Effect of pretreatment concentration on pulp blending between oil palm empty fruit bunch and citronella leaf fibers in terms of pulp and paper properties

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Abstract. Papers are one of the most used materials in food packaging industries. Additives need to be added in order to improve and prolong sensory properties of food. This is to maintain the food quality level before reaching to the end users. A preliminary study has been conducted to produce antimicrobial paper by blending the pulps of Citronella (Cymbopogon nardus) with oil palm empty fruit bunch (EFB). The papermaking was done according to TAPPI Standard Method. Morphological observation using Scanning Electron Microscope was carried out for all samples while physical, optical and mechanical paper properties were also determined. A simple antimicrobial test was also done by using 3 types of fungus namely Aspergillus brasiliensis ATCC 16404, Gram positive Escherichia coli ATCC 25922 and Salmonella choleraesuis (subtype enterica). The morphological observation showed the difference between EFB paper and paper made of blended pulps. Citronella was found to affect the physical, optical and mechanical paper properties as expected. To be specific, the paper opacity was improved while the mechanical properties especially tensile and tear indices were dropped tremendously. However, the presence of Citronella in the paper sheet seems to have good potential as an antimicrobial agent as shown by the antimicrobial test.

1. Introduction
Oil palm has tremendous characteristic that it is commonly recognized as “tree of life”. This is due to all part such as fruits, trunks and leaves can be effectively utilized for living. Oil palm empty fruit banches (EFB) are residues obtained from oil palm plantation area which increasing around 25 million tons by years [1]. The expansion of palm oil industry is expected to reach 80 million tons by 2020. Hence, the abundance of oil palm residues would be increasing especially EFB which is considered as renewable and readily available lignocellulosic materials [2]. EFB is characterized as lignocellulosic material which consists of long fibers in core and rich of silica [3]. It has been applied in many research that producing pulp and paper with high potential of being renewable natural resources in related industry. These fibers which can be transformed into commercial serviettes [4] and corrugated medium board [5] are proper alternative to reduce the dependency on the imported recycled fibers.

Cymbopogon nardus is a scientific name for citronella leaves which carries pleasant aroma and
positive psychological effects on human beings [6]. The leaves are inedible which leads to huge abundance of crops. It is originated in tropical Asia countries such as Malaysia, Thailand, Indonesia, Singapore and India which are economically and physio-geographically rich with biodiversity and seasonal weather [7]. The extracted oil of *Cymbopogon nardus* can be used in some external applications such as, insect repellents [8], pesticide [9], fungicide and bactericide. Moreover, it has huge contribution in economic sector and medical industry which can be applied to synthesize a number of useful aromatic compounds and vitamin A [10]. Besides, *Cymbopogon nardus* is also utilized by soaps, detergent, cosmetics and food flavouring agent producers. In 2015, *Cymbopogon nardus* pulp was successfully prepared by using kraft and soda pulping methods [11]. Therefore, in this study *Cymbopogon nardus* was used by blending it with EFB pulps in order to produce potential antimicrobial paper for food packaging purpose.

Pulping is a crucial and important process in pulp and papermaking technology. It can be carried out by using 3 categories of pulping like mechanical, chemical, and thermal pulping or mixed of these 3 pulping. Acceptable pulps for papermaking are characterized by parameters such as bulky thickness, opacity, and light scattering coefficient [12]. Generally, pulps that are produced through mechanical pulping may obtain good paper opacity property [13]. On the other hand, paper which produced from chemical pulping normally has higher mechanical paper properties [14]. In most cases, pulp was produced mechanically due to lower cost and higher pulp yield factors [4]. Although the mechanical pulp yield was higher compared to chemical pulp, the amount of single fibers would be lesser because an amount of fibers still tend to be in bundle forms.

Mechanical pulp may form high surface roughness and blending paper will have low optical properties paper which can be improved by increasing the number of refining cycles and applying pre-treatment process [16-17]. Zhai et al. (2014) stated that pretreatment which was carried out prior to mechanical pulping process increased porosity, swelling, fibrillation, carbohydrate recovery and reduced lignin content in the papers [18]. In 2016, Johansson et. al, indicated that applying mechanical pulping would be economical and inexpensive method in pulp for paper [19]. Mechanical pulping offers good opacity and high yield of pulps compared to chemical pulping which lead to high profit value obtained by producer. In addition, mechanical pulping requires less chemical consumption compared to chemical pulping. Therefore, this pulping process is known as more eco-friendly production that contributes in forming biodegradable paper and reducing global pollution scale.

