Research Article

Chemical characterization of waste tire pyrolysis products

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ARTICLE INFO	ABSTRACT
	The significance of tire disposal, an attractive waste to convert into burning oil or absorber etc., has been increasing day by day. However, if it does not change into a useful form, it will damage the nature and the living things. Thus, pyrolysis, a well-known method, which is used to convert recycle tire waste into gas, liquid and char. On the other hand, the waste pyrolysis oil or waste tire oil (WTO) has a substantial avaliable calorific value similar to those of fossil fuels. Due to pyrolysis reaction, high amount of sulfur is detected in the WTO; therefore, another step of explosion applied to WTO to decrease sulfur and also re-upgrade quality of oil with such catalysts as Calcium Oxide (CaO) and Natural Zeolite (NZ) at a ratio from 2 to 10 with an increase of 2 for each step, individually. It is noticed that distillation test is a key analysis for separation discrimination of rich or lean quality fuel. As a consequence of mixture of catalyst-WTO reactions, the best curve was observed at a 10% CaO-WTO mixture which was close to diesel#2 and the mixture was separated into two new fuels as light (Gasoline Like Fuel or abbreviated as GLF) and heavy one (Diesel Like Fuel or shortened as DLF) due to temperature differences. According to distillation, FT-IR, NMR and UV–vis were used to analyze WTO, GLF and DLF for defining their characterization as well. Thus, the characterization result data of samples have quasi-equivalent with standard petroleum in open literature, and can be combusted in engine as well.

1. Introduction

The world has come across a renew aspect by lowering traditional energy sources, so the energy gap requires to be filled by waste or natural sources. Unfortunately, waste materials (waste tire, waste oil, garbage etc.) are required to be utilized by minimizing their harmful effects by recycling methods [1-2].

Recently, the amount waste tires (WTs) has increased due to the increase in transportation. However, tires have been worn out or abraded because of road problems or other factors. Thus, high amount of WTs has been discarded to environment, which causes harmful problems throughout human life and ecosystem [3]. The US disposes 500 million tones WT every year [4]. And the amount of tire disposed in Australia for each year is 51 million tones [5].

Owing to high amounts of the residual, the WTs have been used for landfill to minimize ecotoxicology problems [5]. The cement kiln and incinerators use 35 % of total WTs as fuel while 38% is recovered to utilize in civil engineering usage by shredded chips or granulates of WTs. [6]. Mechanical, biological, thermal incineration, chemical and pyrolysis or catalytic pyrolysis methods have been used for WTs in order to recycle them into useful products [7].

Therefore, pyrolysis is a green and clean technique, which can be applied to WTs in order to obtain useful products (non-condensable gas, liquid and solid material) as a result of high temperature and pressure applied at several conditions [8–10]. Based on depolymerization of the WTs, pyrolysis has brought in complex-mixture of organics (paraffin, olefins, terpenes, mono-polyaromatics,

nitrogen and sulfur compounds with heterocycles and oxygenates) like chemical contents of fuels. The initial product of WTs was non-condensable gas which can be burned directly in energy generating systems, because of high heating value. Other one, the solid product, could be activated by nitrogen (N_2) or carbon dioxide (CO_2) flow gases to upgrade as absorbent materials. Besides, the liquid has disgusting odor, low power of hydrogen (pH), low heating value and high sulfur amount which causes air pollution, high corrosion in engine with less burning capacity. Due to tire oil problems, it was treated with catalyst to improve properties of oil for burning [11–14].

Ayanoglu and Yumrutas pyrolyzed WTs particles to oil and then mixed with catalysts to obtain well-quality fuels which have similarity to fuels. Moreover, each sample was analyzed through chemical and physical tests to check their comprehensive with petroleum fuels [15]. Kebritchi et al. [16] studied WTs pyrolysis to compare its yield and properties with other fuels. Furthermore, Ground Tire Rubber (GTR) and Reclaim Rubber (RR) were converted at 400–600 ºC. The alkanes/aromatic ratios of FT-IR results and elemental analyses were close to each other. ^{1}H-NMR and ^{13}C-NMR spectra were declared at different concentrations of aromatics, and they exhibited similarities due to each property. Lopez et al. [17] investigated natural and synthetic rubber of WTs conversion in conical spouted bed reactor between 425-600 ºC. The liquid was tested by gas chromatography and mass spectrometry (GC-MS), two-dimensional gas chromatography (GCxGC) with distillation and elemental tests.

