



## EFFECT OF ACETIC ACID: FORMIC ACID RATIO ON CHARACTERISTICS OF PULP FROM OIL PALM EMPTY FRUIT BUNCHES (OPEFB)

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### ABSTRACT

Oil palm empty fruit bunches (OPEFB) is one of the solid waste which can be used as raw material for pulp. The objective of this research was to determine the effect of acetic acid: formic acid ratio on the characteristics of OPEFB pulp. The result showed that the increasing of the amount of formic acid can reduce levels of cellulose, hemicellulose, lignin and pulp yield. The best results was achieved on the treatment ratio of acetic acid and formic acid = 85:15 which produced 73, 75% cellulose, 7.78% hemicellulose, 1.61% lignin, and 32.57% yield.

**Keywords:** acetic acid, formic acid, OPEFB, pulp.

### INTRODUCTION

Oil palm empty fruit bunches (OPEFB) is one of the majority of solid wastes generated by the oil palm mills. Oil palm empty fruit bunches as 23 percent of the fresh fruit bunches contains 55-60 percent dry weight lignocellulosic materials for [1]. Peak production of palm oil per hectare of 20-24 tons of fresh fruit bunches per year will produce 2.5 to 3.3 tons of lignocellulosic materials. Cellulose content of OPEFB of palm oil is 37.3-46.5%, while hemicellulose and lignin content are 25.3 - 33.8% and 27.6 - 32.5%, respectively [2].

OPEFB is suitable and a potential renewable biomass material for the conversion of other valuable product because it is locally abundant and has high content of lignocellulosic components [3-4]. The average length of fiber: 1.2 mm, the diameter: 15 micrometers, thick walls: 3.49 micrometers and the Runkel Number: 0.87 [5]. An agricultural residue is an important alternative to substitute wood as a raw material in the pulp and paper industry [6] and [7]. Non-wood material is cooked with hybrid chemomechanical and alkali-based chemicals [8] and [9]. The conventional alkaline pulping process such as alkali based chemicals is not suitable for many non-wood raw materials and might cause serious environmental problems. Therefore, many alternative pulping processes have been introduced in the world. Organosolv processes are one of the most promising alternative processes. These cooking methods are based on organic solvents cooking such as alcohols or organic acids. The types of alcohol such as methanol or ethanol and the organic acids such as formic acid or acetic acid usually were used [10].

Pulping organosolve can be used as an alternative method because its investment cost is relatively low; do not pollute the environment, and obtaining pulps from hemicellulose and lignin easily with product properties of high yield, low residual lignin content, high brightness and good strength [11-20]. Moreover, products values included hemicelluloses and sulphur-free lignin fragments due to their high purity, low molecular weight, and easily recoverable organic reagents [21]. Organic acid process is an alternative method of organosolv pulping to delignify

lignocellulosic materials to produce pulp for paper [6], [22-23]. Typical organic acids used in the acid pulping methods are formic acid and acetic acid. The process is based on acidic delignification to remove lignin, a necessary part of the hemicellulose and nutrients, while silicon remains in the pulp. The pulping operation can be carried out at atmospheric pressure. Used acid in pulping can be easily recovered by distillation and re-used in the process [17].

The Formacell process was developed from the Acetosolv process. Organosolv pulping approach in which a mixture of formic and acetic acid is used as the cooking chemical [24] have invented a process where lignocellulosic material is delignified under pressure with a mixture of acetic acid (50-95 w-%), formic acid (< 40 w-%) and water (< 50 w-%) [25]. Formacell pulps from annual plants have better strength properties than corresponding soda pulps [26]. Pulping of organic material for non-timber has made to bark [27], bagasse [28], jute bast [29], cobs cardoon (*Cynara cardunculus*) [30], and wheat straw (*Triticum vulgare* CV. *Horoshiri*) [31]. Delignification of lignocellulosic with low pressure and a mixture of acetic acid (50% 95%), formic acid (<40%), water (<50%), and the temperature between 130°C - 190°C with a ratio of solvent and timber from 1:1 to 12:1 produces the value of Kappa number is low and the strength of the pulp increased in the process formacell with wood raw materials [25].