Many experiments showed that the lemongrass (*Cymbopogon citratus*) essential oil inhibited the mycelial growth of some pathogens: *Collectotrichum gloeosporioides* [20], *Fusarium verticillioides*, *Fusarium proliferatum*, *Fusarium graminearum* [21] and *Fusarium moniliforme* [22]. In 2011, Istianto and Emilda found out that essential oil of citronella grass (*Cymbopogon nardus*) could completely inhibit the mycelial growth of *Fusarium oxysporum* [23], which supported by Samerjai et al. [24]. Based on previous research, *Cymbopogon* family such as *Cymbopogon nardus* has well anti-microorganisms property and can be used in papermaking system. Therefore, this study was carried out in order to utilize the EFB pulp with Citronella pulp to provide a special paper with antimicrobial properties which is great beneficial for packaging paper industries.

2. Materials and Methods

2.1. Raw materials

Oil palm empty fruit bunch fiber was received from a local mill at Pahang in bale form while citronella leaves was harvested from Faculty of Agriculture, Universiti Putra Malaysia. The citronella leaves arrived in fresh forms stacked together in bundles which later dried until moisture content reached 5 % prior to pulp and paper making. Sodium hydroxide was purchased from R&M Chemicals.

2.2. Preparation, Testing and Blending of Pulps

Oil palm empty fruit bunch fibers were cut into 3-4 cm length prior to pre-treatment process. The pre-treatment was carried out by immersing the fibers in sodium hydroxide solution at 1 %, 2 %, 3 %, 4 % and 5 % of concentration. The process was held for 24 hours. The soaked fibers were then washed using tap water until the soapy condition getting lesser for about 30 mins. These washed fibers were then
refined using Refiner Mechanical Pulper (RMP) machine. The refined pulps were screened by using Somerville Screener.

On the other hand, Citronella leaves were cut into 2 to 3 cm prior to refining process using RMP machine. These refined pulps were beaten at a range of beating levels: 0, 1000, 3000 and 3500 revolutions using PFI Beater Mill. These beaten pulps were then tested their pulp freeness using Canadian Standard Freeness apparatus.

The pulp blending was carried out by applying a ratio of 20:80, Citronella on EFB pulps. The papermaking was carried out by using Handsheet Machine based on TAPPI Test Methods,1992 [25].

2.3. Characterization of paper
The characterization of paper was divided into three types namely as physical, optical and mechanical paper testing. The physical tests selected in this study were grammage (TAPPI Standard T410 om-08) [26] and bulk thickness (TAPPI Standard T411 om-97) [27] while optical paper properties consisted of paper opacity (TAPPI Standard T425 om-01) [28] and brightness (TAPPI Standard T 452 om-02) [29]. The mechanical properties were tensile (TAPPI Standard T404 cm-02) [30] and tear strength test (TAPPI Standard T414 om-98) [31].

2.4. Morphological observation
Morphological observation was carried out under Scanning Electron Microscope (SEM) JSM-7900 F, Schottky Field Emission. The samples were coated with gold by using Sputter Coater devise in order to avoid the sample surfaces from creating charging effects during scanning. The observation was done to observe the surface views of each fibers and papers. The magnification levels which were utilized during scanning were 50 to 1000 magnifications.

