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Synthesis of anti-biofoulant green nanoparticles embedded cellulose acetate membranes

Chaired by PROF. DR. ANTONIO PIZZI and PROF. DR. FRANK WIESBROCK
polymers MDPI

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Abstract: Membranes were used in many aqueous applications, including in food processing, *e.g.* clarification of fruit juices. Typical drawbacks of membrane processes are membrane fouling which promotes deterioration of process products. During application of membranes for fruit juice clarification, biofouling occured as the process deals with food subtances. Biofouling is commonly dominated by bacterial attachment and growth on membrane surface, following the deposition of organic molecules from food substances. Natural antibiotics such as *Olea europaea* leaves extract might be used to improve the antibiofouling properties of membranes due to its phenolic contents. In this work, *Olea europaea* substances were obtained by extraction to get the green active solid nanoparticles. Phenolic green nanoparticles then filled into cellulose acetate as membrane matrix. The mixed matrix membrane has therefore a safe antibiofouling properties and suitable for food application. The anti-bioulant effect has been proven by decreasing bacterial attachment down to 23% from initial condition, especially for Gram-negative bacteria such as *Eschericia coli*.

Keywords: mixed matrix membranes; cellulose acetate; green nanoparticle; biofouling; bacterial adhesion

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Introduction

Fruit production in Indonesia and Juice clarification

No.	Fruit	2017 (tons)	2018 (tons)	Annual increase (%)
1	Orange	2,295,310	2,510,420	9.37
2	Mango	2,203,789	1,624,783	19.1
3	Pineapple	1,795,982	1,805,499	0,53
4	Papaya	875,106	887,580	1.43
5	Apple	319,000	481,654	50.99
6	Starfruit	85,318	410,084	12.92
7	Passionfruit	77,190	59,265	-23.22

Fruit juices →

- Value added/profit
- Prevent losses

Data from Dept of Agriculture

Local SME production

Clarification →

Commercial UF Polysulfone

Low permeate → ?

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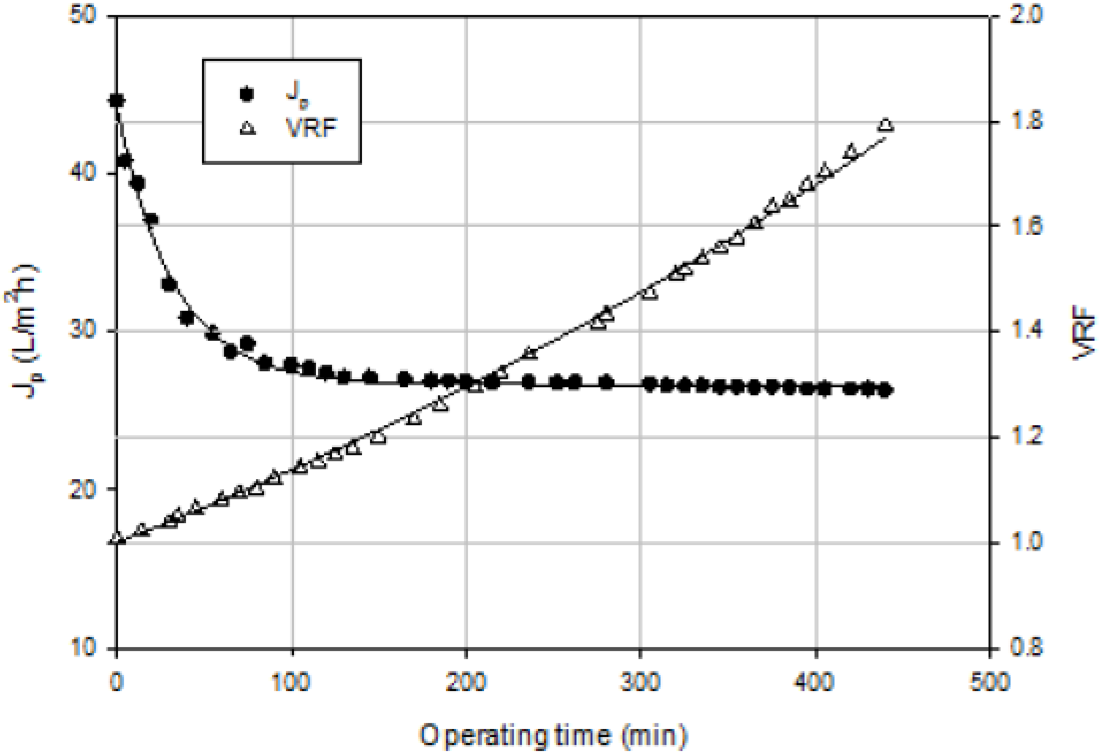
Introduction : Fouling

