Characteristics of Bioplastic Made from Cassava Starch Filled with Fibers from Oil Palm Trunk at Various Amount

FA Syamani¹, Nurjayanti², DJ Pramasari³, WB Kusumaningrum⁴, SS Kusumah⁵, N Masruchin⁶, R Ermawati⁷, G Supeni⁸, AA Cahyaningtyas³

¹Research Center for Biomaterial, Indonesia Institute of Sciences (LIPI)
Jalan Raya Bogor Km. 46, Cibinong, Jawa Barat, Indonesia 16911
²College of Industrial Management (Politeknik STMI) Jakarta
Jl. Letjen Suprapto No. 28 Cempaka Putih, Jakarta, Indonesia 10510
³Research and Development Agency of Industry. Ministry of Industry
Jl. Gatot Subroto Kav. 52-53 Jakarta Selatan, Daerah Khusus Ibukota Jakarta, Indonesia 12950

Corresponding e-mail: firda.syamani@biomaterial.lipi.go.id

Abstract. Petroleum-based plastic causes a threat to the environment at the end of its use because it cannot decompose naturally. Therefore it is necessary to develop bioplastic for food packaging but has the same function as conventional plastic. Starch based bioplastic, generally made from cassava starch. The disadvantage of this bioplastic is its low mechanical strength and damage if exposed to water. One way to increase the mechanical strength of bioplastic is to add fiber. Oil palm trunks are the source of natural fiber that is available in considerable amounts. The purpose of this study was to find out the characteristics of bioplastic from cassava starch with the addition of oil palm trunk fiber, in various amount. Bioplastics are made by heating a solution of cassava starch (tapioca: distilled water = 1:20) at 70 °C for 1 hour while stirring. After starch solution was gelatinized, glycerol (0.3%) and citric acid (1%) was added, and stirring was continued for 15 minutes. Oil palm fiber from oil palm trunk is added to the solution of cassava starch as much as 0%, 1%, 3% and 5% of the weight of cassava starch. The bioplastic solution is then poured into a (20x20) cm acrylic sheet with a thickness of ± 5mm. Furthermore, it is cooled at room temperature for 3 days so that the bioplastic sheet is ready for mechanical strength testing based on ASTM D 882-75b Tensile Properties of Thin Plastic Sheeting, and for thermal properties testing with Thermo Gravimetry Analyzer. The addition oil palm trunk (OPT) cellulose fibers into bioplastic made from modified cassava starch, produce bioplastic with higher tensile strength compare to modified cassava starch bioplastic with addition of OPT pulp fibers. The decomposition temperature of bioplastic made from modified cassava starch with 1% OPT cellulose was higher than of bioplastic with 1% OPT pulp, indicates that bioplastic with 1% OPT cellulose fibers have better thermal stability compare to bioplastic with 1% OPT pulp fibers

1. Introduction

Nowadays, plastics are used almost in every aspect of life. In 2017, the global production of plastics reached 348 million metric tons, with 64 million metric tons produced in Europe alone [1]. However, conventional plastics such as polyethylene, polypropylene, polystyrene, poly(vinyl chloride) and poly(ethylene terephthalate), are made from petroleum which is a non-renewable resource. Moreover,
conventional plastics are non biodegradable and at the end of their used, increasing waste accumulation in the environment then become a threat to the planet. The effort to overcome plastics waste problem was to add some active ingredients that allow plastic to be degraded after usage with the help of sunlight. However, the plastics are only break into smaller particles and becomes a new problem with the emergence of microplastic in the waters.

But the use of plastic, especially in the field of food packaging is inevitable, because conventional plastics packaging is low cost and able to accomplish the function of maintaining the quality of packaged food and facilitate transportation. In addition, plastics in the packaging industry has a large portion due to easy processability, strength and stiffness, barrier to oxygen and moisture, resistance to food component attack and flexibility[2].

