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Biofuel Production from Rubber Seeds by Pyrolysis Method using Natural Zeolite Catalyst

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Abstracts: Rubber seed is rubber plant waste that has the potential as a source of bioenergy. Rubber seed contains 40-50% oil, and rubber seed shell contains lignocellulose (cellulose, hemicellulose, and lignin). Rubber and rubber seed shells can be converted into biofuel through the Pyrolysis Process. Pyrolysis is biomass decomposition with heat assistance without oxygen at 250ºC-600ºC. This research aimed to analyze the characteristics of biofuel products. This research was conducted using a pyrolysis reactor by controlling the temperature at 250ºC, 300ºC, 350ºC, 400ºC, and 450ºC. The characteristics of biofuel produced in this study include density (1.0646 gr/ml), viscosity (5.417 mm^2/s), flash point (88ºC), moisture content (30.4%), calorific value (7451.3997 Cal/g), and cetane number (32.9). Based on the results of biofuel analysis using GC-MS, the C atom chain was dominated by C5-C15 compounds at 44.41%.

Keywords: Rubber seed, Rubber seed shell, Pyrolysis, Biofuel.

1. INTRODUCTION

Petroleum still significantly contributes as the primary source of energy in Indonesia. The global gasoline demand, the influence of the expanding number of humans, and the increasing demand for petroleum energy, particularly in Indonesia, is waning. This possibility is also caused by increased demand for oil energy in Indonesia's transportation and industrial sectors [1]. Therefore, it is necessary to substitute petroleum through renewable energy utilization, such as solar, wind, water, biomass, and so on. Biomass represents several alternative energy sources that may be exploited in Indonesia. Biomass is a non-fossil organic material derived from plants, animals, and microorganisms. Biomass comes from forestry, agriculture, aquaculture, livestock, or industrial and household waste. The rubber plant (*Hevea brasiliensis*) is one of the tropical plantations crops widely cultivated in Indonesia and one of the biomass sources. Rubber trees produce lustrous speckled brown seeds 2.5-3 cm in length and weighing about 3-6 g [2], thereby being surrounded by three elliptical pods (capsules), usually consisting of three seeds (Andrea, 2020).

Rubber seeds are a waste product of rubber plantations that are available and have not been maximally utilized. The oil content of rubber seed can be produced around 40-50% [3]. Indonesia's extensive rubber plantations contribute significantly to the nation's economic growth. Natural rubber generated in 2019 was 3.33 million tons, using a plantation covering 1 million hectares [4]. According to the Indonesian Rubber Commodity Plantation Statistics 2023, the site of rubber plantations in South Sumatra was 722,054 Ha [5]. Four hundred rubber trees can be planted in an area of 1 hectare. Therefore, it is expected to generate 5,050 Kg of rubber seeds annually [6]. The content of rubber seeds will be very capable of being used as fuel, where rubber seeds consist of 40-50% hard brown skin and 50-60% yellowish white kernel. The rubber seed kernel comprises 45.63% oil, 2.71% ash, 3.71% water, 22.17% protein, and 24.21% carbohydrate [7]. The characteristics of the rubber seed shell and the main content of saturated fat in rubber seed oil are shown in Tables 1 and 2. If rubber seed waste is not immediately utilized and appropriately processed, it will cause environmental pollution because the shelf life of rubber seeds is concise. The optimum storage temperature is 7-10°C. At this temperature, rubber seeds have not begun to freeze cells. Thus, they cannot germinate and grow because the constituent cells become damaged [8]. This makes rubber seeds only become waste around the plantation. Rubber seeds are potentially a source of raw materials because they are abundant, renewable, and relatively cheap compared to another biomass [9]. Furthermore, using rubber seeds to be converted into more appropriate products must be done.

