



EFFECT THE ADDITION OF OLEIC ACID ON WATER BARRIER PROPERTIES OF EDIBLE FILMS

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KeyWords

Gelatin, Starch, Oleic Acid, Nano Chitosan, Biocomposite, Edible Film, Hydrophobic

ABSTRACT

The aim of this study was to determine the effect oleic acid addition on characterization of edible film and obtain the optimum concentration to improving the properties of edible film. Completely Randomized Design Method was used for this study consisting of five treatments and five replications. The edible biocomposite films prepared by addition of oleic acid (OA) with different concentration: 0%, OA 5%, OA 10%, OA 15%, and OA 20% based on cassava starch-gelatin total weight (w/w). Characteristic of biocomposite films were assessed by thickness measurement and analyzing water barrier properties. The results observed improving concentration of OA more than 5% (w/w) possessed weaker mechanical properties. The addition OA 5% showed ability films tendency to water absorption about 1,86 g/m Pa s x 10⁻¹⁰ and water transmission rate about 46,80 g/h.m². However because the major content of biocomposite film have hydrophilic nature, the effect of addition of oleic acid through the film were still wettable.

1. INTRODUCTION:

IN OVER LAST YEARS, PLASTICS, AS NON-RENEWABLE MATERIALS PACKAGING HAS CAUSED A SERIOUS ENVIRONMENTAL PROBLEM. THEREFORE, IT IS NECESSARY TO FIND ENVIRONMENT-FRIENDLY ALTERNATIVES TO PLASTIC PACKAGING. BIO-BASED PACKAGING MATERIALS ARE MADE OF NATURALLY RENEWABLE RESOURCES INCLUDING POLYSACCHARIDES, PROTEINS AND LIPIDS THAT OFFER BENEFICIAL EFFECT ON THE ENVIRONMENT IN TERMS OF RECYCLABILITY AND REUTILIZATION COMPARED WITH PLASTIC PACKAGING MATERIALS [1].

EDIBLE FILM AND COATINGS ARE THE FUTURE OF PACKAGING INDUSTRIES [2]. BARRIER FILMS AND COATINGS HAVE BEEN USED TO SUPPRESS MOISTURE MIGRATION BETWEEN PRODUCT AND ENVIRONMENT, OR BETWEEN DIFFERENT PARTS OF MULTICOMPONENT FOODS (WHERE THE PARTS HAVE DIFFERENT WATER ACTIVITIES), TO PREVENT DIFFUSION OF GASES SUCH AS O₂, CO₂ AND ETHYLENE, OR TO PREVENT MICROBIAL SPOILAGE. IN THIS WAY, PROLONGED SHELF LIFE, IMPROVED TEXTURE EXPERIENCE AND PROCESSABILITY ARE ACHIEVED [3].

WATER VAPOUR PERMEABILITY (WVP) AND OXYGEN PERMEABILITY (PO₂) REPRESENT THE MAIN BARRIER PROPERTIES OF MOST OF THE FOOD PACKAGING MATERIALS. INDEED, THE MASS TRANSFER OF THESE GASES PLAYS AN IMPORTANT ROLE IN THE HYDRATION LEVEL OF THE PACKAGED FOOD AND ON THE OXIDATION REACTIONS AND IS THEREFORE A KEY PARAMETER IN DETERMINING THE SHELF LIFE OF THE FOOD PRODUCTS [4].

THE CHARACTERISTIC OF EDIBLE FILMS DEPEND ON MATERIALS USED. AS A GENERAL RULE, FATS ARE USED TO REDUCE WATER TRANSMISSION; POLYSACCHARIDES ARE USED TO CONTROL OXYGEN AND OTHER GAS TRANSMISSION, WHILE PROTEIN FILMS PROVIDE MECHANICAL STABILITY [5].

POLYSACCHARIDES AND PROTEIN ARE TWO MAIN MACROMOLECULES THAT USED FOR EDIBLE FILMS PRODUCTION [6]. STARCH AND GELATIN, HAVE BEEN WIDELY STUDIED DUE TO THEIR EXCELLENT OXYGEN BARRIER PROPERTIES ASSOCIATED TO THEIR TIGHTLY PACKED HYDROGEN BONDED NETWORK STRUCTURE. BUT, DESPITE THESE INDIVIDUAL ADVANTAGES, BOTH POLYSACCHARIDES AND PROTEIN DISPLAY LIMITATIONS DUE TO THEIR POOR MOISTURE BARRIER PROPERTIES, BRITTLINESS, AND LOW TENSILE STRENGTH [7].