Kiwifruit juice using HF PEEK



C. Conidi, *et al.* (*2017*)

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Introduction : Additives

Celulose acetate membrane

+

Nanopowdered Phenolic Extract

As antibiofouling agents

Reduce biofilm formation on the surface of the membranes and prevent biofouling

Natural phenolic source

→ Celulose acetate mixed matrix membrane

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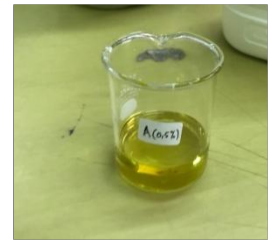
Methods: Phenol extraction

Dried olive leaf powder weighed 25 grams

Extracted with 96% ethanol solvent variation in the ratio of 1: 4, 1: 6 and 1: 8 (w / v) and extraction time 2, 3 and 4 minutes using the Microwave Assisted Extraction (MAE) method

Maceration was carried out for 3 days



The extracted filtrate was concentrated with a rotary vacuum evaporator at 40°C with a rotational speed of 65 rpm

Nanopowder 471-598.5 nm

The concentrated olive leaf extract is dried using a vacuum oven at 40 ° C for 1,5 hours

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Methods: Membrane synthesis

Membrane	Composition Comparison			
	Solvent (20 ml)	Olive Leaf Extract (%)	Casting Thickness (mm)	Cellullose Acetate (gr)
Pristine CA	DMF	0	0.3	4
CA + 0.5%OE	DMF	0.5	0.3	3.98
CA +0.75%OE	DMF	0.75	0.3	3.97
CA+ 1%OE	DMF	1	0.3	3.96

All materials are mixed and stirred using a magnetic stirrer

The solution is poured on the glass plate and casted using a casting knife with an initial thickness setting

Immersed it in a coagulant bath filled with aquades for ± 10 minutes

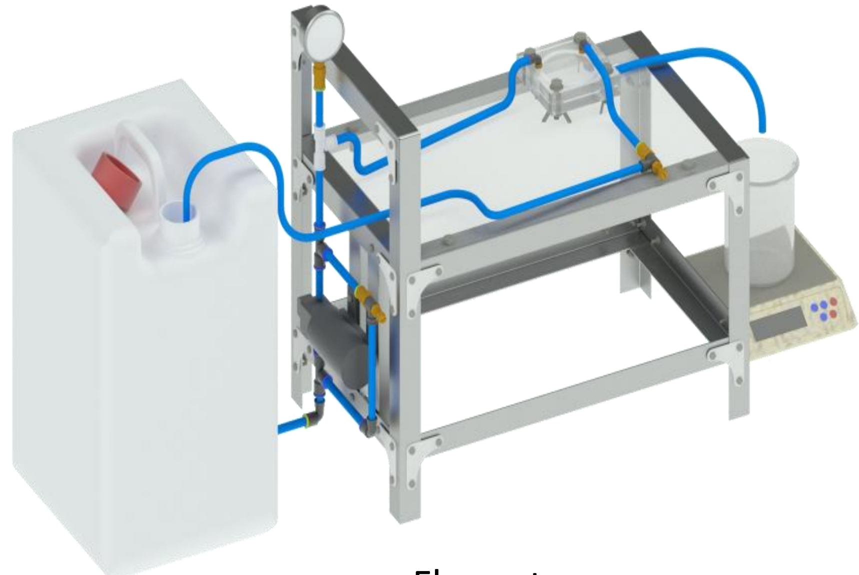
Membrane sheet is dried using nitrogen gas

Cellullose Acetate Mixed Matrix Membrane with Olive Leaf Phenolic Nanopowder Filler

