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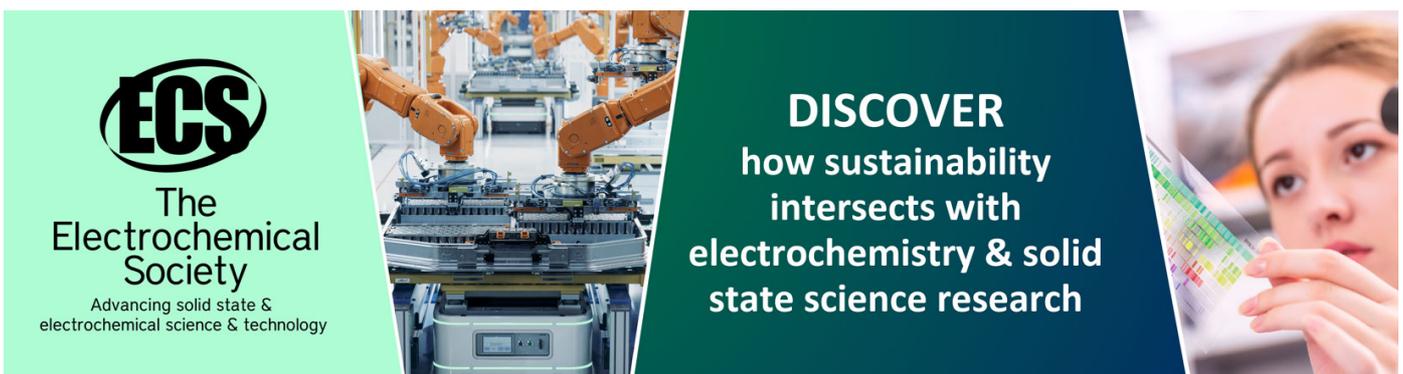
Enhancing the properties of Fischer-Tropsch fuel produced from syngas over Co/SiO₂ catalyst: Lubricity and Calorific Value

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Enhancing the properties of Fischer-Tropsch fuel produced from syngas over Co/SiO₂ catalyst: Lubricity and Calorific Value

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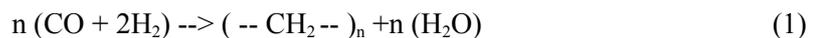
Abstract. Bio-fuel produced from renewable sources is considered the most viable alternatives for the replacement of mineral diesel fuel in compression ignition engines. There are several options for biomass derived fuels production involving chemical, biological and thermochemical processes. One of the best options is Fischer Tropsch Synthesis, which has an extensive history of gasoline and diesel production from coal and natural gas. FTS fuel could be one of the best solutions to the fuel emission due to its high quality. FTS experiments were carried out in 16 different operation conditions. Mini structured vertical downdraft fixed bed reactor was used for the FTS. Instead of Biomass gasification, a simulated N₂-rich syngas cylinder of, 33% H₂ and 50% N₂ was used. FT fuels products were analyzed in GCMS to find the hydrocarbon distributions of FT fuel. Calorific value and lubricity of liquid FT product were measured and compared with commercial diesel fuel. Lubricity has become an important quality, particularly for biodiesel, due to higher pressures in new diesel fuel injection (DFI) technology which demands better lubrication from the fuel and calorific value which is amount of energy released in combustion play very important role in CI engines. Results show that prepared FT fuel has desirable properties and it complies with standard values. FT samples lubricities as measured by ASTM D6079 standard vary from 286µm (HFRR scar diameter) to 417µm which are less than limit of 520µm. Net Calorific value for FT fuels vary from 9.89 MJ/kg to 43.29 MJ/kg, with six of the samples less than EN 14213 limit of 35MJ/kg. Effect of reaction condition on FT fuel properties was investigated which illustrates that in higher pressure Fischer-Tropsch reaction condition liquid product has better properties.

1. Introduction

In previous years, growing concerns about the fast depletion of fossil energy resources and need to reduce greenhouse gas emissions make renewable energy sources much more attractive. The Energy concerns in the world and economic growth in the developing countries are on the drastically rise [1-3] so the energy demands in the field of transportation and manufacturing have increased [1, 2]. Rudolph Diesel, the inventor of the first diesel engine, used vegetable oil (groundnut oil) as fuel in 1900 [4, 5]. His motivation was to improve efficiency and also to down size the price and costs of engines, compared to huge steam engines, so that smaller companies would be able to afford the machinery [6].