Table 1. Characteristics of rubber seed shell [2]

Proximate Analysis	Unit	Experiment Result
Moisture	%	15.97
Ash Content	%	4.15
Volatile Matter	%	47.43
Fixed Carbon	%	32.45
Calorific value	Cal/g	3312.8694

Table 2. The primary fatty acid component found in rubber seed oil [2]

Identified compound	Chemical Structure	%wt.
Palmitat acid	$CH_3(CH_2)_{14}COOH$	7.32
Linoleic acid	$CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COOH$	34.69
Oleic acid	$CH_3(CH_2)_7CH = CH(CH_2)_7COOH$	45.35
Pentadecanoic acid	$CH_3(CH_2)_{13}CO_2H$	8.86

There are several methods of converting biomass into energy sources, especially direct combustion, which produces heat; gasification, which produces gases (such as methane, carbon monoxide, and hydrogen); and liquefaction/pyrolysis, which makes liquid fuels [11]. This plant-based matter has the potential to be transformed into bio-oil. [12], [13] and additional essentials through pyrolysis - high-temperature thermal breakdown of materials in an inert or airless environment [14]. Pyrolysis is a new generation that produces products such as bio-oil, biochar, syngas, and ash that have the potential to generate renewable energy [15]. The pyrolysis process composes organic matter into a solid, liquid, and gas. The amount is determined by the pyrolysis technique, feedstock properties, and reaction variables [16]. Therefore, if the shell receives heat without interacting with air and at a reasonably substantial temperature, the complex chemicals resulting in the structure of the surface will decompose and generate substances in solids, liquids, and gases. [17].

Based on the conventional processes that have been carried out, the pyrolysis thermal cracking process is the best in terms of yield. This process still has shortcomings in terms of heat transfer, including heat transfer between the surface of solids/biomass and gas, which is relatively very small and affects yield and energy consumption more [18]. Heat transfer is somewhat better, and the biomass cracking process is more easily directed into the desired product with the help of a catalyst [19]. The motivation that has the most significant potential is natural zeolite [20]. Natural zeolites are abundant and cheap. Natural zeolite resources will be around 29.5 million tons [21]. Zeolite-type catalysts have vital acidic broasted centers that transfer hydrogen ions to polymer chains [22]. Natural zeolites must first be activated to eliminate impurities such as Na, K, Ca, Mg, and Fe; raise crystallinity; enlarge pores; increase surface area, increase acidity; and increase Si/Al ratio before being employed as a catalyst [23]. In pyrolysis research using zeolite catalysts, it has been applied with palm frond raw materials, producing a yield of 44.4% [24], and pine wood raw materials, creating a yield of 43.77%. [25]. Pyrolysis from 2 Kg coconut shells produced 194 mL of Biofuel [26]. Pyrolysis research with rubber seed shells has been carried out by Chaiya et al. with a yield of 38.22% [27]. Based on the information described, this study utilizes pyrolysis to convert rubber seeds into biofuel using natural zeolite catalysts.

2. MATERIAL AND METHODS

2.1. Raw Material Preparation

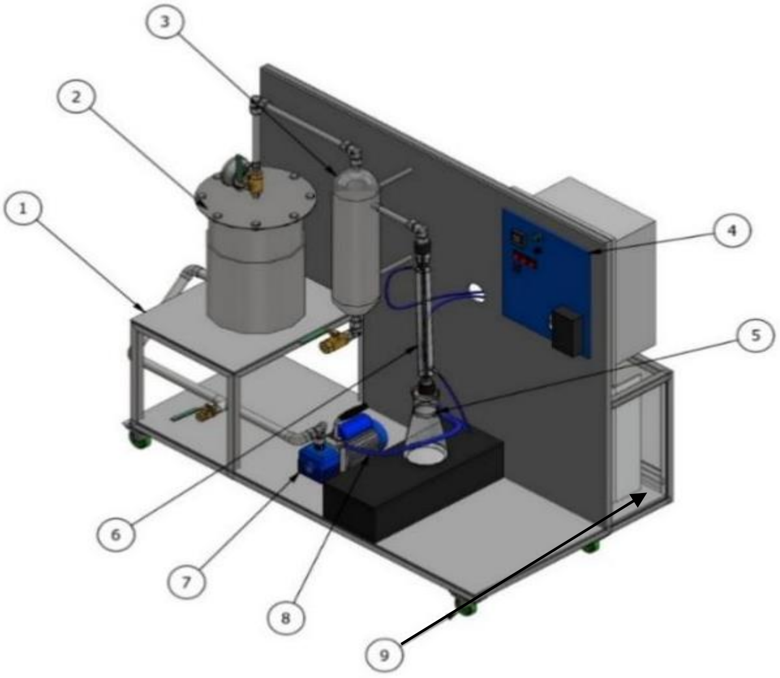
Rubber seeds as the raw material used in this study were collected from Durian Remuk Village, Muara Beliti Sub-district, Musi Rawas Regency, South Sumatra, Indonesia. Subsequently, the rubber seeds were cleaned, and the shell and kernel were separated. The shells and kernels were dried under the sun for 3-4 days to ensure constant water content. The shells and kernels were milled separately using a grinder and sieved to a 50-90 mesh powder size.