The present study aims to characterize GLF and DLF with GF and DF in order to delineate organic compositions by means of FT-IR, NMR and UV-Vis methods which can be utilized in chamber of combustion. Each of analysis determined structure of composition with detection of organic compounds under advanced technology of instrumental analysis.

2. Material and Methods

2.1. Raw Material

WTs were cut into small particles (1mm) which were replaced in the reactor for organic decompositions [18]. The simple schematic representation of cycling paths of WTs is declared at Figure 1.

2.2. Pyrolysis System

The batch pyrolysis system consists of a main reactor, auto control, thermocouple, blender, safety valve, heat exchanger and container (Figure 2). A cylindrical reactor has a dimension of Ø30x40cm with 5 cm glass wool insulation. Additionally, a 5 kW electrical heater is used to verify temperature from room temperature to demanded values. Furthermore, the blender is used for homogenous temperature variations. The last part of the system is heat exchanger which liquefies gas into oil.

2.3. Reaction of Pyrolysis

The main advantages of pyrolysis system are to be cheap according to its parts, which minimized energy consumption and maximized desired products. Furthermore, the pyrolysis has other contribution to decrease residual of solid by increasing condensable (oil) and non-condensable (gas) fractions via heating rates, catalysts, pressure and other parameters for minimizing harmful and polluting effects because of high amount of sulfur [19–21].

WTs were converted into gas, liquid (WTO) and solid at first step of pyrolysis at a heating rate of 5 ºC/min. And then, WTO was reacted with of CaO and NZ individually at mass ratios of 2,4,6,8 and 10. Each sample was distillated to compare with diesel fuel (DF). Consequently, 10% CaO-WTO sample curve was fairly close to DF. However, the sample had a lower boiling temperature than DF.
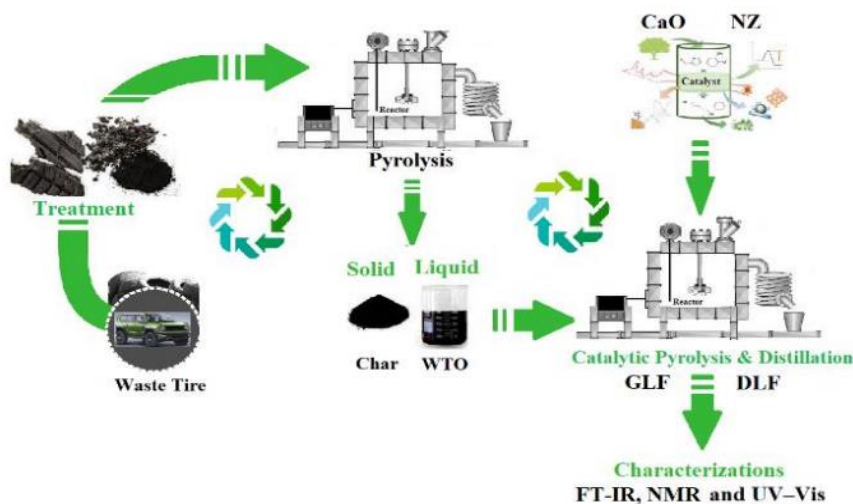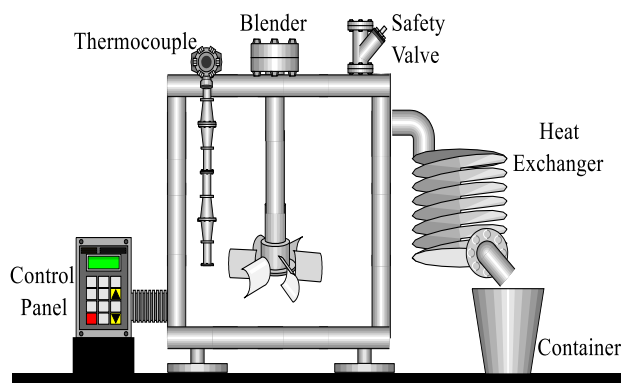