Methods: Properties

1. Mechanical properties
2. Mass transport propertis
3. Antibiofouling properties

    Escherichia coli,
    Bacillus subtilis,
    Staphylococcus aureus



Flux set-up

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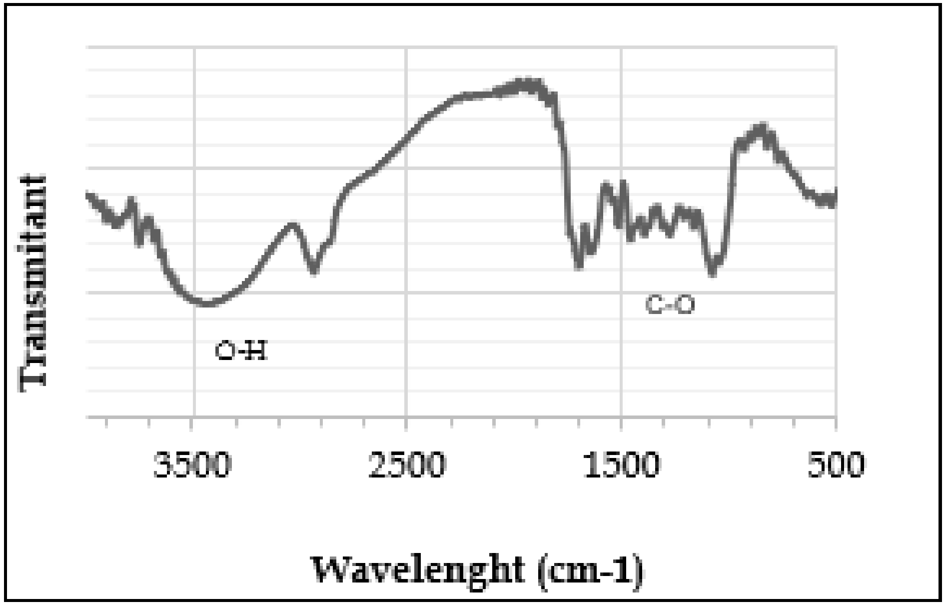
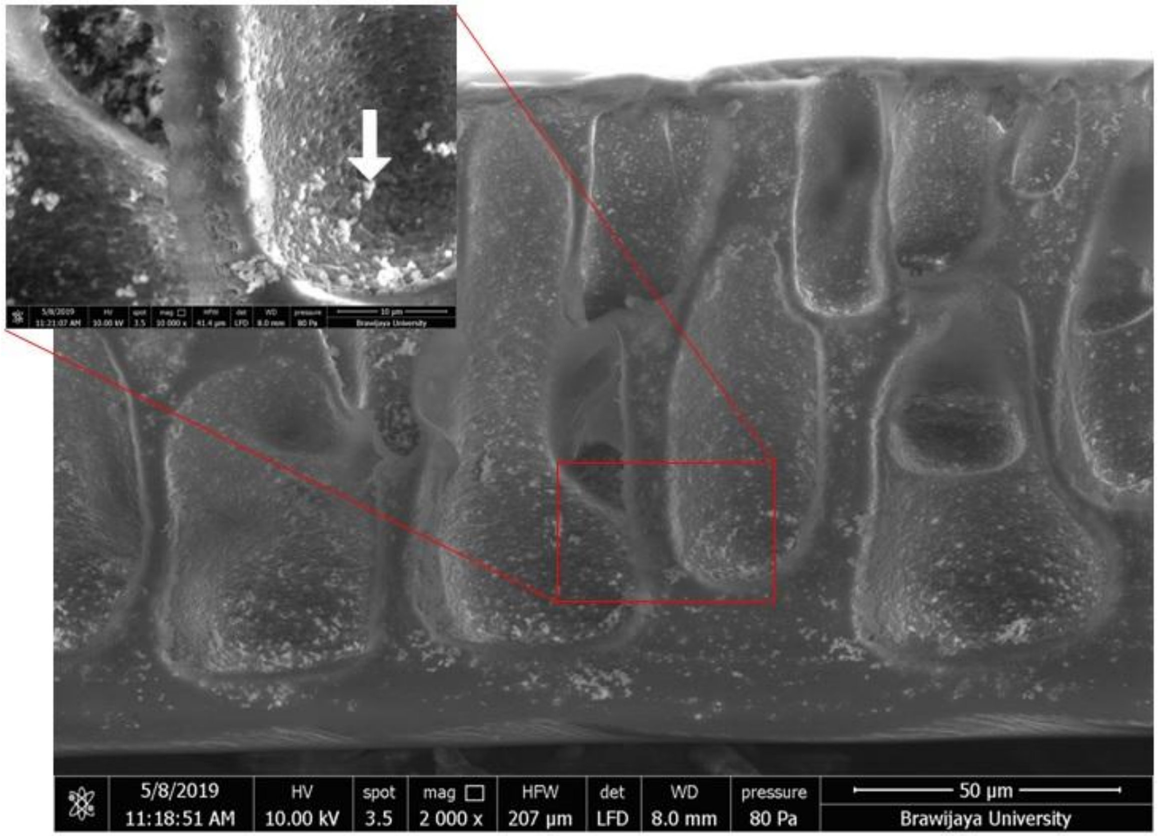
Results: Phenol extraction



