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The Exploration of Alpha Cellulose in Kapok Fruit as Raw Material for Rocket Propellant Production

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Abstract

Rocket propellant is an essential part of weapons systems whose raw materials are largely composed of alpha-cellulose. These chemical compounds can be obtained in a variety of plants and animals, including kapok plants. The objective of this study was to explore the alpha cellulose derived from kapok fruit with a purity level above 90% and meets the qualification as a rocket propellant. Extraction, digestion, and purification were performed to know the presence of alpha cellulose and its quantity. Type Expert MPDn of Philips X-ray diffractometer device according to Indonesian Industrial Standard (SII) was used I this study. Based on the result, it was known that the yield amount of 100 g sample extraction was 28,20g-48,57g with kappa number of 23,63-28,17. The purity level of alpha cellulose and100 g yield of samples obtained was 90.00%-96.89% with the yield of 26.19g-46.67g. The identification of alpha-cellulose in the fiber, center, and peel of kapok fruitmet the specifications as a raw material for propellant and met the standards to support the defence industry to produce nitrocellulose as the main material of rocket propellant.

Keywords: Alpha cellulose; Propellant; Extraction; Kapok plant

Introduction

Rocket propellants are one of the rocket part that serve as a rocket propulsion to the target. These rocket propellant are composed of several chemical compounds such as nitrocellulose, nitroglycerin, ethyl centralite ($C_{17}H_2ON_2O$) and potassium nitrate (KNO_3). Alpha cellulose is a chemical compound used to make nitrocellulose. This cellulose can be derived from various plants. Kapok is one of the plants with limited exploration of its utilization as a propellant raw material in Indonesia. Utilization of kapok fruit by alpha cellulose extraction for explosive raw material is very strategic and preferable than exporting raw materials [1-4].

The exploration of alpha cellulose from kapok fruit through isolation process, purification and identification was conducted in this research to obtain alpha cellulose which has the potential ability as propellant raw material. Thus, several important parameters were used in the isolation process to obtain alpha cellulose with specifications as a propellant material, especially the peel, center and fiber of kapok fruit [5]. The purpose of this research was to identify the potential alpha cellulose isolate as a propellant raw material whose benefit was to accelerate the expansion of kapok plantation so the income of kapok farmers and production of alpha cellulose for commercial purposes or military defense cam be increased [6].

Material and Method

Kapok tree (Ceiba pentandra gaertn)

Figure 1: Muktiharjo Kapok Tree.

Kapok (Ceiba pentandra Gaertn from the family Bombacaceae) or Randu is a multi function tree. Kapok fruit has a more strategic use value in the defence field, especially for rocket propellant materials by utilizing alpha cellulose contained in fiber, peel and center of kapok fruit. Furthermore, the process of alpha cellulose isolation which is a process to obtain alpha cellulose, implemented through several stages. Those stages are: extraction process, mechanical process, chemical process/digesting, semi chemistry process, and advanced oxidation process (purification). Extraction process of extractive substances is the separation of extractive substances from a raw material by using organic solvents such as ethanol and benzene with a ratio of 1: 2, this separation can be performed by a set of socket tools for samples in small quantities, while maceration with ethanol-benzene for ±24 hours was performed for large samples (Figure 1) [7-10].

Extractive substances have a low molecular weight it can easily dissolve in organic solvents compared with other chemical compounds. The mechanism of mechanical process is to decompose the existing fibers in wood and non-wood by mechanical action much grinding and refining. There are several type of this process: Stone Groung Wood Pulping (SGP), Refiner Mechanical Pulping (RMP), Thermo Mechanical Pulping (TMP), and Chemi Thermo Mechanical Pulping (CTMP), the resulting yield is lower than other mechanical processes and the physical features are better with the more and longer fiber fraction [11,12].

