Natural rubber, a potential alternative source for the synthesis of renewable fuels via Hydrous Pyrolysis

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Abstract. Natural rubber is a humid agricultural harvest, which mostly contains hydrocarbon cis-1, 4-Poly isoprene. Through depolymerisation technology, the natural rubber can be changed into liquid product, and then it can be subsequently utilized as a fuel or chemical feedstock. This article aims to provide an outlook on the natural rubber and its sources, which are available globally. Numerous depolymerisation processes, which include pyrolysis, gasification, chemical degradation, catalytic cracking and hydrogenation, were introduced in this paper, while the focus of discussion was emphasized on the hydrous pyrolysis process. Many studies have shown that the use of hydrous pyrolysis able to improve the depolymerisation process, e.g. the raw material can be feed without drying, the process can be carried out at lower temperature, only the water is used as the reaction medium, and it is easy to separate the water from oil product. The effect of operating parameters such as temperature, water to rubber mass ratio, reaction time and type of gases on the product yield and composition were reviewed in this paper. In addition, this paper also highlighted the eco-friendly and economic viability of the hydrous pyrolysis process.

1. Introduction

Shortening of fossil fuels and their effects of greenhouse gas emission has become a very famous and leading problem from the last few years, which subsequently convinced the scientists to develop alternative fuels to substitute these fossil fuels. It is reported that the fossil fuels like gas, coal, and petroleum are available up to next 30, 96, and 29 years respectively [1]. The global consumption of natural gas is around 9,413.69 million m³/day and that of crude oil is estimated around 77.83 million barrel/day [2]. Because of this high consumption and depletion, the prices of fossil fuel are predicted to rise excessively in the next decades. In order to replace the fossil fuels, efforts are being made to invent alternate fuels which are effectual, environmental friendly and economically feasible. The environmental pollution has also become a main issue in the past decades globally due to the excessive use of fossil fuels. The direct combustion of fossil fuels has significantly increased the environmental

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complications due to the release of injurious contaminants such as SOx, NOx, CO₂ and hydrocarbons which consequentially caused global warming, acid rain, and ozone depletion. The utilization of fossil fuels is contributing to 62% of the global CO₂ emissions [3]. In this regard, the United States of America and China are the major emitters (approximately 44% of world’s emission) of the greenhouse gas emissions from conventional fuels. US Energy Information Administration (EIA) stated that, the U.S. had presently produced about 19% of global CO2 emissions [3]. It is expected that by the year 2030, the CO₂ emission levels will rise to 40 billion Mg/year [4], which will be a very shocking situation. Barbosé et al. proposed that to avoid and to combat these environmental issues, the consumption of fossil fuels needs to be minimized and some new renewable fuels would be deployed for their environmental and social benefits [5, 6].

Many alternative energy resources are available worldwide for the substitution of fossil fuels. The user employs fuels to generate mechanical work, power generation, and heat energy, for successive use in daily life. A perfect ideal fuel exhibits good properties such as high hydrocarbon contents, high heating value, low price, less moisture contents, combustion should be controllable, products of combustion shouldn’t be harmful, easy and less transportation and less storage cost. Therefore, an appropriate alternative fuel needs to fulfil the criteria of perfect or an ideal fuel. Several other renewable sources of energy like biomass, bio-diesel, solar energy, hydro-power, and wind-power are available as the substitute of the fossil fuels [3].

Natural rubber (NR) is a biomass generally extracted from Hevea rubber tree, guayule plant, Russian dandelion and rubber rabbit brush [7]. NR is also a unique available source that has a potential to meet upcoming energy requirements. NR is a vigorous, premeditated and exceptional feedstock used in huge quantities globally. It is essential and vital agricultural crop in tropical Asia and is found plentifully in south East Asian tropical countries and several central African countries. Hevea rubber tree cultivates finely in a warm humid climate [8]. NR is a polymeric compound of Isoprene, i.e. Poly-isoprene (C₅H₈)n with small quantity of impurities such as other organic compounds and moisture [7]. In the year of 1997, the worldwide manufacturing of NR was calculated about 5000 thousand metric tons, which is rising progressively on account of its high demand and utilization [3]. Because of its large availability, the NR has attracted the researchers to utilize it as a valuable raw material for the synthesis of valuable chemical commodities such as bio-fuels, fertilizers, and other chemical derivatives by depolymerisation process.

