



**ASD Packaging Congress 2023**  
**2nd International Packaging Industry Congress**



**SAKARYA**  
UNIVERSITY

# **Development of Sustainable Active Packaging Materials from Food Industry Waste**

## **Gıda Endüstrisi Atıklarından Sürdürülebilir Aktif Ambalaj Malzemesi Geliştirilmesi**

**Eylem Karkuş, Güliz Haskaraca, Zehra Ayhan**  
**Sakarya University Food Engineering Department**  
**[zehraayhan@sakarya.edu.tr](mailto:zehraayhan@sakarya.edu.tr)**

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- **Valorization of food waste for sustainable active packaging**

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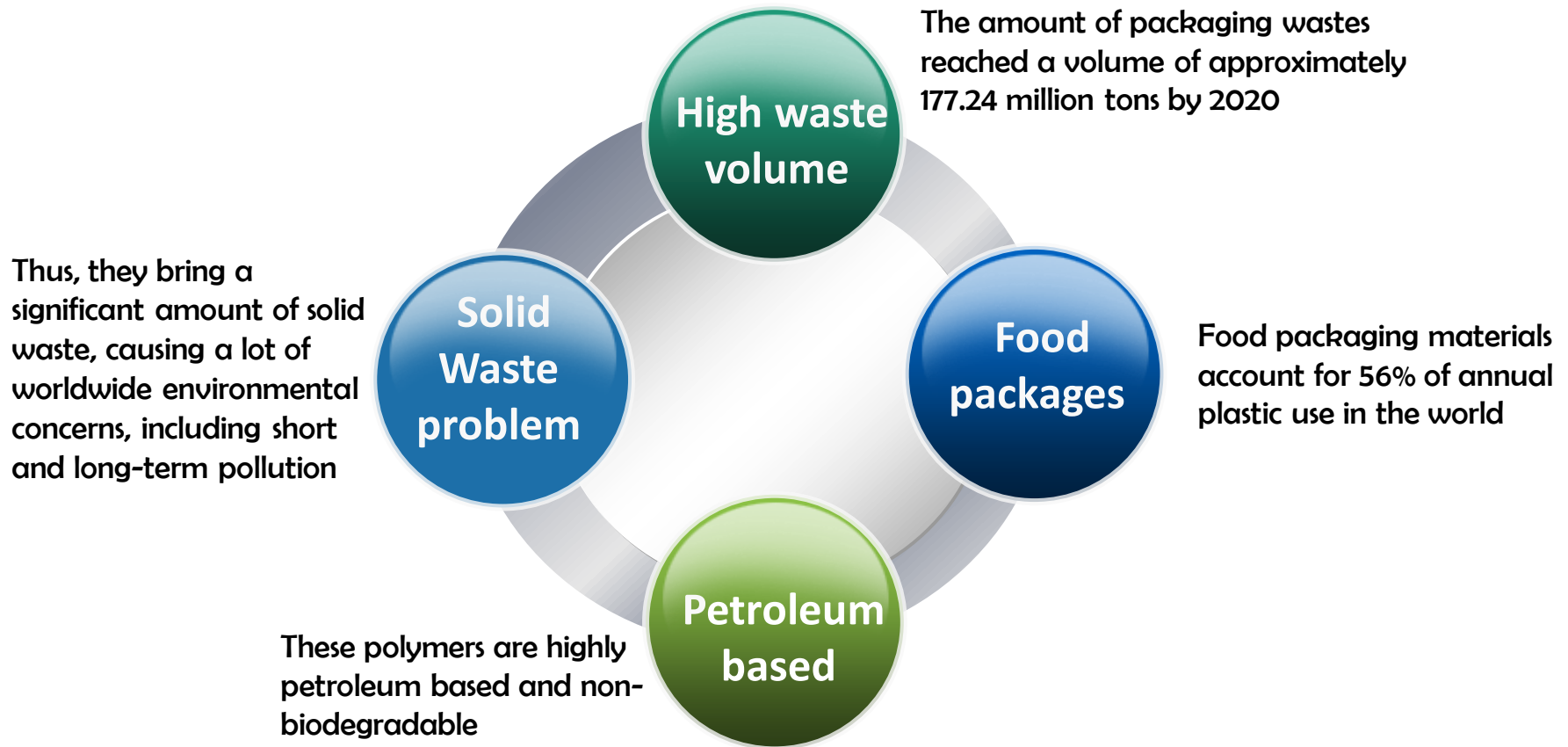
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# Background



**To find alternatives to replace petroleum based materials with more sustainable polymers including agricultural food waste as the most abundant renewable resource on the earth**



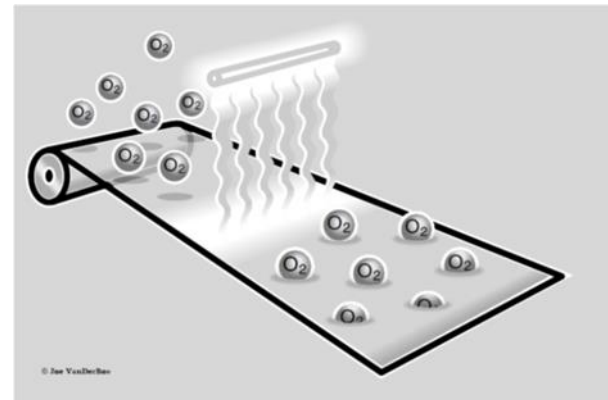
# Sustainable Packaging

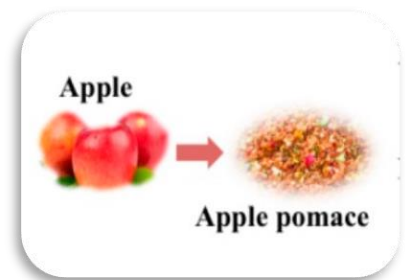
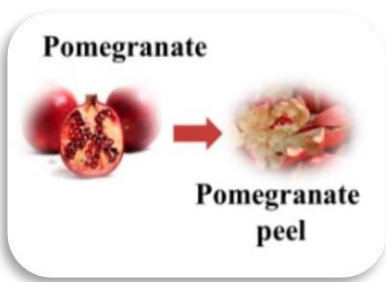
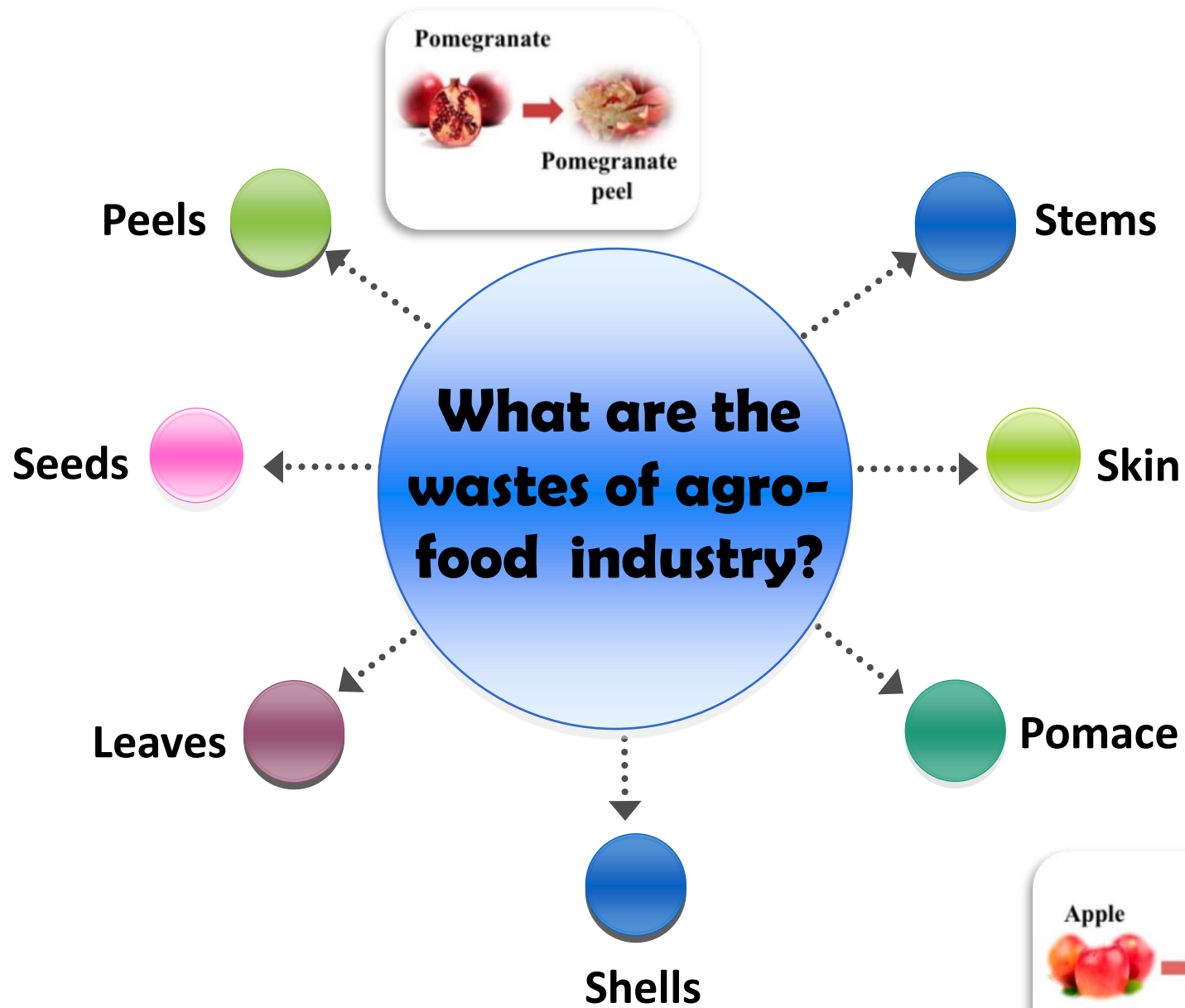
Sustainable food packaging is an optimized, measured (quantified) and validated solution, which takes into consideration the balance of social, economic, ecological and safe implementations of the circular value chain, based on the entire history (life cycle) of the food product-package unit