During a demonstration he said, “The use of vegetable oils for engine fuels may seem insignificant today, but such oils may become, in the course of time, as important as petroleum and the coal tar products of the present time.” [7]. Ethanol fuel from sugar cane was implemented in Brazil after the 1970’s oil crisis, however the advantages began to disappear due to the enhancement of ethanol fuel costs in 1989 [8]. Over the past few decade, there has been a surge in global warming and climate change which has brought about by fossil fuels, mainly petroleum-based liquid fuels, natural gas and coal [9]. This has renewed interest in biofuels from vegetable oils as alternative fuel more than 100 years after the time of Rudolph Diesel’s predictions [4-6]. There are several options for biomass derived fuels production involving chemical, biological and thermochemical processes. Two of the most promising fuels appear to be biodiesel or synthetic fuels such, as Fischer-Tropsch diesel [10]. This is because other potential fuels, such as Ethanol, Methanol and LPG, do not perform as well in modern engines. In addition, in the near future biomass is expected to play an important role and it would be one of the important renewable energy sources [3]. An option for production of renewable fuels from gasified biomass is the Fischer-Tropsch Synthesis (FTS). The FTS is considered as an efficient solution to the problem of finding appropriate substitutes for liquid fossil fuels [11, 12]. Fischer-Tropsch Synthesis is a technology that has an extensive history of gasoline and diesel production from coal and natural gas. The fuel produced by using FT synthesis could be one of the best solutions to the fuels emission due to its high quality. FTS technology devised nearly 90 years ago by Franz Fischer and Hans Tropsch in Germany. Equation 1 represents the main reaction in FTS in which Syngas (Hydrogen and Carbon monoxide) is converted to long chain Hydrocarbons over cobalt catalysts during the reaction [13].



FTS is a surface polymerization reaction in which the reaction between CO and H₂ takes place on the surface of the cobalt catalysts. This process is described as a Carbide formation on the surface of the catalysts, which is discovered, by Fischer and Tropsch nine decades ago. They assumed that the carbide carbons are decomposed by hydrogen with regeneration of the catalysts metal and can be transformed to hydrocarbons. CH₂ entities are formed on the surface of the catalyst and arranged in a row on the surface, which implies the carbide mechanism. Another assumption is freely movement of CH_x units along the catalyst [13, 14]. Two other reactions occur after a hydrocarbon molecule is released from the catalyst surface and reabsorbed to follow other reaction paths. Methane formation as an unwanted product in FTS is shown in the equation 2 and 3 in which are considered as an irreversible reaction. The other important reaction which occurs in FTS process is water gas shift reaction that produces water as co product; this reaction play vital role in reactors which reaction take place over cobalt catalysts and produce carbon dioxide as an undesirable product [15].



Catalysts, reaction conditions, type of reactor, H₂/CO ratio influence on the type of products in FTS [13]. FTS process over cobalt catalyst at normal pressure and temperature of 200-300°C produces linear α olefins C_nH_{2n} as the main products and the small amount of nonlinear products which contain mono methyl branch compounds and also it was considered that by increasing the residence time in reactor, which leads to secondary reactions with the same skeleton of both products, subsequent hydrogenation of olefins forms paraffin C_nH_{2n+2}. High pressure Fischer Tropsch Synthesis over cobalt catalyst produces less olefin in favour of Alkane C_nH_{2n+2} content due to increase of the molecular weight[14].

2. Experiments Methodology

2.1. Fixed Bed Reactor system and operating procedures

Figure 1 shows the schematic diagram of the experimental set-up designed and build by H. Mahmoudi [1] that was conducted in a fixed bed reactor. The experimental work of this project

concentrated on developing a miniaturized version of the plant that could achieve the preliminary investigation regarding the F-T process, before scale up to pilot plant and building of a pilot scale bio-fuel generator. For this purpose, a small scale F-T bio-diesel generator via the Fischer-Tropsch synthesis process was designed, built and commissioned in a laboratory of the School of Mechanical Engineering of the University of Birmingham. A mini structured downdraft fixed-bed reactor was employed in order to find the optimum reaction conditions for maximum production of synthetic fuel, as well as to convert the syngas into the synthetic bio-fuel [1]. Experiments were carried out in 16 different operation conditions that indicated in Table 1. The reactor was fixed in a tube furnace in order to provide the heat temperature and to provide the uniform wall temperature; cast iron jacket was installed between the furnace and reactor. Instead of Biomass gasification, a simulated rich syngas bottle of 17% CO, 33% H₂ and 50% N₂ was used [1, 2, 16]. Two liquid/gas separators were used to separate liquid from gaseous products. A Gas Chromatogram Flame Ionization detector (GC-FID) (HP5890) was employed to analyse the HC₁ – HC₈ online. The heavier liquids products were analyzed offline by using Gas Chromatogram Mass Spectrometry (GC-MS) (Perkin Elmer Clarus 600). Utilization of nitrogen-rich syngas (with 50% of N₂ (volumetric percentage)) leads to reduction in production cost of diesel oil by eliminating the need for application of gas recycling loop after production of syngas by air partial oxidation. Waste generated heat of FT reaction could be removed effectively and temperature runaway probability can be minimized by nitrogen gas [1].