Depolymerisation is a process that decomposes the polymeric organic material into small molecules or into its corresponding monomers. Thermal decomposition (Liquefaction, Pyrolysis, hydrogenation, gasification), Photo-induced decomposition, Chemical decomposition (solvolysis, hydrolysis, ozonolysis, oxidation), and biological decomposition are very renowned depolymerisation processes. Thermal depolymerisation process (TDP), commonly known as hydrous pyrolysis is a suitable technique to convert NR to liquid fuels [9]. During hydrous pyrolysis, carbonaceous organic materials like plastics, rubbers or biomasses etc. are heated in the presence of water to produce valuable chemical commodities. This process has an advantage over the rest of depolymerisation techniques since it uses water that is found in abundance, low cost, and eco-friendly. Furthermore, the separation of product liquid oil from water is easy and inexpensive [10]. This conversion process has been employed on industrial scale to convert the agricultural organic waste into valued products like fuels, fertilizers, and other chemicals [9].

In the present article, the decomposition of NR via hydrous pyrolysis was reviewed. The influence of operating conditions such as temperature, pressure as well as water to rubber mass ratio, on the product quantity and quality were explained. In addition, some essential information associated to NR statistics and its alternative sources were also presented.
2. Natural rubber and its sources

2.1. Natural rubber

Typically, the rubber tree (Heveabrasiliensis tree), rubber rabbitbrush, sunflower, Guayule plant, Fig tree, Goldenrod, and Russian dandelion etc., are the sources to obtain NR. It is a hydrocarbon and also recognized as cis-1,4 Polyisoprene represented by chemical formula (C$_5$H$_8$)$_n$, the molecular weight of 100% cis -1,4 Polyisoprene which ranging from 1 to 2.5e10$^6$ [7, 8]. Rubber is obtained from the liquefied latex through coagulation technique which involves the addition of formic acid. The coagulum is a soft solid slab that is pressed by series of rollers to expel an excessive amount of water and to increase the surface area. After that, smoke ovens are employed to dry the rubber sheets.

Table 1. The available sources of natural rubber [7].

| Rubber Source | Rubber MW (KDa) | Production (Tones/Year) | Rubber Contents (%) | Yield (kg ha$^{-1}$ year$^{-1}$) | Comments |
|---------------|----------------|------------------------|---------------------|---------------------------------|----------|
| Rubber tree H. Brasiliensis | 1,310 | 9,000,000 (2005) | 30 to 50 in latex. 2% of tree dry weight | 500 to 3000 | Usually ripeness period of rubber tree is 6 years. It can live over 100 years, but it is cut mostly after 30 years because tapping reduces its productivity of latex, rebirth of latex takes a few days depending upon the condition of the tree. |
| Guayule shrub P. argentatum Gray | 1,280 | 10,000 (1910) | 3 to 12 of plant | 300 to 1000 | 2-5year production time and regeneration time is 12 to 18 months. |
| Russian dandelion T. kok-saghyz | 2,180 | 3000 (1943) | 0 to 15 of root | 150 to 500 | Its plantation is usually done in early spring. Its takes approximately 85 to 95 days to get mature. Rubber rabbit brush reaches ripeness in 2 to 4 years. It has a life period of 5 to 20 years. It harvests seed at the age of 2 years or more than that. |
| Rubber rabbitbrush C. nauseous | 585 | n.a | less than 7 of plant | n.a | |
| Goldenrod S. virgaurea minuta | 160-240 | n.a | 5 to 12 of root dry weight | 110 to 155 | Low-quality rubber producer. Demonstration Project in 1931. |
| Sunflower Helianthus sp. | 279, 69 | Research stage | 0.1 to 1 of plant | n.a | It contains low molecular weight rubber. |
| Fig tree Ficus carica | 190 | n.a | 4 in latex | n.a | It’s R and D is related to biochemistry. |
| Lettuce Lactuca serriola | 1,380 | Research stage | 1.6 to 2.2 in latex | n.a | It’s R and D is related to genetic engineering and characterization. |
Hydrous pyrolysis of rubber tire was conducted in a 180 mL stainless steel batch reactor using supercritical water as media.