Cellulose

Cellulose $(C_6H_{10}O_5)_n$ is a long-chain polysaccharide carbohydrate polymer from beta-glucose. Several studies of cellulosic chemistry through elemental analysis showed that plant tissue contains a major component that includes 44.4% carbon, 6.2% hydrogen, and 49.3% oxygen. This is similar to the $C_6H_{10}O_5$ empirical formula and the molecular weight of 162. The molecular weight analysis of cellulose indicates a much larger formula weight than 162, cellulose is a high polymer (a molecule composed of many relatively simple repeating units connected by chemical bonds or molecular aggregates simple being united by the secondary forces of the association) (Figure 2) [13-15]

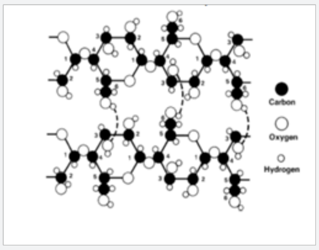

Figure2: Hydrogen Bond in Cellulose (Two-dimensional diagram of intermolecular bonds in part of cellulose).

By using a series of different molecular weights cellulose samples, their intrinsic viscosity was compared with the MW respective molecular weight of each sample as determined by the spread of light or ultra centrifuging. When logs of [μ] are described according to MW logs, then a straight line will be obtained. While the slope and line intercept help evaluate the constants and K which are made in simple equations as follows:

$[\mu] = K . Mwa$

Which μ = Intrinsic Viscosity

MW = respective molecular weight

K = konstanta

Thus, when these constants are known for certain solvent cellulose systems, then simple measurements of the solution viscosity in some concentrations can calculate the average molecular weight of the sample.

Alpha-cellulose is a long-chain cellulose, insoluble in 17.5% NaOH solution or a strong base solution with a DP (polymerization degree) of 600 - 1500. The alpha cellulose is used as a predictor and determinant of cellulose purity. Cellulose with a purity degree of $\alpha > 90\%$ is eligible for use as the main raw material for the propellants or explosives production. While the cellulose quality underneath is used as raw material in the paper industry and fabric industry (rayon fiber) [16-18]. The higher the alpha cellulose level, the better the quality of the ingredients (Figure 3).

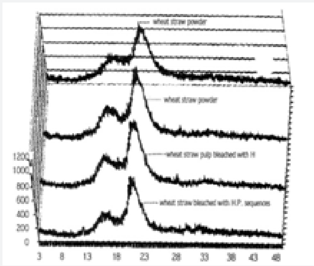

Figure 3: diffraction patterns of wheat straw α-cellulose.

The alpha cellulose can be expressed as planes expressed by peaks of different intensities in X-ray diagrams such as the soft cotton cellulose of the x-ray diffractometer treated with H_2SO_4 in Figure 2.4.

Propellant

In general, there are two types of Solid Rocket propellant which have been developed, namely double base and single base propellant with differences in both the constituent material, function, manufacture, and also the properties. The double base propellant consists of NC (Nitro Cellulose) and NG (Nitro Glyserine). It is produced by extruded or cast process and thus, it's called EDB (Extruded Double Base) or CDB (Cast Double Base). The double base propellant contains a small amount of other chemicals to enhance and modify the ability of the propellant [19]. The basic ingredients of the Double Base Propellant are NC and NG, Catalyst for combustion velocity, Stabilizer, Plasticiser and other additives. While the single base propellant is a type of propellant which is generally used in small caliber ammunition composed of nitrocellulose chemical compounds, K_2SO_4, KNO_3, dinitrotoluene and diphenyllamine [20-22].

Conceptual research frame-work

A research is a systematic process and based on the information obtained, a conceptual research framework (Flow Sheet) can be made as follows (Figure 4).

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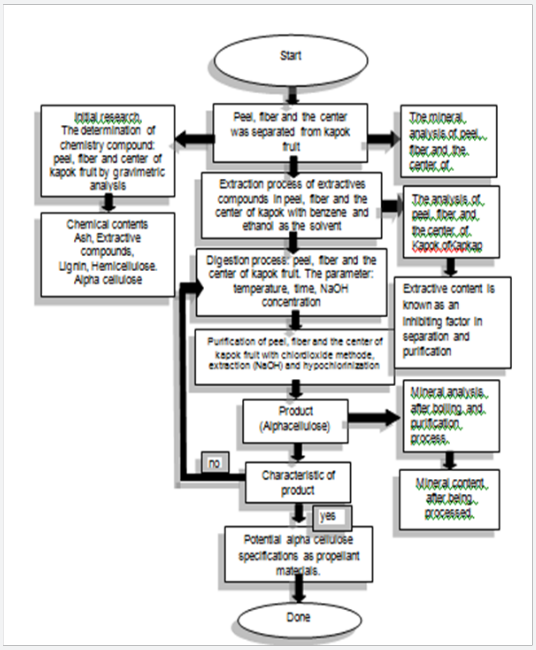

Figure 4: Research Framework.