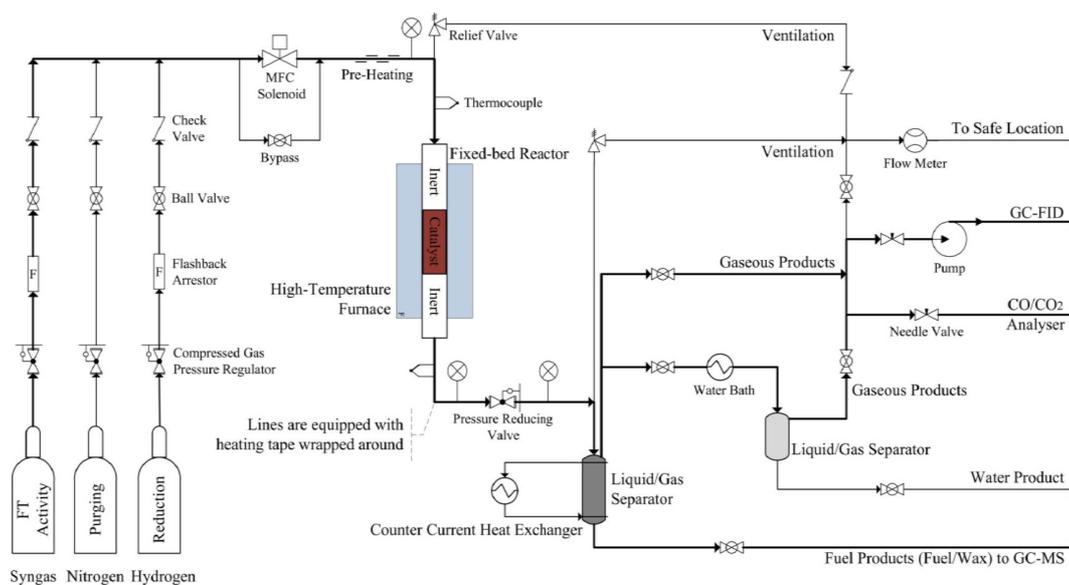


Figure 1. Schematic diagram of small scale bio-diesel generator via Fischer-Tropsch Synthesis (FTS) process and utilized equipment along with Process Path Flow (PPF) implementation conversion of synthesis gas into the liquid heavy by hydrocarbons (Shown with thicker lines) [1].

2.2. F-T Fuel Characterization

Many researchers state that the most important characteristics for fuel application are density, viscosity, heating value and cetane number due to their strong control over emissions characteristics and engine efficiency, ultimately indicating the quality of the fuel [8]. Literature was examined to find key physicochemical properties for biodiesel and synthetic diesel produced by either transesterification or Fischer-Tropsch. In this paper Lubricity and Heat of Combustion of FT fuel was investigated.

2.2.1. F-T Fuel Composition-Gas Chromatography Analysis. Gas Chromatogram Mass Spectrometry (GC-MS) (Perkin Elmer Clarus 600) used in this study for liquid hydrocarbon products analysis which were collected in a cold trap cooled externally at 283 K using a counter current heat exchanger. The product distributions were analyzed off-line employing a DBI column combined with Gas

Chromatogram Mass Spectrometry (GC-MS). A 1 μL portion of each sample containing the mixture of gasoline, diesel and waxes was injected into the GC with a split ratio of 34:1. Non-reactive inert helium gas was used to carry the gases' samples through the instrument. The injection port was heated to 573 K; the oven temperature was increased at 8 K/min from 303 K to 493 K and held at this temperature for 5 minutes. For liquid samples, the qualitative analysis was performed to identify the constituents (elements of functional groups) [1]. 16 samples of FT fuels were analyzed and total values and percentage of paraffin, iso-paraffin, olefins and alcohols within each of the gasoline ($\text{C}_7\text{-C}_{11}$), diesel ($\text{C}_{12}\text{-C}_{22}$) and waxes (C_{23+}) were calculated.

