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Research paper

The Processing of Nitrocellulose from Rhizophora, Palm Oil Bunches (EFB) and kenaf fibres as a Propellant Grade

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Abstract

Nitrocellulose based powders are extensively in military application used as propellant in bullets, shells, and various missiles for tube munitions. In this study, Rhizophora, Palm Oil Bunches- Empty Fruit Bunches (EFB) and kenaf fibres were used as raw materials to produce nitrocellulose suitable for replace gunpowder. All raw materials are converted to dissolving pulps before nitrocellulose are synthesized. The characteristics of both dissolving pulps and nitrocellulose are determined. The result shows that the nitrocellulose from Rhizophora, Palm Oil Bunches- Empty Fruit Bunches (EFB) and kenaf have a tendency as propellant.

Keywords: *Gun powder, nitrocellulose, Rhizophora (Hardwood), Palm Oil Bunches (Empty Fruit Bunches) and Kenaf Bast (Soft Wood)*

1. Introduction

Nitrocellulose based powders are extensively in military application used as propellant in bullets, shells, and various missiles for tube munitions [1]. Nitrocellulose is produced upon esterification reaction of cellulose in the presence of nitronium ions NO2+. NO2+ ions can be produced upon mixing of Nitric acid with a strong acid such as sulfuric acid as shown in Fig. 1.

The cellulose source can be from wood or non-wood sources, but most commonly used is cotton, alfalfa and wood pulp [2]. Rhizophora or mangrove (Bakau) is a tropical species which lives in intertidal zone. Rhizophora is a fast growing species and is often used for conservation or as part of a managed forest to produce timber for construction or charcoal. Prior to 1990, Rhizophora timbers were heavily exploited mainly in Indonesia in the form of roundwood. Nowadays, Rhizophora is traded in the form of wood chips and charcoals. Tannins from Rhizophora bark also used to be a main product but has recently being replaced with synthetic tannins [3].

EFB or empty fruit bunch is a biomass waste generated by the palm oil industry. From the oil palm fresh fruit bunch (FFB) about 20% of crude palm oil (CPO) can be produced and 22-24% of EFB will be generated (FGV, 2014). Using this figure and the Malaysian production of CPO in 2013, more than 20 million metric tonnes of EFB is generated annually. EFB contains 44-48% alpha-cellulose, 16-24% lignin and 1.2-4.8% extractives [4-5]. Conventional use of EFB is as a mulch or compost in the plantation area.

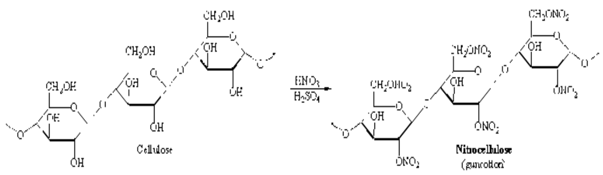

Fig. 1: Nitrocellulose synthesis reaction

Kenaf is an annual herbaceous plant of Malvaceae originating from Africa. It is a fast growing crop and can be harvested for fibres between three to five months [6]. Kenaf consists of two distinct fibres, the long bast fibre and shorter core fibre. In term of composition, the bast consists of 35-40% whereas core constitutes 60%-65%. Although lower in fibre composition, compared to the core fibres, the bast fibres contain higher alpha-cellulose (50% vs. 40%) and lower lignin (13% vs. 22%). Kenaf has been identified as a highly potential fibre source for the pulp and paper industry. However, it is not very economical compared to wood materials for conventional pulp and paper products.

In this study, Rhizophora, EFB and kenaf fibres were used as raw materials to produce nitrocellulose suitable for replace gunpowder. All raw materials are converted to dissolving pulps before nitrocelluloses are synthesized. The characteristics of both dissolving pulps and nitrocelluloses are determined.

2. Cellulose

According to Brittanica Encyclopedia, cellulose is a complex carbohydrate, or polysaccharide, consisting of 3,000 or more glucose units. As the basic structural component of plant cell walls, cellulose comprises about 33 percent of all vegetable matter (90 percent of cotton and 50 percent of wood are cellulose) and is the most abundant of all naturally occurring organic compounds.

