

## The Effect of Kaffir Lime Leaves Distillation Residue Oleoresin Concentration on Active Paper Packaging Characteristics

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**Abstract.** Oleoresin of kaffir lime leaves distillation residue still contains some active compounds such as *Citronellal*,  $\beta$ -*Citronellol*, and *Linalool* which potential to incorporated on the active paper packaging. The purposes of this study were to determine the effect of kaffir lime leaves distillation residue oleoresin concentration on the physical characteristics, sensory characteristics, and antimicrobial activity of the active paper packaging incorporated with kaffir lime leaves distillation residue oleoresin and to determine the functional groups of active paper packaging. The concentration of kaffir lime leaves distillation residue oleoresin were varied at 0%, 2%, 4% and 6%. The result showed that the addition of kaffir lime leaves distillation residue oleoresin increased the thickness and moisture content of the paper and decreased the tensile strengths and folding endurances of active paper packaging. The microbial inhibition tends to increase along with the higher oleoresin concentration addition. Aromatic CH group were found at a wavelength of 897.90 cm<sup>-1</sup> of on paper packaging with 2% oleoresin indicated as functional aromatic functional group allegedly obtained from the kaffir lime leaves oleoresin.

### 1. Introduction

Packaging has an important role in the food industry. The main role is its ability to extend the shelf life of the packaged product. The basic functions of packaging are to contain and protect the product from damage and thus more easily stored, transported and marketed [1]. In addition, there are some additional functions of the use of packaging for food products, namely as the identity of the product, protect the product from bad influences from outside, increase the attractiveness of potential buyers and increase efficiency. To improve the function of the packaging in extending the shelf life, active packaging that can prevent food damage due to the proliferation of microbes, as well as the gas exchange occurs, could be developed.

Active packaging is a system that can actively change the condition of packaged food to extend shelf life, to improve food safety or to maintain sense quality of foods [2]. Packaging will be classified as active packaging if the packaging that have either one of the following function: absorbent materials O<sub>2</sub> (oxygen scavenger), an absorbent material or CO<sub>2</sub> addition, ethanol emitters, ethylene absorber, water absorbent, antimicrobial materials, heating / cooling, absorber which can secret aroma / flavor, or a protective light. Additionally, active packaging can also equip with an O<sub>2</sub> indicator, CO<sub>2</sub> indicator, or indicators of quality defects which reacts with volatile materials produced from chemical reactions, enzymatic and/or damage from the microbial activity in food. Some examples of active packaging type: MAP (Modified Atmosphere



Packaging), plastic packaging with the anti-fog addition [2], and paper packaging with the addition of an antioxidant and antimicrobial compound (active packaging paper) [3].

Active paper packaging is packaging with the addition of an antioxidant or antimicrobial agent that can extend the shelf life of the packaged product [1]. Some substances have been used as active compound in the packaging paper including the uses of essential oils of cinnamon, essential oil of cloves and essential oil of oregano [4]; the uses of potassium sorbate and potassium metabisulphite in coating paper [5]; chitosan and DPCT (o, o'-dipalmitoyl chitosan) as an antimicrobial [6]; protein isolate and carvacrol [7][8], cinnamaldehyde [9] and ginger pulp oleoresin [10]. The concentration of the active substance in the paper packaging can also affect the physical properties of the paper produced. Potassium sorbate and sodium permanganate addition can decrease tensile strength and folding endurance of the paper packaging [5].

Spices and herbs in the form of leaves (fresh or dried), stem, bark or root (rhizome) used as a flavoring of foods and drinks. Spices are also known to have nutritional value, antioxidants, antimicrobial and as medicine. Essential oils extracted from herbs have been known have antimicrobial properties that can be used to control microbial growth in foods. Such as a mixture compounds of carvacrol and thymol in different proportions can reduce the growth of *Pseudomonas aeruginosa* and *Staphylococcus aureus* [9]. Kaffir lime has been used by Indonesian, especially in the form of the fruit and leaves as food seasonings. Kaffir lime leaves are also used as ingredients in traditional medicines because contain alkaloids, polyphenols, essential oils, tannins, and flavonoids [11]. Active compounds produced in the essential oil of kaffir lime leaves such as *Citronellal*, *Isopulegol*,  $\beta$ -*Citronellol*, *Geranyl acetate*, *Neryl acetate*, *Acetic citronellil*, *Charyofilen*, *Nerolidol*, *Diethyl carbitol* and *Fitol* [12]. Whereas other study reported that essential oils lime leaves contain the active compounds in the form of *Citronellal* 64.15%, 10.71%  $\beta$ -*Citronellol*, *Linalool* 5.54%, and *Trans-caryophyllene* 5.31% [13].