Figure 1. Cycling paths of WTs

Figure 2. The batch pyrolysis system

Thus, the sample was divided into two new fuel samples at 54 °C to 150 °C which labelled as gasoline like fuel (GLF) and another one from 150 °C to 360 °C which was stated as Diesel like fuel (DLF) [22-24].

2.4 Fourier Transform Infrared Spectroscopy (FT-IR) Method

The physico-chemical properties with performance and emissions of fuel have been influenced by different composition amount of hydrocarbon. The compositions of fuel include different numbers of carbon which are combined with various elements to form new types of hydrocarbons at different ratios which can be classified as iso-paraffins, aromatics, naphthene, olefins (alkenes): diolefins (alkins) [25]. The FT-IR is used to characterize bond structures of functional groups.

In this study, FT-IR was used to define functional groups and bands by a simple analytical technique which was related with kind of stretching and bending vibrations of substances [26].

FT-IR spectra were recorded between 4000-650 cm^{-1} range with a resolution of 4 cm^{-1} for 4 scans at room temperature by a Perkin Elmer Spectrum-Two spectrometer for WTO, DLF, DF, GLF and GF, and the chemical structure was observed.

2.5 Nuclear magnetic resonance spectroscopy (13C-NMR, 1H-NMR)

NMR has a special characterization analytical technique to satisfy available chemical compound structures of renewable or conventional fuels. Besides, NMR has a good technique to find aromatic and aliphatic hydrogen and carbon atoms in fuel sample. The resonance peaks area were characterized by the amount of ^{1}H or ^{13}C atoms related with chemical compositions at spectra [31]. The qualitative composition of WTO, DLF, GLF, DF and GF were determined in terms of high-resolution technique. Due to high resolution, NMR spectroscopy provided detailed information on chemistry of petroleum fuels as proton and carbon [32]. The ^{1}H, ^{13}C, ^{19}F, ^{31}P, ^{11}B, APT and DEPT core probes were used

by Agilent Premium Compact NMR600 MHz spectrometer. The deuterated chloroform ($CdCl_3$) was used to dissolve sample at internal standard of NMR. ^{1}H and ^{13}C spectra were recorded from -2.00 ppm to 10.00 ppm and 20.00 ppm to 200 ppm, respectively at 600 MHz frequency.

2.6 Ultraviolet–Visible Spectroscopy Analysis (UV-Vis)

UV–vis spectra is used to detect the electronic properties of molecules. Each organic spectrum is taken via light into its structure to determine different aromatic compounds in samples at visible and ultraviolet fields [33].

In this study, UV absorption spectra of WTO, DLF, DF, GLF and GF were analyzed by using Agilent Technologies Cary 60 UV–Vis Spectrophotometer in the wavelength range of 900-200 nm, at 1 nm intervals, at room temperature and scanned by using standard 1 cm quartz cuvettes which are filled with samples for obtaining spectra. Additionally, samples were diluted by ethanol to define the measurable absorbance interval in the spectrophotometer since samples absorbed light in high UV range to obtain spectra.

3. Results and Discussion

3.1 The physical properties of products

A good indicator was physico-chemical analyses that were applied to all samples. The initial product (WTO) was tested to measure calorific value and carbon amount which was close to standard fuel except sulfur ratio. And a second step of pyrolysis was carried out by adding catalysts (2, 4, 6, 8 and 10 % of CaO and NZ separately) to WTO to minimize sulfur and increase features of new mixture samples [18].