Results and Research Analysis

Research design for soda process

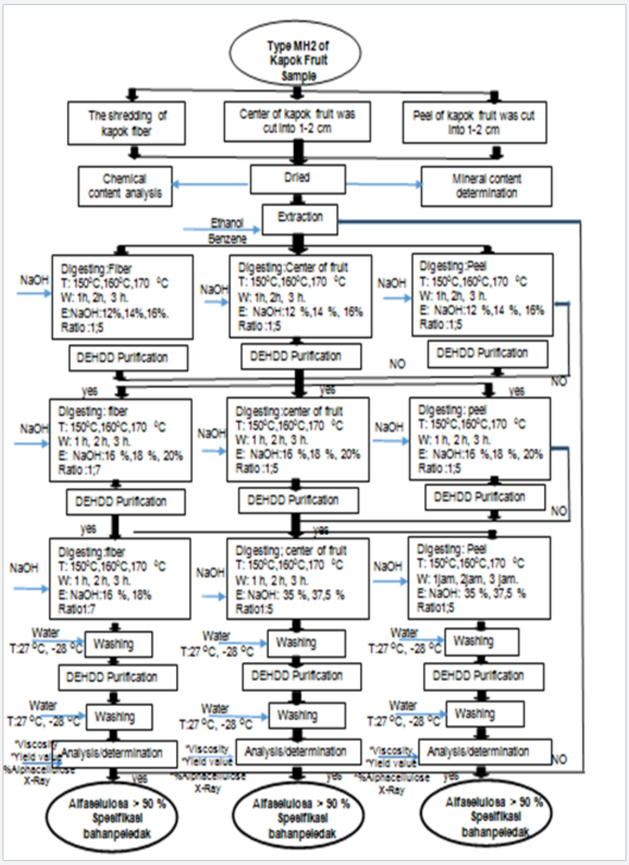

Figure 5: Research flow-chart.

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The experimental design performed in the extraction process was the design of the Box-Behnken experiment. This model was a design to discover and to obtain a large number of variables with less experiment. In this model, there were 3 free variables with 3 experimental design factors that can be implemented. Therefore, the number of Box-Behnken experiment that can be performed was 15 experiments while the Central Composite Design was 27 experiments (Figure 5) [23]. The research flow diagram was showed bellow:

Mineral determination result, kappa number and the yield of kapuk fruit sample

The first stage of mineral content determination in kapok samples which were fiber, liver and skin firstly prepared samples was drying, then the water content was subsequently determined and the samples were stored in a certain room so that the water content did not change. The result of mineral content analysis prior to extraction, cooking and purification process was as follows: Based on the mineral analysis result of each sample there were difference of mineral content. Sample with high mineral content were fiber kapok with 14.498% Calcium (Ca), 12.890% Silicate (Si), 27.608% Magnesium (Mg), while the center of kapok fruit had 12.467% calcium (Ca), 11.357% Silicate (Si), 29.251% Magnesium (Mg), and the peel of kapok fruit had 17.438% Calcium (Ca), 15.881% Silicate (Si), 25.708% Magnesium (Mg) [24-27].

The difference in mineral content especially silicate (Si) was highest in the peel of kapok. This silicate content will affect the level of rust equipment in digester and cellulose purity. Based on the result of mineral analysis after extraction, cooking and purification process, it was known that the mineral content was decreased with the final content described as follows: the mineral content of fiber kapuk were 10.988% Calcium (Ca), 10.660% Silicate (Si) and 25.308% Magnesium (Mg), while in center of kapok fruit were 10.184% Calcium (Ca), 9.597% Silicate (Si) and 21.528% Magnesium (Mg). Finally, there were 9.438% Calcium (Ca), 13.882% Silicate (Si), 23.627% Magnesium (Mg) in kapok fruit peel [28]. The extraction process of extractive substances was the separation of extractive substances contained in the sample prior to the cooking process and purification of the sample [29-32].