2.2.2. Lubricity. Lubricity has become an important quality, particularly for biodiesel, due to higher pressures in new diesel fuel injection (DFI) technology which demands better lubrication from the fuel [17]. Lubricity is simply the ability of the fuel to reduce friction between moving parts within the engine to help it run smoothly. Supposedly, as much as 30% of mechanical energy is consumed by friction [18, 19]. This makes fuel lubrication vital to reduce scarring of components inside the engine. If the fuel does not contain enough lubricating ingredients, it is considered as a "dry fuel" for its incapacity of lubricating the components like fuel delivery and injection system, cylinder liners, etc. [18]. To assess lubricity a tribological test can be performed which measures the size of the wear mark in an HFRR (high frequency reciprocating rig) test [19]. The smaller the wear scar the better the lubricating properties of the fuel, and they must comply with ASTM D6079 maximum value of 520 μm .

Assessment of the lubrication properties of FT fuel was carried out on HFRR (PCS Instrument Ltd) according to the EN ISO 12516-1:2006 standard [20]. This is shown schematically in Figure 2a. The test specimens comprised of 6mm diameter steel ball and steel disc. Fuel temperature maintained at 60 $^{\circ}\text{C}$ and the volume of the sample used set at 2ml. A humidity and temperature controlled cabinet was employed to provide the laboratory air conditions defined by the standard as shown in Figure 2b. These conditions are defined according to the ISO standard defining air conditions are defined according to the ISO standard defining air conditions for testing diesel fuel lubricity [20]. During the test the disc was fully submerged in the tested samples at a reciprocation frequency of 50 Hz lasting 75 min. Optical microscopy (with a 100x magnification lens) was used to assess the size of the wear scar on the upper specimen again to the procedure defined by the ISO standard (including a correction factor to account for standard 1.4 kPa water vapour pressure). All of the lubricity experiments were repeated twice and if a difference in corrected wear scar diameters obtained from the same fuel was higher than 20 μm [21].

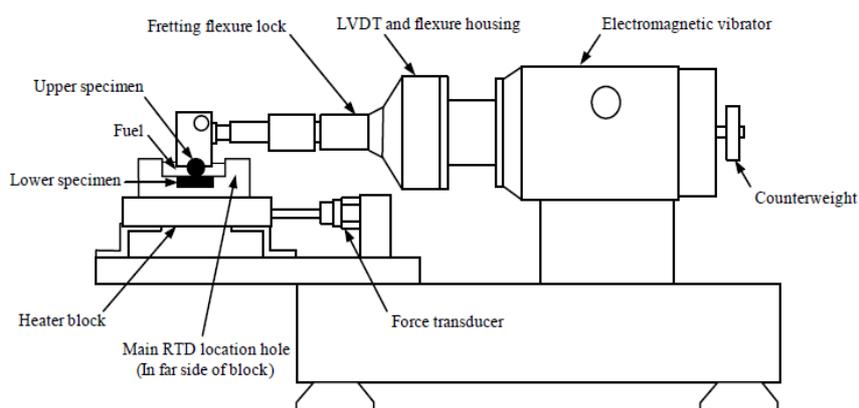


Figure 2a. Schematic diagram of high frequency reciprocating rig (HFRR) [22].

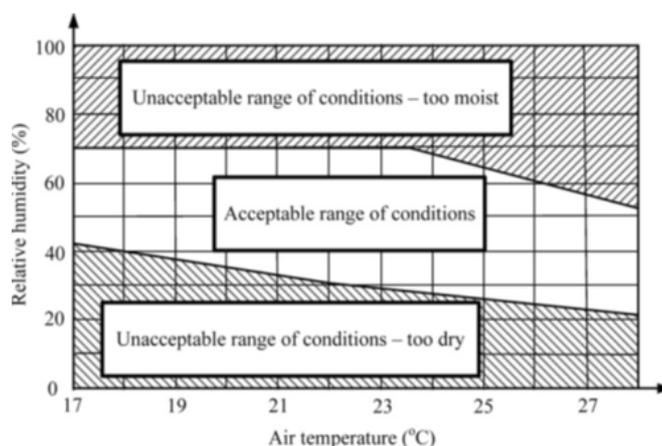


Figure 2b. Laboratory air conditions [23].