The non-renewability and non-biodegradability characteristics of petroleum-based plastics were tried to be solved by developing plastic from bio-based material. Starch is one of biopolymer that can be processed to produce bioplastic because it becomes thermoplastics when properly plasticized with water or other plasticizers [3]. At the beginning of bioplastics development, Griffin et al. used starch granular as a filler in polyolefin (petroleum-based plastics) to be attacked and degraded by microorganism [4]. Starch was blended with low density polyethylene (LDPE) with starch content ranging from 2.5 to 50% were prepared by two methods, namely, solution blending followed by thermopress and extrusion methods. The solution cast LDPE/starch films tensile strength and elongation at break had decreased gradually from 10.78 to 3.92 MPa and 52 to 8 MPa, respectively, with the increase in starch concentration in LDPE from 0 to 50%. However, water vapor transmission rate (WVTR) values increased from 14.9 g/m²/d in plain LDPE film to 75.4 g/m²/d at 90% RH gradient and 38°C in LDPE/starch films with 50% starch content. This result show that LDPE/starch films can be used for packaging of fresh produce to control their moisture evaporation and enhance their shelf life [5].

Addition of nano-filler to starch based bioplastic was one way to induce higher mechanical strength and improve barrier against gases and water vapor. Cellulose nano-filler was considered as environmental friendly and renewable nano-filler [6]. Cellulose microfibers have a diameter of 20–200 Å, while the length can reach several dozen microns. These characteristics are responsible for the interesting mechanical properties of the cellulose microfibril [7]. One of cellulose source is oil palm trunk. Indonesia has the largest oil palm plantation. According to Directorate General of Estate Crops, Indonesia oil palm plantation in 2017 have reached 12,307,677 ha, with production of 35,359,384 tons [8]. Oil palm plantations need to be rejuvenated (cut down and replanted) when oil palm productivity decreased at around the age of 25 years. Based on the Directorate General of Plantations, the area of oil palm plantations is growing at a rate of around 10.31% per year. Oil palm tree, which was first planted on a large scale in Indonesia in 1978, has ended its productive period. According to Nasution [9], there is about 185,000 ha oil palm plantation that should be rejuvenated every year. The average number of oil palm trees is 128 trees per ha, then the dry weight of oil palm trunk is 394.11 kg per tree, so there is about 50.45 tons oil palm trunk per ha [10]. As a result there would be 9,333,250 ton oil palm trunk available every year as potential cellulose source.

It is necessary to develop bioplastic for food packaging with similar characteristics and function as conventional plastic. Starch based bioplastic, generally made from cassava starch. The limitation of starch utilization for bioplastic is its melting temperature and degradation temperature [11]. One way to increase the mechanical strength of bioplastic is to add filler such as pulp or cellulose fibers of oil palm trunk. The purpose of this study was to investigate the characteristics of bioplastic from cassava starch with the addition of oil palm trunk fiber at various amount.

2. Materials and Methods

2.1. Materials
The materials used in this study were modified cassava starch, sodium acetate technical grade obtained from PT Trijaya, acetic acid glacial obtained from Merck and distilled water. Oil palm tree as source of
fiber was cut down from oil palm plantation in Jasinga, Bogor, West Java. The oil palm trunk was taken from 25 years old palm oil tree.

2.2. Modification of Cassava Starch
Modification of cassava starch was carried out using acetate solution (CH_3COOH + CH_3COONa) at pH 7. Preparation of acetate solution was carried out by dissolving as much as 44.52 g sodium acetate in 50 ml of distilled water. After sodium acetate dissolved, acetic acid was added into the solution, gradually until it reaches pH 7. Distilled water was added into solution until solution volume reached 1 l. Modification of cassava starch was carried out by dissolving 100 g cassava starch into 200 ml acetate buffer solution in beaker glass, then stirred and heated on a hot plate stirrer at a temperature of 40 °C to thicken, after which it is dried at temperature room. After drying, the modified starch was mashed and sieved 80 mesh.