These substances were obtained through the extraction process by soxhlet extractor and maceration of the sample they were subsequently analyzed by Pharo 100 spectrophotometer and Cicil 4200 HPLC. The experimental results on the cooking process of the fiber, center and peel of the kapok samples was obtained to determine the kappa number and yield with is were performed under temperature parameter condition, NaOH concentration and different duration of time. The experiment was performed fifteen times of randomized experiments and the variation of each kappa number parameter was obtained between 23.63 and 26.04. This value indicates that the sample was feasible for purification process [33-36].

The yield value obtained was 39.79% to 48.57%, this value had a high enough percentage value and met the requirements for the purification process. The experimental results of fiber, center and peel of kapok fruit which were not preceded by the extraction process with ethanol and benzene were measured by the magnitude of kappa and yield values [37,38]. The difference of kappa and yield value without extraction process were 32.58 and 43.26% respectively in fiber kapok sample with code SR/170/2/20, while kappa number 23.63 and yield value of 40.29% were obtained after the extraction. As for center of kapok sample with code of HT/170/2/, the kappa number obtained was 33.86 with yield value of 34.38% before extraction while, the kappa number of 24.97 with yield value of 32.29% were obtained after extraction [40-43].

Finally, peel of kapok sample with code of KL 170/2/40, the kappa number obtained was 31.24 with yield value of 32.67% before extraction while, the kappa number of 27.87 with yield value of 32.16% were obtained after extraction [44-49]. The purification process on fiber kapok sample was performed fifteen times randomly through five purification steps namely Chlordioxide (D), Extraction (E), Hypochlorination (H), Chlordioxide (D) and Chlordioxide (D), and the yield values were obtained as 37.782% to 46.67% with the viscosity of 2.41 to 5.11ml.pa.s (milli piscal sec), white degree 78.29 to 88.57% and the percentage of cellulose of 86.52% to 96.89%. Alpha cellulose obtained was > 90%, and it was qualified as propellant materials [50-53].

Discussion

Alpha cellulose in purification process

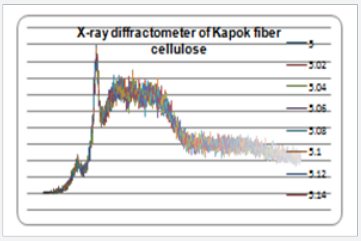

Figure 6: X'Pert X-ray diffractometer of alpha-cellulose fiber kapok sample.

The yield value of kapok fiber were obtained between 38.2 to 46.67%, with viscosity of 4.20 to 5.11ml.pa.s, white degrees 78.29 to 88.57% and the cellulose percentage of 86.52 to 96.89%. The value of the alpha cellulose yield obtained was feasible to be processed as supporting raw materials for stimulus ammunition driven materials on industrial scale [54]. The white degree values were obtained between 78.29 to 88.57% which were close to the 90% ISO standard contained in Mikroflas. When the viscosity values were adjusted to the degree of polymerization in the viscosity table, the degree of polymerization obtained was between 900 and 1300. It means that the alpha cellulose had a degree of polymerization between 1000 and 15000 (Figure 6) [55-57].

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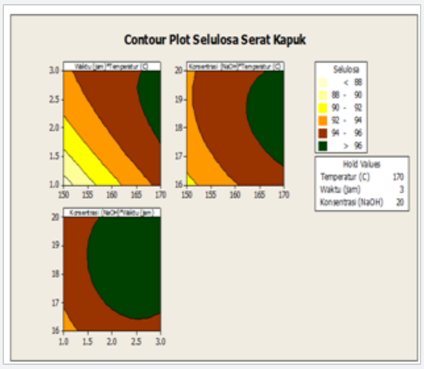

Figure 7: Contour graph of Kapok Peel Cellulose.