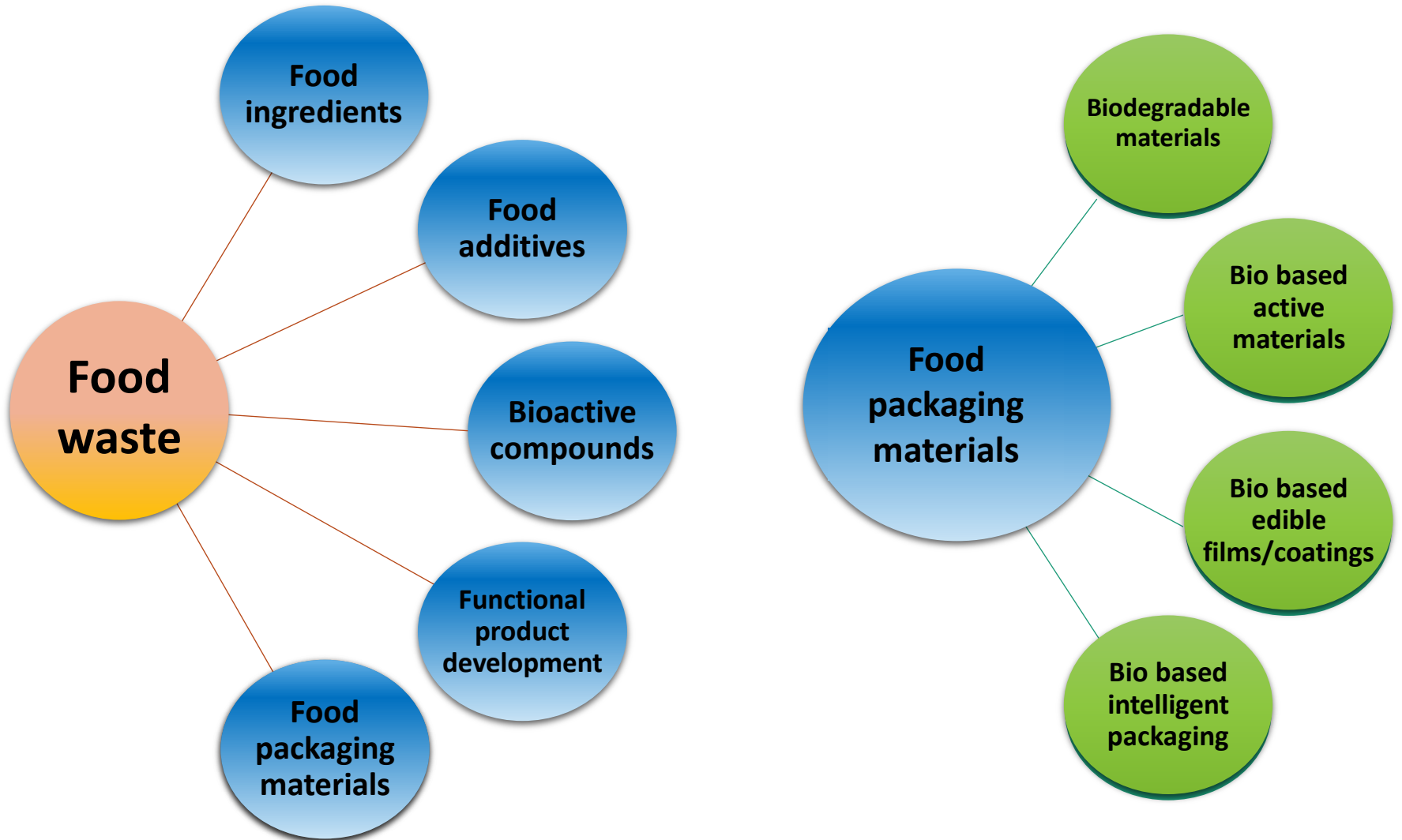
# Active Packaging

- AP systems are designed to “deliberately incorporate components that would release or absorb substances into or from the packaged food or the environment surrounding the food.”
- Active packaging materials are thereby “intended to extend the shelf-life or to maintain or improve the condition of packaged food.” (EC, No 450/2009)

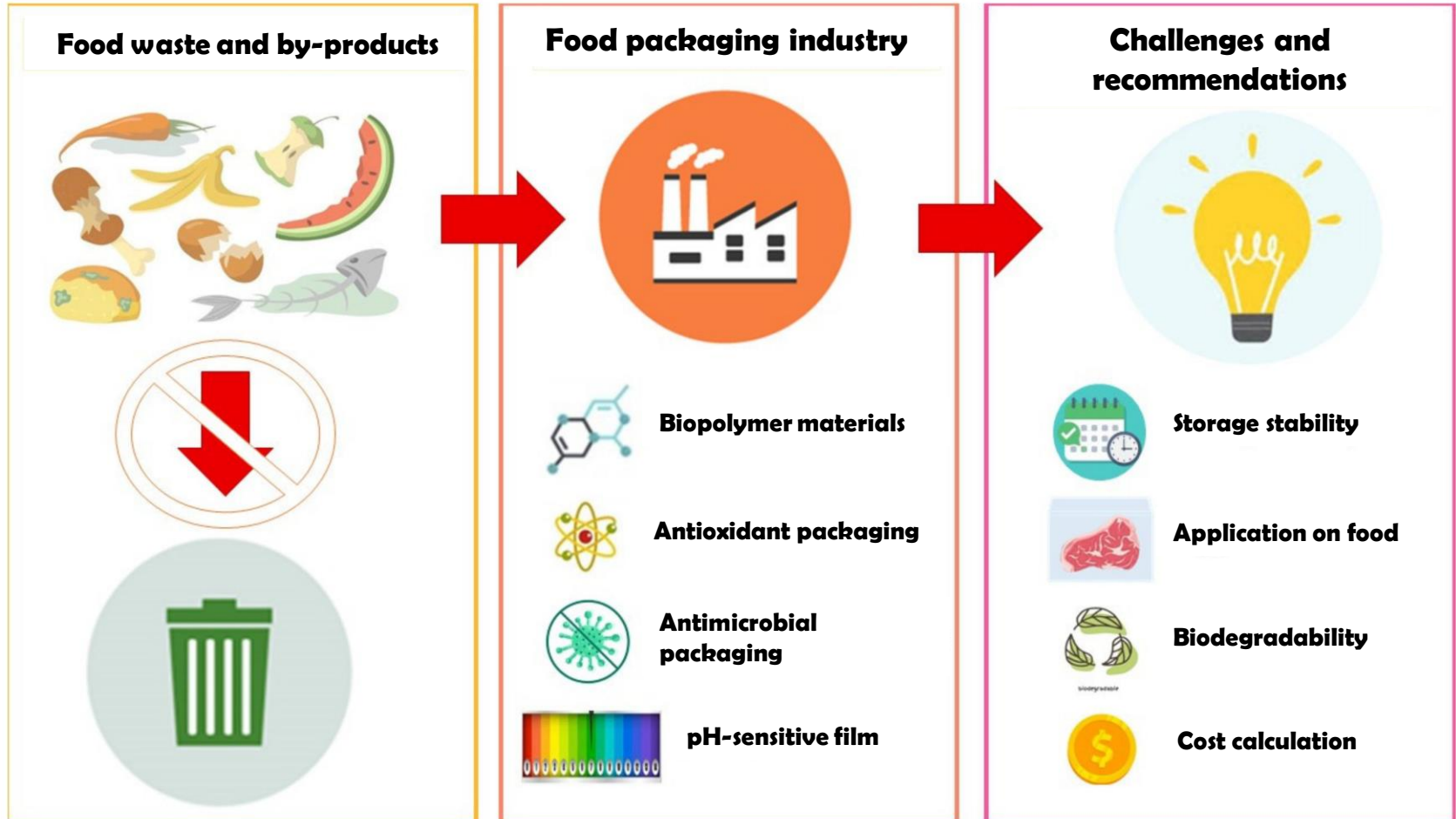




# Potential applications of food wastes



# Applications of food waste and by-products for the food packaging industry



# Aim of the study



**Development of active-edible-bio based films from orange and pomegranate peel waste for sustainable food packaging**

- ✓ **Effects of particle size**
- ✓ **Effect of acid concentration**
- ✓ **Effect of plasticizer concentrations**