- **Temperature**: 450°C
- **Pressure**: 28 MPa
- **Reaction time**: 47.6 and 42.9% respectively
- **Oil yield**: 44.09 MJ/kg, 62.49%, 86.66%
- **Carbon content**: 44 wt.%, 58%, respectively
- **Energy recovery**: 86.66%
- **Calorific value**: 200°C to 430°C
- **Hydrogen and carbonKA0129-899X/334/1/012004

### Table 2. Various reported investigations on hydrous pyrolysis of rubber.

| Ref  | Materials | Process parameters | Product analysis | Comments |
|------|-----------|--------------------|------------------|----------|
| [10] | Rubber tire | | | Pyrolysis efficiency of used rubber tires and the effect of temperature and pressure on sub-critical/super-critical water were studied. Products were analysed by GC, Mass, FTIR, and elemental analyser. |
| [14] | Natural rubber | | | Autoclave as a batch reactor was used to perform the thermal degradation of rubber tire (containing 52.2% wt% natural rubber) by using super-critical water as a pretreatment. Although the magnitude of pressure of super-critical water is higher than the super-critical pressure (critical pressure is 3.57 MPa), water at super-critical condition was as effective as super-critical pentane. |
| [16] | Rubber tire | | | This research mainly aimed to study the effect of temperature and different experimental conditions in hydrous pyrolysis on the chemical composition of products from scrap tires. |
2.2. Natural rubber production statistics and its sources
In 2015, the worldwide production of NR was approximately twice from the year 2000 to 2014. In 2000, it was estimated 6,811 thousand metric tons and in 2014, it was reported to be 12,070 thousand metric tons [11]. In 2000, the global NR consumption was calculated approximately 7,108 thousand metric tons globally [12], and it was increased to 12,159 thousand metric tons in 2014 [11]. Thailand, Indonesia, and Malaysia are the main manufacturers of NR. In 2000, the NR production in Malaysia was reported to be 927,608 tons. Whereas, in the year 2014 the production of NR was estimated about 668,613 tons [11]. In the year 2014, the utilization of NR in Chain, India, USA, Thailand and Malaysia was valued to be 4760, 1015, 932, 541 and 447 tons respectively [13]. Food and Agriculture organization reported that, the production of NR in Indonesia was improved from 1,792.35 to 3,108 metric tons in 2003 and 2013 respectively [12]. The accessible sources of NR are mentioned in Table 1.

3. Hydrous pyrolysis
Numerous methods, like chemical decomposition, pyrolysis, catalytic cracking, hydrogenation, gasification, and hydrous pyrolysis are available to convert the NR into fuels and beneficial products. Among them, hydrous pyrolysis is the most feasible technique for the conversion of organic material (rubbers, biomasses, plastics etc.) into liquid fuels. It is conducted in the presence of water at elevated temperature and pressure conditions which outcomes in the degradation of long-chain polymeric compounds into smaller chain petroleum based products (monomers) [9]. This process is performed in water at elevated temperature (250 to 400°C) and elevated pressure (4 to 22MPa). This process can also be carried under self-generated pressure. The main advantage of this process is that, it can utilize raw material with high water contents, thus no need for drying. Generally, the products of hydrous pyrolysis are liquid-oil, solid residual, and gases. However, the yield of liquid oil achieved is high as compared to other by-products. Furthermore, the oil produced by hydrous pyrolysis shows similar characteristic as naturally occurring crude oil [10]. Although the hydrous pyrolysis process gives high value of liquid yield, the quality of liquid still depends upon the type of raw material used e.g., the oil obtained from scrap tire has good heating value but it also contains high sulfur.

3.1. Utilization of natural rubber in hydrous pyrolysis
There is not much research available on the conversion of 100 percent pure NR into fuel. However, some literature is available on the conversion of tyre rubber (where tyre consists of 40-52.2 wt. % of rubber [14]) using hydrous pyrolysis. Several investigations have been made on the hydrous pyrolysis of tyre rubber to evaluate the effects of different operating parameters for the synthesis of valuable liquid products [10, 14-17].

There are many parameters that affect in hydrous pyrolysis process such as size of particle, residence time, water to rubber mass ratio, heating rate, and reaction atmosphere. In addition, the quality of raw material also has a significant influence on the final product, however, the temperature is found to be the most effective parameter that has an influence on the product quantity and quality. Studies on the hydrous pyrolysis of rubber are shown in Table 2.

3.1.1. Use of water and its effect. During hydrous pyrolysis of rubber tyre, water is utilized as subcritical and supercritical conditions for the decomposition of variety of materials [10]. When temperature of the water is more than its atmospheric boiling point (100°C) but lower than its critical temperature (374°C) in that case the water is in sub condition. However, when temperature as well as pressure of water is equivalent to or more than 374°C and 22.1MPa respectively, it is called as supercritical condition[18]. Supercritical water is extra reactive than subcritical water as at supercritical condition the water transform its structure and most of the hydrogen-bonding associations are fragmented, thus changing the polar forces of attractions into dipole-dipole attractions, which
consequently reduces the dielectric constant of water [19, 20]. Besides water, the degradation of rubber tyre can be carried out using other various compounds. However, water is the most suitable solvent for the decomposition, which is not only eco-friendly but also cost effective and easily accessible.