In 1838, Payen recognized that cellulose is a common material of plant cell walls which occurs in almost pure form in cotton fibre and in combination with other materials, such as lignin and hemicelluloses, in wood, plant leaves and stalks. Some bacteria also can produce cellulose [7].

Cross and Bevan characterised Cellulose as a long chain polymer, made up of repeating units of glucose in the early 1900s. The related plant materials which occur in combination with cellulose are removed by dissolving them in a concentrated sodium hydroxide solution. They categorised the undissolved pulp as an *a*-cellulose. Dissolved pulps are categorised as (*b* - cellulose and ¡ - cellulose) which are later shown not to be celluloses [8].

3. Nitrocelulose

Nitrocellulose was found by Schönbein in 1846 coincidently. He observed the explosive properties of the nitrocellulose after he had mopped up concentrated nitric acid with a cotton towel and left it to dry above the stove [9].The nitrated towel was ignited by the heat of the stove. It ignited almost without the release of smoke . As a result, he found that by applying nitric acid on cotton, which is cellulose, nitrocellulose is formed. Nitrocellulose is produced from cellulose being mixed with a mixture of nitric esters. Because of that, it also can be called as cellulose nitrate. It is the main ingredient of modern gunpowder because of the high flammability of its compound.

In commercial use, nitrocellulose is also being used in making certain lacquers and paints. Chain of up to ten thousands of linked B(1-4) D-glucopyranose units can be formed by Cellulose because it is a linear polysaccharides [10]. In order to produce the nitrocellulose or cellulose nitrate, carbon positions C2, C3 and C6 in Cellulose can be substituted in nitration process which is using nitric acid because it is bonded to hydroxyl groups in the following reaction:

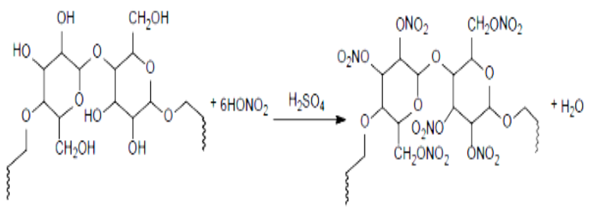

Fig. 2: Nitrating reaction of cellulose by nitric acid [11].

Nitrocellulose serves various purposes based on its nitrogen content. When its nitrogen content is below 12.5%, the nitrocellulose can be applied as components for paints, films and varnishes, and when it is higher than 12.5%, it can be used for various explosives and propellants [12].

This highlights the importance of having the right content of nitrogen in nitrocellulose to match their purposes. For example, to make high quality explosives, it is best to have the highest nitrogen content possible [13]. The maximum percentage of nitrates possible (m/m) is 14.1% [14]. That is because there are only three hydroxyl groups per Dglucopyranose to be substituted by the nitrate groups.

Nitrogen content has a great influence on the performance of the bullets projection. In Fig. 3, it shows that the increase of nitrogen content will increase burning rate. Faster burning rates produce more pressure and release more gas.

The C6 carbon has the fastest rate of substitution followed by C3 and C2 in order of reactivity as shown in Fig. 4. As stated before, the theoretical maximum percentage of nitrogen content is 14.1% and a DS of 3. In practice however, a stable nitrocellulose with nitrogen content higher than 13.5% is not achievable [16].

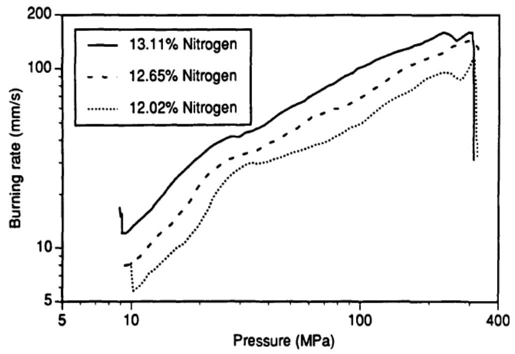

Fig. 3: Burn Rate Curves for Grains with Zero Perforations and Various Nitrogen Levels [13]

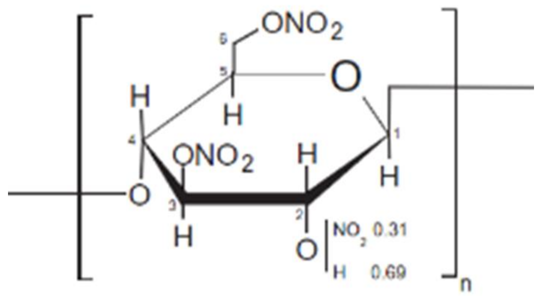

Fig. 4: Chemical structure of a nitrocellulose with a nitrogen content of 12.2% and degree of substitution, DS of 2.3 [15].