The distillation was a good key for fuel properties to notice best curve which was 10% CaO-WTO for the sample, while the mixture had to be extracted into two parts by boiling points of 54 °C-150 °C as GLF and 150 °C-360 °C as DLF. Moreover, the carbon percentages, density, flash point, and HHV of GLF and DLF were

close to fossil fuels. But, DLF sulfur amount require to be decreased by utilizing a novel process. Finally, GLF and DLF features were benefited for conventional engines [18].

3.2 Fourier Transform Infrared Spectroscopy (FT-IR)

The conventional fuels have complex ingredients as paraffins (alkanes), olefins (alkenes), diolefins (alkynes), naphthenic (cycloalkanes), and aromatic hydrocarbons (benzene derivatives), sulfur, oxygen, and nitrogen molecules which cannot directly and easily break into components [30]. Due to conventional fuel ingredients, WTO has same complicated structure as well as aliphatic, aromatic, hetero-atom and polar compounds. For instance, WTO contains aromatic hydrocarbons compounds such as benzene, toluene, xylene, styrene and limonene. And also, the hydrocarbon groups of alkanes which cause an increase in cetane number. Thus, the alkanes amount was preferred to be high in fuel as an advantageous factor [31]. Figures 3 and 4 indicate FT-IR absorbance spectra which aim to show functional group individually while Figures 5 and 6 indicate FT-IR transmittance spectra which target to define conflict functional group of samples. The observed peaks defined alkenes group for each sample. The functional groups, which were found in literature and compared with all samples, were tabulated at Table 1.

As seen in Table 1, IR spectra have been explained deeply in some researches, as represented in Ref. [32–35]. The wave number ranging between 3700 and 3200 cm^{-1} were defined O–H stretching which was shown in the hydroxyl compounds as alcohols, phenols and carboxylic acids. Another absorption between 3000 and 2800 cm^{-1} was determined as -CH3, -CH2, and C-H functional groups to be specified by aliphatic group of alkanes [36-37]. The peak of 3677 cm^{-1} was seen at O-H stretching for GLF, DLF, DF and GF. The peaks of 2962 cm^{-1} and 2925 cm^{-1} were related with C-H stretching which were vibrated to hybridized at sp^3 layers. This aliphatic and aromatic compound were obviously indicated of alkane groups [38]. The peak around 1608 cm^{-1} was allocated at C=C stretching of aromatics and alkenes [39]. Other peaks were observed between 1455, 1376 and 1247 cm^{-1} due to vibration of aliphatic C-H molecules. The C-H stretching (alkanes), C=C stretching (alkenes) and C-H bending (alkanes) were observed at all peaks of samples. Besides, 1150 and 1100 cm^{-1} peaks were described at C-H in plane bending of aromatics. Furthermore, aromatic hydrocarbons were underwent chemical reaction with oxygen at high temperatures more than aliphatic hydrocarbons [40]. The wave number ranges were altered between 1020 and 845 cm^{-1} to define C=C stretching of alkenes.

The peak of 891 cm^{-1} was determined C=C stretching which was presented in GLF, DLF and gasoline. The residual aromatic groups were arranged at 768 cm^{-1}, 742 cm^{-1}, 729 cm^{-1} and 693 cm^{-1} which were seen at sharp medium peaks of C-H cyclic deformations. The peak of C–H cyclic deformations were detected in all samples except DF [40]. The peaks of all samples revealed the main structure of hydrocarbons and their characteristics by FT-IR spectrum analysis.