Several factors affecting the success of pulping acetosolv methods are the ratio of solvent to water, the ratio between the amount of solvent cooker with the material to be cooked, a variety of pulping conditions as regards acetic acid concentration, catalyst concentration, temperature and pulping time [32-34]. Acid catalyst which can be used are HCl and H<sub>2</sub>SO<sub>4</sub>. [32] [9]. The used of formic acid: acetic acid: water 20:60:20 with cooking temperature 107°C for 3 hours with a solution ratios cookers and rice straw 12: 1 to produce pulp with a tensile index of 4.38 mNm<sup>2</sup> / g and index torn kPa<sup>2</sup> 2.52 / g [35]. The concentration of the organic solvent will decrease the yield, hemicellulose, and lignin kappa [9]. The purpose of



this study is to determine the comparative effect of acetic acid: formic acid against chemical characterisation and pulp fibers OPEFB.

## MATERIALS AND METHODS

### Raw material

This study used oil palm empty fruit bunches (OPEFB) from PTPN Business Unit VII Central Lampung Bekri as raw material. Several chemicals used were glacial acetic acid (Merck), formic acid (Bratachem), HCl (Merck),  $H_2SO_4$  (Merck) and distilled water.

### Pulping

Pulping experiments were performed in 1.3 liter autoclaves with 20 g of fibrous strands of OPEFB and a liquor to sample ratio of 15:1 flat-bottom, and wide-mouth boiling flask equipped with a condenser. Solvent comparison of acetic acid: formic acid, namely (K1): 100:0 (K2) 90:10, (K3) 85:15, (K4) 80:20, (K5) 75:25, and (K6) 70:30, and HCl catalyst was given at 0.5% for 1 hour cooking time at 130 °C. After cooking, the spent cooking liquor was separated and collected by filtration and washing with flowing water until reached room temperature or up to the clear water washing results. Then wet pulp leaching results were dried at room temperature in order to obtain dry pulp.

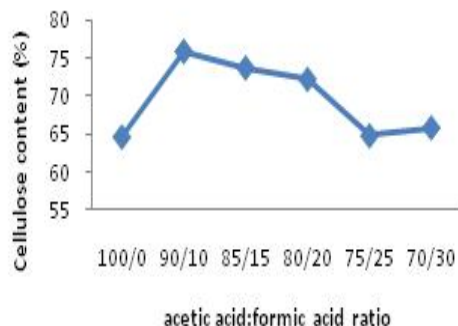
### Pulp characterization

Chemical properties were tested for each pulp consists of yield, cellulose, hemicellulose, and lignin methods Chesson [36]. Scanning electron microscopy (SEM) has been most useful technique for quantification of fibre structural details.

## RESULTS AND DISCUSSIONS

### Cellulose

The results showed that the value of cellulose pulp product ranged from 64.63% to 75.9% (Figure-1). The use of formic acid, acetic acid, and water (30:50:20) with cooking time of 90 minutes were carried out to produce cellulose 56.87% with wood raw materials [37].



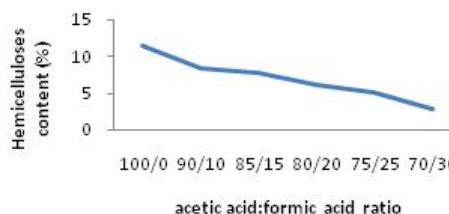
**Figure-1.** Effect of acetic acid: formic acid ratio on the contents of cellulose OPEFB pulp.