The results of the FTIR test on olive leaf extract shows absorption at waves 3401.03 cm^{-1} which indicates the phenol compounds present.

The phenol compound is observed at a wave frequency between 3590-3650 cm^{-1} and 3200-3600 cm^{-1}.

Results: Membrane structure



Surface layer

Macrovoid layer,
nanopowdered
phenolic extract
on membrane surface

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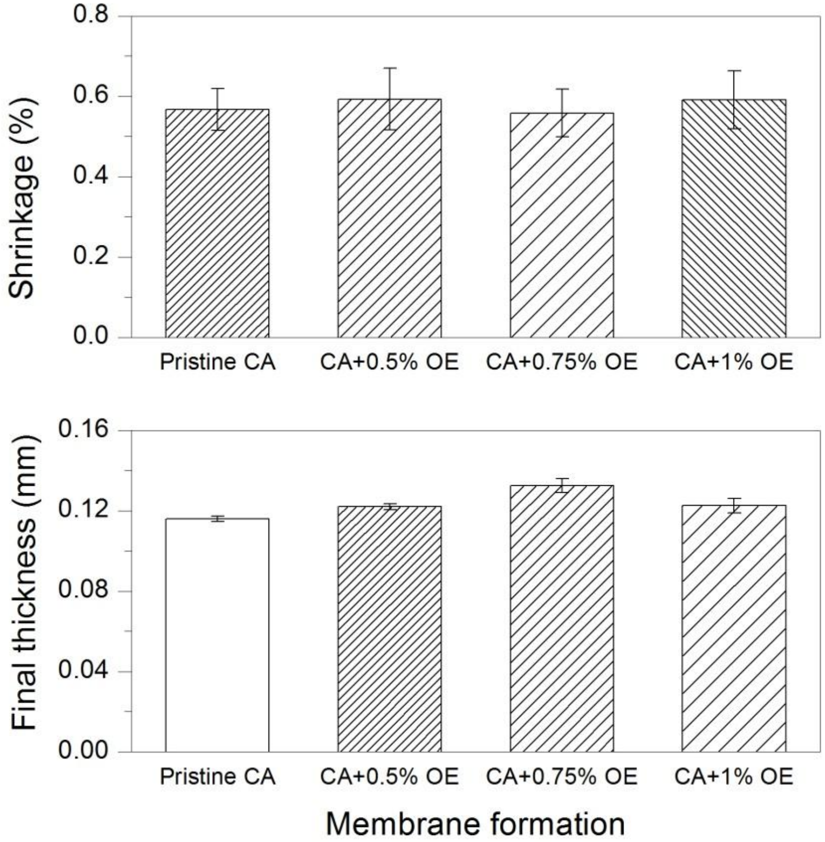
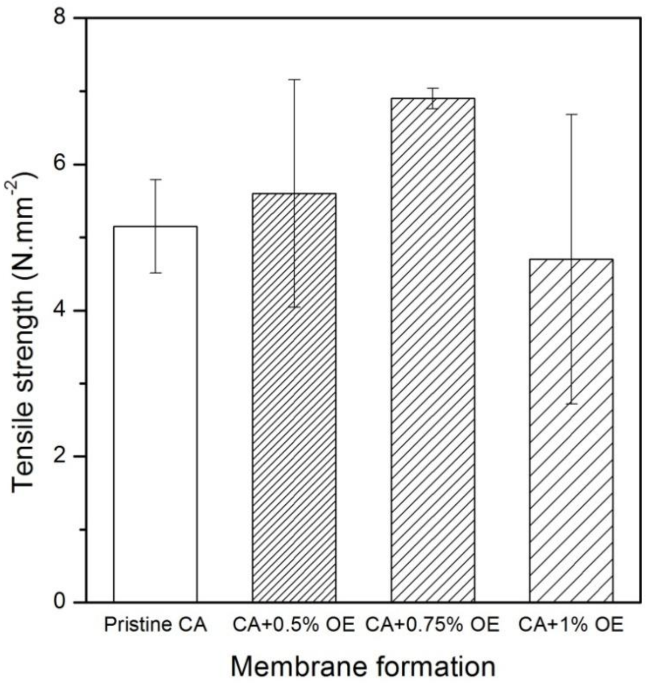
Results: Membrane thickness & tensile strength