3.3 Nuclear magnetic resonance spectroscopy (^{13}C-NMR, ^{1}H-NMR)

NMR principal has been found on average molecular compounds for aromatic, olefin and aliphatic hydrocarbons. The latter structures have a mean of various functional groups number, which were detected at the NMR spectra [41]. The WTO, DLF, DF, GLF and GF were analyzed by ^{1}H-NMR spectra. The characteristic spectral area was defined by aromatic, olefin and aliphatic of proton signals at high and low fields. The ^{1}H-NMR spectra were separated into spectral regions by signals which were determined by aromatic, olefin and aliphatic hydrocarbons, as shown in Figure 7. ^{1}H-NMR spectra defined the aromatic and aliphatic groups in samples.
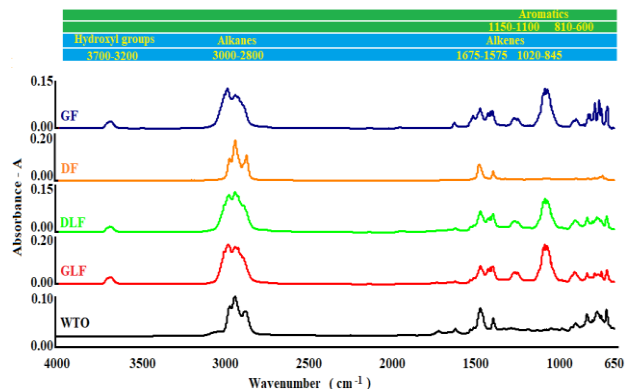

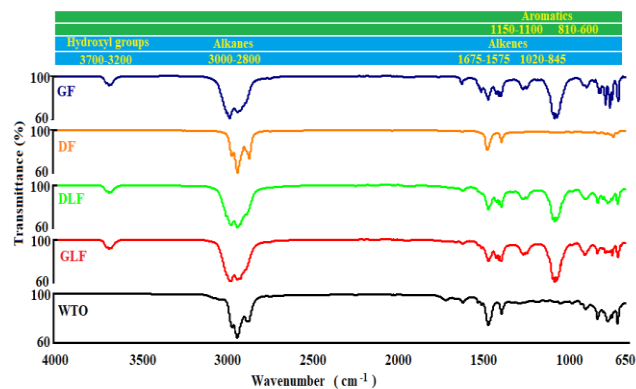Figure 3. FT-IR split absorbance (A) spectra of WTO, DLF, DF, GLF and GF

Figure 4. FT-IR split transmittance (%) spectra of WTO, DLF, DF, GLF and GF
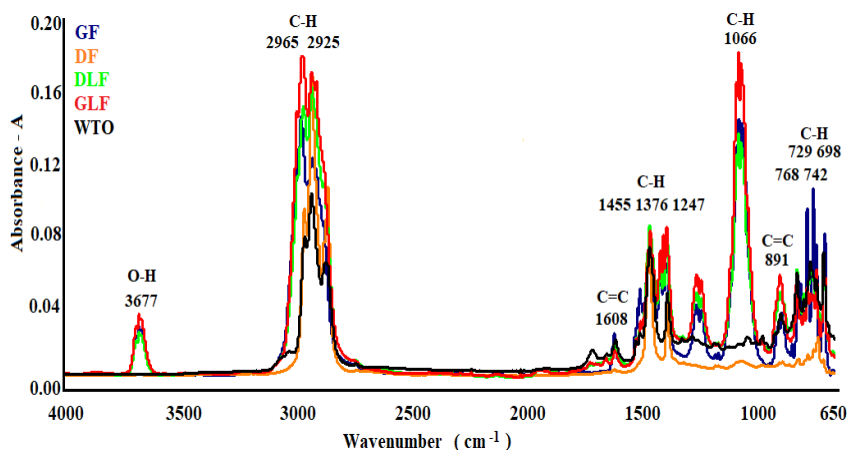
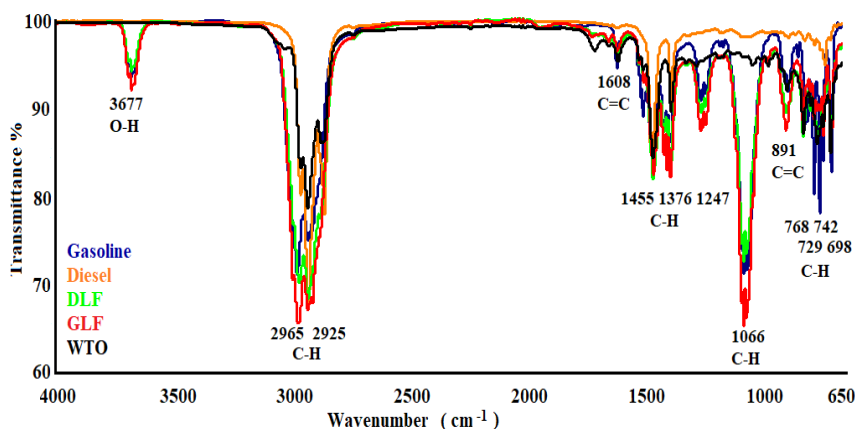Figure 5. FT-IR overlay absorbance (A) spectra of WTO, DLF, DF, GLF and GF