AMONG THE POLYSACCHARIDES THAT PRODUCE NANO-FILLERS WITH ATTRACTIVE FEATURES IS CHITOSAN [8]. CHITOSAN IS A CATIONIC PRODUCT OF ALKALINE DEACETYLATION OF CHITIN, WHICH IS THE MOST ABUNDANT BIOPOLYMER AFTER CELLULOSE [9]. CHITOSAN NANOPARTICLES HAVE BEEN SUCCESSFULLY USED AS FILLERS TO IMPROVE MECHANICAL AND BARRIER PROPERTIES AS WELL AS THE THERMO-STABILITY OF FILMS, DECREASE SOLUBILITY AND PRODUCE MORE COMPACT AND DENSE MATERIALS [10].

MULTIPLE COMPONENTS ARE OFTEN COMBINED TO FORM COMPOSITE FILMS WITH DESIRED PROPERTIES. IN FORMATION OF COMPOSITE FILMS AND COATINGS, TWO OR MORE COMPONENTS ARE COMBINED TO IMPROVE MECHANICAL PROPERTIES, GAS EXCHANGE, ADHERENCE TO SURFACES AND/OR MOISTURE BARRIER PROPERTIES [5]. FATTY ACID WAS INCORPORATED IN HYDROPHILIC POLYSACCHARIDE-BASED FILMS TO IMPROVE THEIR WATER-VAPOR BARRIER PROPERTIES. OLEIC ACID (OA) IS A COMMON LIPID DERIVATIVE THAT CAN EFFECTIVELY IMPROVE THE WATER-BARRIER PROPERTIES OF HYDROPHILIC FILMS. OA CAN BE USED AS HYDROPHOBIC ADDITIVE FOR DECREASING THE WVP OF POLYSACCHARIDE-BASED FILMS.

NEVERTHELESS, NO RESEARCH HAS SPECIALLY EXAMINED THE EFFECT ADDITION OA ON THE CHARACTERIZATION BIOCOMPOSITE FILMS. THE AIM OF THIS STUDY WAS TO DEVELOP A NEW EDIBLE FILM BASED ON STARCH (S), GELATIN (GEL), AND CHITOSAN NANOPARTICLES (CSNPs) AND TO OBSERVED THE EFFECT OF ADDITION OA ON THE CHARACTERIZATION OF BIOCOMPOSITE FILMS, ESPECIALLY WATER BARRIER PROPERTIES.

2. MATERIALS AND METHODS

MATERIALS

S AND GEL WAS PURCHASED COMMERCIALY. CSNPs WAS PRODUCED BASED ON IONIC GELATION METHOD FROM PRINT-G (SUMEDANG, INDONESIA) AND USED AS FILLER IN THIS EXPERIMENTS. GLYCEROL (GLY) PURCHASED FROM BRATACO, Co., LTD. DISTILLED WATER WAS USED IN ALL EXPERIMENTS.

2.1 PREPARATION OF CHITOSAN NANOPARTICLES (CSNPs)

CSNPs PREPARED WITH THE ASSUMPTION CONTAINS 0,1 G OF CHITOSAN (CS) FROM 120 G TOTAL SUSPENSION. CSNPs PRODUCED BY IONIC GELATION WITH THE FORMATION IS CS WITH TPP ANIONS. THE FORMULATION SHOWED IN TABLE 1.