Several studies have been done to use water at subcritical and supercritical conditions for the depolymerisation of rubber tyre [14, 16, 21-23]. Rubber is decomposed almost completely in supercritical water as compared to the subcritical water and the formation of char is prohibited [23]. The amount of water has an influence on the liquid product yield. Zhang et al. observed that the yield of liquid products rose as the water to rubber tyre mass ratio increased during hydrous pyrolysis of rubber tyre. The liquid products yield was improved from 45.9 to 52.9 wt.% when water/rubber tyre mass ratio was increased from 1:1 to 4:1 at 390°C. Furthermore, the large quantity of water increases the nucleophiles concentration, which subsequently elevates the pressure inside the reactor. This is poor condition for gas formation [10]. Similar studies are shown in Table 2.

3.1.2. Influence of temperature. Several investigations have been made to study the influence of temperature on hydrous pyrolysis products. Zhang et al. performed the hydrous pyrolysis of rubber tyre under a temperature range of 200 to 430°C. It was observed that the temperature had high influence on the product quantity. The liquid yield increased from 13.5 wt.% to 52.7 wt.% due to rising in temperature from 200 to 370°C. A further rise in temperature to 400°C did not show any significant increase in the liquid product yield. However, the decrease in liquid yield product was observed at a temperature above 400°C. This decline in yield was recognised due to the decomposition of oil into light fractions and volatiles [10].

Park and Gloyna conducted the depolymerisation of rubber tyre in an autoclave using supercritical water. The maximum oil yield was achieved at 400°C. However, the oil yield reduced within temperature range of 400°C to 450°C due to the transformation of oil into light compounds and volatiles by thermal depolymerisation. At a temperature of 450°C, the conversion of rubber was 5 to 6% greater than that obtained at 400°C. The highest conversion and oil yield of rubber tyre was reported to be 58% and respectively 44 wt.% at 400°C [17]. Same investigations that have been made on rubber are shown in Table 2.

3.1.3. Influence of gas atmosphere. Several gas atmosphere or environment can be adopted to conduct hydrous pyrolysis of rubber such as He, N₂, CO, CO₂, and H₂. However, it was reported that gas atmosphere had very little effect on final product yields [10]. Zhang et al. performed the hydrous pyrolysis under various atmosphere gases such as air, CO₂, CO, H₂ and N₂. The authors observed that gas environment didn’t effect or enhanced the liquid yield. The liquid products obtained with CO and H₂ atmosphere showed the high proportion of light fractions as compared to CO₂ or N₂ [10]. Also, Park and Gloyna carried out the hydrous pyrolysis of rubber tyre under air and He₂ atmosphere. It was found that the gas atmosphere was the second best significant operating variable after temperature that affected the liquid yield. However, atmosphere gas showed very little influence on the oil yield. The usage of He₂ as an alternative of air was more adequate for the recovery of oil. The highest oil yield obtained was 44 wt.% at 400°C under He₂ environment (non-reactive atmosphere). The usage of air as an atmosphere gas affects in the oxidation reaction that enhances the volume of discharge gas [17].

3.1.4. Influence of residence time. Various studies also have been made on the effect of residence time on product yield and composition in hydrous pyrolysis. Zhang et al. investigated the influence of residence time from 20 to 120 minutes at the temperature of 390°C. It was observed that at 390°C the residence time of 20 minute was adequate for the decomposition of rubber tyre and the liquid product yield obtained was 40 wt.%.

Further increase in reaction time to 60 minute with slightly increase in temperature enhanced the liquid product yield to maximum of 53 wt.% [10]. On the other hand, Chen et al. showed that the residence time could be used to control the extent of depolymerisation, which was subsequently useful to control the molecular mass for the synthesis of useful chemicals. The
decrease in molecular mass was observed throughout the time interval of 30 to 180 minutes [16]. Similarly, Funazukuri et al. also described, the decrease in molecular mass was observed as the function of time at 380°C and 22 MPa [14]. Zhang et al. found that high residence time led to low N and S contents in the liquid product [10].

