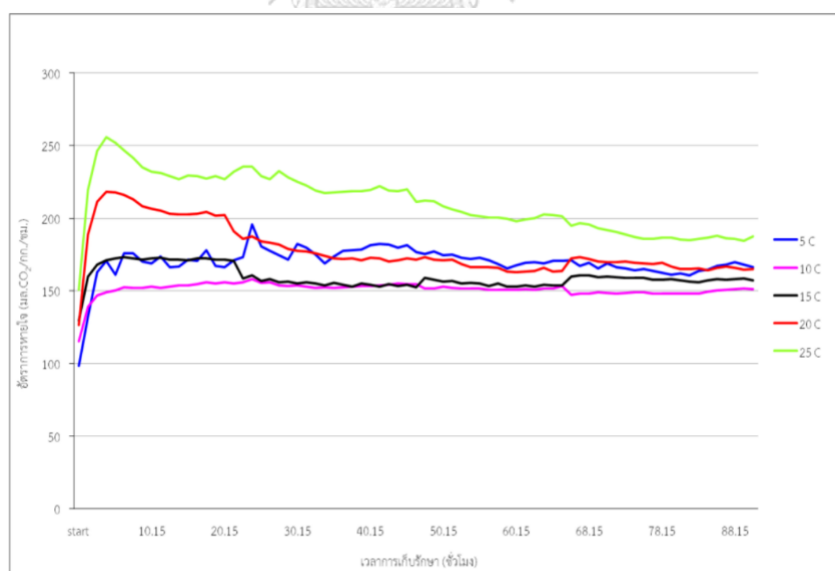


Figure 2.2 Effect of temperatures (5, 10, 15, 20 and 25 °C) on respiration rates of sweet basil [10]

One way to control the respiration and transpiration rate of fresh fruit and vegetable produces to extend the storage life is to use MAP. MAP is a technique that has been studied and utilized widely for a long time. The levels of oxygen and carbon dioxide in the surrounding air are properly managed inside packaging. Currently, not only the gas atmosphere is managed but also relative humidity is properly managed so the modified atmosphere/modified humidity packaging technology (MA/MH) is great attention and developed in the field of fresh produce packaging application.



Figure 2.3 Modified atmosphere packaging application [11]

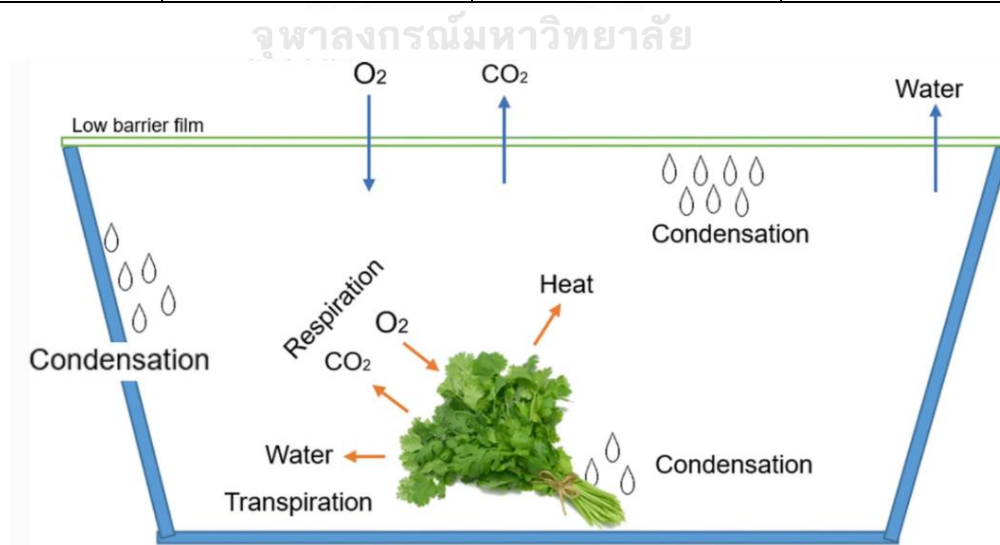
## 2.2 Relative humidity in fresh produce packaging

In fresh fruits and vegetables products, water is the main component about 80-95 % of their mass. The moisture content of some fruit and vegetable was shown in **Table 2.1**. After harvest, fresh fruits products release water through respiration and transpiration that can affect humidity inside fresh fruits and vegetables products packaging [4]. Less humidity can cause much weight loss that cause fresh fruit to deteriorate both commercially and physiologically. In contrast, excess humidity can cause water condensation inside packaging that contribute to infections, both fungal

and bacterial and defect in texture, color, odor and surface structure of fresh fruit and vegetable produce [12]. The moisture condensation mechanism inside packaging was shown in **Figure 2.4**. So, an achieving optimum humidity inside fresh fruit packaging is very essential to prolong shelf life of fresh fruit produce. Typically, for most fresh fruit and vegetable product the storage humidity should be within 85-95% RH [13].

**Table 2.1** The approximate moisture content of some typical food and vegetable [14]

Fruit	Water content (%)	Vegetable	Water content (%)
Apple	84	Asparagus	93
Avocado	76	Beans (green)	89
Banana	76	Broccoli	90
Blue berry	83	Cabbage	92
Grapefruit	89	Lettuce	85
Orange	86	Mushroom	91
Cherry	80	Pepper (sweet)	92

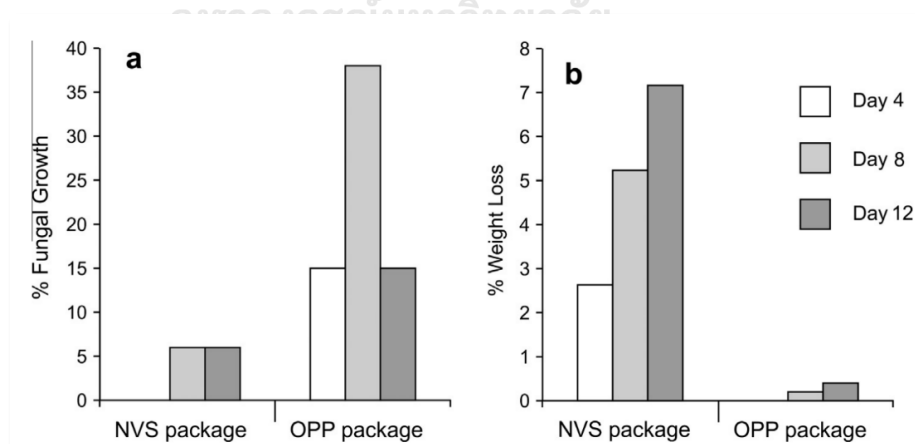


**Figure 2.4** Moisture condensation mechanism in fresh produce packaging [3]

### 2.2.1 Effect of water condensation inside fresh produce packaging

There are many research study effect of water condensation inside fresh produce packaging.

Sousa-Gallagher et al. (2013) [9] studied the effect of the type of packaging on the shelf life of strawberries. Two types of commercial packaging were used in which one is a flexible film “NatureFlex NVS” whose had WVTR 500 g/m<sup>2</sup>day. The other one was polypropylene film whose WVTR was 0.8 g/m<sup>2</sup>day used as control. Weight loss and fungal growth were investigated. The result showed that, NVS films could maintain the highest quality of strawberry. In polypropylene film, severe condensation was detected, with water drops collecting beneath the film, resulting in significant fungal deterioration. There was no condensation in the NVS packaging, the film remained transparent, and no fungal deterioration occurred until the 12<sup>th</sup> days of storage. The fungal growth and weight loss results were shown in **Figure 2.5**. These weight loss figures were found to be below the maximum acceptable limit (10%), and despite the NVS package having a larger weight loss, no significant variations in product quality were detected. As a result, high water vapor permeable films like NVS had the ability to maintain fresh produce quality and extend shelf life.



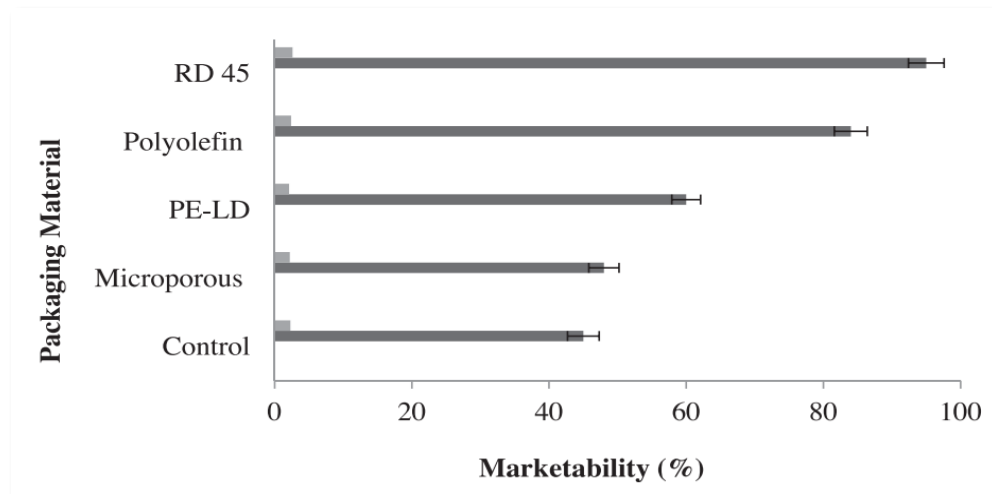
**Figure 2.5** %Fungal growth (a) and %weight loss (b) of strawberry packed in NVS package and OPP package [9]

Chitravathi et al. (2015) [15] studied the effect of passive MAP on postharvest shelf life of green chillies. The characteristics of all plastic films used in this study were shown in **Table 2.2**

**Table 2.2** The characteristics of plastic films used in this study [15]

Type	Thickness ( $\mu\text{m}$ )	Water vapor transmission rate ( $\text{g}/\text{inch}^2/\text{day}$ )	Oxygen transmission rate ( $\text{cc}/\text{m}^2/\text{day}$ )	Carbon dioxide Transmission rate ( $\text{cc}/\text{m}^2/\text{day}$ )
Microporous	37.5	50-70	40,000-50,000	30,000-40,000
LDPE	50.1	1.2	7,000-8,500	28,000-33,000
Polyolefin	35.9	0.9	6,000-8,000	19,000-22,000
Anti-fog (RD45) film	18.7	2.2	10,800-13,800	32,000-36,000

The results showed that microporous film allowed for faster gas exchange than other films after 28 days of green chillies packing. Because of the limited water vapour permeability of the LDPE film, moisture condensation occurred, shortening the shelf life even more. Chillies packaged with anti-fog (RD45) film had a longer shelf-life of 28 days and preserved freshness for a longer duration due to its optimal gas and water vapour permeabilities. Marketability percentage of after 28 days of green chillies packaging in all packaging material was shown in **Figure 2.6**



*Figure 2.6 Marketability percentage of after 28 days of green chillies packaging in all packaging material [15]*

## 2.3 Fresh produce packaging

### 2.3.1 Commercial fresh produce packaging

Commercially plastics used in fresh fruit and vegetable produces packaging are linear low-density polyethylene (LDPE), high-density polyethylene (HDPE) and, polypropylene (PP). Barrier properties to water vapor of all plastic films are shown in **Table 2.3**. Low water vapor transmission rate of all plastic films makes these plastic films not appropriate for fresh produce with high metabolic activity. Condensation of water vapor inside packaging occurs when evaporated water molecules from fresh food are not properly transferred across plastic layers [3] .

**Table 2.3** Water vapor transmission rate of commercially plastic used in fresh produce packaging [2]

Plastic Films	Water vapor transmission rate (g/m <sup>2</sup> /day/atm) at 38 °C 90 %RH
Low density polyethylene (LDPE)	18
High density polyethylene (HDPE)	7-10
Polypropylene, oriented	6-7
Polypropylene, cast	10-12

#### 2.3.1.1 Basil packaging

Basil is one of the popular fresh culinary herbs around the world. It is highly perishable after harvest resulting in short shelf life. It should be kept above 10°C (50°F) to avoid chilling injury at low temperature storage. Browning of the leaves and growing tip, bronzing of the leaf veins, and loss of the glossy aspect of the leaves are all symptoms of chilling injury. The relative humidity for storing basil should be higher than 95 % [5].

Kotepong et al. (2015) [10] studied the effect of packaging type on the shelf life of basil and sweet basil. The characteristics of all plastic films used in this study were shown in **Table 2.4**. Sweet basil and basil were packed and stored at 5, 10, 15, 20 and 25 °C. The packaging test such as weight loss, color change and consumer acceptance were investigated. The result showed that the appropriate storage temperatures of sweet basil and basil were 10 and 15 °C. The suitable packaging materials for basil were LDPE and PE, while the suitable packaging materials for



sweet basil were LDPE and PP. Summary resulted of packaging test was shown in **Table 2.5**.