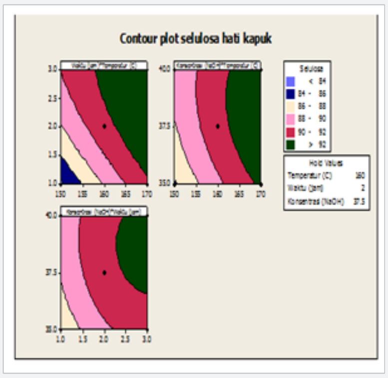

Figure 9: Contour graph of Kapok Center Cellulose.

Similarly, the percentages of cellulose based on the SII (Indonesian Industrial Standards) method and the X-ray diffractometer of the Philip X'Pert MPD brand were obtained from 90.00 to 96.89%, and it met specification as propellant material. As showed in the figure of x-ray diffractometer of alpha cellulose in kapok fiber [58]. The yield values of kapok fruit center were obtained between 26.19 to 35.56%, with viscosity of 4.29 to 4.64ml.pa.s, white degrees 80.89 to 88.26% and the cellulose percentage of 86.20 to 93.10%. The value of the alpha cellulose yield obtained was feasible to be processed as supporting raw materials for stimulus ammunition driven materials on industrial scale (Figure 7) [59,60].

The white degree values were obtained between 80.89 to 88.26% which were close to the 90% ISO standard contained in Mikroflas. When the viscosity values were adjusted to the degree of polymerization in the viscosity table, the degree of polymerization obtained was between 900 and 1300. It means that the alpha cellulose had a degree of polymerization between 1000 and 15000 [61]. Similarly, the percentages of cellulose based on the SII (Indonesian Industrial Standards) method and the X-ray diffractometer of the Philip X'Pert MPD brand were obtained from 86.20 to 93.20%, and it met specification as propellant material. As showed in the figure of x-ray diffractometer of alpha cellulose in center of kapok fruit (Figure 8).

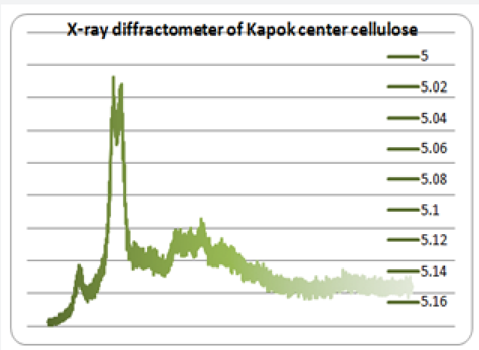

Figure 8: X'Pert X-ray diffractometer of alpha-cellulose center kapok sample.

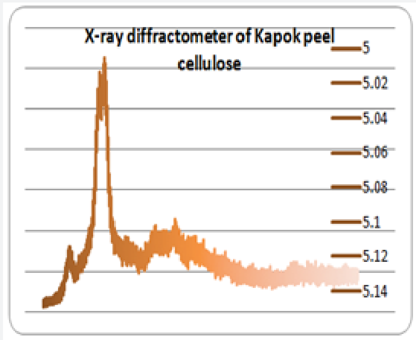

Figure 10: X'Pert X-ray diffractometer of alpha-cellulose peel kapok sample.

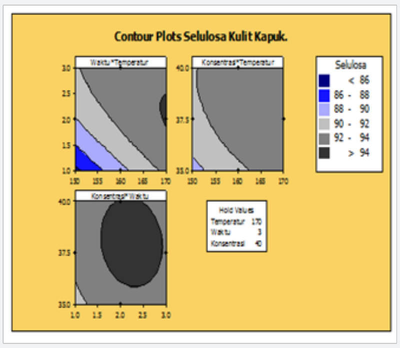

Figure 11: Contour graph of Kapok Peel Cellulose.