**Characterization of developed films**

- ✓ **Physical properties**
- ✓ **Mechanical properties**
- ✓ **Antimicrobial and antioxidant activities**
- ✓ **Thermal properties**
- ✓ **Morphological structure**

# Materials & Methods

- Waste sources
  - Pomegranate peel
  - Orange peel
- Development of films from fruits peels
  - Drying of peels
  - Grinding into powder
  - Solving with water
  - Adding citric acid and glycerol
  - Film forming by solvent casting method
- Characterization of films

# Film production steps from fruit peels



Leaving in water  
for one night



Shaking in  
shaking water  
bath



Dried  
pomegranate  
peels



Grinding



Grinded  
pomegranate  
peels



Particle sizing  
with vibrating  
sieve



Leaving in water  
for one night



Shaking in  
shaking water  
bath



Dried orange  
peels



Grinding



Grinded orange  
peels



Particle sizing  
with vibrating  
sieve

# Film production from fruit peels



Pomegranate



Pomegranate  
Peel



Pomegranate  
Peel Powder



Pomegranate  
Peel Powder  
Film



Orange



Orange Peel

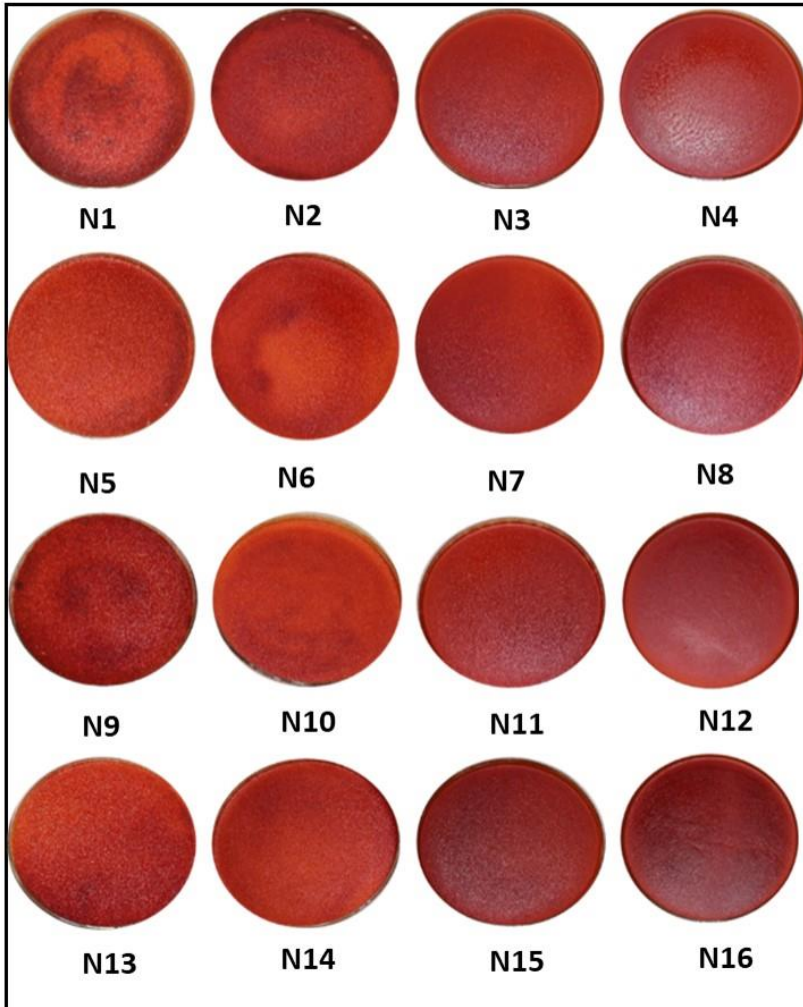


Orange  
Peel Powder

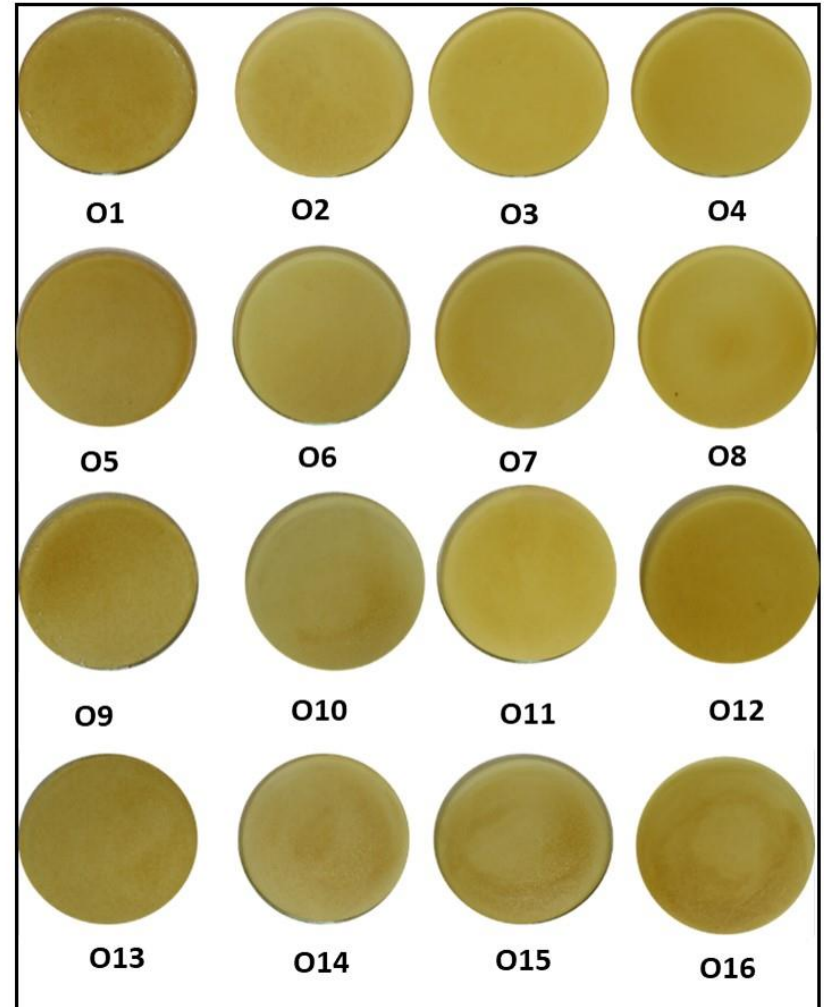


Orange  
Peel Powder  
Film

# Developed Films



(A)