2.3. Oil Palm Cellulose Fiber Extraction
Oil palm trunk was skinned off, cut into 4 cm x 6 cm x 2 m beam, then processed in drum chipper and ring flaker to obtain oil palm rough powder. Oil palm starch was extracted and set aside oil palm fiber as source of cellulose fiber. Oil palm fiber was grounded and sieved to obtain oil palm fine powder that passed through 40 mesh sieve and retained on 60 mesh sieve. The oil palm fine powder was dried in a 60°C oven for 24 hours, until the moisture content was below 5%.

Oil palm trunk cellulose extraction was carried out according to Chieng method with modification in bleaching method [12]. Oil palm trunk fine powder was weighed as much as 20 grams then put into erlenmeyer, then reacted with 200 ml NaOH 4% solution at 80°C for 3 hours. At the end of reaction, as much as 300 ml distilled water was added and leave it overnight. The precipitate was filtered with a filter cloth and washed with distilled water. The alkali extraction is performed to remove alkali-soluble components, in essence, lignin and hemicelluloses that dissolve in the solution and resulted oil palm trunk (OPT) pulp fiber.

The bleaching process was conducted by adding 400 ml distilled water, 10.7 ml sodium chlorite (25% solution) and 0.5 ml acetic acid (100% glacial) into erlenmeyer contain OPT pulp fiber, and heated in water bath at 80°C for 1 hour. After 1 hour, 10.7 ml sodium chlorite and 0.5 ml acetic acid were added again. The total addition of sodium chlorite and acetic acid was 3 times. At the end of bleaching process, the mixture was strained and washed with distilled water using a filter cloth until the filtrate was neutral. The resulting product is oil palm trunk (OPT) cellulose fiber.

2.4. Production of Bioplastic from Modified Cassava Starch Filled with Oil Palm Trunk Pulp Fibers and Cellulose Fibers
Modified cassava starch was dissolved in distilled water with a ratio of 1:20. As much as 2.5 grams of starch was dissolved with 50 ml of distilled water into a beaker glass. The solution was heated in a water bath at 70°C for 1 hour. The solution was stirred at a constant speed of a mechanical stirrer 440 rpm, until it is shaped like a clear gel. After starch solution was gelatinized, glycerol plasticizer 0.3% and citric acid 1% of the dry weight of starch were added into the mixture. Then OPT pulp fibers or OPT cellulose fibers was added at 0%, 1%, 3% and 5% of the dry weight of starch. The mixture was stirred again until homogeneous for ± 15 minutes with the same speed and temperature.

The mixture solution was then poured into a mold (flexi glass) with dimension of 20x20 cm and a thickness of ± 5mm. Spread the mixture solution evenly on the surface of flexy glass. The solution was then cooled to room temperature for ± 3 days, until forms film sheets (bioplastic). The bioplastic was removed from the mold using tweezers and the film is ready to be characterized.

2.5. Analysis of Bioplastic Characteristic

2.5.1. Evaluation of bioplastic mechanical properties. Tensile strength test was carried out accordance with ASTM-882-75b “Tensile Properties of Thin Plastics Sheeting”. Samples were cut in sizes of 1.5x20
cm. Tensile strength was measured with Universal Testing Machine (UTM) Shimadzu Autograph 50kN, speed testing was 2 mm/min. The test results will be read after sampling. This test was carried out 4 times.

2.5.2. Evaluations of functional groups in bioplastic. The bioplastic film sheet was cut into small pieces. Then the sample was placed in a sample container to be tested. Functional group analysis was performed using the Perkin Elmer FT-IR (Fourier Transferred Infrared), Spectrum Two, performed in the range of 400-4000 cm\(^{-1}\), resolution 4 cm\(^{-1}\) and scanned 16 times.