**Table 2.4** Characteristics of plastic films used in Kotepong et al. (2015)'s study [10]

Type	Thickness ( $\mu\text{m}$ )	Water vapor transmission rate ( $\text{g}/\text{m}^2/\text{day}$ )	Oxygen transmission rate ( $\text{cc}/\text{m}^2/\text{day}$ )	Carbon dioxide transmission rate ( $\text{cc}/\text{m}^2/\text{day}$ )
LDPE	26	26.6	14,000	6,240
HDPE	23	14.5	7,250	2,075
PE	62	5.76	2,825	1,215
PP	30	11.6	3,470	984

**Table 2.5** Summary resulted of packaging test [10]

Produce	Type of package	Weight loss	Customer Acceptance (1-5 point)	Color change (1-5 point)	Storage life (Day)
Basil (10 and 15 °C)	LDPE and PE	1.62-2.89%	3-3.3	2.7-2.8	6
Sweet basil (10 and 15 °C)	LDPE and PP	4.23-5.69%	3-3.2	3.3-3.6	9

- Color change (point 1 = 0-20%, point 2 = 21-40%, point3 = 41-60%, point4 = 61-80%, point5 = 81-100%)
- Consumer acceptance (5-point hedonic scale of overall smell, taste, texture and freshness)

Niamthong et al. (2007) [6] studied the effect of temperature and MAP on basil storage. The storage temperature in this study are 5, 10 and 25 °C. The PE-1, PE-2, and PE-3 are the polyethylene films employed in this study. The oxygen transmission rates (OTR) of the PE-3, PE-2, and PE-1 films are 9,000, 13,000, and 18,000 cc/m<sup>2</sup>day, respectively, while the water vapor transmission rates (WVTR) are 80, 130, and 180 g/m<sup>2</sup>day. The result showed that the best temperature for storing holy basil was at 10°C, while holy basil stored at 5°C experienced severe chilling injury symptoms. Basil was yellowing and lost weight very rapid at 25°C due to significant dehydration rates. It was found that modified environment packaging containing PE-1, PE-2, and PE-3 at 10°C can maintain holy basil in good condition and prolong its maximum shelf life by 9 days.

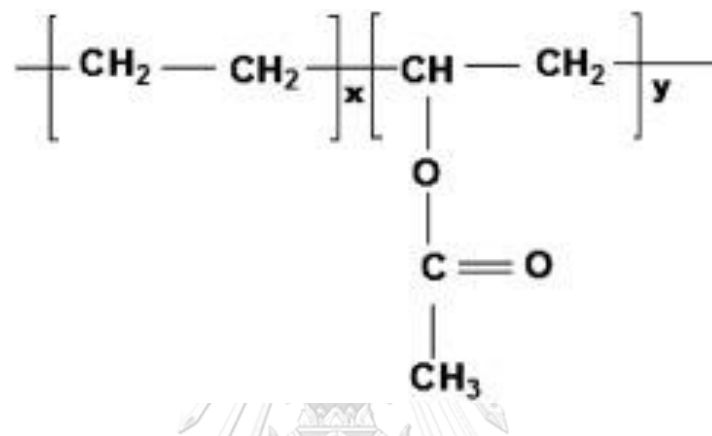
### 2.3.2 Ethylene-vinyl acetate copolymer composite films packaging

Due to the high barrier properties of commercial fresh produce packaging to water vapor, which result in moisture condensation inside packaging, the development of fresh produce packaging with low barrier properties to water vapor and the capacity to prevent moisture condensation inside packaging is interesting.

#### 2.3.2.1 Ethylene-vinyl acetate (EVA)

Ethylene-vinyl acetate or EVA is a copolymer of ethylene and vinyl acetate. It is an elastic and thermoplastic copolymer. The properties of this copolymer depending on vinyl acetate content. Normally EVA contains 1-50% of the vinyl acetate comonomer [16]. When the content of vinyl acetate comonomer increase its results is reducing crystallinity and thus lowering melting point of copolymer, until at 50% of vinyl acetate comonomer the EVA copolymer is totally amorphous [17]. The

barrier properties to gas and moisture of EVA copolymer are lower when compared with commercial fresh produce packaging polymer such as low and high polyethylene (LDPE and HDPE), polypropylene (PP), polyvinylchloride (PVC) [2]. Moreover, the barrier properties to gas and moisture reducing when vinyl acetate comonomer content increase. The chemical structure of EVA is shown in **Figure 2.7**



**Figure 2.7** Chemical Repeating unit of EVA [18]

Qing-ping SHI et al. (2011) [19] studied the effect of EVA (with vinyl acetate group 5%) content on mechanical and barrier properties of LDPE polymer film. EVA / LDPE mass ratios of 0/50, 1/49, 1/19, 4/21, and 1/3 were used to make six distinct films. The gas and moisture barrier properties and mechanical properties of these six films were investigated. The result showed that when the content of EVA copolymer increasing the oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) was increased due to reducing crystallinity. In term of WVTR not only reducing crystallinity but also increasing of polar group of vinyl acetate group in EVA copolymer.

**Table 2.6** Effect of EVA content on gas barrier properties (OTR and WVTR) of LDPE polymer [19]

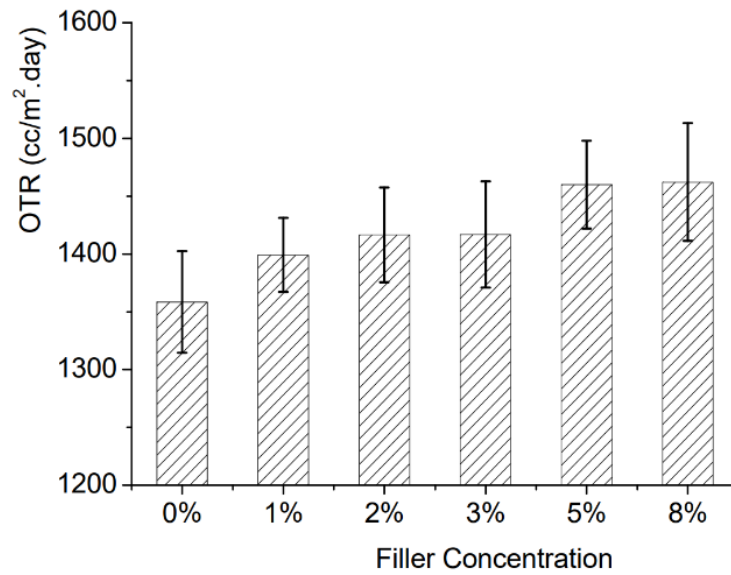
Code	EVA content%	Oxygen transmission rate (cc/m <sup>2</sup> /day)	Water vapor transmission rate (g/m <sup>2</sup> /day)
1	0	2607	7.071
2	2	2618	7.206
3	5	2724	7.419
4	10	2740	7.824
5	16	2798	8.753
6	25	2823	9.318

The additives that are used to improve the properties of the ethylene-vinyl acetate polymer matrix are diatomaceous earth and sorbitan monostearate. Diatomaceous earth acts as a moisture absorber to produce a plastic/desiccant-based moisture-absorbing structure [20]. Sorbitan monostearate acts as an antifogging agent. These two additives are used to produce fresh produce packaging that has low barrier properties to water vapor and has the ability to prevent moisture condensation inside packaging.

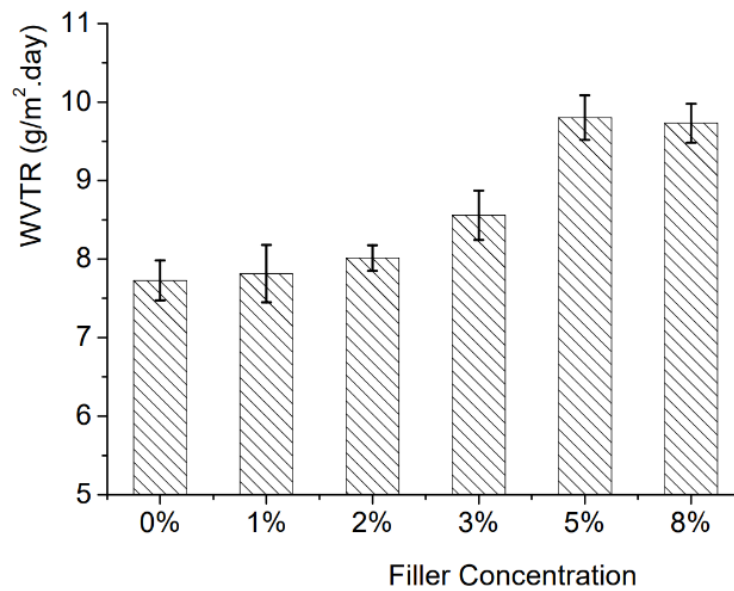
### 2.3.2.2 Diatomaceous earth (Moisture absorber)

Diatomaceous earth is one of silica-based materials. It consists largely of amorphous silicon (mineral silicic acid), 2-4% of alumina and small amount of iron oxides. Due to its high porosity, high silica and natural, abundant and cheap resource so it can widely be used as catalyst, filter aid, absorbent and inorganic filler in polymer matrix [20].

Shao-yun Huang et al. (2010) [21] studied on the preparation of high gas permeable film and its application in fruit preservation by blending diatomite with LDPE polymer matrix. Five films with different diatomite filler concentration were obtained respectively: 1%, 2%, 3%, 5%, 8% and one film with no filler concentration was used as control. The result showed that OTR and WVTR of films increased with increasing diatomite filler concentration these results were shown in **Figure 2.8** and **Figure 2.9** respectively. These happened because Cavity or bubble emerged between two phases when diatomite filler is incompatible with LDPE polymer matrix. Gas molecules can permeate through these flaws. In addition, diatomite had high porous structure so gas molecules can permeate through these pores.

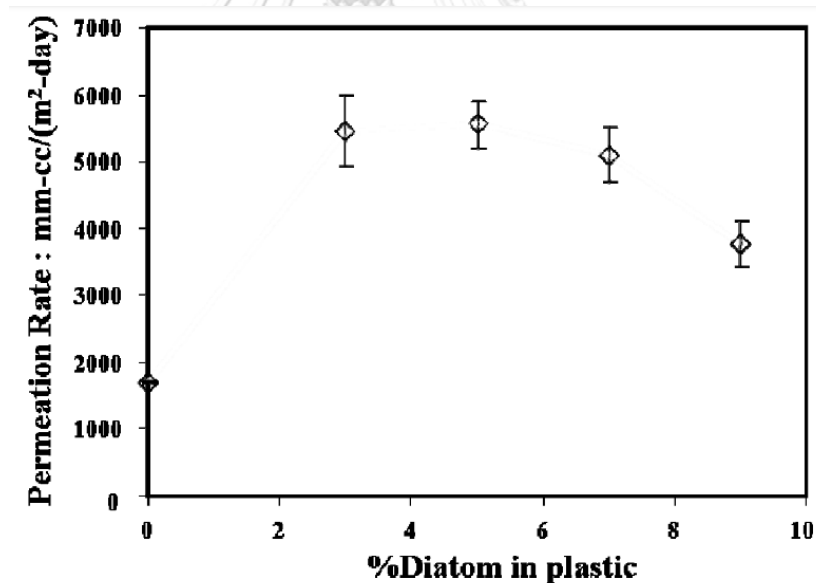


*Figure 2.8 Effect of diatomite concentration (0-8%) on oxygen transmission rate of LDPE polymer [21]*

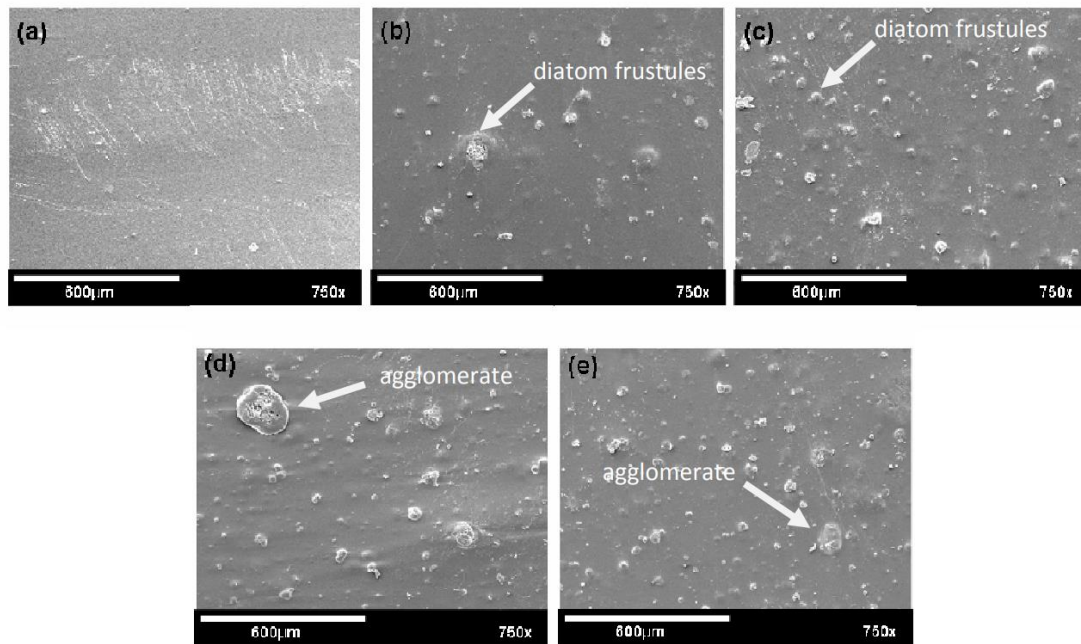


*Figure 2.9 Effect of diatomite concentration (0-8%) on water vapor transmission rate of LDPE polymer [21]*