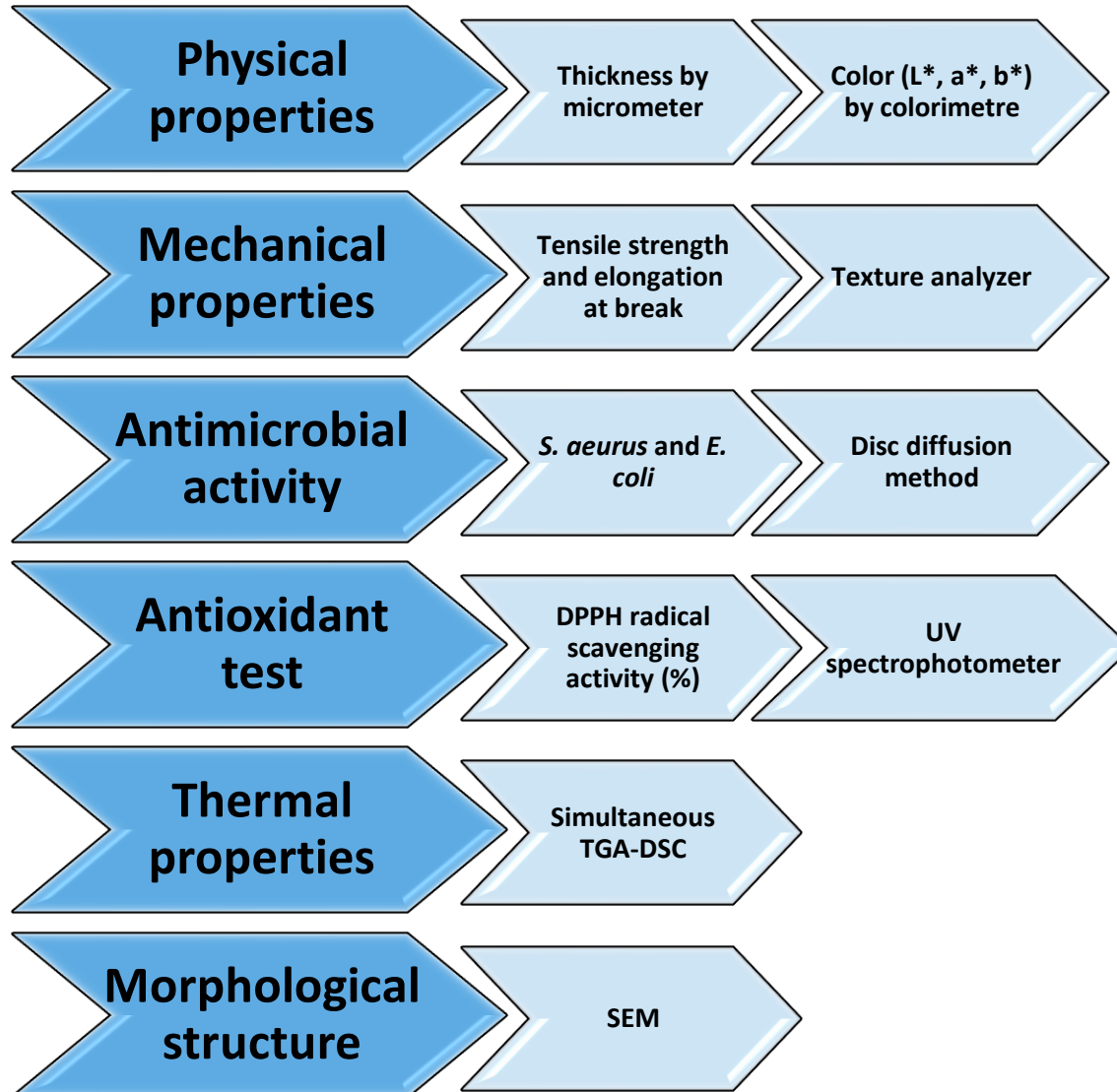


(B)

# Experimental Design

Film Codes	Particle Size ( $\mu$ )	Acid Concentration (%)	Glycerol Concentration (%)
P1/O1	150-125	3	7
P2/O2			10
P3/O3		5	7
P4/O4			10
P5/O5	106-75	3	7
P6/O6			10
P7/O7		5	7
P8/O8			10
P9/O9	75-53	3	7
P10/O10			10
P11/O11		5	7
P12/O12			10
P13/O13	53	3	7
P14/O14			10
P15/O15		5	7
P16/O16			10

# Characterization of the Developed Films



# Results and Discussion



# Physical properties of pomegranate peel based films

<i>Film Codes</i>	<i>Particle Size (<math>\mu</math>)</i>	<i>Acid Conc. (%)</i>	<i>Glycerol Conc. (%)</i>	<i>L*</i>	<i>a*</i>	<i>b*</i>	<i>Thickness (mm)</i>
<i>P1</i>	150-125	3	7	44.89±2.06 <sup>Aax</sup>	24.52±2.42 <sup>Aax</sup>	7.56±2.44 <sup>Aabx</sup>	0.44±0.03
<i>P2</i>			10	47.52±0.38 <sup>Aay</sup>	28.13±0.23 <sup>Aax</sup>	12.35±0.76 <sup>Aax</sup>	0.48±0.02
<i>P3</i>		5	7	50.50±1.67 <sup>Aax</sup>	28.13±1.03 <sup>Aax</sup>	10.83±1.95 <sup>Aax</sup>	0.51±0.06
<i>P4</i>			10	51.21±0.61 <sup>Aax</sup>	28.40±0.79 <sup>Abx</sup>	12.72±1.61 <sup>Abx</sup>	0.55±0.03
<i>P5</i>	106-75	3	7	47.32±0.19 <sup>Aax</sup>	29.03±0.73 <sup>Aax</sup>	12.59±1.16 <sup>Aax</sup>	0.45±0.04
<i>P6</i>			10	48.22±0.16 <sup>Aay</sup>	29.66±0.07 <sup>Aay</sup>	14.20±0.21 <sup>Aay</sup>	0.49±0.04
<i>P7</i>		5	7	49.23±0.77 <sup>Aax</sup>	31.03±0.41 <sup>Aax</sup>	14.81±1.05 <sup>Aax</sup>	0.48±0.03
<i>P8</i>			10	50.52±0.33 <sup>Aabx</sup>	32.29±0.23 <sup>Aax</sup>	16.56±0.09 <sup>Aax</sup>	0.49±0.05
<i>P9</i>	75-53	3	7	44.75±0.89 <sup>Aay</sup>	25.16±1.15 <sup>Aax</sup>	6.28±1.37 <sup>Abx</sup>	0.43±0.04
<i>P10</i>			10	45.01±0.08 <sup>Aby</sup>	25.67±0.40 <sup>Aby</sup>	7.35±0.21 <sup>Aby</sup>	0.45±0.05
<i>P11</i>		5	7	52.34±0.95 <sup>Aax</sup>	30.68±1.03 <sup>Aax</sup>	13.49±1.71 <sup>Aax</sup>	0.45±0.04
<i>P12</i>			10	49.51±0.06 <sup>Abx</sup>	27.40±0.06 <sup>Abx</sup>	9.21±0.30 <sup>Acbx</sup>	0.52±0.06
<i>P13</i>	53	3	7	45.13±0.47 <sup>Aay</sup>	26.43±0.11 <sup>Aax</sup>	7.06±0.49 <sup>Aabx</sup>	0.39±0.04
<i>P14</i>			10	44.66±0.57 <sup>Aby</sup>	25.59±1.05 <sup>Abx</sup>	6.97±0.68 <sup>Abx</sup>	0.48±0.02
<i>P15</i>		5	7	51.10±1.14 <sup>Aax</sup>	29.76±1.24 <sup>Aax</sup>	11.51±2.07 <sup>Aax</sup>	0.43±0.04
<i>P16</i>			10	48.72±0.49 <sup>Abx</sup>	27.37±1.58 <sup>Abx</sup>	8.48±1.24 <sup>Acx</sup>	0.55±0.05

# Physical properties of orange peel based films

<i>Film Codes</i>	<i>Particle Size (<math>\mu</math>)</i>	<i>Acid Conc. (%)</i>	<i>Glycerol Conc. (%)</i>	<i>L*</i>	<i>a*</i>	<i>b*</i>	<i>Thickness (mm)</i>
<i>O1</i>	150-125	3	7	65.56±0.92 <sup>Bay</sup>	12.08±0.85 <sup>Aax</sup>	38.05±0.90 <sup>Acby</sup>	0.41±0.04
<i>O2</i>			10	84.61±0.00 <sup>Aax</sup>	9.54±0.21 <sup>Aax</sup>	42.31±0.56 <sup>Aax</sup>	0.40±0.02
<i>O3</i>		5	7	81.91±0.80 <sup>Aax</sup>	11.34±0.42 <sup>Aax</sup>	42.27±0.08 <sup>Aabx</sup>	0.42±0.05
<i>O4</i>			10	83.84±0.56 <sup>Aax</sup>	9.58±0.34 <sup>Aax</sup>	41.80±0.82 <sup>Abx</sup>	0.43±0.02
<i>O5</i>	106-75	3	7	64.50±4.98 <sup>Aax</sup>	9.85±0.31 <sup>Abx</sup>	36.47±0.33 <sup>Bcx</sup>	0.38±0.03
<i>O6</i>			10	84.77±0.88 <sup>Aax</sup>	9.60±0.18 <sup>Aax</sup>	42.27±0.18 <sup>Aax</sup>	0.42±0.06
<i>O7</i>		5	7	84.33±1.85 <sup>Aax</sup>	9.73±0.48 <sup>Abx</sup>	39.90±1.59 <sup>Abx</sup>	0.40±0.07
<i>O8</i>			10	84.26±0.40 <sup>Aax</sup>	9.38±0.28 <sup>Aax</sup>	39.19±0.12 <sup>Acy</sup>	0.48±0.06
<i>O9</i>	75-53	3	7	68.04±0.25 <sup>Bay</sup>	10.21±0.33 <sup>Abx</sup>	39.12±0.57 <sup>Bby</sup>	0.33±0.01
<i>O10</i>			10	83.05±2.40 <sup>Aax</sup>	11.06±1.45 <sup>Aax</sup>	44.47±0.02 <sup>Aax</sup>	0.39±0.05
<i>O11</i>		5	7	83.19±0.04 <sup>Aax</sup>	10.23±0.00 <sup>Aabx</sup>	45.17±0.06 <sup>Aax</sup>	0.40±0.02
<i>O12</i>			10	84.81±0.47 <sup>Aax</sup>	9.58±0.29 <sup>Aax</sup>	41.59±0.13 <sup>Bby</sup>	0.39±0.05
<i>O13</i>	53	3	7	67.21±0.06 <sup>Bay</sup>	11.21±0.06 <sup>Aabx</sup>	41.23±0.07 <sup>Aay</sup>	0.35±0.02
<i>O14</i>			10	81.56±1.30 <sup>Aax</sup>	11.99±1.33 <sup>Aax</sup>	46.46±3.17 <sup>Aax</sup>	0.37±0.04
<i>O15</i>		5	7	83.55±1.80 <sup>Aax</sup>	10.68±0.52 <sup>Aabx</sup>	46.47±0.93 <sup>Aax</sup>	0.37±0.03
<i>O16</i>			10	82.92±1.56 <sup>Aax</sup>	10.39±1.12 <sup>Aax</sup>	44.57±0.14 <sup>Aax</sup>	0.37±0.03