This study needs to be conducted to determine the addition of a distillation residue kaffir lime leaves oleoresin (*Citrus hystrix* DC.) at some concentration (0%; 2%; 4%; and 6%) on the characteristics of the active paper packaging include water content, tensile strength, folding endurance, thickness, antimicrobial activity and sensory test and to determine the content of the paper active packaging functional group by the addition of kaffir lime distillation residue oleoresin (*Citrus hystrix* DC.) at selected concentration.

## 2. Experimental

### 2.1 Preparation of Kaffir Lime Distillation Residue Oleoresin

The residue of kaffir lime distillation was air-dried using a blower for 8 hours. The prepared materials were extracted by maceration method using 96% ethanol as a solvent in a ratio of materials: solvent = 1: 5 for 5 hours and 14 minutes at 74°C. Furthermore, the extract was evaporated using a rotary evaporator 100 rpm temperature of 60°C [14].

### 2.2 Preparation of Active Paper Packaging

Filter papers were cut into 2 mm x 2 mm. Then, 5 g of cut paper soaked in 250 mL of distilled water for 24 hours. Soaking pieces of paper added with 250 mL of distilled water and crushed with a blender for 15 minutes to become pulp. 0.45 grams of chitosan powder added beaker glass that containing 100 mL of 1% acetic acid and stirred into a solution of chitosan in acetic acid [9]. In another beaker glass, 50 mL of distilled water added to the kaffir lime leaves distillation residue oleoresin that varied at 0 g; 0.3g; 0.6g; and 0.9g and added with tween 80 at 0, 10 drops, 12 drops and 13 drops (respectively) to form an emulsion of oleoresin and Tween-80. Paper pulp, a solution of chitosan in 1% acetic acid, and emulsion oleoresin with Tween-80 mixed using a blender for 5 minutes. In that mixture, distilled water was added until the volume of 1,000 mL and blend for 5 minutes [9]. Solution of 4.5 g of tapioca in 50 mL of distilled water was added to the mixture and blended again. The last mixture was poured into molding paper with screener (20 cm x 30 cm), trimmed rolled and pressed until wet paper sheet formed. The wet paper sheet was dried at room temperature (30°C) for 48 hours with reversal of paper after 24 hours of drying.

### 2.3 Analysis of the Active Paper Packaging

Each active packaging paper was analyzed the physical characteristics such as water content with thermogravimetric method [15], thickness (ISO 536: 2010), tensile strength (ISO 1306: 2009), folding

endurance (SNI 0491: 2009). Antimicrobial activity [16], sensory testing [17] and the functional groups on the packaging paper with the addition of selected oleoresin concentration [18].

#### 2.4 Data Analysis

This research design used to a completely randomized design (CRD), which consists of a single factor, which is a variation of concentration kaffir lime leaves distillation residue oleoresin. Data were analyzed using Oneway ANOVA to determine whether there is a difference of treatment at the 5% significance level was followed by Duncan test DMRT Multiple Range Test at a significance level of 5%.