4. Basic Types of Propellant Grades

MIL-DTL is a United States Details Specification that provides information of material to be used and specific requirements of design. It also provides a guide to construct the item containing performance and detail requirements. Table 1 shows the nitrogen content for each grades of Nitrocellulose subject to the US MIL Details Standard [17] and Fig. 5 shows Nitrocellulose Based Propellants.

Based on Table 1, the nitrogen content of propellant varies where the highest reading is Grade B (13.35%) and the lowest is Grade E with (12.00%). It is proved that nitrocellulose below 12.5% also can be used as a propellant different with the statement of Herman F. Mark et all, [12].

Table 1: Nitrogen content according to propellant grade [17].

Type	Nitrogen content
Grade A, Type I	12.60 + 0.10 %
Grade A, Type II	12.60 + 0.15 %
Grade B	13.35 % min.
Grade C, Type I	13.15 + 0.05 %
Grade C, Type II	13.25 + 0.05 %
Grade D	12.20 + 0.10 %
Grade E	12.00 + 0.10 %

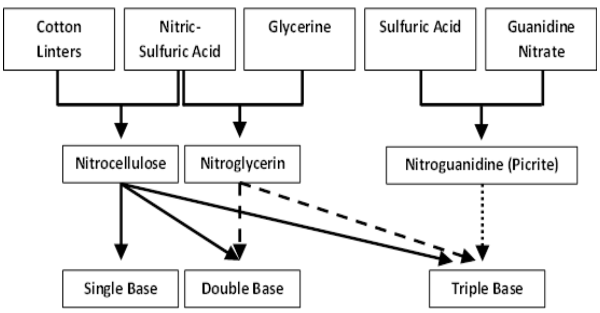

Fig. 5: Nitrocellulose Based Propellants

5. Quality Parameters

Many other quality parameters can be adjusted (nitrogen content, fineness, solubility, viscosity, ash content, insoluble residue in acetone, water content, etc.) to determine the criteria to meet the requirements of its use.

Viscosity is a measurement of its resistance to gradual deformation by shear stress or tensile stress. According to the US Military Standard MIL- DTL-244B [17], viscosity value for nitrocellulose can be measured by applying the Höppler method.

In temperature of 132°C, the method of Bergmann Junk test can be used to measure the stability of nitrocellulose. Bergmann-Junk is the equipment that is being used to test the chemical stability of organic nitrates and nitrocellulose as well. During thermal decomposition at 120 or 132 °C, sample is kept in that temperature to determine the nitrogen oxides evolved quantitatively. Chemical stability of sample is measured by using chemical acidimetric analysis, which is measuring the water extract of the evolved gases.

6. Raw material

6.1. Rhizophora

Rhizopora (Fig. 6) means "root bearer" in Greek which is well known as Pokok Bakau (Mangrove). It can be identify easily be-cause they have arching stilt roots. Rhizophora is 70% - 90% stock in Malaysia.

Rhizophora is mostly used for firewood because of its advantage that is the ease in which the timber can be split. So the seller can split it to meet the needs of the buyer. It also burns evenly and produces good quality heat. It is also used to make charcoal. Ac-cording to Giesen et all, Arab traders had recorded the usage of Rhizophora trees in tanning leather [18]. Fishermen also use the bark of Rhizophora to toughen their fishing lines and ropes. In construction areas, this tree is being used for pilling and structur-ing frames built near mangrove and swamps as well as for build-ing fish traps.
Based on its nature of strength, Rhizophora is chosen to produce Nitrocellulose that is believed to have greater reaction and impact compared to other plants. The availability of this tree can give great benefits to our defence industry especially in the ammunition production sector.