Figure 6. FT-IR overlay transmittance (%) spectra of WTO, DLF, DF, GLF and GF

Table 1. FT-IR functional groups and compounds of WTO, DLF, DF, GLF and GF [32-35]

Wavenumber ranges (cm⁻¹)	Wavenumber (cm⁻¹)	Functional group	Groups	Samples
3700-3200	3677	O-H stretching	hydroxyl	GLF, DLF, gasoline
3000-2800	2962,2925	C-H stretching	alkanes	Whole samples*
1675-1575	1608	C=C stretching	alkenes	Whole samples*
1525-1115	1455,1376,1247	C-H bending	alkanes	Whole samples*
1150-1100	1066	C-H in plane bending	aromatics	GLF, DLF, gasoline
1020-845	891	C = C stretching	alkenes	Whole samples*
810-600	768,742,729,693	C–H cylic deformations	aromatics	DLF, GLF, WTO, gasoline

*Whole samples: WTO, DLF, diesel, GLF and gasoline.

As seen in Table 2, DLF, GLF and GF were analyzed by 1H-NMR that aromatic peaks were observed between 9.00-6.00 ppm of 1H chemical shift. The aliphatic alkane and aliphatic alkene peaks were detected at a range of 1.74-0.88 ppm and 6.00-1.70 ppm, respectively. Moreover, 1H chemical shift was identified in all samples (WTO, DLF, DF, GLF and GF). The samples were also tested by 13C-NMR spectroscopy to obtain more detail of their chemical structure. 13C nuclei of NMR sample spectra are seen in Figure 8.

13C-NMR spectroscopy presents more detail on broad chemical shifts interval data (0-200 ppm) [41]. 13C-NMR spectra have large numbers of distinguishable signals to define carbon groups (i.e. methyl, methylene, methylene) and information about their locations which were assigned at various typical regions [42]. Thus, 13C-NMR informs about presence of aromatic and aliphatic carbon in samples which have an agreement with FT-IR spectra. In Table 2, 13C-NMR analysis was identified aromatic peaks from 150.00 to 125.00 ppm by 13C chemical shift for DLF, GLF, DF and GF. And also, alkane and alkene of aliphatic compounds were seen in all samples which were detected via 13C chemical shift range of during interval of 27.99-13.69 ppm and 150-110 ppm, respectively.

Table 2. ^{1}H-NMR and ^{13}C-NMR spectral regions for single components and structural groups of WTO, DLF, DF, GLF and GF [43, 44]

Class	^{1}H chemical shift (ppm)	^{13}C chemical shift (ppm)	Formulas	Samples
Aromatics	9.00-6.00	150.00-125.00	Ar-H	GLF,DLF, GF
Deuterated chloroform	7.27	77.36	CDCl3	Whole samples*
Aliphatics Alkenes	6.00-1.70	150-110	C=C	Whole samples*
Aliphatics Alkanes	1.74-0.88	27.99-13.69	C-C	Whole samples*

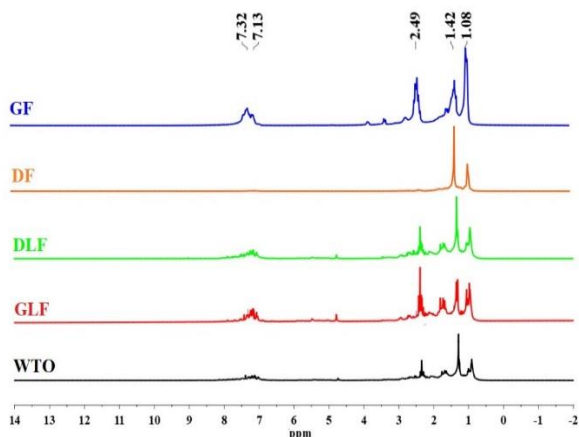*Whole samples: WTO, DLF, diesel, GLF and gasoline.