### 3. Result and Discussion

#### 3.1 Physical Characterization of Paper Packaging

**3.1.1 The water content.** The addition of oleoresin not significantly affected the water content of the paper packaging (Table 1). The addition of ginger pulp oleoresin up to 6% also showed insignificant effect on the water content of paper [10]. However, the increasing trend of the water content observed by the addition of oleoresin. Water content increased along addition of oleoresin in the paper. The higher concentration of oleoresin, the higher proportion of the tween-80 as emulsifier were needed at the paper packaging production. Tween-80 can role function as an emulsifier or surfactant that can stabilize the emulsion [19]. Tween-80 is hygroscopic and provide a porous structure [20]. In the packaging paper with the addition of oleoresin have a higher water content than the control paper packaging due to the porous structure was formed allowing the packaging paper re-absorb water prior water content testing process after drying. This is not consistent with the previous study [21], which states that in the manufacture aloe vera powder, the addition of Tween-80 reduced the water content of the flour because tween-80 can expand the surface so that it will speed up the drying process because the transportation system is accelerated in removing water contained in the material on the evaporation process. The results obtained on testing the water content of paper packaging of this study fulfilled the requirement by ISO 0123: 2008 on cardboard duplex that maximum 10% of water content.

**Table 1.** Physical characterization test active paper packaging with an addition of kaffir lime leaves distillation residue

Oleoresin concentration	Water content (wb)%	Thickness (cm)	Tensile strength (N/mm)	Folding endurance
0%	8.893 <sup>a</sup> ±0.901	0.791 <sup>a</sup> ± 0.005	1.007 <sup>b</sup> ±0.152	0.430 <sup>a</sup> ±0.220
2%	9.921 <sup>a</sup> ±0.245	0.879 <sup>ab</sup> ±0.061	0.492 <sup>a</sup> ±0.155	0.240 <sup>a</sup> ±0.020
4%	9.981 <sup>a</sup> ±0.334	1.014 <sup>c</sup> ±0.023	0.446 <sup>a</sup> ±0.249	0.095 <sup>a</sup> ±0.095
6%	10.142 <sup>a</sup> ±0.433	0.935 <sup>bc</sup> ±0.040	0.298 <sup>a</sup> ±0.040	0.070 <sup>a</sup> ±0.000

Note: Notation with same letters in the same column showed no significant difference at a significance level of 5%

**3.1.2 Thickness.** There was a significant difference in the thickness parameter (Table 1) of all samples. The thickness of paper packaging ranged between 0.7-1 mm. Paper packaging with the addition of oleoresin tends to have a higher thickness than the control paper packaging (without oleoresin addition). Several studies reported that the higher total solid of the material the higher thickness of the film. The thickness of the edible film increased with the addition of corn starch and white saffron filtrates [22] and several essential oils [23].

**3.1.3 Tensile Strength.** From Table 1 can be seen that the addition of oleoresin can affect tensile strength value. This is because higher water content value and a porous structure resulting from the addition of tween 80 as an oleoresin emulsifier that can affect the physical properties of the packaging paper becomes more brittle, so that impact to the decreasing of tensile strength. This is consistent with the other study [24] which stated that the addition of tween 80 as a plasticizer can improve the flexibility of the mixture but may decrease the value of Young's modulus in the film. The tensile strength of the pulp sheet derived from

several types of wood in Indonesia ranged between 42-61 Nm / g [25]. Lowering of tensile strength value also performed by the addition of ginger pulp oleoresin on paper materials [10].

**3.1.4 Folding Endurance.** Folding endurance value of paper packaging decrease with the increasing of oleoresin addition (Table 1). The highest folding endurance value was performed by control paper packaging. However, the statistical analysis at a significance level of 5% of the folding endurance analysis results showed no significant difference. Folding endurance value is influenced by the structure of the packaging paper sheet that rigid and porous. Folding endurance decrease because it's affected by moisture content [26]. Active paper with lower water levels tends to show much lower folding endurance values. Water molecules affected stretching on the cellulose fibers, due to the reduction in hydrogen bonding intramolecular while flexibility cellulose fibers affect the durability of paper folding. However, in this study the results obtained in contrast to the results of the research. Higher water content samples showed much lower folding endurance value.

### 3.2 Sensory Test of Active Paper Packaging

The color of packaging paper with the addition of 2%, 4% and 6% significantly different from the control paper packaging (Table 2). It can be seen that the color of 2% addition of oleoresin concentration showed significantly different with the that of 4% and 6% addition. Paper packaging control showed the highest assessment score of 4.31 which can be interpreted that paper packaging was preferred by the panelists.