Comparable membrane thickness



Small increase on membrane mechanical strength

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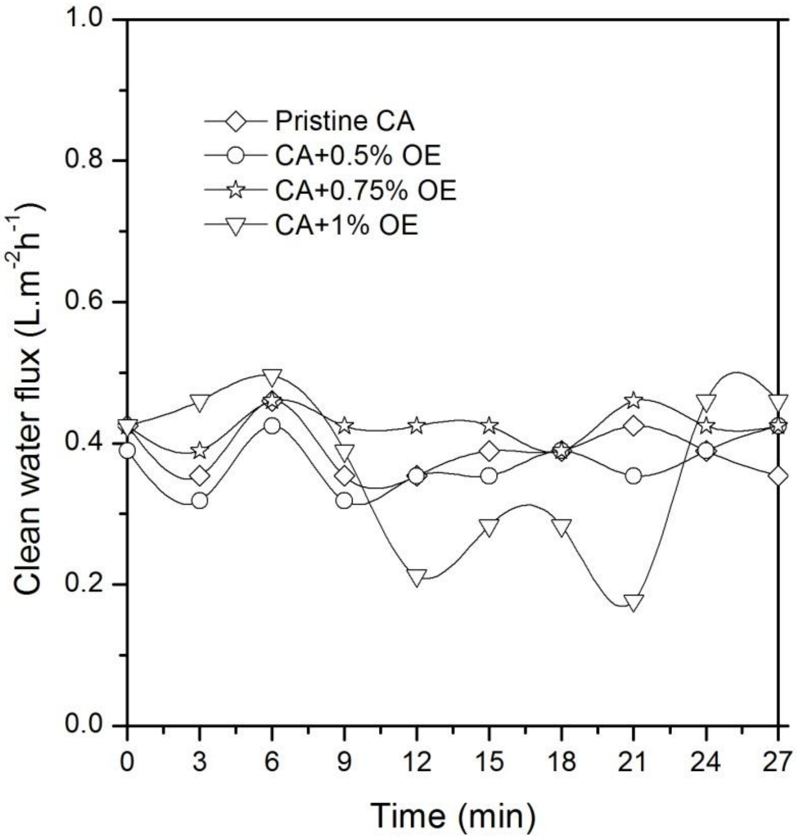
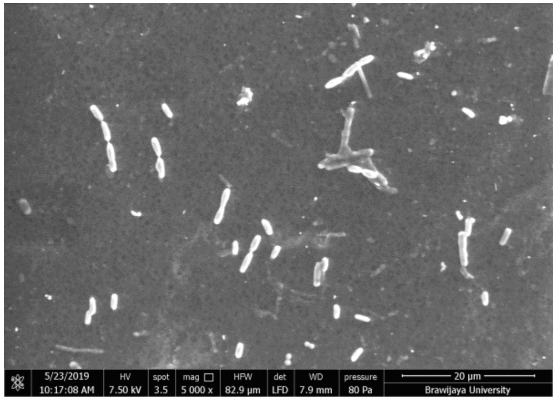
Results: Clean water flux