2.2. Zeolite Activation Procedure

Activation of zeolite in this study involves a sulfuric acid solution with a concentration of 1.2. Zeolite was activated by impregnation with sulfuric acid at 78ºC and stirred at 125 rpm for 1 hour. Zeolite was then separated using Whatman 42 filter paper, and then the zeolite sample was rinsed with water from a distilled process until it reached a pH of approximately neutral. The zeolite sample was then dried at 100ºC for two hours using an oven. Zeolite is cooled in a desiccator and weighed until the mass is constant.

2.3. Pyrolysis Apparatus Testing Procedure

The pyrolysis process to convert rubber seed into biofuel uses the pyrolysis apparatus shown in Figure 1. Raw materials in the form of rubber seeds that have been dried as much as 2 Kg were inserted into the reactor. The catalyst in natural zeolite was also included in 3% or 60 g. Then the reactor heating mantle was turned on, and the reaction temperature was set at 250ºC, 300ºC, 350ºC, 400ºC, and 450ºC. The heating process lasted for 2-3 hours. The smoke formed then entered the separator to separate the heavy and light fractions. Furthermore, the results from the separator entered into the condenser and condensed to get maximum results. The biofuel products were deposited in a holding tank, and the smoke from the process was precipitated to get tar.



Descriptions

1. Framework
2. Reactor
3. Separator
4. Control panel
5. Erlenmeyer
6. Condenser
7. Vacuum Pump
8. Air Hose
9. Containment basin

Figure 1. Pyrolysis Apparatus

3. RESULTS AND DISCUSSIONS

3.1. Characteristic Analysis of Biofuel Product from Rubber Seeds from Pyrolysis

Biofuel produced by rubber seeds with pyrolysis process requires high temperature. Researchers used a natural zeolite as a catalyst with a temperature variation of 250ºC to 450ºC with a time of 120 min to determine the performance of the pyrolysis apparatus to determine the product of the physical and chemical properties.

3.2. Physical Properties Analysis Results of Rubber Seeds by Pyrolysis

The biofuel obtained from pyrolysis has color and odor characteristics, as shown in Table 3.

Table 3. Physical Properties of Biofuel Products

No	Samples	Color	Odor
1	Rubber seed	Cloudy black	Pungent
2	Rubber seed shell	Translucent black	Pungent
3	Rubber seed + Rubber seed shell	Brownish black	Pungent

3.2. Temperature Influence on Biofuel Yield

Yield is a percentage that states the product produced from the amount of raw material fed. In this study, the percent yield calculated was the amount of biofuel against temperature variation.