**Table 2.** Sensory test of active paper packaging

Oleoresin concentration	Colour	Texture	Odor	Overall
0%	4.31 <sup>c</sup> ±0.604	3.34 <sup>a</sup> ±0.614	3.66 <sup>b</sup> ±0.769	3.93 <sup>b</sup> ±0.593
2%	3.63 <sup>b</sup> ±0.615	3.33 <sup>a</sup> ±0.547	2.90 <sup>a</sup> ±1.094	3.13 <sup>a</sup> ±0.730
4%	2.93 <sup>a</sup> ±0.828	3.20 <sup>a</sup> ±0.805	2.83 <sup>a</sup> ±1.053	3.00 <sup>a</sup> ±0.695
6%	2.83 <sup>a</sup> ±0.952	3.20 <sup>a</sup> ±0.761	3.13 <sup>ab</sup> ±1.137	3.07 <sup>a</sup> ±0.785

Note: Notation with same letters in the same column showed no significant difference at a significance level of 5%

The texture values of papers were 3.34; 3.33; 3.20; 3.20 for 0%, 2%, 4% and 6% samples, respectively (Table 2). This value can be interpreted that neutral assessment given by the panelists. This was apparently due to the method of making the manual paper, packaging paper, causing the similar texture.

The odor of control active paper packaging showed significantly different values with packing paper with the addition of 2%, 4%, and 6% oleoresin. Control paper packaging has the highest value compared to other papers packaging. The higher oleoresin concentration addition generated stronger odor.

Control paper packaging showed the highest overall value as shown in Table 2. The value of control paper packaging significantly different from the packing paper with the addition of 2%, 4%, and 6% oleoresin. Paper packaging control was preferred by the panelists (3.93). The packaging paper with the addition of 2%, 4%, and 6% oleoresin showed the neutral values those were 3.13; 3.00; and 3.07, respectively. The additional of ginger pulp oleoresin also reduced sensory characteristic (color, texture and overall) of paper packaging [10].

### 3.3 Antimicrobial Activity of Active Paper Packaging

The result of antimicrobial activity testing against *Pseudomonas fluorescens* and *Aspergillus niger* showed that the addition of 2%, 4%, and 6% oleoresin concentration were not significantly different from the control paper packaging (without the addition of oleoresin) (Table 3). However, it can be seen that the inhibition tends to increase along with the higher concentration addition.

**Table 3.** Antimicrobial activity of active paper packaging

Oleoresins concentration	<i>Pseudomonas fluorescens</i> (mm)	<i>Aspergillus niger</i> (mm)
0%	5.72 <sup>a</sup> ±0.035	9.47 <sup>a</sup> ±0.274
2%	6.09 <sup>a</sup> ±0.579	12.65 <sup>a</sup> ±2.227
4%	6.46 <sup>a</sup> ±0.071	13.58 <sup>a</sup> ±0.389
6%	7.07 <sup>a</sup> ±1.034	14.27 <sup>a</sup> ±3.662

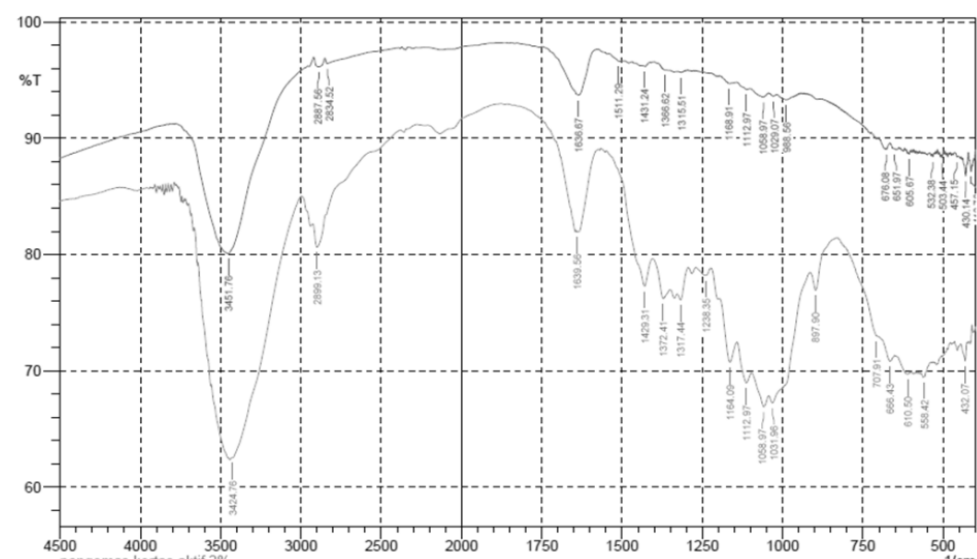
Notes: Notation with same letters in the same column showed no significant difference at a significance level of 5%  
Calculation includes paper packaging diameter