TABLE 1 : THE FORMULATION FOR MAKING CHITOSAN NANOPARTICLES SUSPENSION

Materials	Quality (g)
Chitosan	0,1
Acetic Acid	99,90
Tripolyphosphate (TPP)	0,02
Aqueous Solution	19,98
Total (g)	120

THE METHODOLOGY FOR THE PREPARATION OF THE PRODUCT IS AS FOLLOWS:

CS POWDER (MESH 200) WAS WEIGHTED 0,1 G THEN DISSOLVED IN 99,90 G OF ACETIC ACID SOLUTION (1% v/v). TPP POWDER WAS OBTAINED BY DISSOLVED 0,2 G IN 19,98 OF AQUEOUS SOLUTION. THE BOTH OF SOLUTION WAS UNDER VIGOROUS MAGNETIC STIRRING FOR

2 HOURS AT ROOM TEMPERATURE. SUBSEQUENTLY CS SUSPENSION WAS ADDED DROP-WISE TO TPP SOLUTION UNDER VIGOROUS MAGNETIC STIRRING AT ROOM TEMPERATURE. THE FINAL SUSPENSION WAS ULTRA-SONICATED (HIELSCHER UP 2005, GERMANY) FOR 60 MINUTES IN ORDER TO BREAK ANY AGGREGATIONS AND REDUCE PARTICLE SIZE.

2.2 PREPARATION OF BIOCOMPOSITE FILMS

BIOCOMPOSITE FILMS WERE PREPARED BY DRY-CASTING METHOD WITH SOME MODIFICATIONS. THE FORMULATION FOR BIOCOMPOSITE FILMS SHOWED IN TABLE 2.

TABLE 2 : THE FORMULATIONS OF EDIBLE BIOCOMPOSITE FILMS WITH THE TREATMENT OF THE ADDITION OF OLEIC ACID

Materials	Treatment of the Addition of Oleic Acid (OA %)				
	0%	OA 5%	OA 10%	OA 15%	OA 20%
Gelatin (g)	1,33	1,33	1,33	1,33	1,33
Starch (g)	0,67	0,67	0,67	0,67	0,67
Glycerol (g)	0,4	0,4	0,4	0,4	0,4
Aqueous solution (g)	73,6	73,5	73,4	73,3	73,2
Chitosan nanoparticle (g)	24	24	24	24	24
Oleic acid(g)	0	0,1	0,2	0,3	0,4
Total (g)	100	100	100	100	100

FILM FORMING MATERIALS 2 G, SUCH AS STARCH (1,33 G) AND GELATIN (0,67) G WERE MIXED. CSNPs SUSPENSION 24 G (0,2 WT%) AND OA WITH DIFFERENT TREATMENTS, (0%, 5%, 10%, 15%, AND 20% W/W, BASE ON S-GEL WEIGHT) WERE ADDED AS FILLER AND HYDROPHOBIC MATERIAL, RESPECTIVELY. ALL MATERIALS DISSOLVED IN 100 G DISTILLED WATER AT 80 °C FOR 30 MINUTES. GLY (0,2 G) WAS ADDED AS PLASTICIZER IN MINUTES-20 UNDER CONTINUOUS STIRRING. AFTERWARD, THE FILM FORMING WAS HOMOGENIZED BY ULTRASONIC UNDER CONTINUOUS STIRRING FOR 15 MINUTES. FINALLY FILM FORMING WAS CAST INTO PLATE (22 x 15 CM) AND DRIED AT 50 °C FOR 5 H IN DRYING-OVEN. DRIED FILMS WERE PEELED FOR FURTHER ANALYSIS. BIOCOMPOSITE FILMS DENOTED AS CONTROL, OA 5%, OA 10%, OA 15%, AND OA 20%. ALL TREATMENTS HAVE FIVE REPLICATIONS AND DUPLO.

3. FILM CHARACTERIZATION

3.1 THICKNESS MEASUREMENT

THICKNESS OF THE FILM WAS DETERMINED USING A MICROMETER TO THE NEAREST 0.001 MM AT 10 SPOTS AROUND THE FILM AND THE VALUES WERE USED IN CALCULATIONS.

3.2 WATER BARRIER MEASUREMENT

WATER VAPOR PERMEABILITY WAS MEASURED BY ASTM E96-80 METHOD. SAMPLE WERE CUT IN SQUARE PIECES (4 x 4 CM). THE SQUARE PIECES WERE SEALED IN CUPS CONTAINING 3 G OF CaCl₂ AND PLACED IN WATER VAPOR TESTER AT TEMPERATURE 26 ± 2°C AND RH 90 ± 2%. THE WEIGHT OF THE CUPS WAS MONITORED EVERY HOUR OVER 8 H. THE SLOPE OF EACH LINE WAS CALCULATED BY LINEAR REGRESSION (R² > 0.99) OF WEIGHT CHANGE VS. TIME. WATER VAPOR TRANSMISSION RATE (WVTR) AND WVP WERE CALCULATED BASED ON EQUATION :