2.5. Antimicrobial test
Pathogenic microorganisms were obtained from the Institut Biosains, Universiti Putra Malaysia. The determination of antimicrobial properties of blended paper was carried out using two Gram-negative strains, Escherichia coli ATCC 25922 and Salmonella choleraesuis, and one filamentous fungi strain Aspergillus brasiliensis ATCC 16404. According to the Manual of Antimicrobial Susceptibility testing, the culture used for the antimicrobial test Gram-negative bacteria was Mueller Hinton broth (MHB), and Mueller Hinton Agar (MHA) fungus strains. Meanwhile, an inhibition growth assay using the technique of agar diffusion was applied to the plates containing MHA medium which were inoculated with Gram-negative strains, Escherichia coli ATCC 25922, Salmonella choleraesuis and one filamentous fungi strain Aspergillus brasiliensis ATCC 16404. The agar was drilled to form holes of about 0.1 cm, and then 10 g of raw fibers of EFB and citronella leaves was added into each hole separately. The incubation time for both Escherichia coli ATCC 25922, and Salmonella choleraesuis was 5 days. On the other hand, Aspergillus brasiliensis ATCC 16404 took 7 days. The antimicrobial activity was tested by the formation of a halo showing the inhibition zone.

3. Results and Discussions
3.1. Effect of sodium hydroxide concentration on EFB pulp properties
The pulp yield and freeness results were shown as in Table 1. Pretreating the EFB fibers with 5 % sodium hydroxide could produce the highest percentage of pulp yield. An increment of 15.87 % pulp yield was obtained compared to non-treated EFB fibers. The results approved that sodium hydroxide would increase the lignocellulose biomass pulp yield [32]. Besides that, pulp freeness results a decline pattern. Higher concentration of sodium hydroxide during pre-treatment process had successfully dropped the reading of pulp freeness about 60.53 %. This huge decrement showed that sodium hydroxide provides great effect in helping the defibrillation of fibers during refining process [34]. The fibers absorbed acceptable amount of water by draining 300-700 ml of water that is economical for papermaking process.
3.2. Effect of beating level on citronella pulp property
The pulp freeness of Citronella after a series of beating level was presented in Table 2. The results proved that the Citronella leaves were more firm compared to EFB fibers. The pulp freeness of the Citronella showed 510 ml CSF reading after undergone 3,500 revolutions of beating level. A decrement of 37.42 % in terms of pulp freeness was achieved compared to the earliest sample without any beating. In every 500 revolutions of beating level, an amount of 50 ml pulp freeness value decreased. Kamoga (2015) reported that Citronella mechanical fibers which have been beaten until 1000 PFI revolutions recorded pulp freeness as high as 740 ml [36]. This showed that the fibers have less tendency to absorb water which may be related to less fibrillation occurred on the fiber surfaces. In other words, the citronella fibers were quite rigid and less flexible. Beating the pulp fibers using a laboratory blender can improve the structural or physical properties of paper [37].

Table 2. Pulp freeness of Citronella without and with a series of beating level.

| Beating level (revolutions) | 0    | 1000 | 2000 | 3000 | 3500 |
|----------------------------|------|------|------|------|------|
| Canadian Standard Freeness, ml | 815  | 740  | 630  | 520  | 510  |

3.3. Effect of blending on the physical, optical and mechanical paper properties
Table 3 exhibited the results for physical, optical and mechanical paper properties which were made from EFB and blended pulps. The results of physical paper properties were divided into grammage, apparent density and bulk thickness. There was a decrement of 9.95 % in grammage which can be related to the less incorporation of citronella which might having fewer bonding with EFB pulps. As stated in earlier explanation, the citronella pulps were quite rigid and firm which difficult the bonding process. Hence in 1 meter square of paper, the weight of fibers in blended paper was lighter than in EFB paper due to pretreatment process that was applied onto EFB pulps [38] and improved its mechanical properties. In conjunction with grammage results, paper apparent density was found to have moderate difference between EFB and blended paper. There was slightly different of paper thickness between paper made from EFB and blended pulps. The thickness for both papers was 412.3 μm and 912.4 μm respectively. The citronella might be having coarser size of pulps which need wider areas to attach among the pulps due to free treatment onto pulps unlike the alkaline pretreated pulp [39].

The opacity of paper made from blended pulps was found out to be better than EFB paper which may be due to the presence of citronella pulps which filled the voids between fiber to fiber [40]. However, the brightness decreased 3.21% after adding the citronella. Different materials in terms of pulps will provide significant effect to the formation of paper included paper brightness.