2.2.3. Heat of Combustion(Heating Value, Gross Calorific Value). One of the very important factor in the fuel economy and power deliverability is the calorific value of a fuel. The presence of oxygen in fuel improves combustion properties and emissions but reduces the calorific value. A fuel heating value is a gauge of the amount of thermal energy it releases during its burning and is an influential factor in the fuel economy and power deliverability [24]. There are several values to consider when concentration on the available energy within a fuel, the lower heating value (LHV-net) and the higher heating value (HHV-gross) included. These differ, as the lower HV does not include energy in the combustion of water vapour, whereas the higher does. The net heating value is the appropriate quantity for comparing fuels as the engine exhaust water in gas phase [25]. A diesel engine desires fuels of higher calorific value because it facilitates the heat release during combustion and improves engine performance during combustion [4, 9].As was stated earlier a high heating value is desired, though a minimum value is not specified in the biodiesel standards ASTM D6751 but is prescribed in EN 14213 at 35 MJ/kg [26].

IKA C200 oxygen bomb calorimeter according to the ASTM D240 standard was carried out to measure heating value of the FT fuels. The use of gross or net calorific value varies with type of industry. Engine manufacturers use net calorific value and UK boiler manufacturers use gross [27].Heat of combustion is determined in this test method by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions. The heat of combustion is computed from temperature observations before, during and after combustion with proper allowance for thermochemical and heat transfer corrections. Gross calorific value is gathered from instrument. If the percentage of hydrogen in fuel sample is not known, the net heat of combustion may be calculated as follows[28]:

$$Q_n = 10.025 + (0.7195)Q_g \quad (4)$$

where:

- Q_n Q_n = Net heat of combustion at constant pressure, MJ/kg,
- Q_g Q_g = Gross heat of combustion at constant volume, MJ/kg.

3. Results and Discussion

3.1. F-T Fuel Composition-Gas Chromatography Analysis

Hydrocarbon distribution of FT synthesis on liquid phase of products measured and analysed by PerkinElmer Clarus 60 Gas Chromatography Mass Spectrometry. GC analysis is performed for identifying the hydrocarbon compounds. Table 1 indicates the total values and percentage of paraffin, iso paraffin, olefins and alcohols within each of the gasoline (C₁-C₁₂), diesel (C₁₂-C₂₂) and waxes (C₂₃₊) which were calculated by analysing the result of GCMS results.

Table 1. F-T Fuel Characterization.

FT Sample	Fischer Tropsch Experiment Reaction Conditions			W _i Liquid Product (%)			Composition of products in Diesel Fraction (%)			FT Characterization		
	Temperature (K)	Pressure (bar)	(NL/gramWHSV)	W (C ₇ -C ₁₁)	W (C ₁₂ -C ₂₂)	W (C ₂₂₊)	Paraffin	Olefin	Alcohol	Gross Calorific Value (MJ/kg)	Net Calorific Value (MJ/kg) % H unknown	Lubricity (μm)
FTS 1	503	10	1.8	17.66	81.35	0.99	97.4	2.18	0.42	18.8	13.54	389
FTS 2	503	15	2.4	21.06	77.55	1.39	92.83	4.63	2.53	56.95	40.99	342
FTS 3	503	20	3	17.52	81.53	0.95	92.27	3.39	4.34	51.45	37.03	403
FTS 4	503	25	3.6	20.69	78.19	1.12	90.42	4.5	5.08	48.33	34.78	360
FTS 5	518	10	2.4	26.64	71.96	1.4	98.11	1.1	0.79	49.37	35.53	296
FTS 6	518	15	1.8	21.12	78.08	0.08	98.68	0.81	0.51	29.15	20.98	346
FTS 7	518	20	3.6	21.98	76.53	1.49	95.42	2.47	2.11	50.9	36.64	339
FTS 8	518	25	3	14.91	81.7	3.39	96.78	2.04	1.18	51.8	37.28	417
FTS 9	528	10	3	32.76	66.76	0.48	97.11	1.82	1.07	36.2	26.06	286
FTS 10	528	15	3.6	24.35	70.89	4.76	97.7	1.84	0.46	54.13	38.96	294
FTS 11	528	20	1.8	23.58	75.14	1.28	98.35	1.22	0.43	43.76	31.5	336
FTS 12	528	25	2.4	21.56	76.78	1.66	92.83	3.59	3.58	50.62	36.43	340
FTS 13	543	10	3.6	24.85	73.09	2.06	97.45	2	0.55	13.75	9.89	320
FTS 14	543	15	3	25.035	73.93	1.04	98.96	0.57	0.48	38.16	27.47	330
FTS 15	543	20	2.4	17.62	80.12	2.26	96.35	2.73	0.92	60.14	43.29	372
FTS 16	543	25	1.8	24.59	73.84	1.57	98.09	1.4	0.51	57	41.02	325