Fig. 6: Rhizophora (Hardwood)

6.2 Kenaf

Kenaf bast (Fig. 7) consists of the skin that contains fibre and wood parts. For the purpose of producing fibres, it is necessary to plant fibres without branches. Kenaf branches are not needed because of the low fibre production, while small seedlings with higher fibre production help to enhance photosynthesis. The colour of the bast could be green, red, or a mixture of red and green. Kenaf bast diameter can reach up to 25 mm depending on the variety and growing environment. Kenaf bast's surface is smooth, downy, hairy rough and also thorny.

The highest fibre content is at the bottom of the bast of1 to 1.25 m tall. Kenaf fibre contains 44%-62% α-cellulose, 14%-20% hemicellulose, pectin 4%-5%, 6%-19% lignin and 0%-3% ash. Being stem intact holocellulose contains 77%-79%, 37%-50%, α-cellulose, lignin 16%-20%, and 2%-4% ash.

Fig. 7: Kenaf Bast(Softwood)

6.3 Empty Fruit Bunches (Oil Palm)

Empty Fruit Bunches (EFB) (Fig. 8) are the products after the fruit bunches produce steamed, separated palm fruit for oil production. EFB are obtained from mills every day when the process is carried out. It is estimated that a total of 0:20% to 0:24% of EFB can be obtained from each ton of Fresh Fruit Bunches (FFB) that are processed. Malaysia is the world's leading producer of palm oil and had produced over 69 million tons and 74 million tons of oil FFB in 2004 and 2005[19]. This means that more than 13.8 and 14.8 million tons of EFB are produced from factories each year. EFB will increase the total production in the future, due to higher oil output of the country.

At the factory, the EFB will be separated and dumped in the space provided. In the past, most of the EFB will be burnt by the man-agement as a solution to get the ash from the bunches. This mate-rial could be used by farmers including palm plantations as a source of fertiliser because it contains nutrients, especially potas-sium (K, about 40% of the dry weight of EFB and some small phosphate (Pazos) and magnesium (MgO). In addition, ash from bunches is also very beneficial to the soils that are acidic or acid soils. This is because the nature of the bunch ash is alkaline (pH 10-12) and it can help to reduce soil acidity effectively. Increased pH disbursed will indirectly improve soil fertility area. Generally, the ICC contains about 0.80% nitrogen (N), 0.22% phosphate (Pazos), 2.90% potassium (~ O), 0.30% magnesium (MgO) and some small micro nutrients.

Fig.8: Empty Fruit Bunches (Oil Palm)

7. Methodology

In the process to produce nitrocellulose, the first step is the selection of raw material. The three types of material, which reflect each category of fibers. Rhizhophora bast (hardwood), kenaf bast (softwood) and oil palm bunches (empty fruit bunches) were selected as samples to be used in producing nitrocellulose. The stages involved are pulping, pulp bleaching, dissolving pulp and nitrating process. The overall process flow for EFB is shown in Fig. 9, flow for Rhizophora in Fig. 10 whereas for Kenaf Bast in Fig. 11.