# Mechanical properties of the pomegranate peel based films

<i>Film Codes</i>	<i>Particle Size (<math>\mu</math>)</i>	<i>Acid Conc. (%)</i>	<i>Glycerol Conc. (%)</i>	<i>TS (MPa)</i>	<i>EB (%)</i>
<i>P1</i>	150-125	3	7	0.35±0.03 <sup>Adx</sup>	23.28±1.96 <sup>Abx</sup>
<i>P2</i>			10	0.27±0.02 <sup>Abx</sup>	22.73±1.30 <sup>Abx</sup>
<i>P3</i>		5	7	0.31±0.01 <sup>Acx</sup>	22.03±0.26 <sup>Abx</sup>
<i>P4</i>			10	0.28±0.01 <sup>Acx</sup>	22.78±0.89 <sup>Acx</sup>
<i>P5</i>	106-75	3	7	0.56±0.01 <sup>Acy</sup>	23.05±0.13 <sup>Aby</sup>
<i>P6</i>			10	0.31±0.01 <sup>Bby</sup>	22.64±1.46 <sup>Abx</sup>
<i>P7</i>		5	7	0.61±0.00 <sup>Abx</sup>	29.06±1.27 <sup>Aax</sup>
<i>P8</i>			10	0.39±0.01 <sup>Bbx</sup>	24.96±1.37 <sup>Acx</sup>
<i>P9</i>	75-53	3	7	0.72±0.01 <sup>Abx</sup>	27.20±1.82 <sup>Aabx</sup>
<i>P10</i>			10	0.45±0.02 <sup>Bax</sup>	23.53±1.05 <sup>Abx</sup>
<i>P11</i>		5	7	0.84±0.13 <sup>Abx</sup>	28.81±1.95 <sup>Aax</sup>
<i>P12</i>			10	0.51±0.06 <sup>Aax</sup>	29.19±1.47 <sup>Abx</sup>
<i>P13</i>	53	3	7	0.87±0.05 <sup>Aay</sup>	29.87±1.56 <sup>Aax</sup>
<i>P14</i>			10	0.33±0.00 <sup>Bby</sup>	30.08±0.22 <sup>Aay</sup>
<i>P15</i>		5	7	1.28±0.02 <sup>Aax</sup>	31.55±0.15 <sup>Bax</sup>
<i>P16</i>			10	0.52±0.01 <sup>Bax</sup>	40.29±0.52 <sup>Aax</sup>

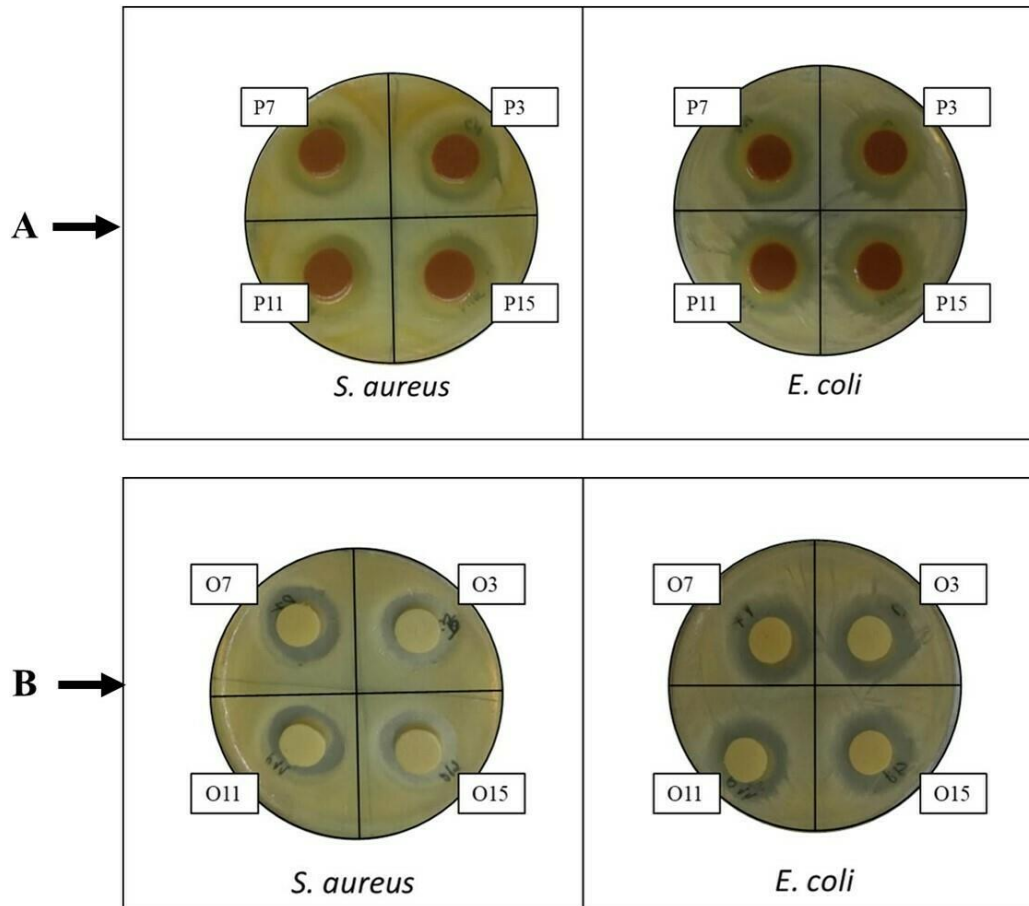
**Reducing the particle size improved the mechanical properties**

# Mechanical properties of the orange peel based films

<i>Film Codes</i>	<i>Particle Size (<math>\mu</math>)</i>	<i>Acid Conc. (%)</i>	<i>Glycerol Conc. (%)</i>	<i>TS (MPa)</i>	<i>EB (%)</i>
<i>O1</i>	150-125	3	7	0.72±0.06 <sup>Abx</sup>	32.84±1.70 <sup>Aax</sup>
<i>O2</i>			10	0.46±0.01 <sup>Bax</sup>	30.47±2.13 <sup>Aax</sup>
<i>O3</i>		5	7	0.58±0.02 <sup>Acx</sup>	34.02±2.45 <sup>Abx</sup>
<i>O4</i>			10	0.46±0.02 <sup>Aax</sup>	35.42±0.61 <sup>Acx</sup>
<i>O5</i>	106-75	3	7	0.86±0.05 <sup>Aabx</sup>	34.38±0.71 <sup>Aay</sup>
<i>O6</i>			10	0.46±0.02 <sup>Bax</sup>	38.92±5.37 <sup>Aax</sup>
<i>O7</i>		5	7	0.68±0.00 <sup>Abx</sup>	40.08±0.20 <sup>Aax</sup>
<i>O8</i>			10	0.46±0.01 <sup>Bax</sup>	38.76±0.11 <sup>Bbx</sup>
<i>O9</i>	75-53	3	7	0.87±0.01 <sup>Aax</sup>	35.48±2.70 <sup>Aax</sup>
<i>O10</i>			10	0.48±0.01 <sup>Bax</sup>	29.55±0.25 <sup>Aay</sup>
<i>O11</i>		5	7	0.59±0.03 <sup>Acy</sup>	37.16±0.32 <sup>Babx</sup>
<i>O12</i>			10	0.49±0.02 <sup>Aax</sup>	42.79±1.07 <sup>Aax</sup>
<i>O13</i>	53	3	7	0.85±0.02 <sup>Aabx</sup>	38.59±0.17 <sup>Aax</sup>
<i>O14</i>			10	0.45±0.00 <sup>Bay</sup>	29.33±0.11 <sup>Bay</sup>
<i>O15</i>		5	7	0.76±0.01 <sup>Aax</sup>	37.20±0.72 <sup>Aabx</sup>
<i>O16</i>			10	0.49±0.01 <sup>Bax</sup>	38.97±0.62 <sup>Abx</sup>