Siraratprapa et al. (2010) [22] studied the effect of diatomite filler concentration on gas barrier properties ( $\text{CO}_2$  permeation) and morphology of EVA (with 18% vinyl-acetate group) copolymer composite films. Four films with different diatomite filler concentration were obtained respectively: 3%, 5%, 7%, 9% and one film without diatomite was used as control. Gas barrier properties resulted in **Figure 2.10** show that all EVA/diatomite polymer composite films had higher  $\text{CO}_2$  permeation rate than EVA pure film due to the creation of micron-scale defects at the EVA/diatomite interface. Moreover, the diatom frustule filled in EVA matrix induces disruption of the EVA chain packing as increase in the size of free volume. However, as can be further confirmed in SEM images in **Figure 2.11**, when diatomite filler concentration is higher than 5 wt%, its  $\text{CO}_2$  permeation significantly decreases because of the agglomeration of diatomite.



*Figure 2.10* Effect of diatomite concentration (0, 3, 5, 7 and 9%) on carbon dioxide transmission rate of EVA polymer [22]



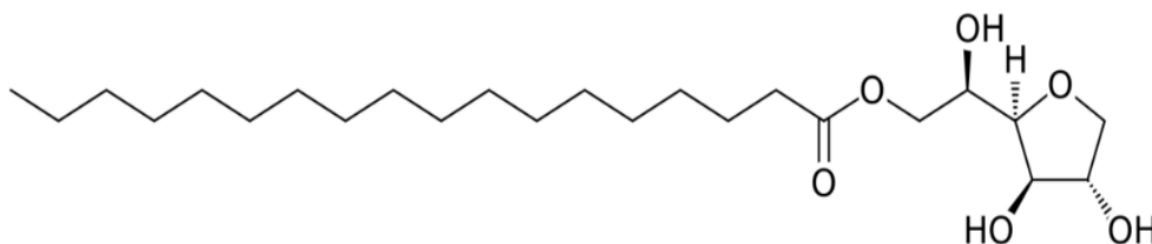
*Figure 2.11 SEM images of EVA-diatomite polymer composite film at 0, 3, 5, 7 and 9% respectively (a-e) [22]*

### 2.3.2.3 Sorbitan monostearate (Antifogging agent)

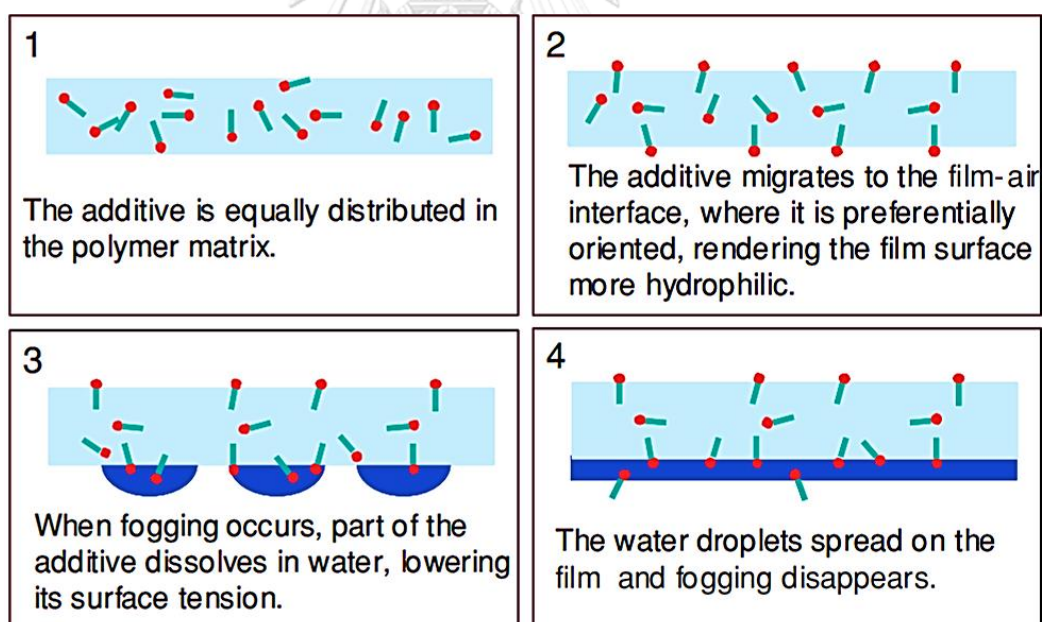
Sorbitan monostearate (SM) or Span 60 is an emulsifier esterified from sorbitol and stearic acid with the European food additive number E491. It is one of non-ionic surfactant that has wetting, dispersing and emulsifying properties [23]. It can be used as food additive, cosmetic and plastic additive in normally plastics such as ethylene-vinyl acetate (EVA) and linear low-density polyethylene (LDPE). In term of plastic additive, due to its structure, which consists of two main parts: a hydrophilic head and a hydrophobic tail when incorporate to polyethylene polymer matrix that has non-polar in nature the hydrophilic head of the antifogging agent migrates to the film surface and decreases the interfacial tension difference between the non-polar polymer and the water droplets. As a result, it plays an important role as antifogging properties—the properties that have the ability to spread water droplets on the films surface in order to prevent water droplets condensation on the films surface [24].



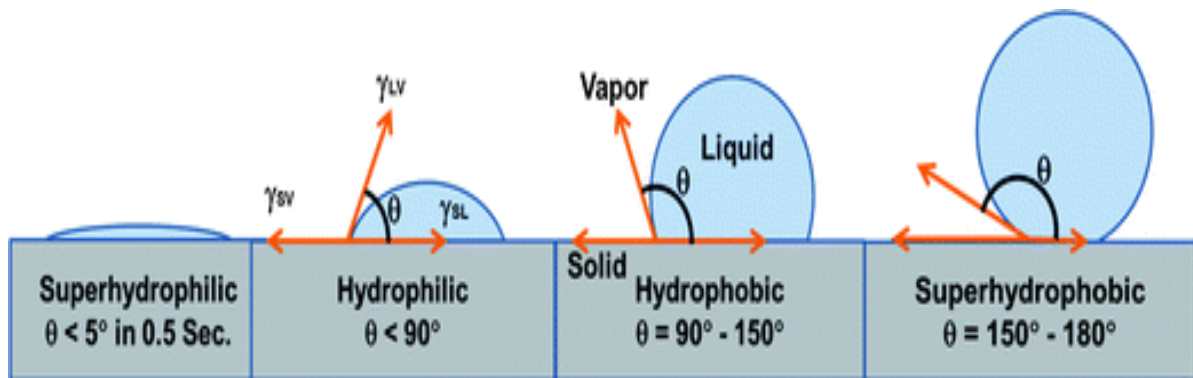
The chemical structure of SM was shown in **Figure 2.12**. Mechanism as antifogging agent and type of water contact angle on solid surface range from superhydrophilic to superhydrophobic were shown in **Figure 2.13** and **Figure 2.14** respectively.



**Figure 2.12** Chemical structure of sorbitan monostearate (SM) ( $C_{18}H_{34}O_2$ ) [23]



**Figure 2.13** Antifogging agent mechanism in polymer matrix [24]



*Figure 2.14 Water contact angle of solid surface from superhydrophilic to superhydrophobic [25]*

Waldo-Mendoza et al. (2017) [26] Studied effect of non-ionic surfactant on fogging control of LDPE/EVA bilayer films. Seven types of non-ionic surfactant which are Glycerol ester (GE), Octadecanoic acid (OA), Polyglycerol ester (PE), Polyglycerol stearate; 1,2,3-Propanetriol (PS), Polyglycerol monostearate (PM), Sorbitan monostearate (SM) and Glycerol monooleate (GM) were used. The bilayer film which layer A made of EVA (15% of vinyl acetate groups) with 3% all types of non-ionic surfactant and layer B made of pure LDPE with 1% all types of non-ionic surfactant was made. Water contact angle measurements of each layer of all bilayer films were recorded. The resulted in **Table 2.7** was shown that all non-ionic surfactant can significantly decrease water contact angle of plastic films so the wettability of all plastic films increasing. Regarding the membranes with nonionic surfactants, It was a non-significant difference between layer A and B for the non-ionic surfactant compound SM, GM, GE and OA but it was a significant difference between layer A and B for the non-ionic surfactant compound PE, PS and PM. The molecular weight of a non-ionic surfactant compound that show in **Table 2.8** can be used to explain this. Because the diffusion rate to the surface of plastic films was sluggish for PE, PS, and PM due to their large molecular weight, their antifogging performance was good, as it was for SM, GM, GE, and OA. When compare the content of non-ionic surfactant

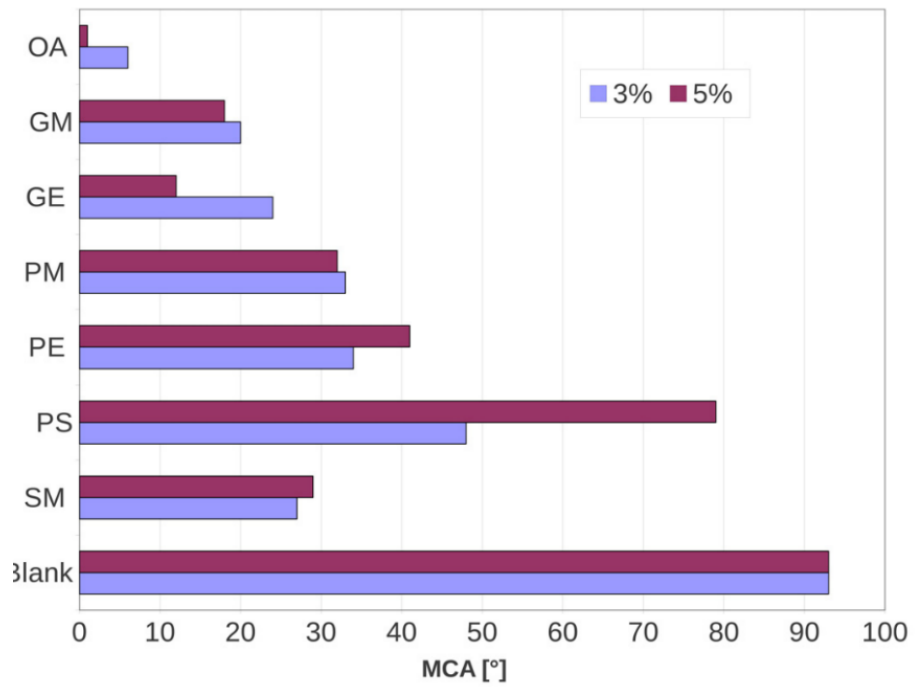
for 3% and 5% of non-ionic concentration in layer A (EVA) the resulted of water contact angle measurement in **Figure 2.15** show that it was not significantly different.

**Table 2.7** Mean contact angle (MCA) of EVA (layer A) with 3% nonionic surfactant and pure LDPE (layer B) with 1% nonionic surfactant [26]

Film type	Layer A	Layer B
	MCA	MCB
Blank	93±2	99±2
OA	6±1	0
GM	20±2	27±3
GE	24±2	17±2
SM	27±6	28±4
PM	33±3	60±5
PE	34±8	64±8
PS	48±2	89±4

**Table 2.8** Molecular weight of all non-ionic surfactants [26]

Type of surfactant	Molecular weight (g/mol)
OA	284.48
GM	358.56
GE	356.55
SM	430.62
PM	506.72
PE	420
PS	726.48



*Figure 2.15 Effect of 3 and 5% all non-ionic surfactant on mean contact angle of layer A (EVA 15% of vinyl acetate groups) [26]*



## CHAPTER III

### METHODOLOGY

In this chapter, the researching process include materials, film preparation, film characterization and basil packaging test, are described as follows.