The results showed that the use of formic acid which was higher than 10% can reduce levels of cellulose pulp of OPEFB. This was caused by the hydrolysis of polysaccharides during pulp cooking process. Chemical hydrolysis in the production of pulp with an acid environment degradation reaction is the most distinctive of the glycoside-glycoside bond glycosidic di-oligo and polysaccharides. Thus, the long chains of cellulose will be short and compounds degradation products such as hydroxy acids will dissolve cellulose when washing hence that the levels will be lower [38]. The high concentration of formic acid was not only dissolves the lignin into an organic solvent, but cellulose was also degraded [39].

### Hemicellulose

The results showed that the average value of hemicellulose generated in this study ranged from 2.71% to 11.44% (Figure-2). The use of formic acid and 2-hour cooking time ranges produced 7, 8% of hemicelluloses [9]. The concentration of formic acid can decrease levels of hemicellulose due to the degradation experienced by hemicellulose [39].

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**Figure-2.** Effect acetic acid: formic acid ratio to the contents of hemicellulose OPEFB pulp.

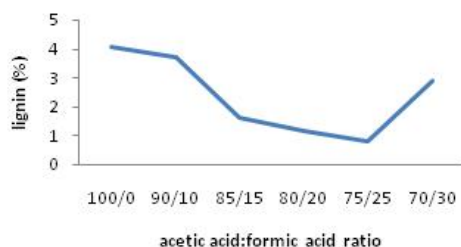
Formic acid concentration which is greater than 10% can reduce levels of hemicellulose produced. This is presumably due to the nature of hemicellulose compounds which are amorphous and rapidly degraded by the acidic solution. Hemicellulose is bonded with polysaccharides, proteins and lignin and more soluble than the cellulose [40]. Polysaccharide compounds such as cellulose and hemicellulose are glycoside bond linking chains of these compounds. Glycoside bond was easily hydrolyzed by the acid through a chemical reaction and this situation accelerated by warming [41]. Hemicellulose will experience a reaction oxidation and degradation beforehand than cellulose, hemicellulose for shorter molecular chains and branched. Hemicellulose is insoluble



in water but soluble in aqueous alkaline solution and more easily hydrolyzed by acid than cellulose [38].

### Lignin

The results showed that the average value of lignin produced in this study ranged from 0.811% to 4.055% (Figure-3).



**Figure-3.** Effect acetic acid: formic acid ratio to the contents of lignin OPEFB pulp.

The decreasing of lignin in pulp cooking process OPEFB was allegedly due process of delignification during cooking. Delignification process was caused by the acid hydrolysis of  $\alpha$ -aryl ether bond [42-43]. The dissolution of lignin has been ascribed to the cleavage of  $\alpha$ -aryl and aryl-glycerol- $\beta$ -aryl-ether bonds in its molecule [44]. Hydrogen ion concentration plays a very important role in solvent pulping. This is because lignin dissolution is expected to be preceded by the acid-catalyzed cleavage of  $\alpha$ -aryl and  $\beta$ -aryl ether linkages in the lignin macromolecule, and becomes soluble in the pulping liquor. The use of formic acid is less than 10% and its cooking time less than 1.5 hours causes the lignin is still high. This because of the cooking process duration is not sufficient for lignin delignification. Other reported that the palm fronds pulp lignin content generated by cooking use formic acid solution tends to decrease with increasing the cooking time at each concentration of formic acid is used. The lignin content after cooking use formic acid and the cooking time 1 hour - 3 hours [45]. OPEFB obtained pulp lignin content generated at 11.20% - 19.12%. The formic acid and acetic acid can delignificate of lignin because it is acidic so it can degrade lignin well. The lignin diminishing is suspected because of the condensation of lignin in the pulp. A kinetic model made is lignin dissolution followed by lignin condensation [46-47].

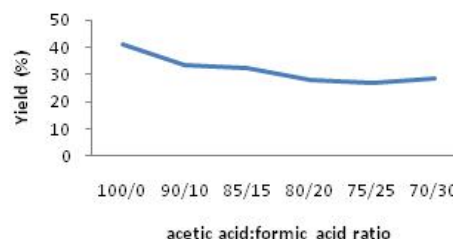
Condensation during pulping occurs to a greater extent with formic acid than with acetic acid, and to a greater extent with acetic acid than with propionic acid. This happens was due to the use of organic acid higher concentration and tend to encourage the re-lignin polymerization reaction that has been dissolved in the cooking liquid, so that the lignin content of pulp increased again [17].