**Reducing the particle size improved the mechanical properties**

# Antibacterial activity of pomegranate & orange peel based films



- P3/O3 150-125  $\mu$
- P7/O7 106-75 $\mu$
- P11/O11 75-53  $\mu$
- P15/O15 53  $\mu$

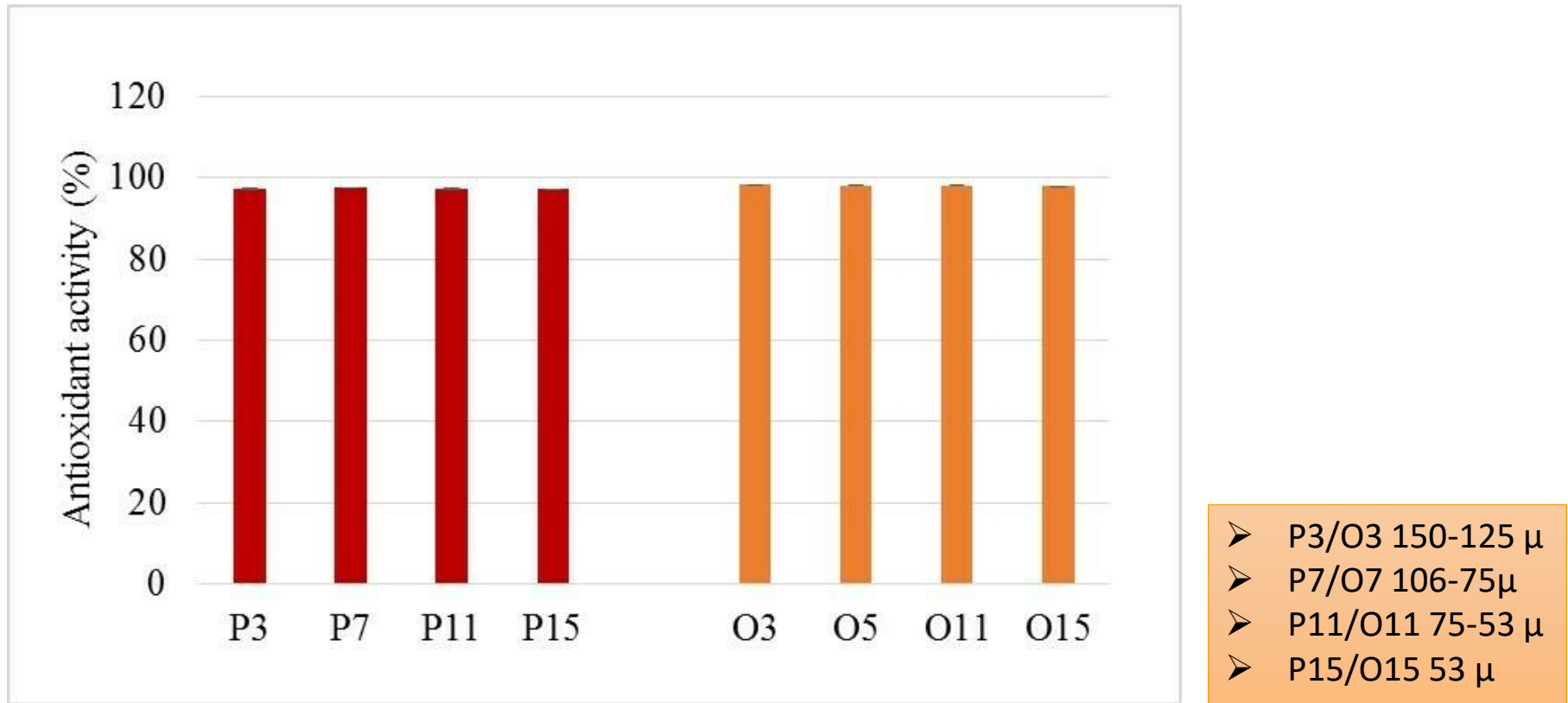
# Antimicrobial activity of pomegranate & orange peel based films

Film Codes	Particle Size ( $\mu$ )	Diameter of inhibition zone (mm)	
		<i>S. aureus</i>	<i>E. coli</i>
P3	150-125	34.00 $\pm$ 1.83 <sup>A</sup>	30.50 $\pm$ 0.58 <sup>A</sup>
P7	106-75	33.75 $\pm$ 2.75 <sup>A</sup>	30.25 $\pm$ 0.96 <sup>A</sup>
P11	75-53	33.75 $\pm$ 1.26 <sup>A</sup>	30.50 $\pm$ 1.12 <sup>A</sup>
P15	53	34.00 $\pm$ 0.82 <sup>A</sup>	31.25 $\pm$ 0.96 <sup>A</sup>
O3	150-125	26.00 $\pm$ 0.00 <sup>B</sup>	27.00 $\pm$ 0.00 <sup>A</sup>
O7	106-75	26.00 $\pm$ 0.00 <sup>B</sup>	27.00 $\pm$ 1.41 <sup>A</sup>
O11	75-53	26.00 $\pm$ 0.00 <sup>B</sup>	25.50 $\pm$ 0.71 <sup>A</sup>
O15	53	29.00 $\pm$ 2.00 <sup>A</sup>	29.00 $\pm$ 1.41 <sup>A</sup>

A-B: Mean values in the same column with different letters are significantly different ( $p \leq 0.05$ ) for the 5% acid and 7% glycerol concentrations (P: Pomegranate, O: Orange).

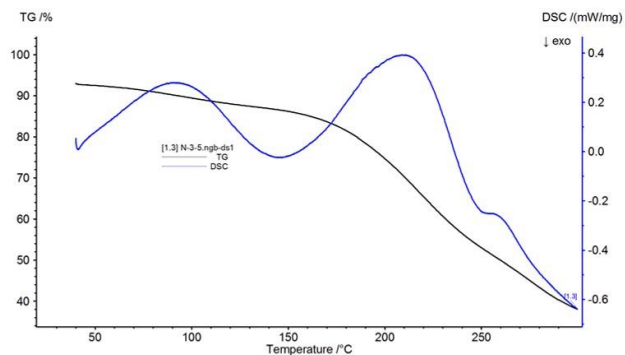
Pomegranate and orange peel based films showed very high antimicrobial activity against *E. coli* and *S. aureus* irrespective of particle size.

# Antioxidant activity of pomegranate & orange peel based films

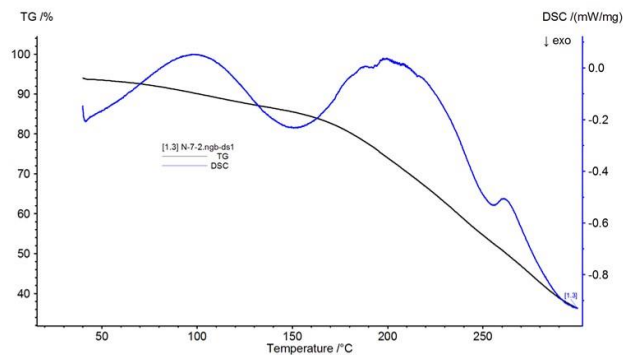


Pomegranate and orange peel based films also showed very high antioxidant activity (97–98%).