3.2. Lubricity

The tribological lubricity results for 16 Fischer Tropsch samples are gathered in Table 1. Wear Scar Diameter varies between 286 μm to 417 μm. From figure 3a, it can be seen that the change of FT reaction conditions effect on the lubricity of the fuels. Increasing the pressure affects the poor lubricity property and in high temperature lubricity is better. WHSV does not have significant effect on lubricity property. By validating the result with Mahmoudi [1] which stated that optimum condition for fuel production via FTS is high pressure reaction condition, it can conclude that lubricity in higher pressure indicates poor quality.

Hydrocarbon distribution of F-T synthesis on liquid products which were measure by GCMS as a function of the lubricity in figure 3b represents that decrease in production of the olefins and gasoline products (C₇-C₁₁) could increase the lubricity by controlling the initiation, growth of carbon chain and enhancing the chain termination. From figure 3b it can clearly see that in heavier hydrocarbons by increasing the percentage of the heavier hydrocarbons in product lubricity increases. So by increasing value of the Diesel in liquid product lubricity become poor. In contrast presence of the light hydrocarbons improves lubricity of the F-T fuel. Figure 4 clearly states that all fuel samples are below the ASTM D6079 Maximum limit (520 μm) with comparison of the Ultra-Low Sulphur Diesel fuel which was tested in the same condition by HFRR. This contradicts Alleman et al. [29] and Norton et al. [30] who stated that the lack of aromatic content and Sulphur in FT diesel produces unacceptable lubricity fuels. Sulphur compounds in diesel fuels create some natural lubricity which is counterproductive to current aims to reduce the Sulphur content and pollutants in fuels. Additives could be a solution to this and it is thought that biodiesels from transesterification (FAME) can have superior lubricity than other alternative fuels. Best Lubricant fuel is FT 9 which is in low pressure of

10bar and temperature of 528K, however poor lubricant fuel is FT 8 with reaction condition of high pressure 25bar and low temperature 518K.

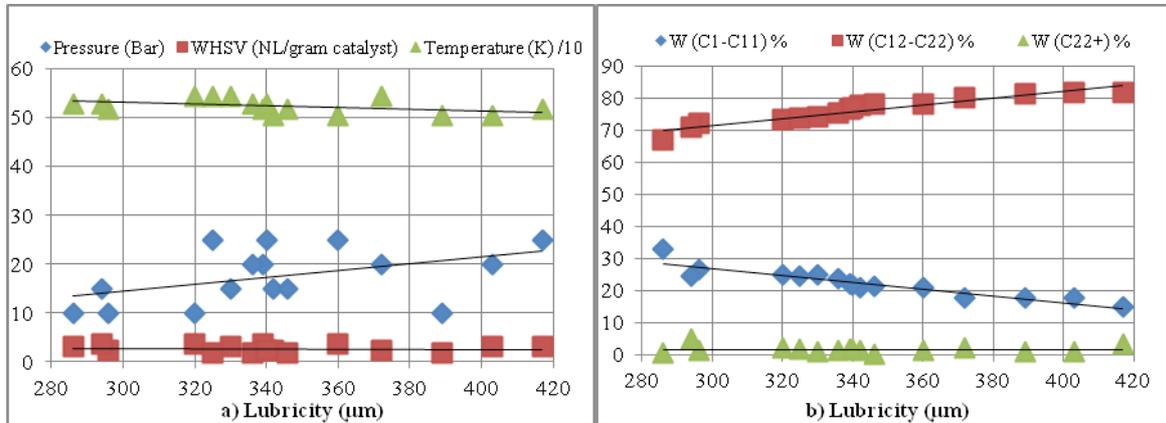


Figure 3a. Relation between Lubricity and FTS Reaction Conditions (Pressure, Temperature and WHSV) **Figure 3b.** Relation between Lubricity and FT Fuel mass fraction.