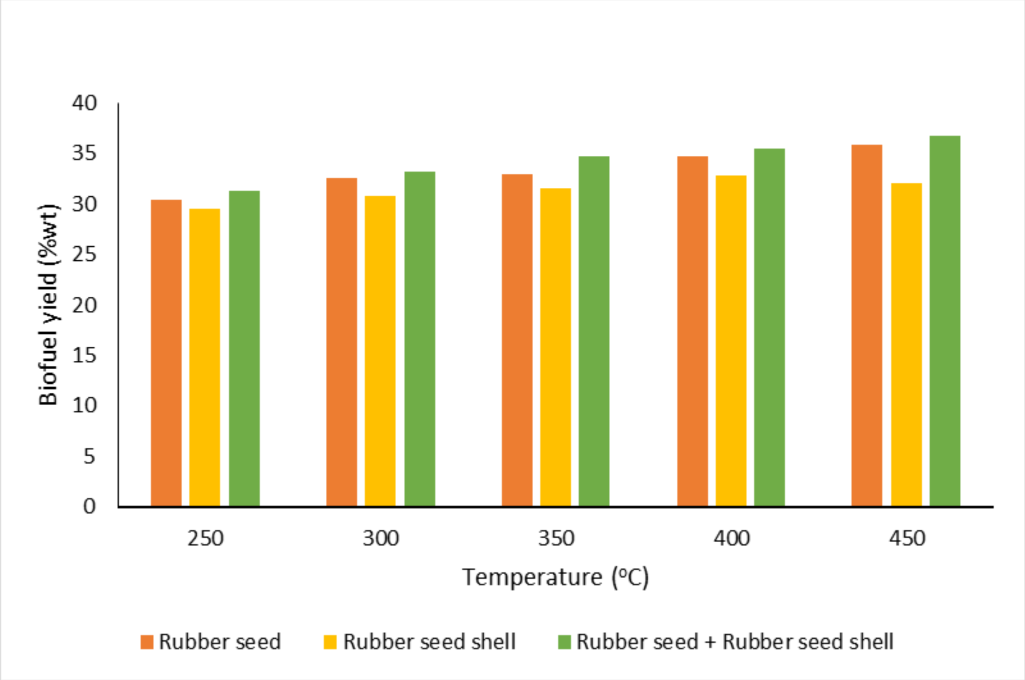


Figure 2. Temperature Influence on Biofuel Yield (%wt.)

Figure 2 shows that the yield on rubber seed raw materials was 30.4 - 35.9 %wt., the rubber seed shell had a yield of 29.5 - 32.8 %wt., and a mixture of rubber seeds with rubber seed shells produced a yield of 31.3 - 36.8 %wt. The increase in pyrolysis temperature, the composition of biomass (cellulose, hemicellulose, and lignin) that decomposes more and more so that the biofuel yield increases with increasing pyrolysis temperature. The temperature rise enables particles to advance more rapidly, increasing the likelihood of interaction among particles. The rise in temperature at an axis boosts the reaction velocity, increasing the yield percentage achieved [28]. This finding suggests that degradation of samples occurs significantly more often at higher temperatures [29]. According to the data, the mixture of shells and rubber seeds produced more than the unmixed material. This result showed that the mix of seeds and shells was the most optimal raw material in yield. Therefore, the combination of rubber seed and rubber seed shell provides a higher yield of biofuel than using either of them alone because rubber seed shell has a low ash percentage and a heating value equivalent to virgin wood, making it an appealing attribute for a solid biofuel [30]. Rubber seed kernel is widely recognized for its oil content, with a 33.1 wt.% oil output that can potentially be used to produce biofuel [2].

3.3. Temperature Influence on Biofuel Density

Density is the amount or quantity of a substance. The density value is influenced by temperature. The higher the temperature, the density of a fluid decreases because the cohesion force of the fluid molecules decreases [31]. Density is one of the physical properties that can be used to indicate the type of product or certain compounds. The specific gravity of the product obtained in this study ranged from 1.0553 to 1.0991 g/mL. This result was approximately the density of water, which is 1000 Kg/m^3. The overall effect was almost close to the ASTM D7544-12 Standard value, which has a range of (1.1 - 1.3 g/mL). The factor causing the density to be inconsistent was the presence of other liquids in the pycnometer during the analysis process and many other compounds in biofuel products, such as tar compounds. The density of biodiesel samples reduces as temperature rises. In addition, as the temperature increases, the density decreases. [32]. In contrast, the density of biodiesel blends falls as the temperature increases [33]. As a result, the temperature has an inverse connection with biofuel density, which means that as the temperature rises, biofuel density correspondingly rises [34].