#### 3.1 Materials

Ethylene vinyl acetate (EVA) with 15% vinyl acetate group was obtained from TPI polene under the trade name EVAN8036. Diatomaceous earth was obtained from Chems R Us Co., Ltd under the trade name Celite Super Floss. The median particle size and specific gravity of diatomaceous earth were 7.5  $\mu\text{m}$  and 2.3 respectively. The chemical composition of diatomaceous earth was shown in **Table 3.1**. Sorbitan monostearate (SM) was obtained from Krungthepchemi Co., Ltd under trade name SPAN 60. It has melting temperature at 52-54  $^{\circ}\text{C}$ .

**Table 3.1** Chemical composition of diatomaceous earth

Type of chemical	(% by weight)
$\text{SiO}_2$	89.6
$\text{Al}_2\text{O}_3$	4.0
$\text{Fe}_2\text{O}_3$	1.3
$\text{P}_2\text{O}_5$	0.2
CaO	0.5
MgO	0.6
$\text{Na}_2\text{O} + \text{K}_2\text{O}$	3.3

#### 3.2 Film preparation

Film preparation is consisting of binary blend of EVA /diatomite, binary blend of EVA/SM and, tertiary blend of EVA/diatomite/SM. The blending process of all plastic films was prepared by melt blending using twin screws extruder, Thermo Hakke Rheomex, Germany. The screw speed was 60 rpm, and the temperature profile along the barrel was 150-170  $^{\circ}\text{C}$ . Before melting, raw materials except SM

were dried in an oven at 60 °C at least for 24 hours. The weight ratio for each blending was shown in **Table 3.2**, **Table 3.3** and, **Table 3.4**. When mixing completed, Mixed compounds was blown into film by using a single screw extruder, Collin, Blown film line BL 180/400E, Germany with temperature profile along barrel 120-170 °C and the screw speed was 85 rpm. The thickness of all plastic films was  $40 \pm 2 \mu\text{m}$ .

**Table 3.2** Weight ratio of binary blend of EVA / Diatomite

Sample name	EVA (wt%)	Diatomite (wt%)
EVAD3	97	3
EVAD5	95	5

- The weight ratio of diatomite was varied at 3 and 5% wt. This is because, as discussed in Chapter II, the highest weight ratio performance of diatomaceous in EVA and LDPE polymer matrix was 5% wt.

**Table 3.3** Weight ratio of binary blend of EVA / SM

Sample name	EVA (wt%)	SM (wt%)
EVASM3	97	3

- The weight ratio of SM was fixed at 3% wt. This is because, as discussed in Chapter II, the weight ratio of SM in EVA polymer matrix was 3 % wt and when increasing the weight ratio of SM to 5 % wt, the contact angle resulted was not significant difference.

**Table 3.4** Weight ratio of tertiary blend of EVA / SM/ Diatomite

Sample name	EVA (phr)	Diatomite (phr)	SM (phr)
EVADSM	100	3	3

- The weight ratio in tertiary blend of EVA / SM/ Diatomite was carried out in unit of part per hundred (phr) because it is easier for processing when mixing compound more than 2 component. The content of SM and diatomite is 3 phr (approximately 2.83%wt) because it may be good optimizing performance between gas and moisture barrier properties, mechanical properties, water contact angle, and clarity of EVA copolymer composite films.

### **3.3 Properties of the copolymer composite films**

#### **3.3.1 Analysis of water vapor permeability**

Water vapor transmission rate (WVTR) of all EVA copolymer composite films was measured by using water vapor permeation analyzer; PERMATRAN-W Model 398, Mocon, USA at 37.8 °C and 90% RH (relative humidity) according to ASTM E-398. The copolymer composite films were cut into a circle mold of equipment with a 50 cm<sup>2</sup> surface area. In order to achieve the precise value, four films were tested.

#### **3.3.2 Analysis of oxygen permeability**

Oxygen transmission rate (OTR) of all EVA copolymer composite films was measured by using oxygen permeation analyzer; OX-TRAN 2/21, Mocon, USA according to ASTM D3985. at 23°C, 0% relative humidity, and a 40 cm<sup>3</sup> /min oxygen flow rate. The copolymer composite films were cut into a circle mold of equipment with a 100 cm<sup>2</sup> surface area. In order to achieve the precise value, four films were tested.

#### **3.3.3 Mechanical analysis**

Tensile test of all EVA copolymer composite films was measured according to ASTM D882 using Universal Testing Machine (Instron 5567, NY, USA). Tensile properties such as tensile strength at break and percentage of elongation at break were determined using it. All of the EVA polymer composite films were cut into rectangular shapes with a 20 mm width and a 100 mm length. With a 1 kN load cell, the gauge length and grip separation rate are 50 mm and 50 mm/min, respectively. To get the exact value, five films were tested.

### 3.3.4 Contact angle analysis

Water contact angle of all EVA copolymer composite films was measured by using contact angle tester (Kruss/DSA 10MK2). All of EVA copolymer composite films was placed on a glass slide. A water droplet was dropped slowly on glass slide and contact angle was measured. To get the exact value, five films were tested.

### 3.3.5 Cold fog analysis

Two-thirds of a 250 mL beaker is filled with tap water. The beaker is covered with selected EVA copolymer composite films before being refrigerated for three hours (5 °C). A photograph is taken of the water condensation on all of the testing films.

### 3.3.6 Opacity analysis

Opacity properties of all EVA copolymer composite films was measured by using Oakland RT-6000 Opacity meter. The Oakland Instrument Model RT-6000 is a visible-light source/photoelectric-detector-based off-line film opacity meter. It measures the amount of visible light absorbed/reflected by a film sample when that sample is placed between the light source and detector. The copolymer composite films were cut into a A4 shape and placed between the light source and detector. To get the exact value, five films were tested.

## 3.4 Application of the copolymer composite films on the storage of holy basil

Fresh and pre-washed holy basil was obtained from PDI trading Co., Ltd., Thailand. The 80 g of the holy basil with uniform in size, color, shape, and free of damage were packed into a selected copolymer composite pouch or commercial perforated polypropylene pouch (15×40 cm<sup>2</sup>). The sample pouches were kept in a temperature-controlled cabinet at 10±0.5 °C for 3 weeks. Relative humidity and temperature of the atmosphere inside the pouches during storage was recorded by the temperature-humidity data logger packed in each pouch. Sampling was done periodically for the following quality determination.



#### 3.4.1 Weight loss of holy basil during storage

The weight loss of basil packed in all test packaging was analyzed by periodically weighed to calculate the percentage weight loss by using equation

$$\% \text{Weight loss} = \frac{(\text{initial weight} - \text{present weight})}{\text{initial weight}} \times 100 \quad \text{Equation 3.1}$$

#### 3.4.2 Appearance of holy basil during storage

Images of the holy basil packed in all test packaging at specific storage time were taken to consider the overall appearance of the holy basil.

#### 3.4.3 Deteriorated proportion of holy basil during storage

Parts of holy basil which notably deteriorated such as color change, and wilting in each test packaging were sorted out and weighed to calculate the percentage of deteriorated basil by using the following equation.

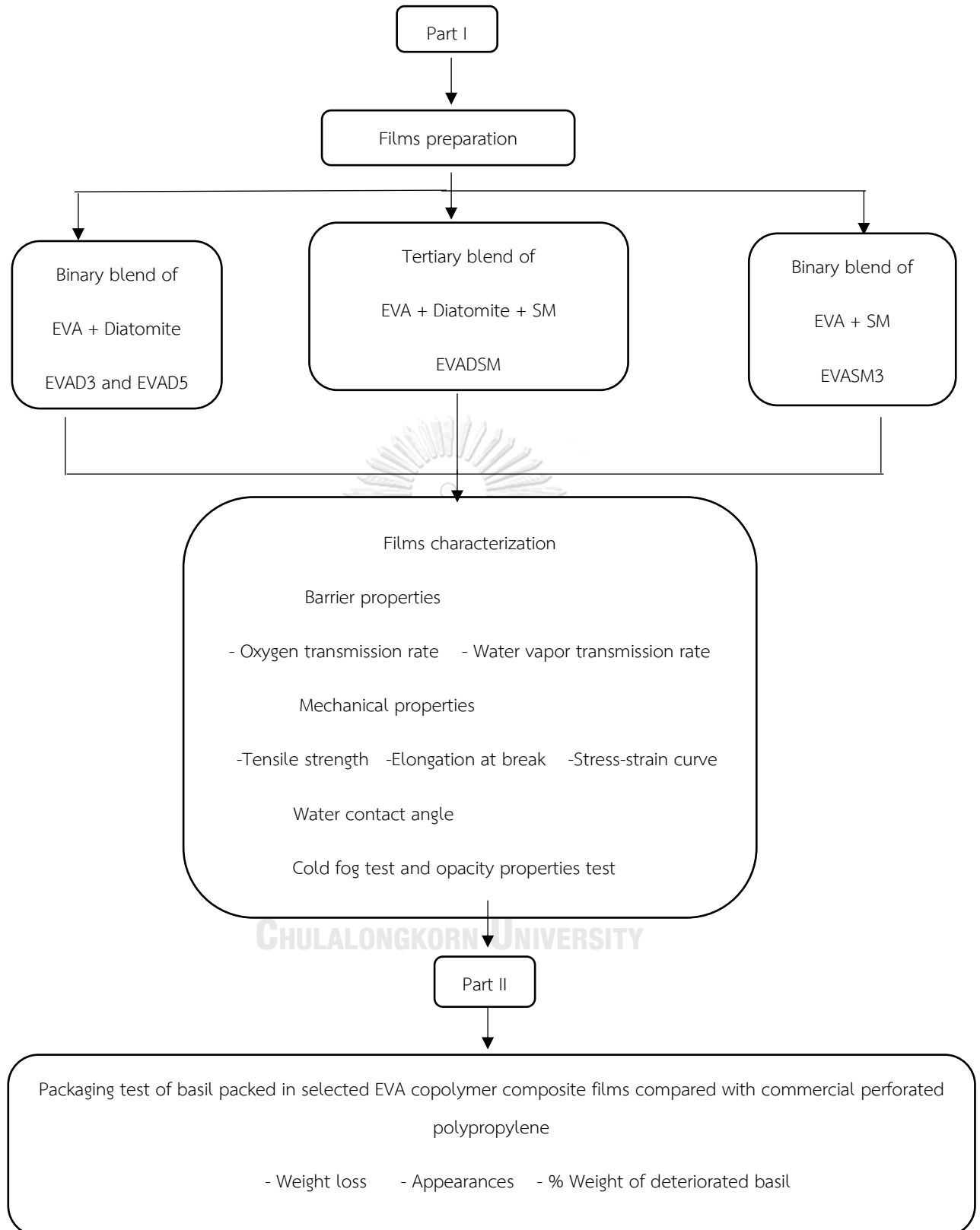
$$\% \text{ Deteriorated basil} = \frac{\text{Weight of deteriorated basil}}{\text{Weight of all basil}} \times 100 \quad \text{Equation 3.3}$$

#### 3.4.4 Temperature and relative humidity during storage

Temperature and relative humidity inside packaging during storage were recorded by using temperature and relative humidity data logger

### 3.5 Methodology

In this research, experiment was separate in two parts. Part I is the preparation of EVA copolymer composite films and characterization including gas barrier, mechanical, opacity and water contact angle properties. Part II is comparison of the efficiency of selected EVA copolymer composite films from part I and commercial perforated polypropylene films for storage of basil.