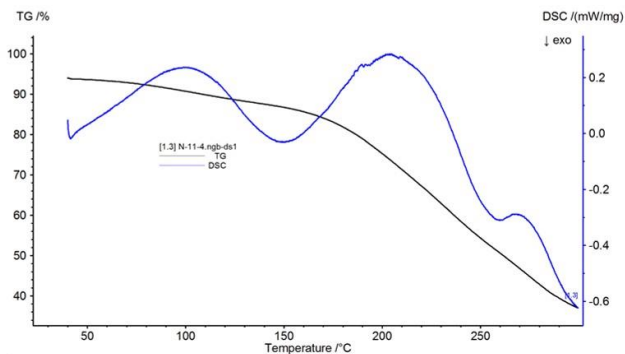
# Thermal properties of pomegranate peel based films



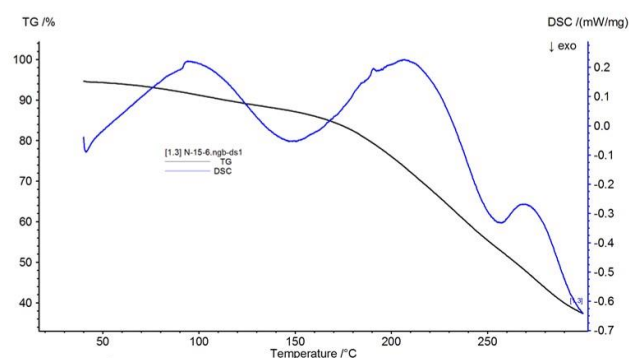
P3



P7



P11

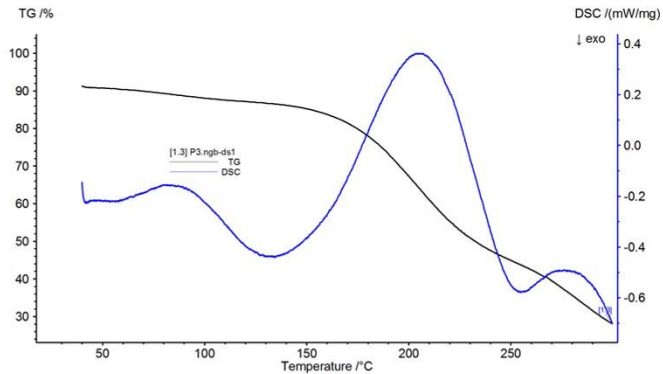


P15

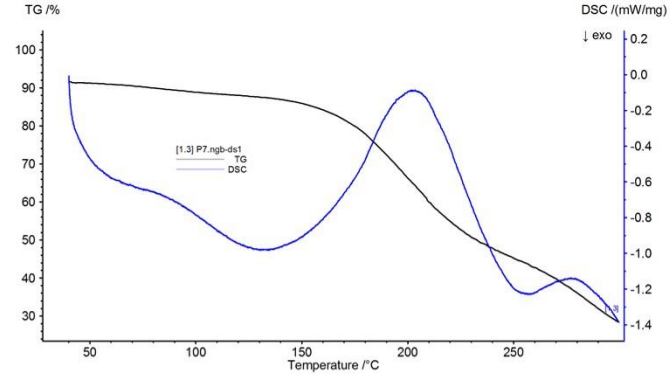
P3/O3 150-125  $\mu$ , P7/O7 106-75 $\mu$ , P11/O11 75-53  $\mu$ , P15/O15 53  $\mu$

Major thermal degradation (>50%) was observed between 160 and 300 °C for pomegranate and orange peel based films irrespective of particle sizes

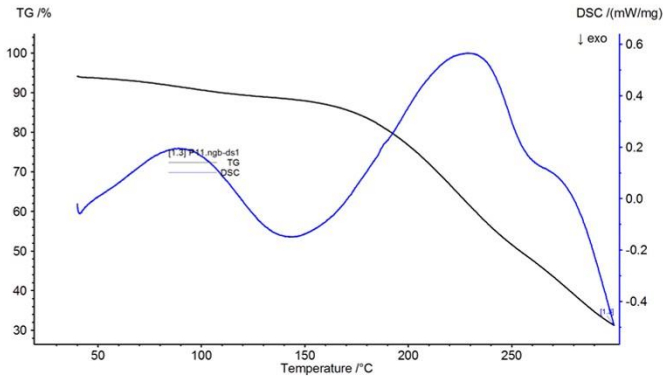
# Thermal properties of orange peel based films



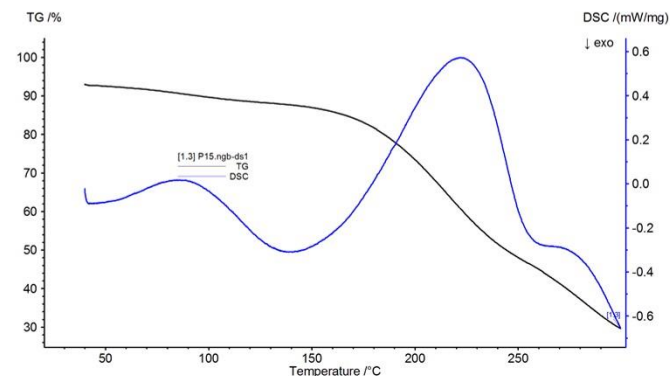
O3



O7



O11



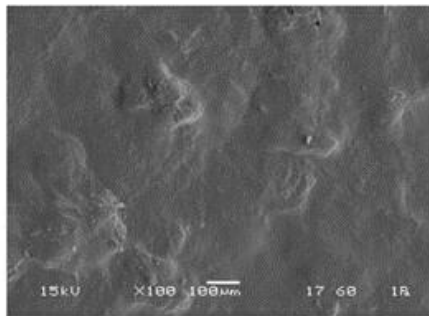
O15

P3/O3 150-125  $\mu$ , P7/O7 106-75 $\mu$ , P11/O11 75-53  $\mu$ , P15/O15 53  $\mu$

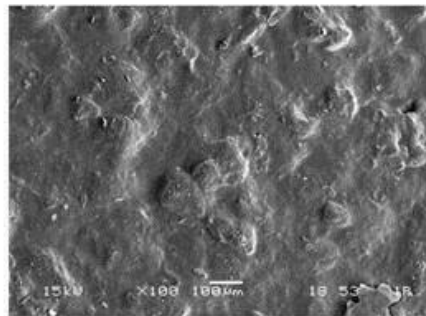
Thermal behavior of both types of films showed very similar thermal properties which were not highly affected by the particle size.

# Morphological structure of pomegranate & orange peel based films

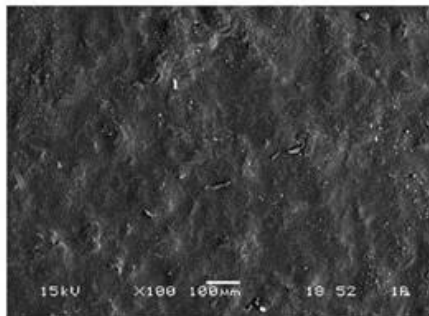
## Pomegranate peel based films



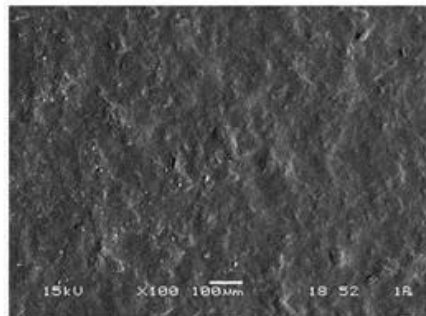
P3



P7

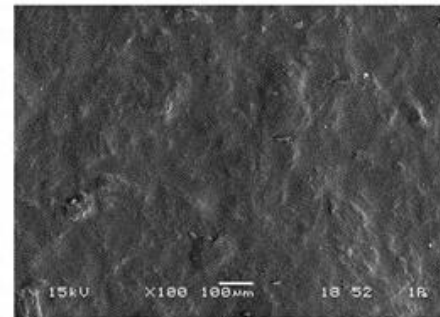


P11

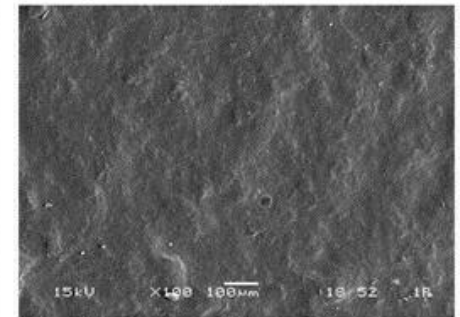


P15

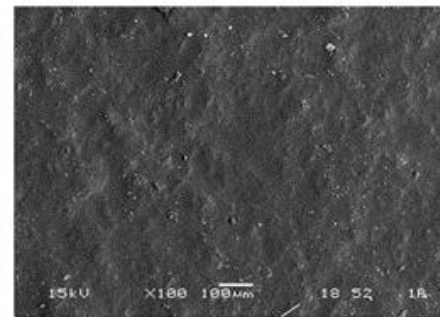
## Orange peel based films



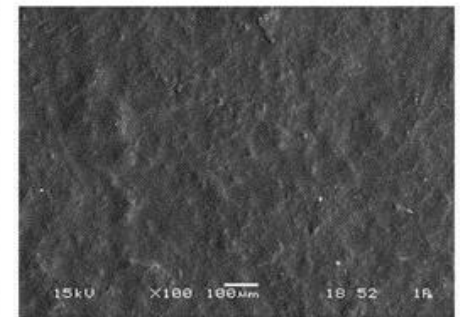
O3



O7



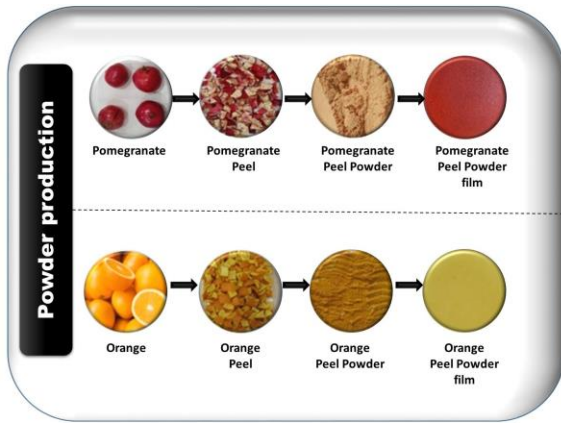
O11



O15

P3/O3 150-125  $\mu$ , P7/O7 106-75 $\mu$ , P11/O11 75-53  $\mu$ , P15/O15 53  $\mu$