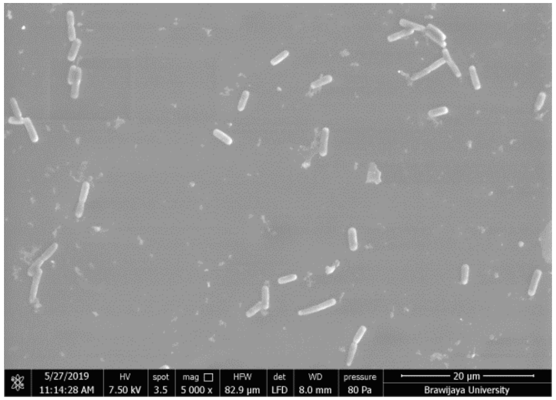


Comparable mass transport over the membrane

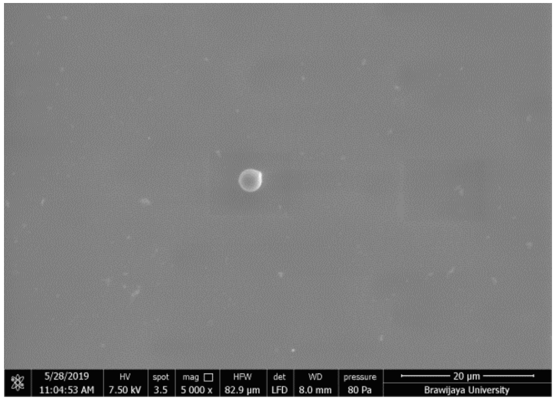
Results: Bacterial adhesion



Escherichia coli

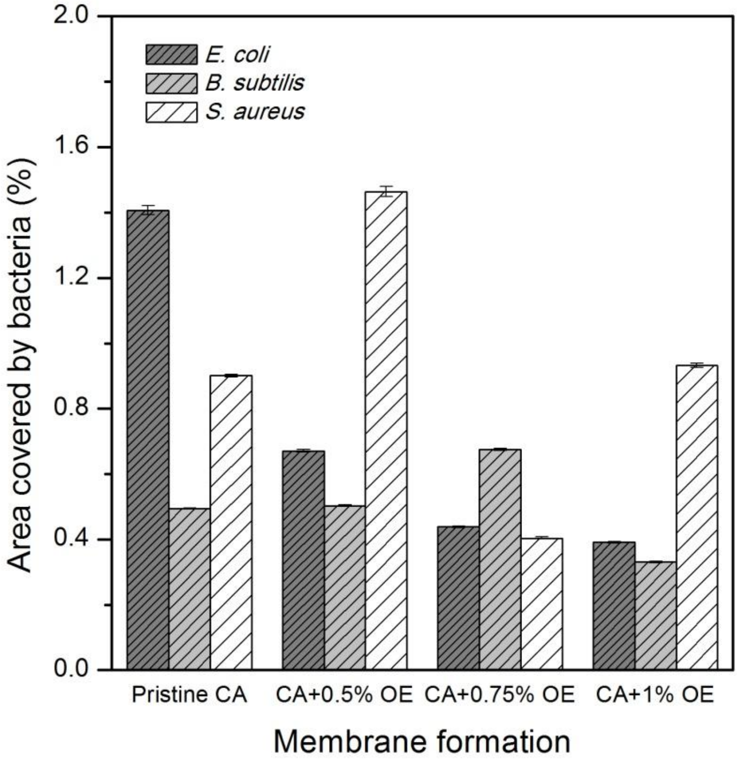


Bacillus subtilis



Staphylococcus aureus

Results: Bacterial adhesion



Escherichia coli are Gram negative bacteria, while *Bacillus subtilis* and *Staphylococcus aureus* are Gram positive bacteria.

Gram-negative bacteria have a low affinity, has thin membrane cells of 1-3 nm. Gram-positive bacteria have membrane cell thickness of 25-50 nm.

Phenolic compound might easily diffused through thin membrane cells of *Escherichia coli* rather than *Bacillus subtilis* and *Staphylococcus aureus*. Thus damaging the bacterial cells.

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Conclusions

1. Phenolic nanopowder extracted from *Olea europaea* leaves.
2. Nanopowder extract impregnated into CA matrix via phase inversion process.
3. The membranes show comparable mechanical properties and mass transport compared with pristine CA membrane.
4. Antibacterial properties observed for Gram-negative bacteria with thin membrane cells.

Thank you