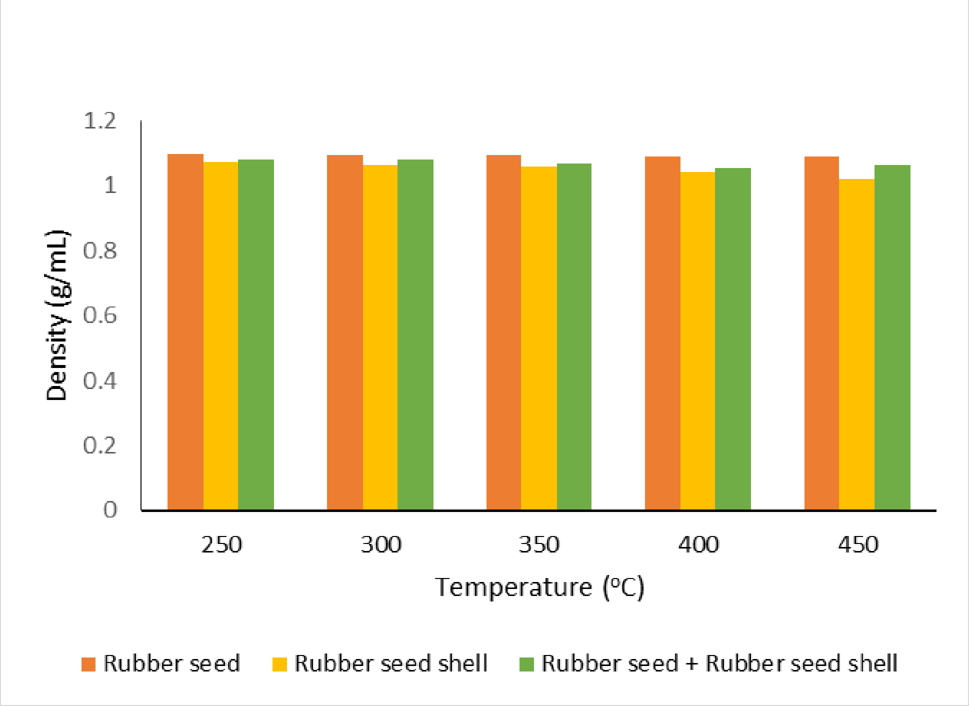


Figure 3. Temperature Influence on Biofuel Density

3.4. Temperature Influence on Biofuel Viscosity

Viscosity is defined as the internal friction or friction of the fluid against the container in which it flows. It exists in liquids or gases and is essentially the friction between adjacent fluid layers as they move across each other or the friction between the fluid and the container in which it flows [31]. Kinematic viscosity is a critical fuel attribute that determines the atomization quality and dimension of the fuel molecule in spray application [35]. The kinematic viscosity of biodiesel-diesel blends is a highly influential influence on their intrinsic properties [36], dictating fuel atomization and complete combustion in the diesel engine [36]. The kinematic viscosity of the product produced from this analysis is 5.016-5.738 mm^2/s. The difference in viscosity was caused because when testing the biofuel produced, the falling ball was not hampered by the viscosity of the biofuel. Measurement of the viscosity of biofuel oil was aimed at determining the oil's viscosity at a specific temperature so that the oil can flow at that temperature. In addition, the temperature rises, the kinematic viscosity of biodiesel specimens falls [37], [38]. Despite rising temperatures, the chain oxidation reaction accelerates, and the degree of oxidation increases substantially, impacting biodiesel kinematic viscosity [39]. The dynamic viscosity of biodiesel and its mixes increases with decreasing temperature and exhibits Newtonian behavior to the pour point [40].