$$WVTR : \frac{m_1 - m_2}{t \cdot A}$$

$$WVP : \frac{WVTR}{\Delta P} \cdot d$$

4. STATISTICAL ANALYSIS

DATA FROM MEASUREMENTS WERE ANALYZED USING F-TEST ANALYZE AND ONE WAY ANNOVA. THE DIFFERENCES IN THE MEAN VALUES WERE ANALYZED USING DUNCAN'S MULTIPLE RANGE TEST (DMRT) WITH SIGNIFICANCE LEVEL OF 0,05 AND A CONFIDENCE INTERVAL OF 95% (P<0,05).

5. RESULTS AND CONCLUSIONS

5.1 THICKNESS ANALYSIS

THICKNESS GREATLY INFLUENCES THE WVP AND TRANSPARENCY OF THE FILM. PRECISE AND ACCURATE MEASUREMENT OF THIS PARAMETER IS REALLY IMPORTANT TO AVOID THE EFFECT TO BARRIER PROPERTIES [2]. THE MEASUREMENTS IN 10 SPOTS OF FILM OBTAINED MEAN OF THICKNESS ABOUT 43,58 MM-74,3 MM. BASED ON JIS (JAPANESE INDUSTRIAL STANDARD), EDIBLE FILM THICKNESS MAXIMUM WERE 250 MM AS GOOD CATEGORIZED FOR FOOD PACKAGING [12]. FOR COMPARATION RESULTS, THE VALUES FOUND BY CECCHINI ET AL. (2017)[13] AROUND 153-208 MM WORKING WITH BEESWAX AND BREA GUM. FIGURE 1 SHOWED THE DIFFERENCES THICKNESS BY THE ADDITION OF OLEIC ACID IN BIOCOMPOSITE FILMS.

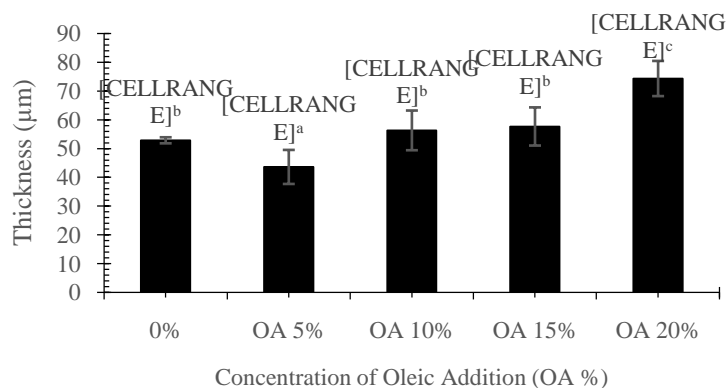


FIGURE 1 : GRAPHIC OF THE EFFECT OF ADDITION OA TO EDIBLE FILM’S THICKNESS

THE AVERAGE THICKNESS VALUE IS STILL SIMILAR TO THE AVERAGE THICKNESS FROM THE RESEARCH HASSAN AND NORZIAH (2017)[14] USES THE RAW MATERIAL FOR SAGO-GELATIN STARCH IS 50-70 MM. EDIBLE FILMS THICKNESS WERE INFLUENCED BY SUSPENDED MATERIALS, SIZE OR FILM’S PLATE DIMENSION. THE OTHERS FACTOR ARE OLEIC ACID NOT FILLS THE ENTIRE MATRIX. OLEIC ACID IS THOUGHT TO GATHERING AND AGGLOMERATION AT A CERTAIN AREA SO THAT THE AREA HAVE HIGHER VALUE THAN THE OTHERS AREA.