**Figure 3.1** Methodology of this research

## CHAPTER IV

### RESULTS AND DISCUSSION

#### 4.1 Properties of the copolymer composite films

##### 4.1.1 Gas barrier properties

The resulted of water vapor transmission rate (WVTR) and oxygen transmission rate (OTR) of all EVA copolymer composites were shown in **Table 4.1** and **Figure 4.1**. It can be seen that when the concentration of diatomite additive increases the OTR and WVTR are increased. This is due to the porous and amorphous structure of diatomite additive itself and the cavity emerging in interface between diatomite additive and polymer matrix that enhance the overall free volume of the polymer. In terms of SM additive content, it can be seen that when incorporating SM additive in the polymer matrix the WVTR results are not significantly different from neat polymer and the OTR results are much lower than that of neat polymer. In terms of WVTR, although hydrophilicity strongly increases, the physical adhesion by SM additive to polymer matrix that enhance the dense structure of overall polymer made the difficulty of water vapor molecule to transmitted. In terms of OTR, SM additive in the EVA copolymer composite films decreased OTR dramatically

Table 4.1 Gas barrier properties of all EVA copolymer composite films

Film	WVTR (g/m <sup>2</sup> /day)	OTR (cc/m <sup>2</sup> /day)
EVA15	73.88±3.49	9983.47±149.37
EVAD3	86.07±1.98	10959.53± 163.54
EVAD5	87.60±3.88	11342.8±149.30
EVASM3	73.90±3.23	3662.48±93.69
EVADSM	85.83±3.20	3785.25±197.40

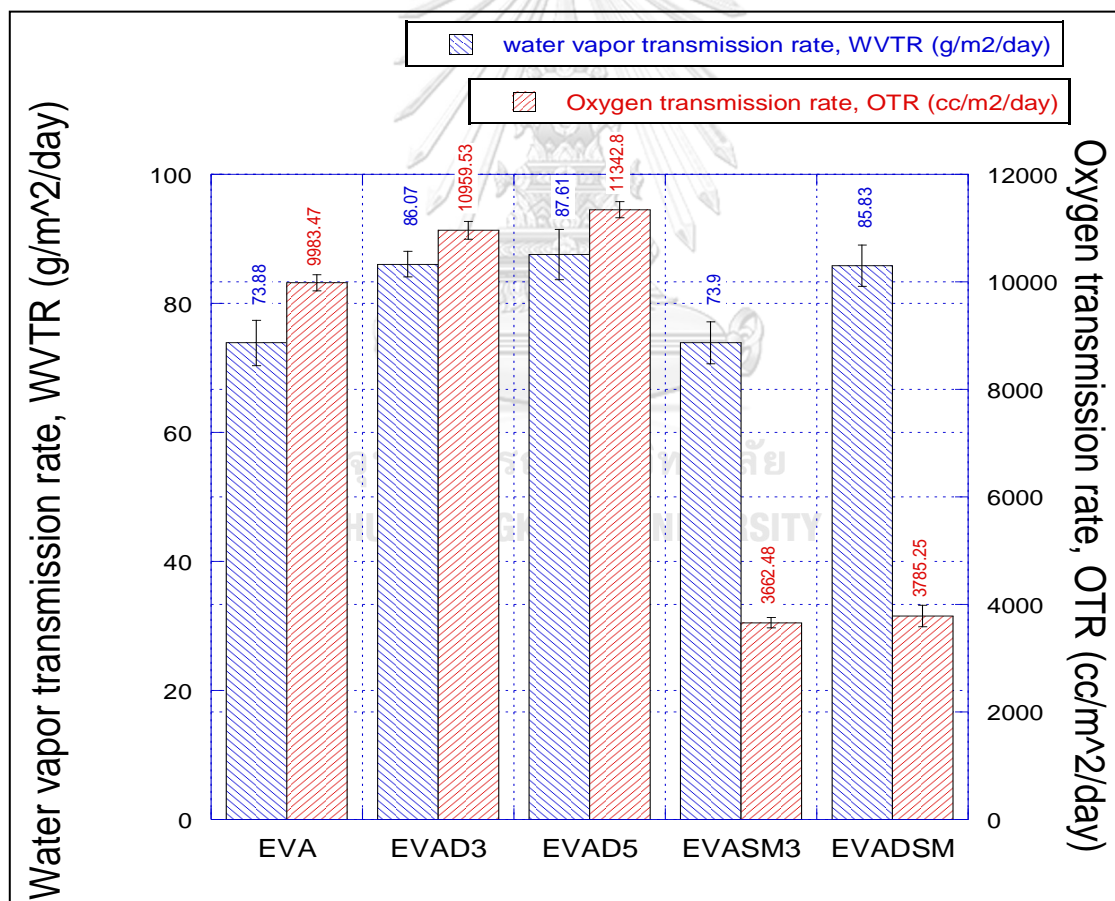


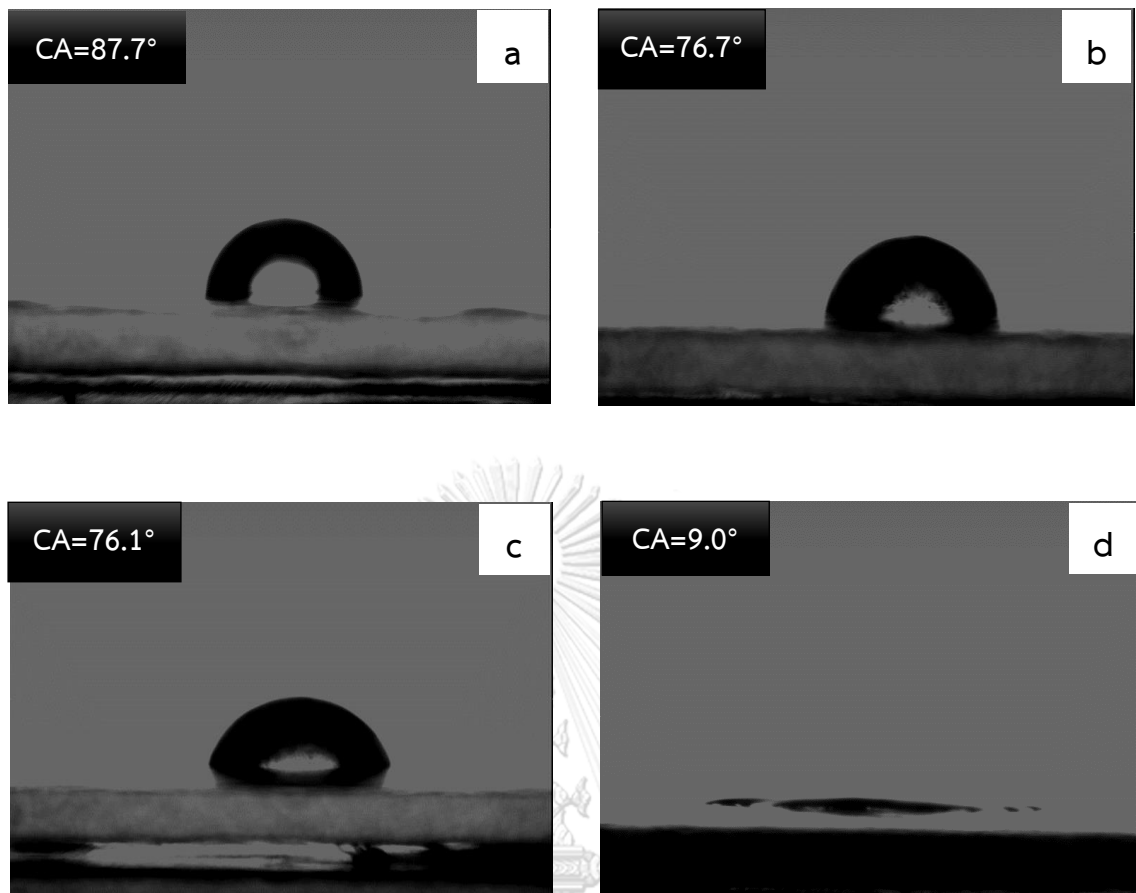
Figure 4.1 Effect of diatomite and SM additives on oxygen and water vapor transmission rate of all EVA copolymer composite films

#### 4.1.2 Water contact angle properties

The water contact angle results of all EVA copolymers composite were shown in **Table 4.2** and **Figure 4.2**. It can be seen that by loading the diatomite to the EVA polymer matrix the water contact angle was reduced. This is due to the hydrophilic silanol group (Si-OH) on the diatomite surface that leads to a better interaction of water droplets with the surface of EVA copolymer than that of neat EVA copolymer. When focusing on the SM content, the results show that by loading the SM to the EVA polymer matrix the water contact angle was dramatically reduced. Many strong hydrophilic functional groups of SM structure lead to a great polarity of EVA copolymer surface resulting in high hydrophilic surface properties.

**Table 4.2** The contact angle of all EVA copolymer composite films

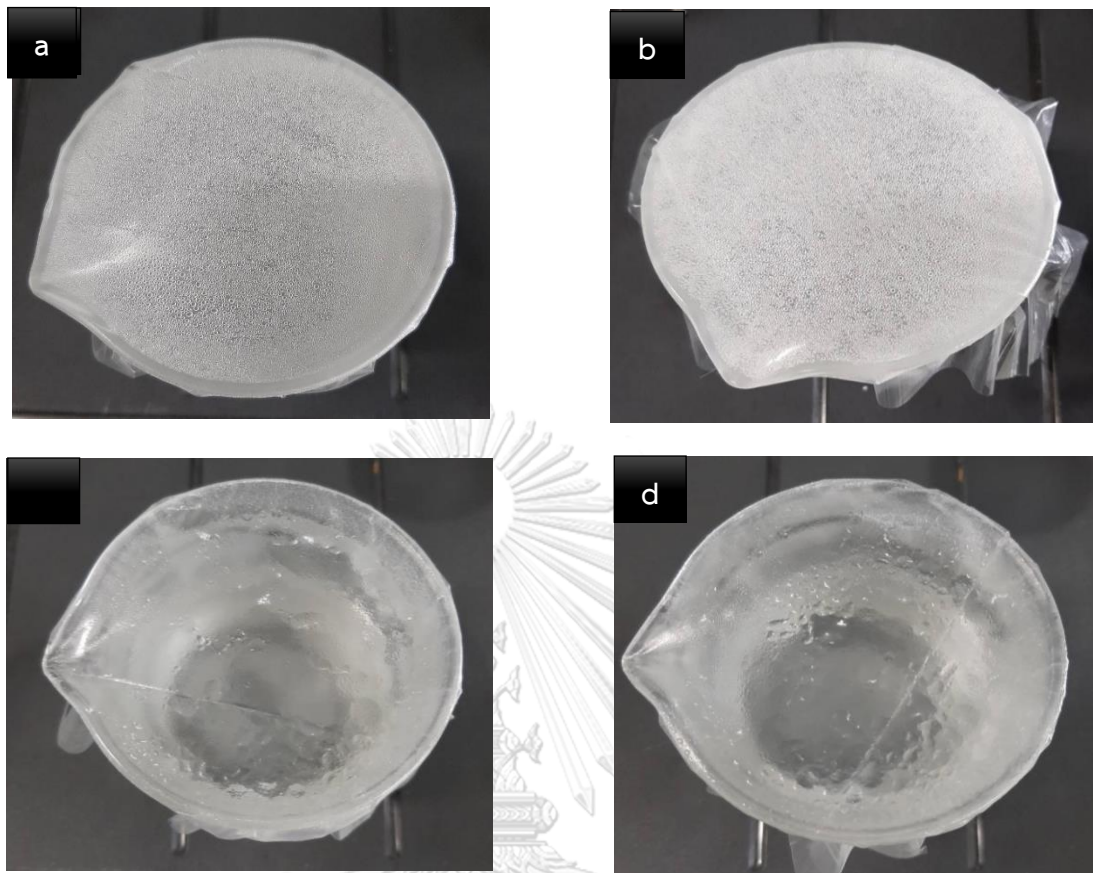
Film	Contact angle (°)
EVA15	87.7±0.10
EVAD3	76.7±0.10
EVAD5	76.1±0.10
EVASM3	9.0±0.10
EVADSM	NA



*Figure 4.2 Effect of diatomite and SM additives on water contact angle of EVA copolymer composite films*

(a) EVA15 (b) EVAD3 (c) EVAD5 (d) EVASM3

#### 4.1.3 Cold fogging properties



**Figure 4.3** Effect of diatomite and SM additives on cold fogging properties of EVA copolymer composite films

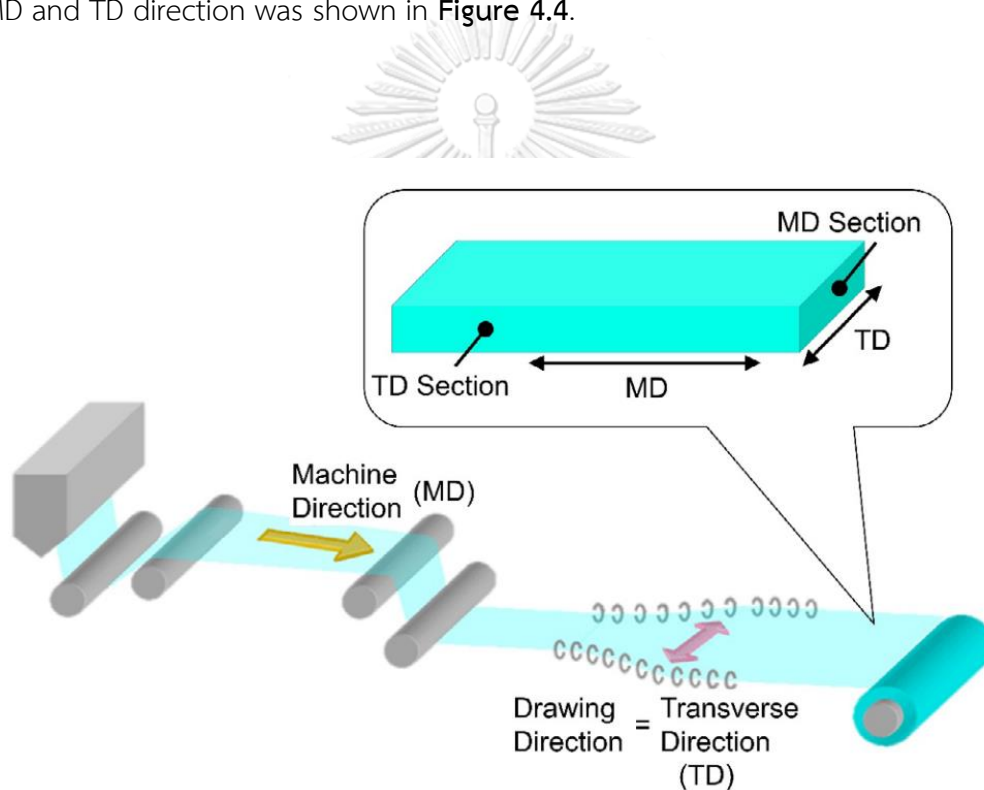
(a) EVA15 (b) EVAD3 (c) EVASM3 (d) EVADSM

The cold fogging properties resulted of all EVA copolymers composite were shown in **Figure 4.3**. It can be seen that EVA pure and EVA blend with diatomite showed very poor cold fogging properties - an opaque layer of small water droplets [27]. This indicates that the wettability enhanced by diatomite is not enough to spread water droplets on the film surface. In contrast, the samples that have SM content (EVADSM and EVASM3) showed acceptable cold fogging properties – some drops are randomly scattered [27]. This indicates that the wettability enhanced by SM can spread water droplets on the film surface and not only the strong wettability

enhanced but also the structure of SM which consists of a hydrophilic head and a hydrophobic tail made it easier to migrate from the polymer matrix to the film surface so the antifogging performance is higher than diatomite.