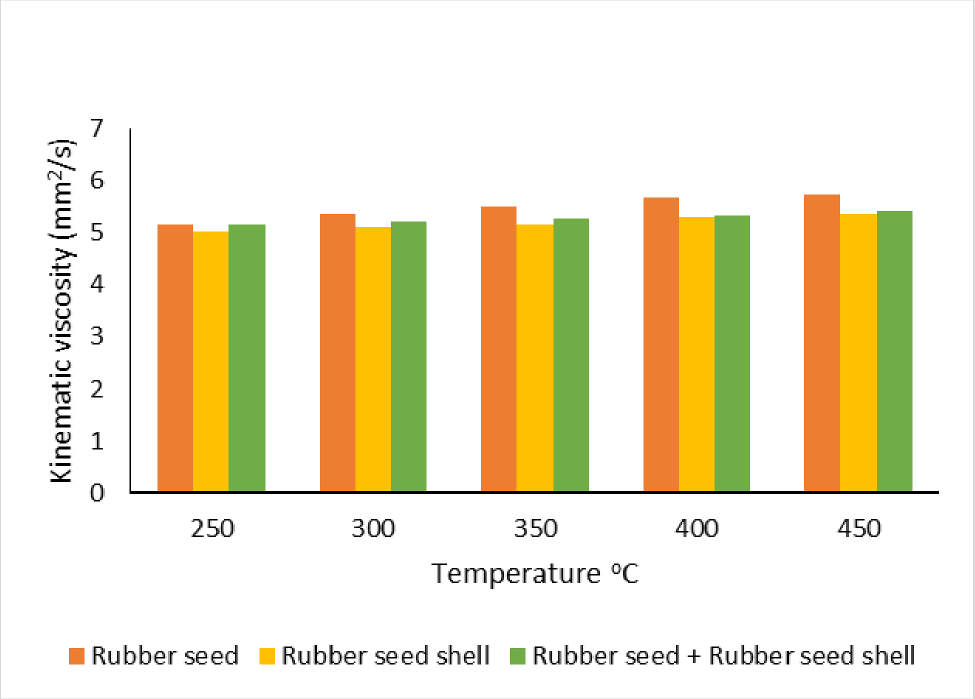


Figure 4. Temperature Influence on Biofuel Viscosity

3.4. Temperature Influence on Biofuel Water Content

Water content is the amount of water content in a substance. In this study, water content affects the effectiveness when burning to determine the amount of water in Biofuel products. The greater the water content, the worse the quality of biofuel. Otherwise, the biofuel quality is better if the water content is negligible. Figure 5 shows the moisture content in each variation, which ranged between the considerable water content caused by the hydro processing process's by-products and hydrodeoxygenation. The oxygen released from the process reacted with hydrogen to form water vapor. From the study results, the moisture content was obtained in the amount of 15.3-30.4%. These results have met the ASTM D7544-12 Standard, which has a max of 30% for the moisture content contained in the product. However, the lowest value was found in rubber seeds with a temperature of 250°C, and the highest was found in a mixture of seeds and rubber seed shells at 450°C. This water content indicated the formation of water during the reaction. This occurred during biofuel pouring, where water content was incorporated into the biofuel during condensation. The amount of water quantity in biofuel was also affected by the raw biomass's moisture content and the oxygen quantity in the pyrolysis organic matter [41]. In the pyrolysis of pine bark as raw material, the proportion of biofuel output decreased when the pyrolysis temperature was raised [42]. This suggests that higher temperatures can lead to the production of biofuels with lower water content.