The yield values of kapok fruit peel were obtained between 29.96 to 36.92%, with viscosity of 4.27 to 4.66ml.pa.s, white degrees 79.56 to 86.26% and the cellulose percentage of 87.02 to 94.05%. The value of the alpha cellulose yield obtained was feasible to be processed as supporting raw materials for stimulus ammunition driven materials on industrial scale. The white degree values were obtained between 79.56 to 86.26% which were close to the 90% ISO standard contained in Mikroflas (Figure 9). When the viscosity values were adjusted to the degree of polymerization in the viscosity table, the degree of polymerization obtained was between 900 and 1300. It means that the alpha cellulose had a degree of polymerization between

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1000 and 15000 [62]. Similarly, the percentages of cellulose based on the SII (Indonesian Industrial Standards) method and the X-ray diffractometer of the Philip X'Pert MPD brand were obtained from 87.02 to 94.05%, and it met specification as propellant material (Figure 10). As showed in the figure of x-ray diffractometer of alpha cellulose in kapok fruit peel (Figure 11) [63].

Conclusion

Alpha cellulose parameter

Important parameters in the isolation and purification of alpha cellulose with specifications as a propellant material in a sample of peel, center and fiber of kapok were:

a. Pre-conditioning of samples prior to the research process were specific raw material specifications, and non-humid sampling room conditions by maintaining the humidity. The poor sample storage conditions caused the sample to be damaged, the sample water content would be different and the sample damage could lead to the larger consumption of the solvent (NaOH).

b. The density and sample flakes dimensions determine the content of the digester. The long-slice of sample causes the contents of the digester tube to be uneven lead to an uneven on the solvent chemicals and resulting in the immaturity of the sample. Shredding was done to prepare kapok fiber sample, while cutting was the method for prepare peel and center of kapok fruit sample. The size of cut was 1 to 2cm. The flakes dimension affected the penetration time of the solution and the diffusion of liquid ions. Thick flakes made penetration and diffusion process became difficult so that delignification reaction occurred slowly. The flakes thickness of center and peel of kapok fruit was 2mm.

c. The extraction process should be performed before the boiling process because in extraction process, because extractive substances such as resins, tannins, phenols, fatty acids and terpenes was only soluble in organic solvents such as ethanol and benzene in a ratio of 1: 2, while extractive substances such as sugar, pectin, organic salts and dyes can be extracted in water. When the extraction process was performed before boiling process, the cellulose obtained would be easier to purify.

d. The ratio for boiling kapok fiber samples was 1:7. In this ratio, the solvent can be evenly distributed in the kapok fiber sample so that the penetration and diffusion of the solvent can be homogeneous in the kapok fiber sample. The ratio of 1:5 was chosen for center and peel of kapok fruit. In this ratio, the solvent can be evenly absorbed in the center and peel of kapok fruit. If the ratio is in appropriate during the boiling process, the sample will be dark and black and difficult to purify. High level of alkaline can cause fiber damage and the decrease of yield value. If the ratio is incorrect, it can cause an imperfect lignification process and an uneven level of sample maturity.

e. The purification process on fiber, center and peel of the kapok fruit samples to obtain pure alpha was performed using DEHDD stage (Chlordioxide, Extraction, Hypochlorination, Chlordioxide and Chlordiocide).

Influence factors

Parameter condition of time and NaOH concentration which significantly affect the purity and yields value of alpha cellulose

a. The purity level of alpha cellulose for kapok fiber samples was 96.89% with yield value of 37.78%, at temperature of 170 oC, duration of 3 hours and NaOH concentration of 18%. When the time was reduced to 2 hours with temperature of 170 oC, NaOH concentration of 20%, the percent purity was 96.11% with yield value of 38.27%. The purity of alpha cellulose obtained in the above conditions meets the specification as a propellant material.

b. The purity level of alpha cellulose for the center of kapok samples was 93.90% with yield value of 26.10%, at temperature of 170 oC, duration of 3 hours and NaOH concentration of 37.5%. When the time was reduced to 2 hours with temperature of 170 oC, NaOH concentration of 35%, the percent purity was 92.71% with yield value of 27.28%. The purity of alpha cellulose obtained in the above conditions meets the specification as a propellant material.

c. The purity level of alpha cellulose for kapok fiber samples was 94.05% with yield value of 29.6%, at temperature of 170 oC, duration of 3 hours and NaOH concentration of 37.5%. When the time was reduced to 2 hours with temperature of 170 oC, NaOH concentration of 35%, the percent purity was 93.11% with yield value of 30.11%. The purity of alpha cellulose obtained in the above conditions meets the specification as a propellant material.

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