2.5.3. Evaluations of bioplastic thermal properties. The thermal properties test was performed using the Thermogravimetric Analysis (TGA) 4000 Perkin Elmer. A 8 mg bioplastic was placed on a ceramic crucible. The test was carried out by heating the sample at a temperature of 25-500\(^{\circ}\)C with a heating speed of 10\(^{\circ}\)/min.

3. Results and Discussion

3.1. Oil palm trunk cellulose fibers
Cellulose extraction from oil palm trunk was conducted by two steps, namely alkali treatment and bleaching process. After alkaline treatment and bleaching process, 20 grams oil palm trunk fine powder, can be converted into 9.059 grams cellulose. The OPT cellulose fibers produced from this study was presented in Figure 1. Alkali treatment was used to remove lignin and hemicellulose contained in OPT fine powder. The bleaching process caused the change of fiber color from brown to white.

Bleaching is applied after alkaline treatment to remove all elements containing chromophore groups and phenolic compounds as lignin and hemicellulose [13]. Bleaching agents convert coloured impurities (containing chromophore groups) into colourless particles [13]. This study used sodium chlorite and acetic acid as bleaching agent therefore bleaching process in this study could be classified as acid-chlorite delignification. Acid-chlorite delignification is selective in the removal of lignin with only trace solubilization of glucan and xylan [14]. Phenolic compounds comprise one (phenolic acids) or more (polyphenols) aromatic rings with attached hydroxyl groups in their structures [15]. During bleaching process, sodium chlorite oxidation cleaved the phenolic ring [16]. Therefore after alkaline and bleaching process, the OPT pulp fibers will be converted to OPT cellulose fibers which its color is white.

![Figure 1](image1.png)

Figure 1. (a) Oil palm trunk powder, (b) Cellulose fibers isolated from oil palm trunk powder.

3.2. Bioplastic mechanical properties
The effect of the addition of oil palm trunk (OPT) pulp or cellulose in modified cassava starch on bioplastic tensile strength is presented in Figures 2 as follows.
OPT cellulose produced from oil palm trunk fine powder was applied as a filler in modified cassava starch films with the purpose of improving bioplastic mechanical properties. The tensile strength values show that the addition of 1% OPT cellulose will increase the tensile strength of bioplastics, up to 184.24%. However the addition of 3% and 5% OPT cellulose fibers can not increase the tensile strength value more than the addition of 1% OPT. Overall, the tensile strength of bioplastic with the addition of OPT cellulose was higher than of bioplastic with OPT pulp. This was because the size of the OPT cellulose fiber is quite smaller than OPT pulp (Figure 3). The average diameter of OPT pulp fibers in this study was 20.924 µm and the average diameter of OPT cellulose fibers was 11.974 µm. The smaller the fibers diameter will increase the total fibers surface area and allows OPT cellulose fiber to enlarge contact on the film matrix of modified cassava starch to produce higher tensile strength. On the other hand, the addition of OPT pulp fibers reduce bioplastic tensile strength. Pulp fibers still contain impurities that could become barrier of stress transfer during force loading on tensile strength testing.

Bioplastic film with addition of 1% OPT pulp fibers has a slightly higher tensile strength value than bioplastic film without any filler, but bioplastic with addition of 3% and 5% OPT pulp fibers show lower tensile strength when compared to bioplastic without any filler. Bioplastic tensile strength analysis show that bioplastic with addition 1% OPT cellulose was the best tensile strength value (22.37 N / mm²). While the lowest bioplastic tensile strength value was 3.05 N / mm².
**Table 1.** Mechanical properties of LDPE and modified cassava/OPT pulp or cellulose bioplastics

| Parameter          | LDPE   | Bioplastic with OPT cellulose fibers | Bioplastic with OPT pulp fibers |
|--------------------|--------|--------------------------------------|---------------------------------|
| Tensile strength (N/mm²) | 8-12   | 10.47-22.37                         | 3.05-11.30                     |
| Elongation (%)     | 600-650| 1.43-2.09                           | 0.70-1.88                      |

The tensile strength of bioplastic with addition of OPT cellulose were 10.47-22.37 N / mm², this shows that bioplastics are able to compete with the tensile strength of LDPE (low density poly ethylene) synthetic polymers which is between 8-12 N / mm² so that it is still suitable for use as packaging material. Table 1 show mechanical properties of LDPE and this result summary.