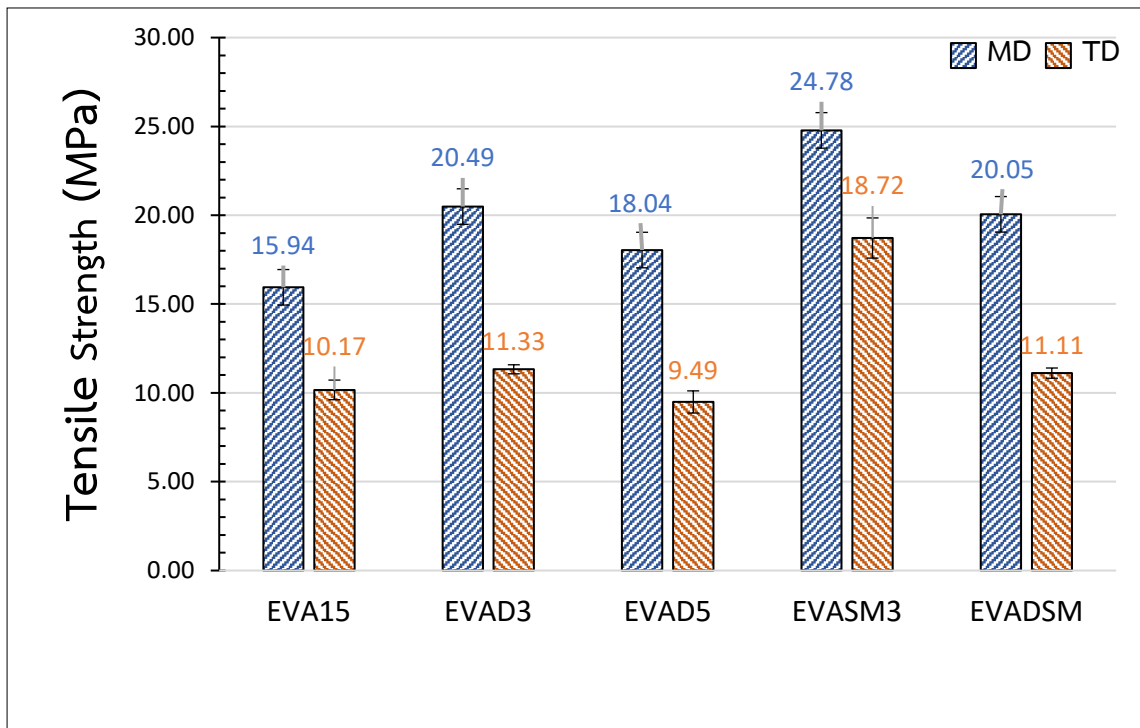
#### 4.1.4 Mechanical properties

The mechanical properties results including tensile strength, stress-strain curve and elongation at break in both machine direction (MD) and transverse direction (TD) were shown in **Figure 4.5**, **Figure 4.6** and **Figure 4.7** respectively. The MD and TD direction was shown in **Figure 4.4**.



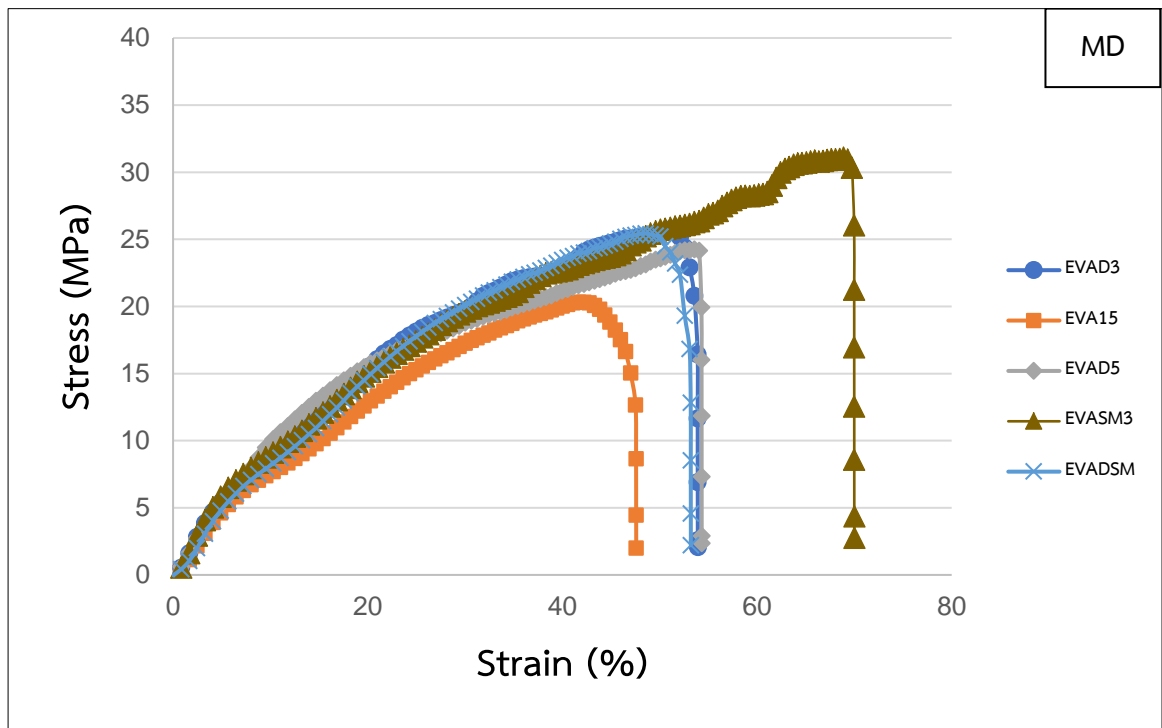
**Figure 4.4** Direction for measuring mechanical properties including MD and TD directions



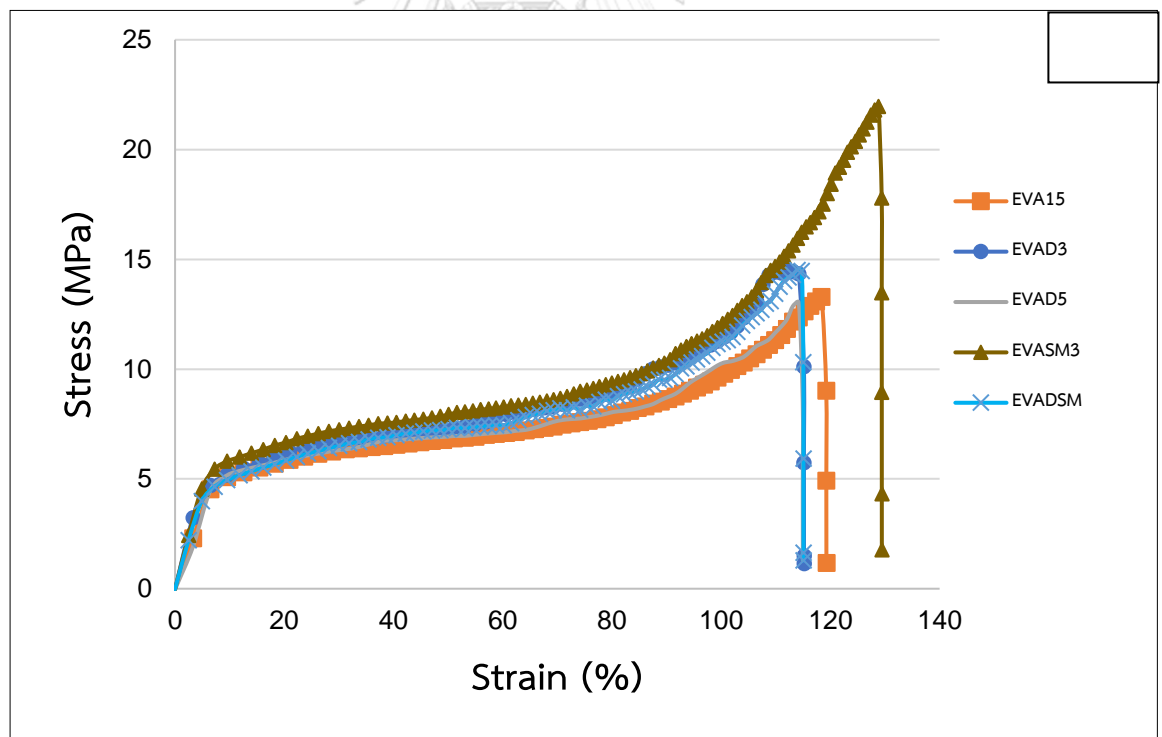


*Figure 4.5 Effect of diatomite and SM fillers on tensile strength of EVA copolymer composite films*

It can be seen that EVA copolymer composite films had a higher tensile strength compared to neat EVA pure this is because of physical adhesion improvement by the interaction between polymer matrix with diatomite and SM additives. When comparing diatomite and SM additives, the resulted show that EVA copolymer composite with SM additive (EVASM3) had highest tensile strength this can explain in two ways. One was due to the more polar groups of SM additive that can be made to have stronger interactions with polar C=O of the EVA polymer matrix. Another one was EVASM3 had much higher in strain that high strain can lead to high stress and tensile strength which is corresponding to strain hardening effect. This strain hardening effect happened in both MD and TD directions-especially in the TD direction, which can be seen in the stress-strain curve in **Figure 4.6**.