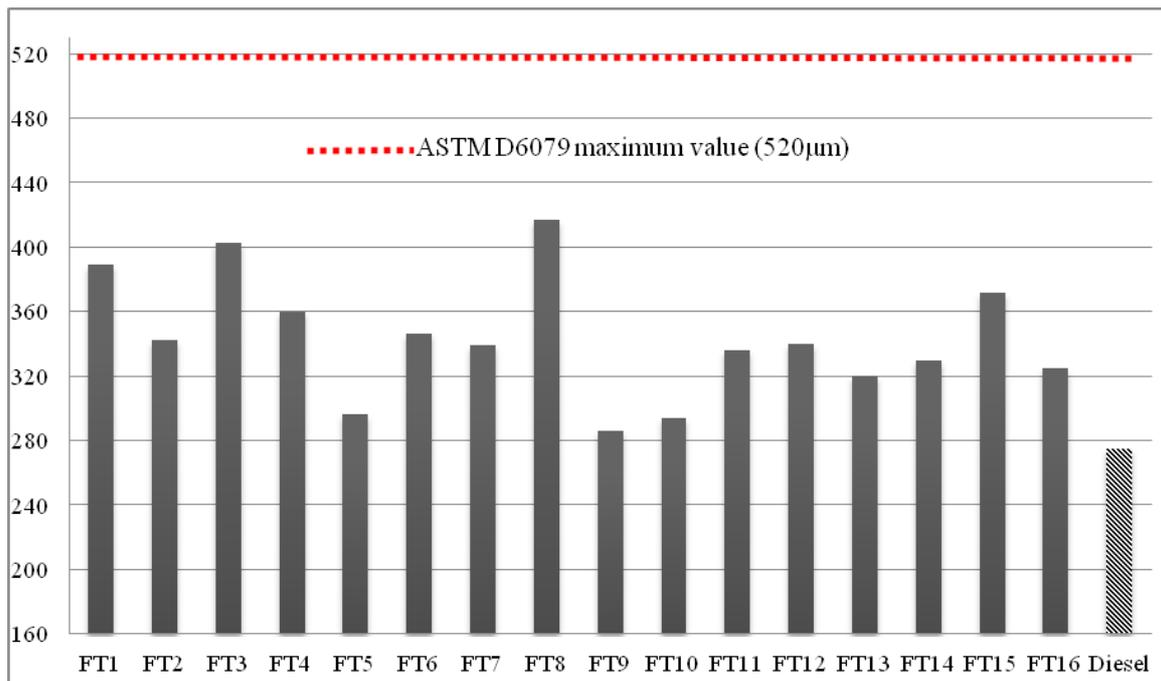


Figure 4. FT Fuel Lubricity (µm).

3.3. Heat of Combustion

Heating value is the amount of heating energy released by the combustion of a unit value of the fuels. Figure 5a indicates effect of FT reaction condition on Calorific value of fuels. It is desirable to have higher calorific value around 45 MJ/kg for diesel fuel. So in high pressure reaction condition FT fuels have appropriate heating value. Temperature and WHSV do not have any significant effect on Calorific value. Figure 5b shows effect of presence of hydrocarbon distributions on calorific value. Presence of light hydrocarbons causes decrease in calorific value but heavier hydrocarbons does not have any significant effect on amount of heat of combustion. Figure 6 shows comparison between the heating values of the Fischer-Tropsch Fuels produced in School of Mechanical Engineering at

University of Birmingham and also ULSD fuel were tested in IKA C200 oxygen bomb calorimeter. Six of these samples are in below limit of 35 MJ/Kg which needed some additives to increase their heat of combustion. FT15 in high pressure 20bar and high temperature 543 K is the fuel with highest net calorific value of 53.29MJ/kg.