**Figure 4.** Elongation of bioplastic with varied of oil palm trunk (OPT) pulp or cellulose content

Percent elongation is the elasticity of a material when pulled up to break. Glycerol is used as a plasticizer so that the plastic is more elastic. Based on the data obtained, the addition of OPT pulp or cellulose had no significant effect on the bioplastics elongation. Elongation percentage obtained were ranging from 0.70 to 2.09% (Figure 4). This result shows that up to 3% of fiber addition, has not reached a level that can interfere the mobility of the polymer chain [17]. The elongation of bioplastics in this study was smaller than of LDPE synthetic polymer elongation (600-650%). The comparison of bioplastic elongation in this research with elongation of synthetic polymers can be seen in Table I.

### 3.3. Functional Groups in Bioplastic

The FT-IR spectrum was measured using the Perkin Elmer Spectrum Two. The functional group analysis was conducted on bioplastic with the addition of 1% OPT cellulose, due to its highest tensile strength properties. Functional group analysis is performed to determine the presence of functional groups in bioplastics from modified cassava starch as a matrix with the addition of OPT cellulose. The FT-IR spectrum was in the wave range of 400-4000 cm⁻¹. The results of the functional group analysis can be seen in **Figure 5**.
Figure 5. FTIR spectra of bioplastic with no filler (A), bioplastic with oil palm trunk (OPT) pulp 1% (B), and bioplastic with OPT cellulose 1% (C).

The absorption area at a wavelength of 3200-3600 cm\(^{-1}\) indicates the O-H functional groups of hydrogen bonds. Absorption area at wavelengths of 2850-2970 cm\(^{-1}\) indicates the presence of alkane C-H functional groups [18]. The analysis shows that the spectrum of modified cassava bioplastic with no filler and bioplastics with OPT cellulose have OH stretching functional groups at 3291.39 cm\(^{-1}\) and 3292.44 cm\(^{-1}\). While CH stretching appears at wave numbers 2925.60 cm\(^{-1}\) and 2924.39 cm\(^{-1}\). Spectra peak indicates OH stretching and CH stretching functional groups at wave numbers 3289.40 cm\(^{-1}\) and 2926.02 cm\(^{-1}\) were more sharply shown in bioplastics with OPT cellulose.

Table 2. Functional groups of bioplastic from modified cassava starch with no filler, bioplastic from modified cassava starch with 1% OPT pulp or 1% OPT cellulose

| Functional groups & bonding type | Wave number (cm\(^{-1}\)) |
|---------------------------------|--------------------------|
|                                 | Modified cassava starch  | OPT pulp 1% | OPT cellulose 1% |
| O-H Stretching                  | 3291.39                  | 3292.44     | 3289.40          |
| C-H Stretching                  | 2925.60                  | 2924.39     | 2926.02          |
| C=C Stretching                  | -                        | -           | 1642.64          |
| C=C Stretching                  | 1560.43                  | 1560.64     | -                |
| C-H Bending                     | 1408.58                  | 1409.79     | 1411.46          |
| C-H Bending                     | -                        | 1363.42     | 1363.42          |
| C-O Stretching                  | 1149.80                  | 1149.81     | 1149.95          |
| C-O Stretching                  | 1078.36                  | 1078.24     | 1078.26          |
| C-H Stretching                  | 993.07                   | 993.50      | 994.94           |

The spectra peaks of bioplastic with OPT cellulose were sharper than of bioplastic with no filler. The absorption area at wavelength 1500-1600 cm\(^{-1}\) which is assumed to be the functional group C=C aromatic ring [19]. The spectra peaks at wave numbers 1560.43 and 1560.64 cm\(^{-1}\) of the modified cassava starch bioplastic with OPT pulp, indicated C=C stretching functional groups, but this spectra did not appear again in bioplastic with OPT cellulose. These conditions can occur due to chemical treatment given [14].