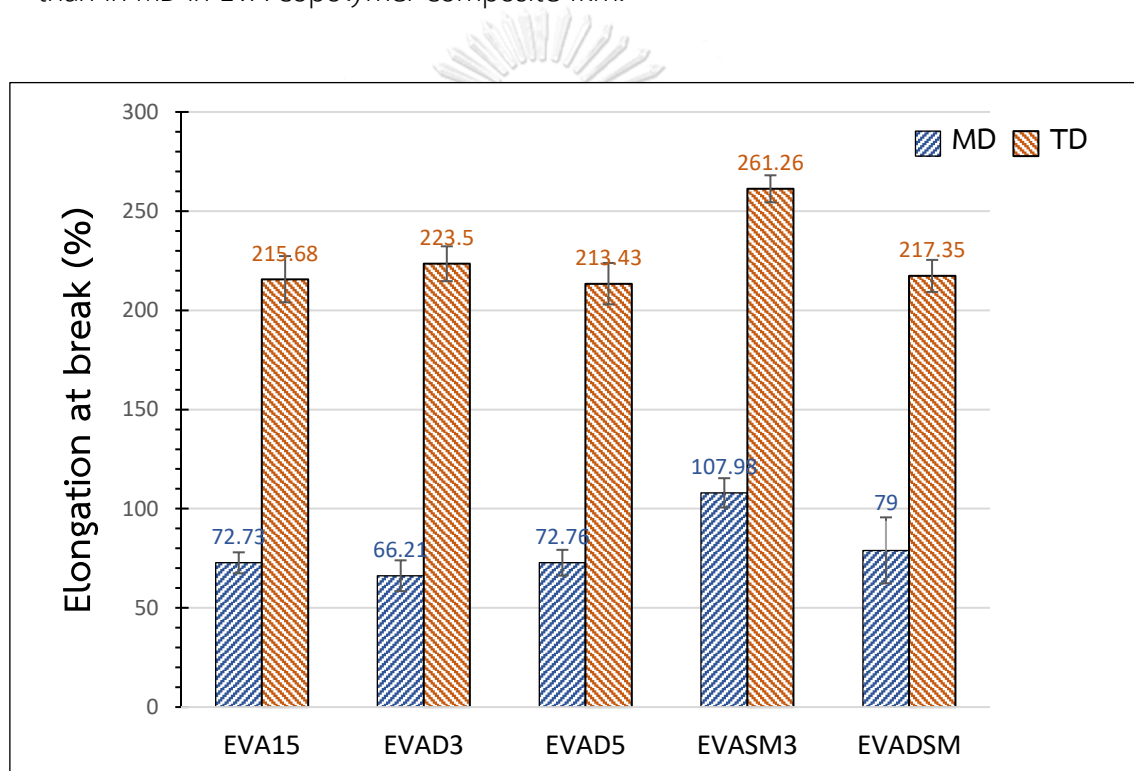
(a)



(b)

Figure 4.6 Stress-strain curve of all blends in both (a) MD and (b) TD

the stress-strain curves of all blends in both MD and TD were shown in **Figure 4.6**. It can be seen that all copolymer films had a ductile deformation behavior. All EVA copolymer composite films had a higher maximum load compared to neat EVA. The EVA blend with diatomite (EVAD3 and EVAD5) showed lower strain and lower stress when compared to EVA blend with SM. The EVASM3 film showed the highest stress and strain in both MD and TD directions. The strain hardening effect occurred in TD direction which could be caused by dislocation of atom in structure in TD more than in MD in EVA copolymer composite film.



*Figure 4.7 Effect of diatomite and SM fillers on elongation at break of EVA copolymer composite films*

The elongation at break of all blends in both MD and TD were shown in **Figure 4.7**. It can be seen that the elongation at break of EVA blend with diatomite does not show significant difference compared with that of neat EVA film, so the flexibility of EVA copolymer does not improve by diatomite additive. On the contrary,

the elongation at break of EVA copolymer blend with SM (EVASM3) in both MD and TD direction were much higher than that of neat EVA so the flexibility of neat EVA copolymer has increased by blending with SM additive.

#### 4.1.5 Opacity properties

**Table 4.3** *The opacity properties of all EVA copolymer composite films*

Film	% Opacity
EVA15	5.80
EVAD3	5.40
EVAD5	6.40
EVASM3	5.80
EVADSM	7.40

The opacity properties results that showed in **Table 4.3** indicates that the EVA copolymer with additives content (EVAD5 and EVADSM) had higher percentage of opaque properties when compared with neat EVA. It indicates that the additives content decrease the transparency of neat EVA. The EVA copolymer with both additives (EVADSM) showed the highest reduction in transparency, but the clarity of this EVA copolymer composite is applicable for use as fresh holy basil packaging.

## 4.2 Application of the copolymer composite films on the storage of holy basil

**Table 4.4** The summary properties of EVA copolymer composite films

Type	WVTR (g/m <sup>2</sup> /day)	OTR (cc/m <sup>2</sup> /day)	Antifogging properties	Opacity properties (%)	Tensile strength (MPa)	
					MD	TD
EVA15	73.88±3.49	9983.47±149.37	unacceptable	5.80	15.94±0.94	10.17±0.55
EVAD3	86.07±1.98	10959.53±163.54	unacceptable	5.40	20.49±1.07	11.33±0.25
EVAD5	87.60±3.88	11342.80±149.30	unacceptable	6.40	18.04±1.33	9.49±0.62
EVASM3	73.90±3.23	3662.47±93.69	acceptable	5.80	24.78±1.05	18.72±1.13
<b>EVADSM</b>	<b>85.83±3.20</b>	<b>3785.25±190.40</b>	<b>acceptable</b>	<b>7.40</b>	<b>20.05±0.79</b>	<b>11.11±0.29</b>

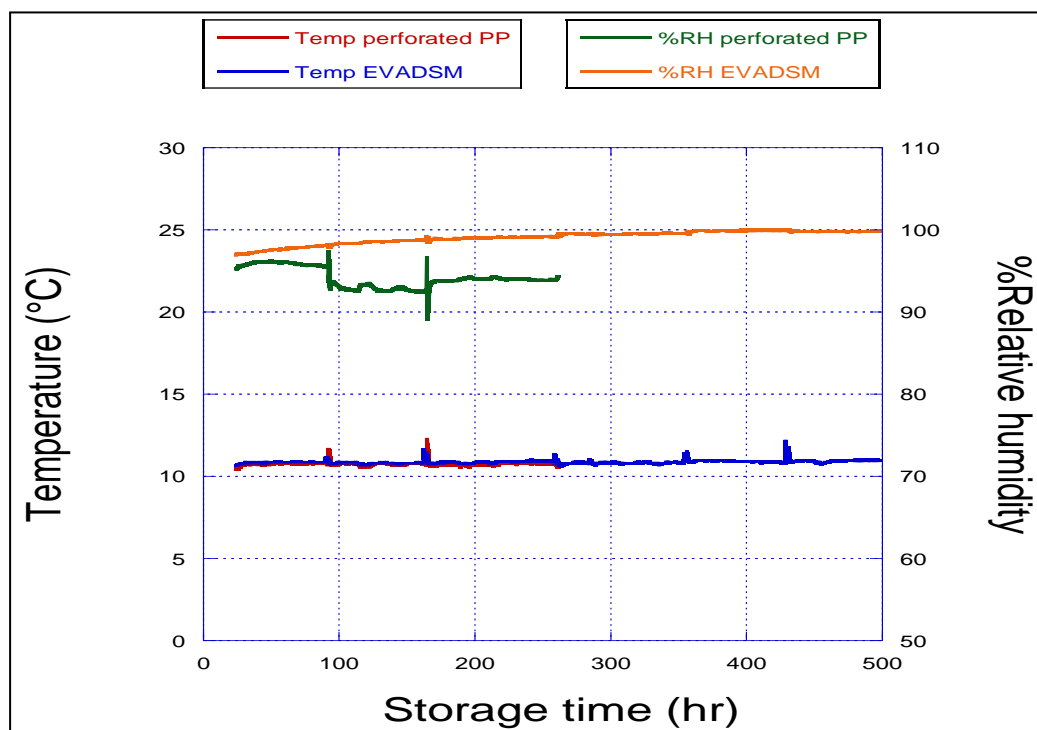
From OTR, WVTR, antifogging properties, opacity properties as well as mechanical properties which were shown in **Table 4.4**, even though EVADSM had some drawbacks, such as the highest percentage of opaque properties, the important parameters are suitable for holy basil storage included optimum gas barrier properties-high WVTR and not too high OTR that can reduce respiration rate of holy basil and good antifogging properties. The EVADSM pouch was chosen to compare the efficiency of shelf-life extension of holy basil with the commercial perforated polypropylene pouch from PDI factory. The reason for not choosing EVASM3, which is good for cost savings, had one additive content, and had most of the same summary properties as EVADSM except for lower WVTR and higher mechanical properties, is that it did not work well in the real-used application. There is difficulty to open the EVASM3 packaging film. The reason that opening the EVASM3 bag is difficult could be due to the existence of SM additive which enhances the polarity of two surfaces, which makes strong interaction between two high polar surfaces difficult to separate. In contrast, EVADSM had diatomite with SM additive. The diatomite additive can disturb and decrease the effect of SM lead to easily open the packaging film after

blown film extrusion. Therefore, the fresh holy basils were packed in both EVADSM and commercial perforated polypropylene as shown in **Figure4.8**.



**Figure 4.8** Example of basil 80 g packed in test packaging A (EVADSM) and B (commercial perforated PP).

## 4.2.1 Temperature and relative humidity during storage



**Figure 4.9** Temperature and %Relative humidity in all test packaging at different storage time

The temperature and relative humidity during storage were shown in **Figure 4.9**. The average temperature at equilibrium condition in perforated polypropylene packaging pouch was  $10.72 \pm 0.08$  °C and the average temperature at equilibrium condition in EVA copolymer composite packaging pouch was  $10.86 \pm 0.11$  °C. Both average temperatures are corresponding to the setting storage temperature of the temperature-controlled cabinet at  $10 \pm 0.5$  °C. Due to the hole that allows water vapor to readily flow from inside to outside air, the relative humidity in perforated polypropylene fluctuated and lower than the relative humidity in EVA copolymer composite. The relative humidity in the selected EVA copolymer composite gradually increased and reached an equilibrium condition of approximately 98.9%. The fluctuation of signal around 100, 150, 250, 350, and 450 hr might be caused by

opening the temperature-controlled cabinet to get the samples, at the sampling time of 3, 7, 10, 14, and 20 days, respectively.

#### 4.2.2 Weight loss of holy basil during storage

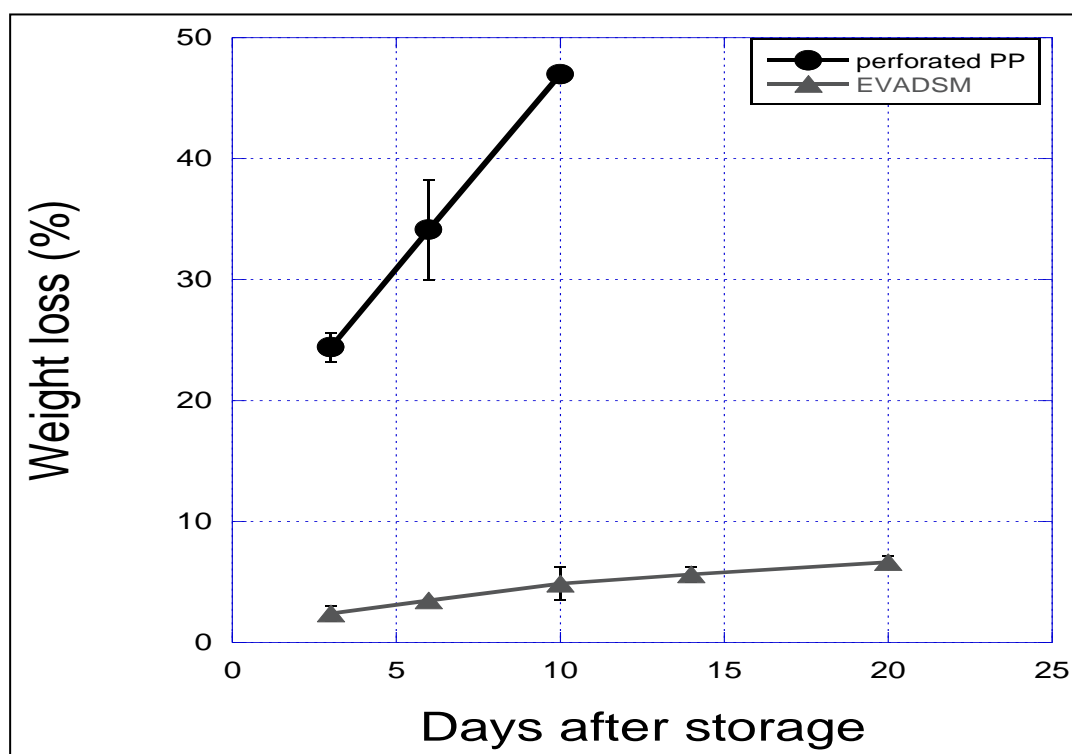


Figure 4.10 Weight loss of holy basil in all test packaging at different storage time