THE DIFFERENCES IN THICKNESS CAN BE CAUSED BY THE NATURE, COMPOSITION, AND RAW MATERIALS USED IN MAKING EDIBLE FILMS. THE NATURE OF THE BASIC INGREDIENTS OF HYDROPHILIC EDIBLE FILMS IS DIFFICULT TO BLEND WITH OLEIC ACID WHICH IS HYDROPHOBIC. THE THICKNESS OF THE EDIBLE FILM IS AFFECTED BY THE CONCENTRATION OF DISSOLVED SOLIDS ON THE EDIBLE FILM AND THE SIZE OR DIMENSIONS OF THE FILM PRINTING CONTAINER

5.2 WATER BARRIER PROPERTIES

WVP (WATER VAPOR PERMEABILITY) WAS USED TO EVALUATE THE PACKING ABILITY OF THE FILMS TO PREVENT WATER TRANSFER BETWEEN FOOD AND AIR, WHICH INCREASE THE SHELF LIFE OF FOOD (MA ET AL. 2016). THE RESULTS OBTAINED MEAN 1,86-3,36 G/M PA. S. X 10-10 - 3,36 G/M PA. S. X 10-10 . THE RESULTS OF WVP ANALYSIS SHOWED IN FIGURE 2.

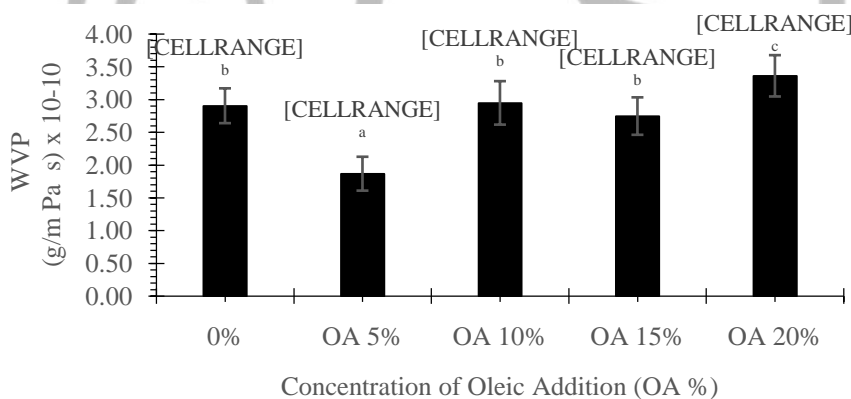


FIGURE 2 : GRAPHIC OF THE EFFECT OF ADDITION OA TO EDIBLE FILM’S WVP

THE EFFECT OF ADDING FATTY ACIDS TO THE FILM FORMULATION ALSO WAS CONSIDERED. AT HIGH CONCENTRATIONS OF FATTY ACIDS, A DISRUPTION OF THE POLYMER MATRIX IS CAUSED BY THE HYDROPHOBIC MOLECULES, INCREASING WVP VALUES (THESE COMPOUNDS WOULD PRESENT A LOWER COMPATIBILITY WITH THE MATRIX). AT LOW AMOUNT OF FATTY ACIDS, THE MATRIX DISRUPTION DOES NOT OCCUR AND THE HYDROPHOBIC EFFECT OF THESE COMPOUNDS IS MORE SIGNIFICANT. A HETEROGENEOUS DISTRIBUTION OF LIPIDS AND LOW STABILITY OF THE FORMED EMULSION WERE OBSERVED FOR FILMS COMPOSED OF HIGH FA CONCENTRATION [15].

THE LOWER PERMEABILITY VALUE INDICATES THE ABILITY OF EDIBLE FILM TO PASS WATER VAPOR FROM THE ENVIRONMENT TO FOOD PRODUCTS, SO THAT THE SAFETY OF FOOD PRODUCTS INCREASES AND EXTENDS THEIR SHELF LIFE. THE RATE OF TRANSMISSION OF EDIBLE FILM MOISTURE IS INFLUENCED BY SEVERAL FACTORS, NAMELY THE STRUCTURE OF EDIBLE FILMS (HOMOGENEITY, EMULSIONS, MULTILAYERS), CRYSTAL TYPE, SHAPE, SIZE, AND LIPID DISTRIBUTION. THE STRUCTURE OF EDIBLE FILM WITH THE ADDITION OF OLEIC ACID IS GETTING MORE ROUGH AND UNEVEN WHICH CAUSES THE PRESENCE OF PORES IN THE FILM MATRIX SO THAT MOISTURE CAN ABSORB EASILY [16].