The C-O ester functional group appears at wave number 1050-1300 cm\(^{-1}\) and wave number 675-995 cm\(^{-1}\) indicates the existence of the alkene functional group [18]. In bioplastic with OPT pulp and OPT cellulose, the spectra peaks were found at successive wave numbers 1149.80-1078.36 cm\(^{-1}\); 1078.24-1149.81 cm\(^{-1}\); and 1078.26-1149.95 cm\(^{-1}\), indicates C-O stretching functional groups, whereas spectra
peaks were appear at wave numbers 993.71 cm\(^{-1}\) and 993.50 cm\(^{-1}\), indicates the alkene C-H functional groups.

The FT-IR spectra of bioplastics with OPT pulp and bioplastics with OPT cellulose (Table 2), shows an absorption of new functional groups at 1365 cm\(^{-1}\) which was C-H bending. The FT-IR spectra of bioplastic with OPT cellulose, shows an absorption of new functional groups at 1642 cm\(^{-1}\), which was C=C stretching. However, sharper spectra peaks were presented in FTIR spectra of bioplastic with OPT cellulose.

3.4. Bioplastic thermal properties
Thermal degradation testing is carried out by Thermo Gravimetry Analysis (TGA) which aims to determine the change in mass of bioplastic on temperature rise. This test can also provide information on the results of thermal decomposition of bioplastics produced in this study. Curve changes in the mass of modified cassava bioplastic with the addition of 1% OPT pulp or 1 % OPT cellulose (Figure 6 and Figure 7) to the temperature rise.

![Image](image1.png)

**Figure 6.** Thermogram TGA bioplastic from modified cassava starch with no filler, 1% OPT pulp and 1% OPT Cellulose

![Image](image2.png)

**Figure 7.** Thermogram DTG of bioplastic from modified cassava starch with no filler, 1% OPT pulp and 1% OPT Cellulose
Based on the obtained data, the TGA curve of the three bioplastics to the onset decomposition temperature is not much different (Figure 6). The initial weight reduction at temperatures below 100°C is caused by evaporation of water [20]. Modified cassava bioplastic decomposition began to occur at 258.12°C, at the addition of 1% OPT pulp the decomposition temperature occurred at 255.59°C, whereas at 1% addition of OPT cellulose decomposition temperature occurred at 258.21°C. DTG curve (Figure 7) show that bioplastic with 1% OPT Pulp have lower decomposition peak temperature, also at temperature for 5% and 50% of decomposition than modified starch and bioplastic 1% of OPT Cellulose as seen at Table 3. Char residue which correspond for solid content after decomposition along determined temperature, tend to be the same as decomposition temperature trend. Overall, bioplastic 1% of OPT cellulose have better thermal stability than bioplastic 1% of OPT Pulp.

| Table 3. TGA data of modified starch with no filler, 1% OPT Pulp, and 1% OPT Cellulose |
|---------------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Sample                         | T onset (°C)    | T max (°C)      | T 5 (°C)        | T 50 (°C)       | Char residue at 450 °C |
| Modified starch                | 258.12          | 280.25          | 76.23           | 302.64          | 27.05            |
| Bioplastic 1% OPT Pulp         | 255.59          | 260.7           | 74.91           | 294.57          | 25.72            |
| Bioplastic 1% OPT Cellulose    | 258.21          | 282.3           | 83.49           | 304.08          | 26.62            |

4. Conclusion
The addition oil palm trunk (OPT) cellulose fibers into bioplastic made from modified cassava starch, produce bioplastic with higher tensile strength compare to modified cassava starch bioplastic without addition any fibers. On the other hand, addition of 1% OPT pulp fibers, could slightly increase bioplastic tensile strength. This is because the size of the OPT cellulose fiber diameter is slightly smaller than OPT pulp diameter so that it can produce higher bioplastic tensile strength. The decomposition temperature of bioplastic made from modified cassava starch with 1% OPT cellulose was higher than of bioplastic with 1% OPT pulp, indicates that bioplastic with 1% OPT cellulose more thermally stable.