Pomegranate and orange peel based films had homogeneous, smooth and crack-free surfaces showing high potential for film forming property by decreasing the particle size.



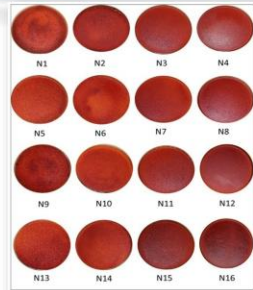
16 different films were formulated according to following parameters

- Four different particle size (150-125, 106-75, 75-53 and 53  $\mu\text{m}$ )
- Two different acid concentration (3% and 5%)
- Two different glycerol concentration (7% and 10%)

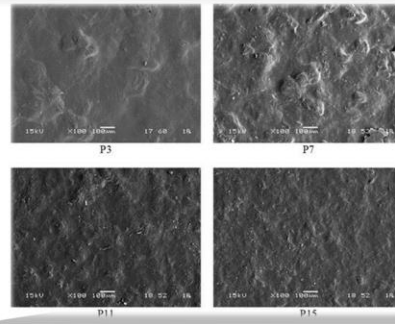
Biofilm Codes	Particle Size ( $\mu$ )	Acid Concentration (%)	Glycerol Concentration (%)
P1/O1	150-125	3	7
P2/O2			10
P3/O3		5	7
P4/O4			10
P5/O5	106-75	3	7
P6/O6			10
P7/O7		5	7
P8/O8			10
P9/O9	75-53	3	7
P10/O10			10
P11/O11		5	7
P12/O12			10
P13/O13	53	3	7
P14/O14			10
P15/O15		5	7
P16/O16			10

Optimum acid and glycerol concentration was selected among the 16 formulation, by analysing the physical and mechanical properties [concentrations for acid 5% and for glycerol 7% were selected ]

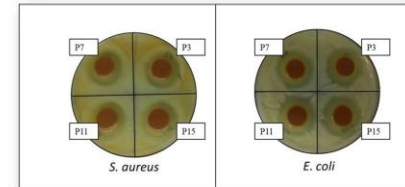
TGA-DSC, SEM, antioxidant and antimicrobial analyzes were made to determine and compare the characteristics of selected films



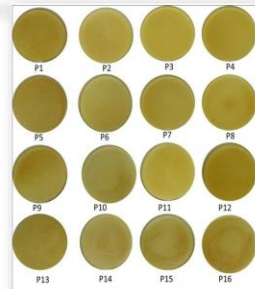
16 different formulated pomegranate biofilms



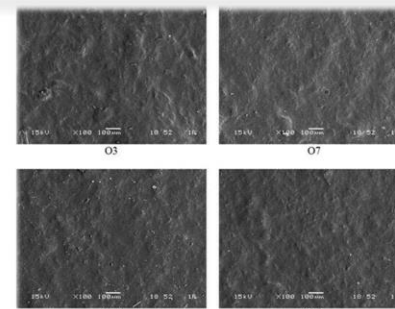
SEM images of selected Pomegranate biofilms



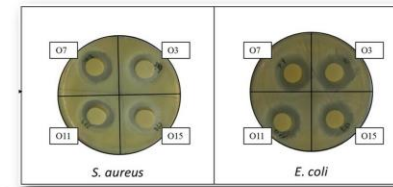
Agar disk diffusion assay results: Inhibitions zones for *S. aureus* and *E. coli* of pomegranate biofilms



16 different formulated orange biofilms



SEM images of selected orange biofilms



Agar disk diffusion assay results: Inhibitions zones for *S. aureus* and *E. coli* of orange biofilms

# Conclusions

- Pomegranate and orange peels could be good potential as antimicrobial and antioxidant biopolymer sources to be used in the polymer matrix as sustainable and active food packaging material.
- Having edible property, both peel based polymers could be used
  - as a coating for food due to high antioxidant and antimicrobial activity,
  - as a separator between layered meat products such as hamburger patties to improve shelf life.



# THANK YOU VERY MUCH FOR YOUR ATTENTION!

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Development and characterization of sustainable-active-edible-bio based films from orange and pomegranate peel waste for food packaging: Effects of particle size and acid/plasticizer concentrations

Eylem Karakuş, Zehra Ayhan<sup>\*</sup>, Guliz Haskaraca

Sakarya University, Faculty of Engineering, Department of Food Engineering, Sakarya, Turkey

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Bio based polymer  
Pomegranate peel  
Orange peel  
Food waste  
Sustainable food packaging

## ABSTRACT

Sustainable packaging materials especially from food processing waste are of getting interest. The aim of the study was to develop edible bio based films from processing wastes of pomegranate and orange peels. For this purpose, pomegranate and orange peels were dried and processed into flour in different particle sizes. The effects of particle size, concentration of acid and plasticizer on physical and mechanical properties were investigated. Antimicrobial, antioxidant, morphological and thermal properties were further determined for the selected film formulations. Results showed that the particle size significantly affected mechanical properties of the films ( $p < 0.05$ ), with improvement of the mechanical properties for the reduced particle size. Pomegranate and orange peel based films showed very high antimicrobial activity against *E. coli* and *S. aureus* irrespective of particle sizes. The pomegranate and orange peel based films had homogeneous, smooth, and crack-free surfaces. Pomegranate and orange peel based films showed very similar thermal properties and major thermal degradation was observed between 160 and 300 °C for both type of films irrespective of particle sizes. Pomegranate and orange peels could be good potential as antioxidant and antimicrobial biopolymer sources for sustainable and active food coating/packaging.

## 1. Introduction

The global packaging market has shown steady growth in recent years and as a result of this growth, the amount of packaging wastes reached a volume of approximately 177.24 tons by 2020 (Eurostat, 2022). Food packaging materials account for 56% of annual plastic use in the world (PAGEV, 2018). These polymers are highly petroleum based and non-biodegradable. Thus, they bring a significant amount of solid waste, causing a lot of worldwide environmental concerns, including short and long-term pollution (Motelica et al., 2020; Rhim et al., 2013; Zhong et al., 2020).

The trend in current studies is to find alternatives to replace petroleum based polymers with more sustainable polymers (Ali et al., 2019; Hanani, 2018) including agricultural food waste as the most abundant renewable resource on the earth (Motaung & Linganiso, 2018). Previous research on the bio-based film development from food wastes was based on using apple peel, banana peel, potato and lemon peels as a polymer source or reinforcement material in the polymeric matrix (Borah et al.,

bio-based polymers using food wastes such as, apple peel with chitosan (Sablani et al., 2009), chitosan-based film incorporated with apple peel polyphenols (Riaz et al., 2018), banana peel and banana starch film (Silva et al., 2020), potato and sweet lime peels (Borah et al., 2017), pomegranate peel as reinforcement into starch film (Ali et al., 2019), pomegranate peel in fish gelatin or zein films (Hanani et al., 2019) have been studied to explore the possibility of using these polymers as food packaging materials. The cellulose and starch contents are limiting factors in selecting agro waste precursors. High cellulose concentration is required for high strength applications but limits the biodegradation rate (Maraveas, 2020).

There are only limited studies using agro waste directly for development of edible bio based films (Borah et al., 2017; Silva et al., 2020; Yaradoddi et al., 2022). That's why pomegranate and orange peels were selected to develop bio based films in the present study. Pomegranate peel is a by-product of pomegranate juice and syrup. Processing of pomegranate generates a large volume of peel, accounting for 50% of the total fruit weight (Ahmad et al., 2015). Also, pomegranate peel is a