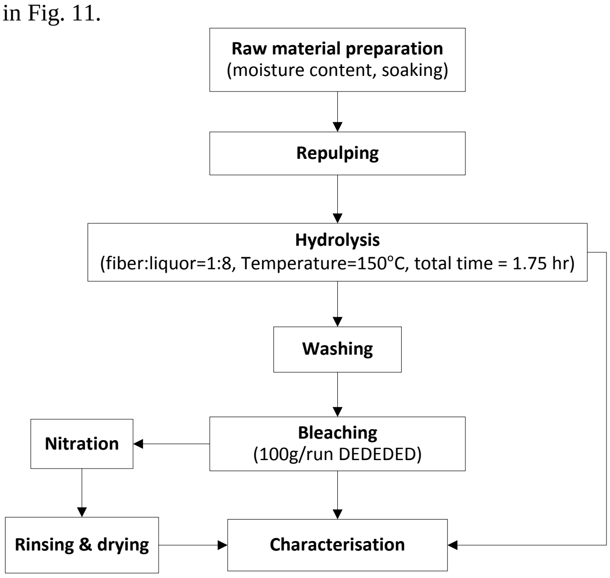

Fig.9: Overall nitrocellulose process flow for EFB

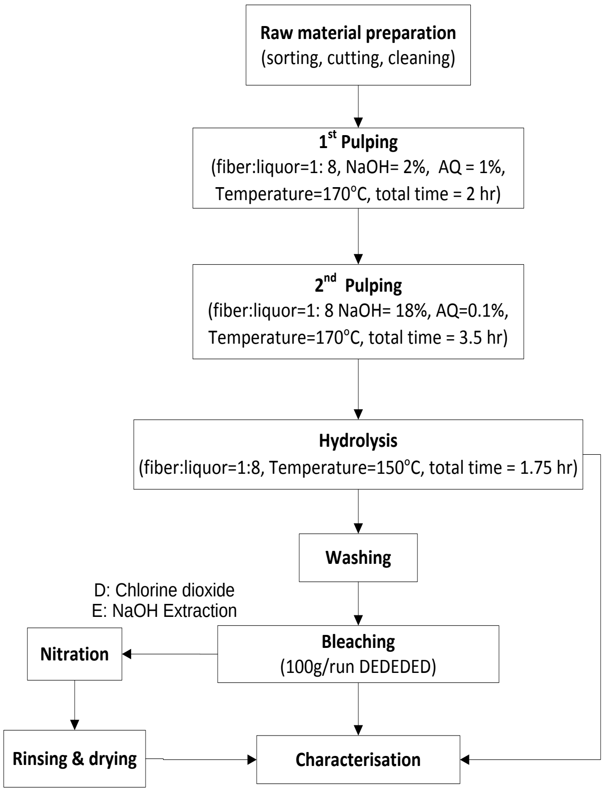

Fig.10: Overall nitrocellulose process flow for Rhizophora

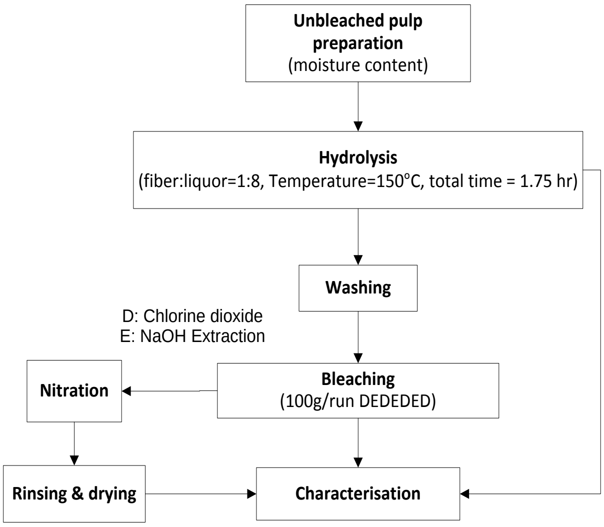

Fig.11: Overall nitrocellulose process flow for Kenaf Bast

8. Characterisation and Analysis

At different stages of the work, analysis and characterisation of product were carried out according to methods listed in Table 2. The analysis of titration with ammonium iron sulfate $(NH_4)_2Fe(SO_4)_2$ was carried out to determine the content of nitrogen in nitrocellulose. The degree of substitution was calculated according to Vaca Garcia et al [20].

Table 2: Characterisation and analysis

	Properties	Method
Unbleached pulps	Kappa number	TAPPI, T 236 om-99 1999
Dissolving pulps	Hemicellulose α-cellulose Lignin Ash	Wise 1946 TAPPI, T 203 om-93 1993 TAPPI, T 222 om-02 2002 TAPPI, T 211 om-02 2002
Nitrocellulose	Ash Moisture Nitrogen Degree of substitu-tion	TAPPI, T 211 om-02 2002 ASTM D4795-94 & Metrohm Ti Application Note No. T-37 Walsroder® Nitrocellulose

8.1. Pulping yield and Kappa number

The table 3 and Fig. 12 shows the percentage of the pulping yield and Kappa number for every 100grams of all three samples. The highest yield for pulping is Kenaf Bast which is 54%, followed by Rhizophora which is 31.2% and the last one is EFB without yield for 100 grams.