High lignin content pulp cooking results may be due to the condensation process so that lignin settles on the surface of the pulp so that the color becomes darker [48]. The process of condensation formed by the merger of

the chains of carbon which form the chains are longer, where the compound formed is an intermediate carbocation [38]. Useformacell process will produce pulps with properties such as high-yield, low residual lignin content, high brightness and good strength can be produced [14] [15].

### The yield of pulp

The results showed that the average value of the yield generated in this research ranged from 27.05% to 41.02%. Increasing the amount of formic acid lowering the yield is presented in Figure-4.



**Figure-4.** Effect acetic acid: formic acid ratio to the contents of yield OPEFB pulp.

Formacell pulping is resulted in intensive hydrolysis of lignin carbohydrate bonds because the acid solution has reactive properties which can break bonds lignin-polysaccharide complexes thereby lowering the yield. The increasing of chemicals concentration was tending to decrease the yield of total pulp due to the degradation of polysaccharides and lignin. This happens because of the concentration the higher the amount of lignin is degraded more and more. There is also an increase in the degradation of wood components such as cellulose and wood extractive substances. Delignification by organic solvents has been ascribed to the hydrolysis of  $\alpha$ -aryl-ether and lignin-cellulose bonds, and to conform to a *pseudo* first-order kinetics like yield losses [49].

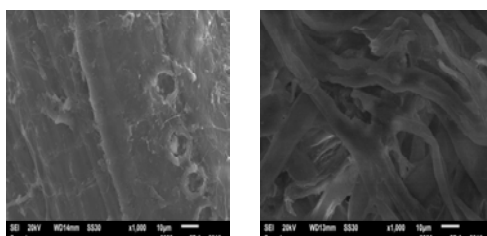
### Assessment of the structure of OPEFB pulp fibres

OPEFB pulp fibres have several important characteristics which determine their structural and mechanical properties. The fibre wall thickness and the degree of fibrillation are just a few examples of the characteristics that describe a fibre structure and behaviour [50]. Figure 5 shows the morphology of treated OPEFB pulp. It was observed that the morphology of the fibers differs with the treatment process. Treatment acetic acid:formic acid ratio separated the fiber bundles into individual fibers with a significant decrease in fiber diameter.

OPEFB raw fiber surface uneven and matted with a binder lignin. OPEFB raw Fibers composed of bundles with individual cells are continuously bonded together by cementedlignin and hemicellulose components. Irregular surface appears with this cementing and candles materials and their circle indicates silica. According to [51] and



[52], the white granules are silica bodies which are known as phytoliths and they are embedded on the surface of OPEFB fibres. Figure-5 shows that there is a change of the fiber structure becomes smaller in the pulping process. Figure-6 SEM analysis results OPEFB before and after in the pulping (magnification 1000x). Scanning electron microscopy (SEM) has been an important technique for assessment of fibre structures since its introduction



(a) EOFEB raw fiber

(b) acetic acid:formic acid 85:15

**Figure-5.** Morphology of treated OPEFB pulp.

The results of the analysis used Scanning Electron Microscope (SEM) with a magnification of 1000x indicate a change of crystalline form and the large vessel diameter into irregular shapes and black dots that indicate silica began to disappear. Other reported that test results showed that the fiber OPEFB SEM has a long vascular and fiber with large diameter [53].

## CONCLUSIONS

The results of the study showed that the increasing of the amount of formic acid added can reduce levels of cellulose, hemicellulose, lignin and pulp yield TKKS. The best result of this research was the ratio of acetic acid: formic acid = 85:15 which produced level of cellulose 73, 75%, hemicelluloses 7.78%, lignin content 1.61%, and yield of 32.57%.

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