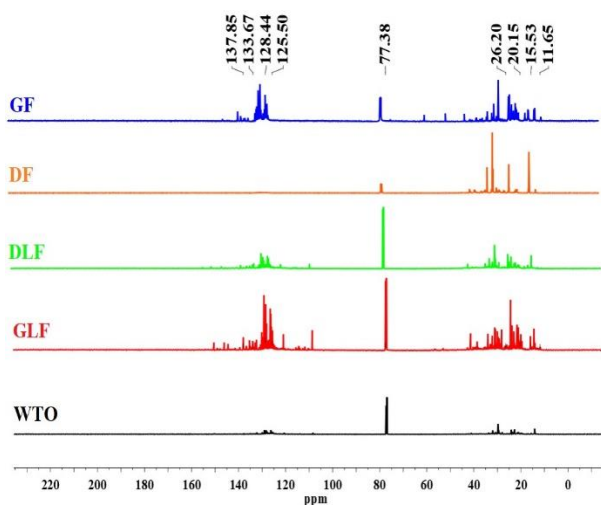Figure 7. ^{1}H-NMR spectra of WTO, DLF, DF, GLF and GF

Figure 8. ^{13}C-NMR spectra of WTO, DLF, DF, GLF and GF

3.7. Ultraviolet–Visible Spectroscopy Analysis (UV-Vis)

UV-vis spectrometry is used for high accuracy quantity analysis for limited application interval due to range of absorption. Thus, organic compounds were enlightened on visible and ultraviolet fields to observe absorption spectrum for functional sample groups. The absorbance curves of diluted samples were observed at wavelength of 700–200 nm as seen in Figure 9. The curves were plotted by UV-Vis spectra data to compare samples' shape and slope which have similarity with chemical compounds of fossil fuels [45]
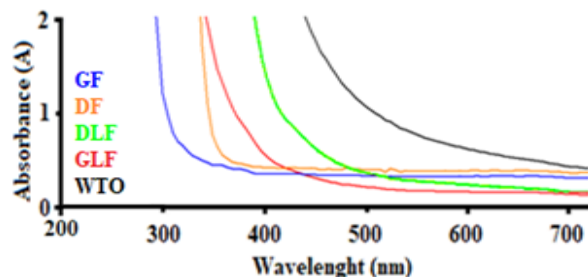

Figure 9. UV-vis absorbance spectra of WTO, DLF, DF, GLF and GF

3. Conclusions

WTs was liquefied into affluent organic products as gas, oil and solid. Especially, the physical and chemical features with calorific value of WTO were near to DF, except for sulfur. Thus, CaO and NZ catalysts reacted with different mass ratios to decrease sulfur quantity. Then, the new products compared with DF. 10 % CaO-WTO mixture curve was close to DF. Due to boiling points, the mixture sample was separated into light (GLF) and heavy (DLF) fractions. Both of GLF and DLF were very similar to GF and DF. However, some available characterization analyses were done for GLF and DLF via FT-IR, NMR and UV–Vis which was explained as items:

- FT-IR spectrum analyses of GLF and DLF had peaks which demonstrated fundamental structure of hydrocarbons, similar to standard fuels.
- ^{1}H-NMR and ^{13}C-NMR spectra of GLF and DLF had aromatic and aliphatic carbon groups which were compatible with FT-IR spectra which were close to gasoline and diesel fuel.
- UV-Vis spectra GLF and DLF had a similarity as visible and ultraviolet fields with standard petroleum fuels.

Declaration

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors also declare that this article is original, and was prepared in accordance with international publication and research ethics, and no ethical committee permission or special permission is required.

Author Contributions

G. Uguz and A. Ayanoglu have equal contribution and responsibility for the study.

Acknowledgment

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