Table 3: Pulping and Kappa results

	Unit	Rhizophora	Kenaf bast	EFB
Pulping yield	%	31.2	54.0	-
Kappa no	-	3.24	8.0	15.2

The data (Fig.12) shows the highest pulping yield is Kenaf Bast. The result gives two possibilities; it can produce more α-cellulose or it contains other elements such as lignin and carbohydrates more than α-cellulose. Samples must go through other processes to measure the volume of α-cellulose.

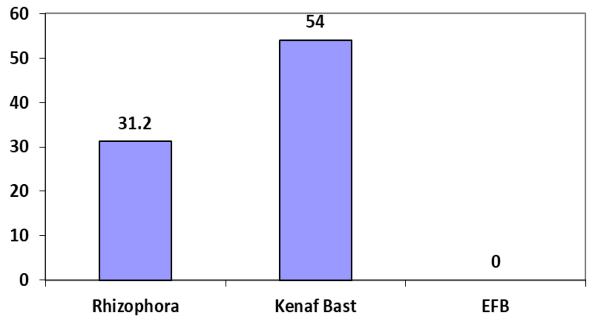

Fig.12: Pulping Yield

8.2. Pulp bleaching and dissolving pulp characteristics

Pulp bleaching was carried out according to the conditions shown in Table 3. The treatment of each stage was mild to prevent cellulose degradation. The dissolving pulp characteristics produced in this work are shown in Table 4. The alpha-cellulose contents for all pulps are higher than 91% with the highest alpha cellulose shown by EFB dissolving pulp. The lignin content was low, except for Kenaf dissolving pulp. The ash contents for all dissolving pulps were below 1.2%.

Table 4: Characteristics of dissolving pulp produced

Properties	Unit	Rhizophora	Kenaf bast	EFB
Hemicellulose	%	8.7	1.5	0.0
α-cellulose	%	91.3	91.6	98.9
Lignin	%	0.0	6.26	0.1
Ash	%	0.1	0.6	1.0

8.3. Hemicellulose

Hemicellulose exists along with cellulose in almost all plant cells wall. It is little and weak when cellulose is strong. The attachment of microfibrils with hemicellulose is strengthened by lignin. So based on percentage of hemicellulose, the volume of α-cellulose can be produced from each sample can be predict. The highest content of hemicellulose is Rhizophora (8.7%) followed by Kenaf Bast (1.5%) and EFB (0%) as shown in Fig. 13.

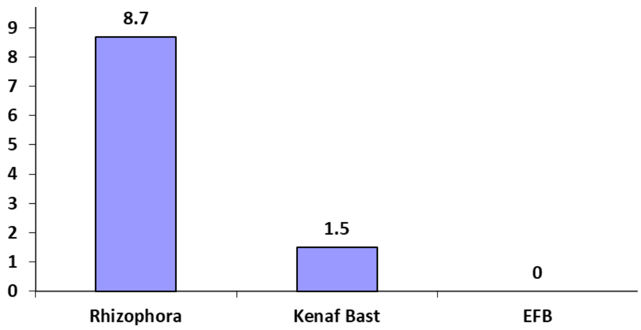

Fig. 13: Hemicellulose content

8.4. α-cellulose

α-cellulose is a pure cellulose yield that does not comprise other element through chemical pulping and bleaching process. The volume of α-cellulose compared to hemicellulose is the opposite. Highest hemicellulose is the lowest α-cellulose. The data obtained from this experiment shown EFB was the highest α-cellulose (98.6%), followed by Kenaf Bast (91.6%) and Rhizophora (91.3%) as shown in Fig. 14.