3.2. Characteristics of products
The various investigations have been made on the characteristic of products obtained by pyrolysis and hydrous pyrolysis of rubber. Zhou et al. stated that at optimum conditions, HHV of oil achieved was 44.09-45.09 MJ/kg [10]. The HHV achieved was very similar to petroleum obtained from crude oil like diesel (44.8MJ/kg). Zhou et al. reported, the ultimate analysis of oil product obtained at optimum conditions contained C and H contents of 85.96wt% and 11.9wt% correspondingly [10]. Also, Funazukuri et al. performed the elemental analysis of oil products consisting about 85wt.% and 10wt.% of carbon and hydrogen contents at 380°C and 22MPa [14]. For solid products, Funazukuri et al. reported the elemental analysis of solid residue consisting about 90.9 wt.% and 1.49 wt.% of C and H contents at 380°C and 22MPa [14]. Also, Yi et al. described that the C contents were high in solid residual. The mean value of C contents were estimated greater than 80wt.% [23]. Yi et al. studied that most of the product gas were comprised of CH₄, H₂, CO, CO₂ and hydrocarbons with lower molecular weight. The concentration of CH₄ and H₂ was high in the product gas. The yield of CH₄ increased between the temperature range of 400 to 550°C. At 500°C & 22.2MPa, CH₄, H₂ and CO₂ were approximately 38, 38 and 16%. NOx and SOx were not produced in gas products [23]. Chen et al. conducted the NMR study of the oil products, which showed the presence of aromatics, olefin and aliphatic groups. The reported composition of HC, aromatic rings, and olefins in products were 80.9 wt.%, 18.8 wt.%, and 0.39 wt.% correspondingly at 380°C and 27.6MPa [16]. Similar investigations made on the characteristic of products obtained by the hydrous pyrolysis can be seen in Table 2.

3.3. Environment and economical viability
Hydrous pyrolysis is a feasible and eco-friendly technique for the conversion of waste into useful liquid fuels. Liquid oil products, gas products and solid residue are three major products that obtained in hydrous pyrolysis. In hydrous pyrolysis of rubber tyre, most of the S and N contents are taken by solid residue thus considerably reducing the SOx and NOx emission in gas products [16, 17, 23]. However, further developments are essential for the decrease of N and S contents in the solid residual. Similarly, for liquid products little developments are required for the reduction of N and S contents [10]. Hydrous pyrolysis process of rubber tyre gives high quality and quantity of oil products and this method can play a vital role in the development of energy industry. It has been reported that the hydrous pyrolysis of rubber tyre is a cheap method to transform rubber tyre into fuel [15]. Normally, the pyrolysis of rubber tyre is carried out at elevated temperatures above 400°C, which consequently increases the operational cost of pyrolysis. Therefore, the investigations have been made to optimize this process using H₂O below 400°C. Rushdi et al. have studied the economic feasibility of the hydrous pyrolysis of rubber tyre and it was shown that the yearly profit of $1.19–1.77M could be made by the hydrous pyrolysis of 30MT rubber tyre/day. This process can be made extra profitable by utilizing solid residual and product gases through process upgrading [15].

4. Conclusions
NR is one of the perfect alternative sources to substitute fossil fuels, which can potentially resolve the issues of energy crisis. The review article showed that there were several methods available for the decomposition of NR such as pyrolysis, chemical decomposition, gasification, catalytic cracking, hydrogenation and hydrous pyrolysis. Among them, hydrous pyrolysis of NR was found to be a low cost process because it gave high liquid yield at lower temperature conditions. From literature survey, it was seen that most of the research work have been done on rubber tyre. The highest liquid yield of about 44 wt.% was achieved under optimal conditions, which were shown to be at temperature of 400°C, a H₂O to rubber mass ratio of 2:1, and a residence time of 60 minutes. Additionally, this
technique could utilize a raw material with high H₂O contents without the need for drying. Furthermore, the usage of H₂O as a reactive medium has attained much attention since it makes easy on the separation of H₂O from oil products and much cost-effective as compared to other organic solvents. For environmental concern, it is important to note that during the hydrous pyrolysis of rubber tyre the NOx and SOx were not obtained in the gas product. In addition, the usage of pure NR as a raw material is eco-friendly as pure NR doesn’t contain S contents. A very little literature is available on the hydrous pyrolysis of NR so, a lot of works are needed to be performed in the future on the hydrous pyrolysis of NR to evaluate the effect of parameters on product yield and composition. Moreover, the NR is more environmentally friendly as compared to rubber tyre.

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