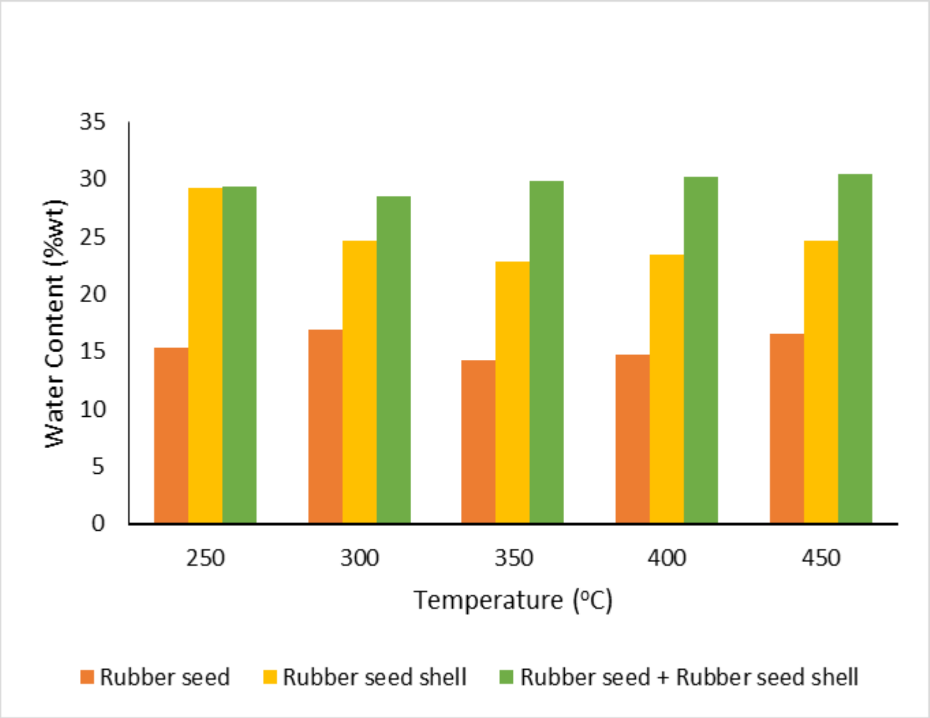


Figure 5. Temperature Influence on Biofuel Water Content

3.5. Temperature Influence on the Flash Point of Biofuel

A fuel's flash point is determined simply by the temperature when the energy generates enough vapor for combustion through air at the pre-spark stage [43]. The influence of temperature on biofuels' flash point varies based on each biofuel's kind and composition. A fuel's flash point has no immediate impact on its burning. Meanwhile, the flash point is a significant property regarding safety and handling because it denotes the maximum temperature at which the fuel may burn when in the presence of a heat source [44]. The effect of temperature on the flash point of biofuels is complex. It can be influenced by various factors, including the fuel's composition and the specific conditions of the testing method. Understanding the flash point of biofuels is essential for safety and handling purposes, but its direct impact on combustion is not as significant as other physical properties. However, the composition of biofuels can affect their flash point. For example, the number of double bonds and carbon atoms in the fuel can influence the flash point. The flash point of biofuels can be influenced by temperature. Higher temperatures can lead to a lower flash point. Furthermore, the flash point is one of several fuel quality factors that can influence the combustion and emission characteristics of biofuels [45]. In this study, the flash point measurement was carried out using a flash point tester. The higher the flash point, the easier the fuel storage process. Figure 6 showed the flash point with temperature variations ranging from (86-93) ºC. This result has met the ASTM D7544-12 Standard, which has a minimum flash point value of 450ºC. The thing that can affect the magnitude of the flash point of a product is volatile matter. The less volatile biofuel content, the more difficult it is to ignite the product.

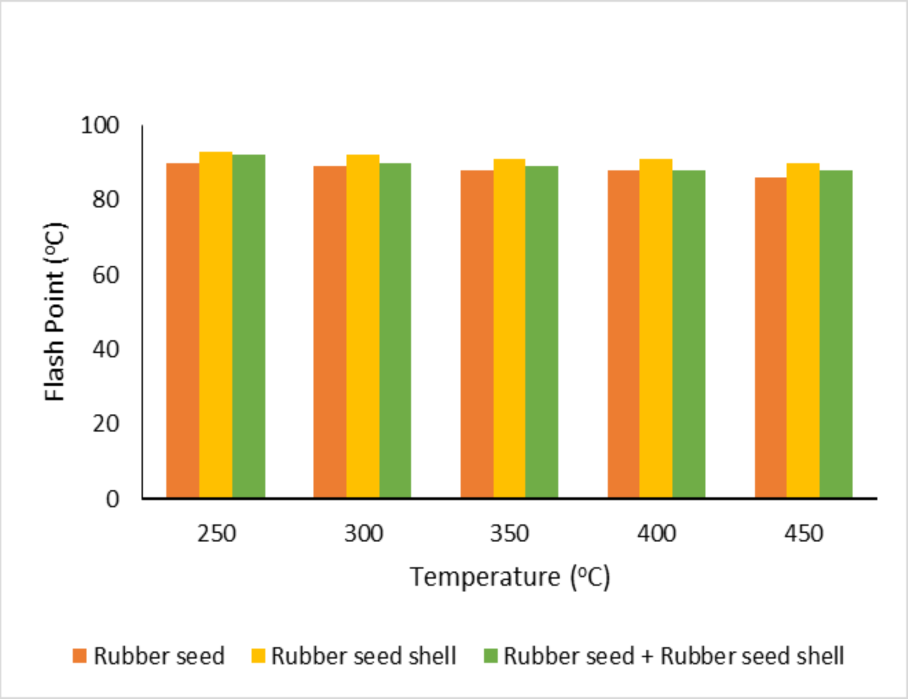


Figure 6. Temperature Influence on the Flash Point of Biofuel

3.6. Biofuel Calorific Value Analysis

Calorific value is the energy generated when a fuel is entirely burnt in a constant flow process. Calorific value is an essential criterion in assessing fuel quality. The calorific value obtained was 7451.399 Cal/g or 31.197 MJ/Kg. The calorific value has met the calorific value of the American Standard ASTM D7544-12, which is a minimum of 15 MJ/Kg. The temperature of the process along substantial solid residence time affects the calorific value of pyrolysis oil [46]. The more the fuel's calorie content, the more efficient the energy produced because it generates more heat with less mass. The high energy value reflected the carbon-to-hydrogen ratio in the Biofuel [47].