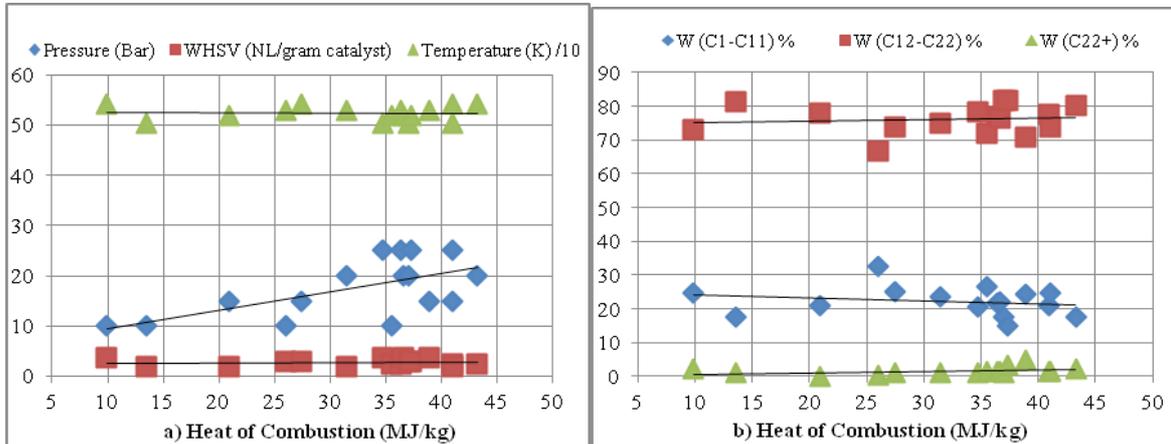


Figure 5a. Relation between Heat of Combustion and FTS Reaction Conditions (Pressure, Temperature and WHSV).

Figure 5b. Relation between Heat of Combustion and FT Fuel mass fraction.

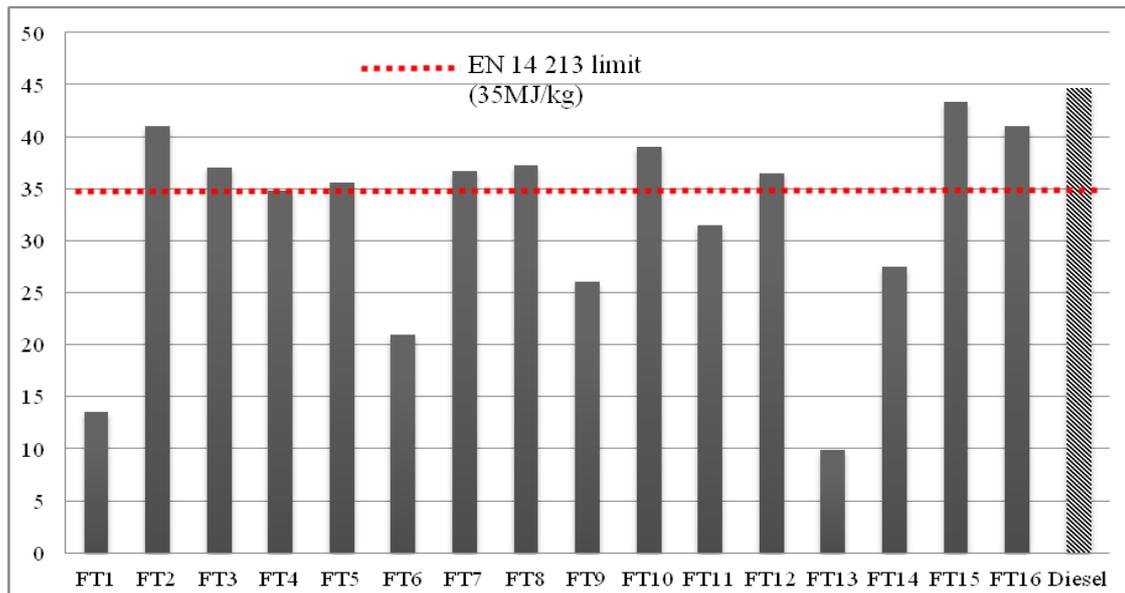


Figure 6. FT Fuel Net Calorific Value (MJ/kg).

4. Conclusions

In this paper, Fischer Tropsch Synthesis as one of the best options for biomass derived fuels production is used which has extensive history of gasoline and diesel production from coal and natural gas. FTS fuel could be one of the best solutions to the fuel emission due to its high quality. FT fuel produced in fixed bed reactor over Cobalt catalyst and it has been characterized in GCMS to determine its hydrocarbon distributions. Lubricity and calorific value measured in HFRR and IKAC200 respectively. Results show that prepared fuels in School of Mechanical Engineering at University of Birmingham have good lubricities which are more than Diesel fuel but it comply the ASTM D6079 Standard 520 μ m. Calorific value of only six samples are below of the limit. And result from graphs

show that high pressure reaction condition improves calorific value of fuels. Because all of the samples have good lubricity so high pressure Fischer Tropsch Synthesis reaction condition produce more desirable fuels. Also Mahmoudi [1] in his PhD thesis used Taguchi method to find an optimum reaction condition which is operating at Temperature 528 K, Pressure 20-25 bar and WHSV 3-3.6NL/gram catalyst. Similarly in high pressure FTS condition, FT fuel has desirable lubricity and calorific value.

Acknowledgments

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