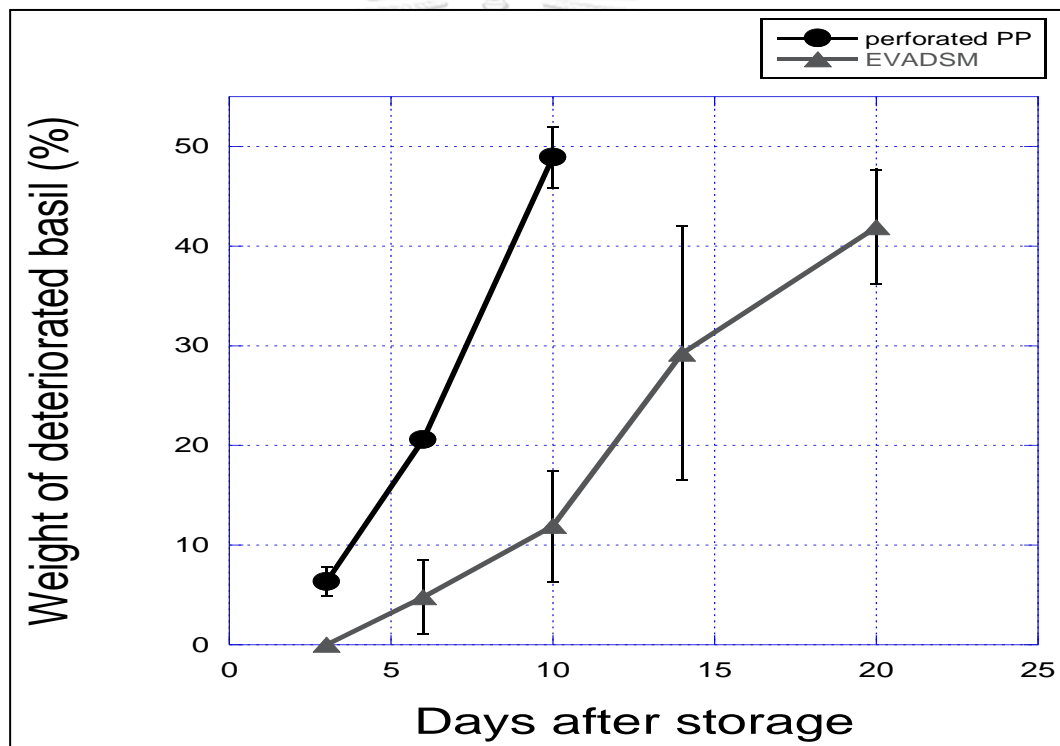
จุฬาลงกรณ์มหาวิทยาลัย  
CHULALONGKORN UNIVERSITY

Weight loss of holy basil in all plastic pouches was shown in **Figure 4.10**. At similar storage time, holy basil packed in perforated polypropylene pouch showed much higher weight loss compared to those packed in copolymer composite packaging pouch. It can be explained that, in a perforated polypropylene pouch, the oxygen from outside can easily come across the hole to the inside resulting in high respiration and dehydration rates. In term of relative humidity, the relative humidity at equilibrium inside the perforated polypropylene pouch was approximately 93.9% which can expedite weight loss of holy basil. In contrast, the copolymer composite packaging pouch had optimum water vapor and oxygen transmission rates. Migration of water vapor and oxygen across this packaging film occurred in a lower extent, in



comparison to that of the perforated polypropylene pouch, resulting in lower respiration and dehydration rates. The relative humidity at equilibrium inside the selected copolymer composite pouch was approximately 98.9%, which was comparable to the relative humidity recommended for leafy green vegetables [28]. This value was higher than that found in the perforated polypropylene pouch, and could retard the moisture loss from fresh holy basil during storage. Even if the relative humidity inside the selected EVA copolymer composite pouch was quite high. There was no water condensation and fogging occurred.

#### 4.2.3 Weight of deteriorated holy basil during storage



*Figure 4.11* Weight of deteriorated (%) of holy basil in all test packaging at different storage time

The weight of deteriorated holy basil in all plastic pouches was shown in **Figure 4.11**. It can be seen that holy basil packed in perforated polypropylene pouch showed much higher weight of deteriorated holy basil in all storage times compared with those packed in copolymer composite packaging pouch, which correspondent to the weight loss results.

#### 4.2.4 Appearances

The appearance of holy basil in all plastic pouches was shown in **Figure 4.12**. It can be seen that in perforated polypropylene packaging, The wilting of leaves occurred after 3 days of storage its indicates that the freshness of holy basil was slightly diminished after 3 days of storage. After 6 days of storage the noticeable drop in freshness and the browning of leaves occurred. Holy basil packed in perforated polypropylene was in unacceptable condition after 10 days of storage. In EVA polymer composite packaging the freshness of holy basil was maintained in good condition until 14 days of storage. After 14 days of storage, the browning of leaves is occurring, and the holy basil packed in EVA polymer composite packaging was in unacceptable condition after 20 days of storage.















	Perforated PP Test 1	Perforated PP Test 2	EVADSM Test 1	EVADSM Test 2
DAY 3				
DAY 6				
DAY 10				
DAY 14				
DAY 20				

Figure 4.12 Apperences of holy basil in all test packaging pouches at different storage times

## CHAPTER V

### CONCLUSIONS

In this research, the binary blend of EVA copolymer with diatomite, the binary blend of EVA copolymer with SM and the tertiary blend of EVA copolymer with diatomite and SM were successfully mixed and blown into copolymer composite packaging films. The copolymer composite film properties showed that diatomite content increased WVTR and OTR, slightly reduced water contact angle, and improved mechanical properties of the EVA polymer matrix. The cold fogging properties of neat EVA copolymer and EVA copolymer blended with diatomite showed unacceptable cold fogging properties. In term of SM content, the SM content did not change WVTR but dramatically reduced OTR, greatly reduced water contact angle, and much improved mechanical properties of the EVA polymer matrix. Moreover, EVA with SM content showed acceptable cold fogging properties. The opacity density results showed that the additives' content of both SM and diatomite slightly decreased the transparency of copolymer composite films. For the application test, it was found that holy basil packed in a selected EVA copolymer composite pouch (EVADSM) had much lower weight loss and deteriorated proportion weight and longer shelf life compared to holy basil kept in commercial perforated polypropylene pouch. At a storage temperature of 10 °C, the commercial perforated polypropylene could prolong the shelf life of holy basil at a satisfactory quality for 6 days. However, the selected polymer composite pouch (EVADSM) could extend the shelf life of holy basil at a satisfactory quality for at least 14 days in this study.

## REFERENCES

1. Wu, C.-T. *An overview of postharvest biology and technology of fruits and vegetables*. in *Technology on Reducing Post-harvest Losses and Maintaining Quality of Fruits and Vegetables Proceedings of 2010 AARDO Workshop*. 2010. 農業試験所.
2. Sandhya, *Modified atmosphere packaging of fresh produce: Current status and future needs*. *LWT-Food Science and Technology*, 2010. **43**(3): p. 381-392.
3. Bovi, G.G., *Regulation of Humidity in Fresh Produce Packaging*. Reference Module in Food Science ed. G.W. Smithers. 2017, Amsterdam: Elsevier, ©2017. 1-6.
4. Bovi, G.G., O.J. Caleb, M. Linke, C. Rauh, and P.V. Mahajan, *Influence of packaging and storage conditions on biochemical quality and enzymatic activity in relation to shelf life enhancement of fresh basil leaf*. *Biosystems Engineering*, 2016. **150**: p. 24-39.
5. Cantwell, M. and M. Reid. *Herbs (Fresh Culinary)*. Recommendations for Maintaining Postharvest Quality Feb 11, 2022]; Available from: [https://postharvest.ucdavis.edu/Commodity\\_Resources/Fact\\_Sheets/Datastores/Vegetables\\_English/?uid=17&ds=799](https://postharvest.ucdavis.edu/Commodity_Resources/Fact_Sheets/Datastores/Vegetables_English/?uid=17&ds=799).
6. Niamthong, T., S. Sittipod, and V. Chonhenchob, *Development of Holy Basil Storage Using Low Temperatures and Modified Atmosphere Packaging*. *Agriculture and Natural Resources*, 2007. **41**: p. 286-293.
7. Sharma, R., S. Bhatia, and P. Kaur, *Influence of packaging and storage conditions on biochemical quality and enzymatic activity in relation to shelf life enhancement of fresh basil leaf*. *Journal of food science and technology*, 2018. **55**(8): p. 3199-3211.
8. ศิริพานิช, จ. and ธ. ร่มโพธิ์ภักดิ์, การจัดการหลังเก็บเกี่ยวผักและผลไม้. 2549, โรงพิมพ์ศูนย์ส่งเสริมและฝึกอบรมการเกษตรแห่งชาติ มหาวิทยาลัยเกษตรศาสตร์ วิทยาเขตกำแพงแสน: นครปฐม.
9. Sousa-Gallagher, M., P. Mahajan, and T. Mezdad, *Engineering packaging design accounting for transpiration rate: Model development and validation with*

- strawberries*. Journal of Food Engineering, 2013. **119**(2): p. 370-376.
10. โศตรพงษ์, ภ., การจัดการคุณภาพผลิตผลสดหลังการเก็บเกี่ยวเพื่อการส่งออก. 2558, กรมวิชาการเกษตร: กรุงเทพฯ.
  11. พรเฉลิมพงศ์, พ. *Modified Atmosphere Packaging (MAP) / การบรรจุแบบดัดแปรบรรยากาศ*. Available from: <http://www.foodnetworksolution.com/wiki/word/0828/modified-atmosphere-packaging-map>.
  12. Linke, M. and M. Geyer, *Condensation dynamics in plastic film packaging of fruit and vegetables*. Journal of Food Engineering, 2013. **116**(1): p. 144-154.
  13. Bei, B., C. Bryant, K.-M. Gilson, J. Koh, P. Gibson, A. Komiti, H. Jackson, and F. Judd, *A prospective study of the impact of floods on the mental and physical health of older adults*. Aging & mental health, 2013. **17**(8): p. 992-1002.
  14. manitoba. *Water Content and Water Activity: Two Factors That Affect Food Safety*. Available from: <https://www.gov.mb.ca/agriculture/food-safety/at-the-food-processor/water-content-water-activity.html>.
  15. Chitravathi, K., O. Chauhan, and P. Raju, *Influence of modified atmosphere packaging on shelf-life of green chillies (Capsicum annum L.)*. Food Packaging and Shelf Life, 2015. **4**: p. 1-9.
  16. Sadiku, E.R., A.B. Reddy, D. Gnanasekarana, B. Oboirien, B. Aderibigbe, K. Varaprasad, and S. Goddeti, *Nanostructured polymer blends for gas/vapor barrier and dielectric applications*, in *Design and Applications of Nanostructured Polymer Blends and Nanocomposite Systems*. 2016, Elsevier. p. 239-259.
  17. Emblem, A., *Plastics properties for packaging materials*, in *Packaging Technology*. 2012, Elsevier. p. 287-309.
  18. Sisanth, K., M. Thomas, J. Abraham, and S. Thomas, *General introduction to rubber compounding*, in *Progress in rubber nanocomposites*. 2017, Elsevier. p. 1-39.
  19. Shi, Q.P., D.L. Li, and W.C. Xu, *LDPE/EVA Modified film prepared by melt blend method and study of its mechanical performances and barrier property*. Materials Science Forum, 2011. **689**: p. 321-327.
  20. Bellotti, N. and C. Deyá, *Natural products applied to antimicrobial coatings*, in

- Studies in Natural Products Chemistry*. 2019, Elsevier. p. 485-508.
21. Huang, S.-y., D.-l. Li, W.-c. Xu, and Q.-p. Shi. *Study on the Preparation of High gas Permeable Film and its Application in Fruit Preservation*. in *Proceedings of the 17th IAPRI World Conference on Packaging*. 2010. China.
  22. Siraratprapa, S., C. Aumnete, W. Phompan, S. Kongniam, and J. Kruenate, *Development of Composite Film for Improving Gas Permeable Properties*. *Advanced Materials Research*, 2010. **93-94**: p. 647-650.
  23. Han, J. *What is Sorbitan Monostearate (E491) in food and yeast? Uses and Safety*. Available from: <https://foodadditives.net/emulsifiers/sorbitan-monostearate/>.
  24. Plasman, V., T. Caulier, and N. Boulos, *Polyglycerol esters demonstrate superior antifogging properties for films*. *Plastics, Additives and Compounding*, 2005. **7(2)**: p. 30-33.
  25. Charalambous, L. *Learning from Lotus Leaves*. Available from: <https://medium.com/five-guys-facts/learning-from-lotus-leaves-424d74cb0667>.
  26. Waldo-Mendoza, M.A., Z.V. Quiñones-Jurado, J.C. Pérez-Medina, B. Yañez-Soto, and P.E. Ramírez-González, *Fogging control on LDPE/EVA coextruded films: wettability behavior and its correlation with electric performance*. *Membranes*, 2017. **7(1)**: p. 11.
  27. Potenza, M. and B. Nielsen, *Antifogging Agents for Polyethylene Films*. *Handbook of Industrial Polyethylene and Technology: Definitive Guide to Manufacturing, Properties, Processing, Applications and Markets*, 2017: p. 865-875.
  28. Paull, R., *Effect of temperature and relative humidity on fresh commodity qualit*. *Postharvest biology and technology*, 1999. **15(3)**: p. 263-277.

## VITA

**NAME** Nuttinan Boonao

**DATE OF BIRTH** 30 Aug 1997

**PLACE OF BIRTH** Bangkok province

**INSTITUTIONS ATTENDED** Graduated high school from Suankularb wittayalai Nonthaburi school in 2016.  
Graduated Bachelor's Degree in Industrial Chemistry, Faculty of Science, Kasetsart University in 2020.

**HOME ADDRESS** 30  
Soi Nonthaburi 13 Bang Krasor Subdistrict Mueang Nonthaburi District Nonthaburi province