The tensile strength of blended paper reduced tremendously while tear strength dropped 2.7 units. This decrement was expected due to the presence of citronella leaves which may have less quality bonding among each other. As stated in Table 2, the pulp freeness was considered as high that indicates the rigidity of fibers in absorbing water. Hence, this will affect the chances in bonding among the fibers and also with EFB fibers. Mechanical strength is affected by amount of fibers on one square meter paper [41]. As explained earlier, the grammage of blended paper is lesser than EFB paper. Therefore, it is proved that blended paper will obtain lower value of tear strength compared to EFB paper alone as shown in Table 3.
Table 3. Physical, optical and mechanical properties of EFB and blended paper.

| Property                  | EFB paper | Blending paper |
|---------------------------|-----------|----------------|
| **Physical properties**   |           |                |
| Grammage (g/m²)           | 36.38     | 32.76          |
| Apparent density (g/cm³)  | 0.243     | 0.223          |
| Thickness (μm)            | 412.3     | 912.4          |
| **Optical properties**    |           |                |
| Opacity (%)               | 44.60     | 50.24          |
| Brightness (%)            | 36.09     | 32.88          |
| **Mechanical properties** |           |                |
| Tensile index (KNm/g)     | 28.58     | -              |
| Tear index (mN.m²/g)      | 10.95     | 8.25           |

3.4. **Effect of blending on paper morphological property**

The cross cut views that blended paper was thicker than EFB paper as shown in Figure 1(a) and (b). As shown in Table 3, the thickness of blended paper was 2.21 times than the EFB paper itself. Figure 1(c) and (d) presented that blended paper has less uniform focally on fiber shapes and sizes than EFB paper. This is because of the presence of citronella pulps in the paper systems.

![Figure 1](image1.png)

**Figure 1.** SEM observation of (a) cross cut of EFB paper at 100X magnification (b) cross cut of blended paper at 50X magnification (c) surface of EFB paper at 100X magnification, and (d) surface of blended paper at 100X magnification.

3.5. **Effect of pulp blending on paper antimicrobial property**
EFB fiber and EFB paper were investigated having low antimicrobial defense against *Aspergillus brasiliensis* ATCC 16404 but better resistant onto *Escherichia coli* ATCC 25922 and *Salmonella choleraesuis*. However, blended paper has low antimicrobial defense against *Escherichia coli* ATCC 25922 and *Salmonella choleraesuis* but vice versa onto *Aspergillus brasiliensis* ATCC 16404. Meanwhile, citronella fibers from raw materials have low stage of antimicrobial defences onto *Aspergillus brasiliensis* ATCC 16404, *Escherichia coli* ATCC 25922 and *Salmonella choleraesuis* but changed after blended with EFB pulp. The efficacy of the antimicrobial agent will depend on water activity of the sample [45]. These results are very important because they showed that blended paper had high potential acting as antimicrobial paper as long as improvement in ratio portion or particle size of citronella is revised. The citronella fiber is showing antimicrobial agent property [42; 43]. In 2015, Nassar applied *Escherichia coli* ATCC 25922 on blended paper between lignocellulosic materials with recycled old newspaper having similar results [44]. Therefore, the blended paper needs to be improvised later on regarding on its ratio portion of blending and technique of applying the anti-microbial agent onto the paper.

| Type of sample  | Fungus                              | Gram negative strains                           |
|-----------------|-------------------------------------|-------------------------------------------------|
|                 | *Aspergillus brasiliensis* ATCC 16404 | *Escherichia coli* ATCC 25922 | *Salmonella choleraesuis* |
| EFB paper       | L                                   | M                                               | M |
| Blended paper   | M                                   | L                                               | L |
| EFB fiber       | L                                   | M                                               | M |
| Citronella fiber| L                                   | L                                               | L |

L: low anti-microbial activity
M: medium of anti-microbial activity

4. Conclusion

Preliminary results showed that paper produced from citronella and EFB has high chances to be developed as antimicrobial paper provided some adjustments to the formulation can be revised and improvised. The pretreatment of EFB fiber gives impact onto pulp yield and permeability of water onto pulp produced as well as beating pulps by using PFI Mills. Blended paper of efb and citronella shows effectiveness against fungal attack and can be revised to improve the best formula for blending paper for waste material to make antimicrobial paper which will be useful in food packaging industry.

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