#### 3.4. Analysis of Active Paper Packaging Functional Groups

In this study, the cellulose can be demonstrated at a wavelength of 1366.62 cm<sup>-1</sup> (CH) and 1029.07 cm<sup>-1</sup> (CO) on the control packaging paper and 1372.41 cm<sup>-1</sup> (CH), 1058.97 cm<sup>-1</sup> (CO) in the 2% oleoresin packaging paper (Table 4). Based on FTIR analysis, at 2% oleoresin packaging paper can be found OH groups (3224.76 cm<sup>-1</sup>), CH (2899.76 cm<sup>-1</sup>, 1429.31 cm<sup>-1</sup> and 1372 cm<sup>-1</sup>). CN group can be known from the uptake 1258.55 cm<sup>-1</sup> and 1317.44 cm<sup>-1</sup>, while the CO group showed at 1058.97 cm<sup>-1</sup> and group C = C absorption showed at 1,639 cm<sup>-1</sup>. In addition, at the selected paper packaging was performed any aromatic CH group at a wavelength of 897.90 cm<sup>-1</sup> (Figure 1). This functional aromatic functional group allegedly obtained from the kaffir lime leaves oleoresin. Tween 80 indicated by absorption bands at 2938 and 2869 cm<sup>-1</sup> to CH, 1730 cm<sup>-1</sup> to C = O, and 3462 cm<sup>-1</sup> to OH [27]. In this study, the CH contained in uptake 2899.76 cm<sup>-1</sup>, and 3224.76 for the OH group. The spectrum of chitosan can be identified at a wavelength of 895 cm (bond NH) and also at a wavelength of 1,654 cm (stretching of C = O group of the amide of N-acetyl) [6]. While on paper packaging with a concentration of 2% chitosan oleoresin can be identified from the wavelengths of 897.9 cm.

**Table 4.** Functional Groups Analysis

Functional Groups	Wavelength (cm <sup>-1</sup> )	
	Control	2% Oleoresin
O-H	3451.76	3424.76
C-H	1431.24;1366.62; 2887.56	2899.13;1429.31; 1372.41
C-N	1315.51;1168.91; 1058.97	1258.55;1317.44; 1238.35
C-O	1029.07	1058.97
C=C	1636.67;1511.29	1639
C-H (aromatic)	-	897.90



**Figure 1** Functional groups of active paper packaging  
(Note : Control: top spectrum line; 2% Oleoresin:bottom spectrum line)

#### 4. Conclusion

The result showed that the addition of kaffir lime leaves distillation residue oleoresin increased the thickness and moisture content of the paper and decreased the tensile strengths and folding endurances of active paper packaging. The microbial inhibition tends to increase along with the higher oleoresin concentration addition. Aromatic CH group were found at a wavelength of  $897.90\text{ cm}^{-1}$  of on paper packaging with 2% oleoresin indicated as functional aromatic functional group allegedly obtained from the kaffir lime leaves oleoresin. Generally, the paper packaging with 2% oleoresin showed similar physical characteristics with the control samples except for the tensile strengths properties. However, the antimicrobial activity of the paper packaging with 2% oleoresin was slightly higher than that of control sample indicating the improvement of the paper packaging properties.

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