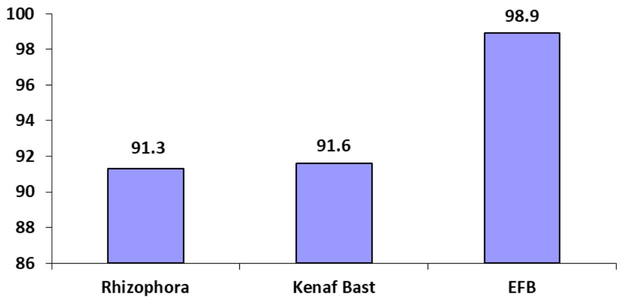

Fig. 14: α-cellulose content

8.5. Lignin

Lignin is the element that binds cellulose, hemicellulose, carbohydrates and other elements in the plant. Kenaf Bast contain the highest lignin content which is (6.26%) followed by EFB (0.1%) and Rhizophora (0%). From this experiment, it indicates for every 100 grams of Rhizophora,the reading is nil. Although Rhizophora is from the hardwood category, it had a low volume of lignin as shown in Fig 15. This shows that it is linked and bound strongly by its form of fibre.

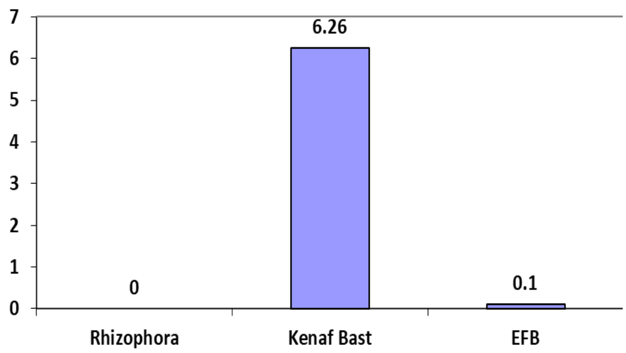

Fig. 15: Lignin content

8.6. Ash

EFB had the highest volume of ash (1%), followed by Kenaf Bast (0.6%) and Rhizophora (0.1%) as shown in Fig. 16. The higher ash content will produce more ash residue from the firing. This residue can make our weapon dirty and needs more maintenance.

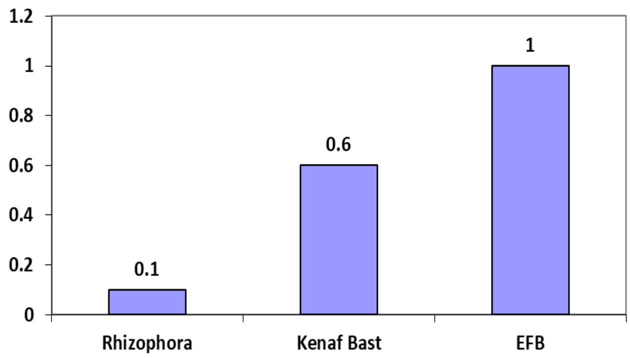

Fig. 16: Ash content

8.7. Nitrocellulose characteristics

The characteristics of nitrocelluloses produced are summarised in Table 5. The highest yield was obtained from Rhizophora dissolving pulp, followed by EFB and kenaf bast. Rhizophora nitrocellulose also showed the highest nitrogen content (11.4%) whereas kenaf bast and EFB nitrocellulose showed slightly lower nitrogen (11.3% and 10.8%, respectively). There was no alcohol content in the nitrocellulose as they were processed, stored and analysed directly without alcohol. The yields were higher than 100% due to the additional weigh contributed by the -NO2 functional group.

Table 5: Results of nitrocellulose produced

Properties	Unit	Rhizophora	Kenaf bast	EFB
Nitrocellulose yield	%	144.2	100.3	128.1
Moisture content	%	55.7	62.5	62.2
Nitrogen content	%	11.4	11.3	10.8
Degree of substitution	DS	2.09	2.06	1.90
Alcohol content	%	0	0	0

8.8. Calorimetry test

Calorimetry test was also conducted to measure the gross heat value of the sample. All the data collected uses joules as a unit of measurement as shown in Table 6.