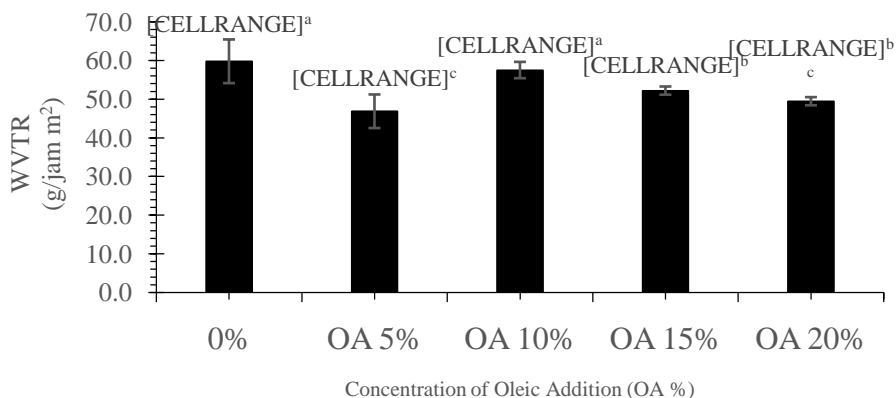


FIGURE 3 : GRAPHIC OF THE EFFECT OF ADDITION OA TO EDIBLE FILM'S WVTR

THE AVERAGE AMOUNT OF WVTR (WATER VAPOR TRANSMISSION RATE) VALUE AROUND 46,80 G/H.M2 UP TO 59,72 G/H.M2. THE STANDARD OF WVTR BY JAPANESE INDUSTRIAL STANDARD (JIS) MAXIMUM WERE 200 G/M2.24 HOURS. THE EFFECT OF ADDITION OLEIC ACID SHOWN NOT EFFICIENTLY DECREASE THE TENDENCY TO WATER ABSORPTION. THE EFFECT OF ADDING FATTY ACIDS TO THE FILM FORMULATION DID NOT HAVE A SIGNIFICANT EFFECT ON THE WVP. AT HIGH CONCENTRATIONS OF FATTY ACIDS, A DISRUPTION OF THE POLYMER MATRIX IS CAUSED BY THE HYDROPHOBIC MOLECULES, INCREASING WVP VALUES (THESE COMPOUNDS WOULD PRESENT A LOWER COMPATIBILITY WITH THE MATRIX). THIS COULD BE DUE TO THE HYDROPHILIC-HYDROPHOBIC RATIO IN THE FILM STRUCTURE. FATTY ACID IN FILM MATRIX IMPARTS THE HYDROPHOBIC CHARACTER RESULTING IN TO MORPHOLOGICAL ALTERATIONS IN THE FILM STRUCTURE [2]. THICKNESS OF THE FILM MATRIX IS STRAIGHTLY RELATED TO STARCH CONCENTRATION WHICH DESCRIBES THE INCREASING SOLID DENSITY OF FILM MATRIX AND PROMOTED THE RESISTANCE TO THE WATER VAPOUR PERMEABILITY [17].

FILM INSOLUBILITY IS ASSOCIATED WITH A HIGH INTERACTION DENSITY AND, MORE SPECIFICALLY, THE PRESENCE OF INTERMOLECULAR COVALENT BONDS OR "PHYSICAL KNOTS" (I.E., CHAIN ENTANGLEMENTS). IN ADDITION TO INTRA- AND INTERMOLECULAR FORCES AMONG THE FILM PROTEINS OR AMONG PROTEINS AND PLASTICIZER, THE SOLUBILITY OF PROTEINS ALSO DEPENDS ON THEIR ISOELECTRIC POINT (PI) AND IONIC STRENGTH OF THE MEDIUM. THE INTERACTION BETWEEN THE CHARGED MOLECULES AND THE MOLECULES OF THE POLAR SOLVENT INCREASES WITH THE INCREASING CHAIN CHARGE [15].

6. CONCLUSIONS

THE RESULT CONCLUDE THAT THE EFFECT ADDITION OF OA 5% TO THE EDIBLE BIOCOMPOSITE FILMS BASED ON STARCH, GELATIN, AND CHITOSAN NANOPARTICLES WAS THE BEST TREATMENT WITH MEAN VALUE OF THICKNESS 43,58 MM, WVTR 46,80 G/H.M2, AND WVP 1,86 G/M PA S X 10-10.

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