5. Acknowledgment
Authors would like to thank Ministry of Research Technology and Higher Education of the Republic of Indonesia for research fund though INSINAS 2019, functional food flagship LIPI.

6. References
[1] Garside M 2019 Global plastics production from 1950 to 2017 Available at: https://www.statista.com/statistics/282732/global-production-of-plastics-since-1950/ Accessed on 24 July 2019
[2] Silvestre C, Duraccio D, Cimmino S 2011. Progress in Polymer Science 36 pp 1766–1782
[3] Wang XL, Yang KK, Wang YZ 2003. J Macromol Sci Pol 43(3) pp 385-409
[4] Griffin GJL 1974 Biodegradable filler in thermoplastics. Advanced in Chemistry Series 134 pp 159-170
[5] Raj B, Sankar KU, Siddaramalah 2004 . Advances in Polymer Technology 23(1) pp 32–45
[6] Gadhave RV, Das A, Mahanwar PA, Gadekar PT 2018 . Journal of Polymer Chemistry 8 pp 21-33
[7] Pérez-Pacheco E, Canto-Pinto JC, Moo-Huchin VM, Estrada-Mota IA, Estrada-León RJ and Chel-Guerrero L 2016. Composites from Renewable and Sustainable Materials, IntechOpen
[8] Hendaryati DD, Arianto Y, 2016 Tree crops estate statistics of Indonesia 2015-2017: Palm Oil, hhDirectorate General of Estate Crops, Ministry of Agriculture
[9] Nasution D 2018 Pemerintah Remajakan 9.109,29 Hektar Sawit Rakyat Sumatera Utara http://kur.ekon.go.id/pemerintah-remajakan-910929-hektar-sawit-rakyat-sumatera-utara
[10] Siswoko E, Mulyadi A, Thamrin, Bahruddin. Jurnal Ilmu Lingkungan 11(2) pp 154-163
[11] Abdullah ZW, Dong Y 2018. *J Mater Sci*. 53 (5):3455-3469
[12] Chieng BW, Lee SH, Ibrahim NA, Then YY, and Loo YY 2017 Isolation and characterization of cellulose nanocrystals from oil palm mesocarp fiber *Polymers* 9 355 doi:10.3390/polym9080355. www.mdpi.com/journal/polymers
[13] Choudhury AKR 2011 *Pretreatment and preparation of textile materials prior to dyeing in Handbook of Textile and Industrial Dyeing: Principles, Processes and Types of Dyes* Vol 1 in Woodhead Publishing Series in Textiles. Pages 64-149
[14] Ahlgren, P.A., Goring, D.A.I., 1971. *J. Chem*. 49, 1272–1275.
[15] Minatel IO, Borges CV, Ferreira MI, Gomez HAG, Chen CO, Lima GPP 2017 *Phenolic Compounds: Functional Properties, Impact of Processing and Bioavailability*, IntechOpen
[16] Collings GF, Yokoyama MT, Bergen WG 1978 *J Dairy Sci* 61:1156-1160
[17] Wicaksono R, Syamsu K, Yuaisih J, Nasir M 2013. *J Tek Ind Pert*. 23(1) 38-45
[18] Sutan SM, Maharani DM, Febriari F 2018. *Jurnal Keteknikan Pertanian Tropis dan Biosistem* 6(2) 157-171
[19] Zulferienni, Marniza, Sari EN 2014. *Jurnal Teknologi dan Industri Hasil Pertanian* 19(3) 257-273
[20] Jiugao Y, Ning W, Xiaofei M 2005 . *Starch/Stärke* 57 494–504