Table 6: Data obtained from calorimetry test

Categories	NC Rhizophora	Ball M196
Method	Dynamic	Dynamic
Benzoic Acid (grams)	0.3119	0.5330
Energy Equivalent, EE Value	10,421,220	10,451,900
Weight (grams)	0.312	0.533
Fuse	15	15
Sulfur	Nil	Nil
Initial Temperature (^{0}C)	26.6028	27.2175
Jacket Temperature (^{0}C)	29.9758	29.9696
Spike Weight	Nil	Nil
Acid	10	10
Temperature Rise	0.1979	0.2683
Gross Heat J/gm	**6,612,900.29**	**5,260,853.39**

From the data obtained, gross heat produced from nitrocellulose is 6,612,900.29 J/gm and ball M196 5,260,853.39 J/gm. This shows that the nitrocellulose produced a 1,352,046.9 J/gm (25.7%) greater gross heat than ball M196 gunpowder as shown in Fig. 17.

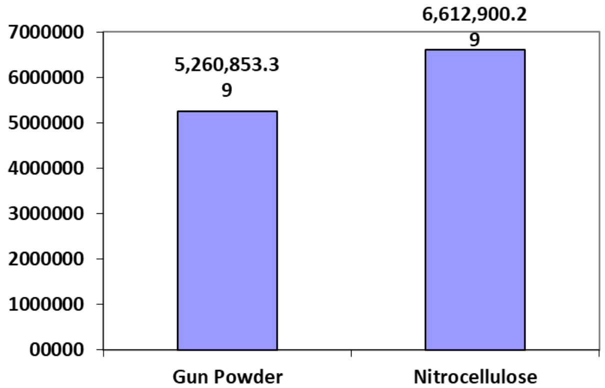

Fig. 17: Gross Heat (J/gm)

8.9. Energy Equivalent Value (W)

Energy equivalent or heat capacity of the calorimeter is determined to represents the sum of the heat capacities of the components in the calorimeter. Represented by this value is notably the bucket, the water in the bucket and the metal bomb.

$$W = \frac{\text{Gram of Benzoic Acid x Heat of combution}}{\text{Temperature rise}} = \frac{J}{°C} \qquad (1)$$

8.10. The Fuel Test (Gross Heat of Combustion)

The gross heat of combustion (Hg) can be calculated using the following formula after the EE Value is determined:

$$W = \frac{\text{Temperature rise x Energy equivalent}}{\text{Sample weighing}} = \frac{J}{gm} \qquad (2)$$

Table 7: Energy Equivalent Value (W) and The Fuel Test (Gross Heat of Combustion) results.

Item/ characterization	Calculation	Calometric value	difference
Energy Equivalent (W) in J/°C			
Nitrocellulose (Rhizophora):	10,422,252	10,421,220	1032
Ball M196 gun powder:	10,451,118	10,451,900	758
The Fuel Test (Gross Heat of Combustion) in J/gm			
Nitrocellulose (Rhizophora):	6,610,126.40	6,610,126.40	2,773.89
Ball M196 gun powder:	5,261,247.22	5,261,247.22	393.83

In Table 7, EE value collected from the calorimetry test for Nitrocellulose (Rhizophora) is 10,421,220J/°C while based on calculation the value is 10,422,252 J/°C. The difference is -1032J/°C whilst EE Value collected from the callorimetry test for Ball M196 gun powder is 10,451,900J/°C while based on calculation the value is 10,451,118J/°C. The difference is +758J/°C.

Gross heat collected from the calorimetry test for Nitrocellulose (Rhizophora) is 6,612,900.29 J/gm while based on calculation the gross heat is 6,610,126.40 J/gm. The difference is +2,773.89J/gm, whilst Gross heat collected from the calorimetry test for Ball M196 gun powder is 5,260,853.39 J/gm while based on calculation the gross heat is 5,261,247.22 J/gm. The difference is -393.83J/gm where the error is -0.04%.

9. Conclusion

This paper discussed the process of nitrocellulose as gunpowder. As a conclusion, nitrocellulose can be produced using Rhizophora, Kenaf Bast and Oil Palm Bunches (Empty Fruit Bunches). Nitrogen content of these three samples ranges from 10.8% to 11.4%. This nitrogen content can be increased by extending nitrating process. Higher nitrogen content can be obtained by increasing the nitration time. However, precautions should be taken to avoid hydrolytic degradation of cellulose or side reactions to take place. It is found that dissolving pulps from Rhizophora, EFB and kenaf successfully produced with purity higher than 91% and nitrocellulose from Rhizophora showed the highest yield and nitrogen content.

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