3.6. Biofuel Chemical Compound Fraction and Composition

In this study, the analysis of chemical compounds and biofuel composition using Gas Chromatography-Mass Spectrometry (GC-MS) was carried out on samples using 3% catalyst temperature of 450ºC. From testing using GC-MS, it is assumed that the analysis of chemical compounds and biofuel composition results represent the entire sample, as shown in Figure 7. The chromatography peaks showed the types of components contained in biofuel. It can be seen that the number of identified features reached 88 kinds of details, illustrated by the formation of 88 peaks. The results of the identification of these components were tabulated in Table 4. Biofuel from the biomass pyrolysis process contains a composition of hydrocarbon compounds with diverse C chains, as seen in Table 4. It can be seen that the resulting product has the C15-C18 chain as the dominant composition of 34.8%, where this chain defines the diesel fraction. In comparison, other compositions are gasoline, kerosine, 44.41% naphtha (C5-C15), 3.86% lubricating oil (C18-C20), 1.28% paraffin (>C20), 7.95% fatty acid compounds, and 5.78% other compounds. The presence of gasoline, naphtha, and kerosine (C5-C15) is due to the continuous cracking process so that more light hydrocarbon chains are produced. In addition, the content of C18-C20 (Lubricating Oil) and >C20 (Paraffin) was found to be low since the biomass had not fully cracked into the desired product, diesel with a range of C15-C18. The biofuel produced has no fractions < C5 because hydrocarbons below C5 were gas-phase hydrocarbons uncondensed, evaporating into the air.

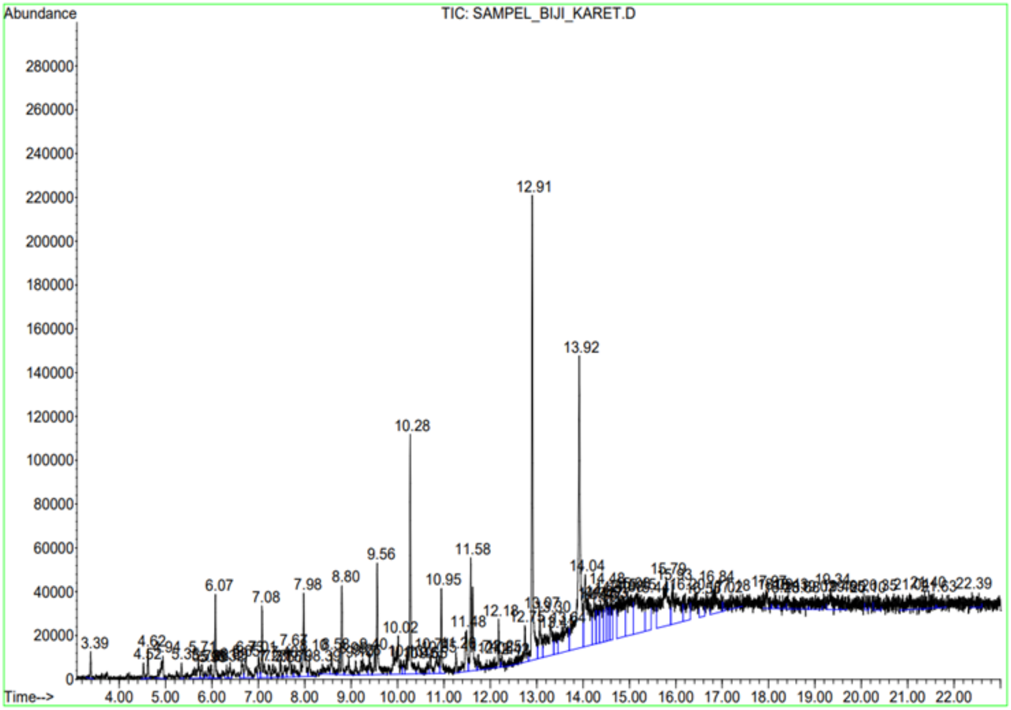


Figure 7. GCMS Graph of Rubber Seed-Based Biofuel

Table 4. Characteristics of Biofuel

Fraction	Component	Composition (%)
<C5	Gas	0
C5-C15	Gasoline, Kerosene, naphtha	44.41
C15-C18	Diesel	34.8
C18-C20	Lubricating Oil	3.86
>C20	Residue	1.28
CxHxOx	Metil ester, alcohol, fatty acid	7.95
-	Others	5.78

CONCLUSIONS

Utilizing the results, it is possible to deduce that the properties of rubber seed raw materials, for the physical properties of biofuels, were as follows: the optimum liquid product at a temperature of 450ºC obtained for rubber seeds received density (1.0646 g/mL), viscosity (5.417 mm^2/s), flash point (88ºC), moisture content (30.4%), calorific value (7451.3997 Cal/g), and cetane number (32.9). Based on the results of biofuel analysis using GC-MS, the C atom chain was dominated by C5-C15 compounds at 44.41%.

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