Peat and lignite leaching process with tetralin in autoclave to produce oil

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Abstract: Instead of directly burning a lignite having low calorific value and peat, the Elbistan lignite (*L*) and the Adiyaman peat (*P*) were mixed and leached in an autoclave to obtain an oil to not only provide the demand for energy but also protect the environment. The effects of the peat ratio in the mixture on the properties of co-liquefaction products (oil, char, asphaltene, and preasphaltene) and oil yield were investigated in details. The products were characterized by XRD, FTIR, and elemental analysis. In addition, the composition of the oil was identified by GC/MS, showing that the peat ratio did not affect the chemical composition of the oil due to the high lignin content and the nearly same elemental compositions. On the other hand, the oil yield for a co-liquefaction process was found as 34.3% to be higher than the average value of oil yields obtained from the individual feeds (24.3% for lignite and 28% for peat), showing the synergistic effects between the lignite and peat. The obtained oil was paraffinic-low waxy oil with 5138.62 kcal/kg of calorific value and 0.94 g/cm³ density. Finally, it was suggested that the production of a more valuable product using the peat and the lignite having low calorific value will not only contribute more to the country's economy in future but also be better for the environment, instead of directly burning them.

Keywords: leaching, Adiyaman peat, Elbistan lignite, paraffinic-low waxy oil

1. Introduction

Liquefaction is a kind of leaching process, and one of the clean energy production way obtained from coal, having low calorific value and high ash content (Wang et al., 2014; Xiaohong et al., 2015). This technique becomes a viable option for the production of transportation fuels in the early part of the next century (Karaca and Koyunoglu, 2010). This process not only provides the demand for energy which is rising depending on industrialization, but also protects the environment. Recently, the cost of a liquefaction process has become very high to be able to obtain a liquid fuel because of the low petroleum price. In order to decrease the liquefaction cost and increase oil yield, coal is generally mixed with biomass, which is locally available, cheap, and renewable energy source. The main idea is to compose of synergism effect between coal and biomass which can be used as a hydrogen donor for a system (Stiller et al., 1996; Karaca and Bolat, 2002; Xiao-hong et al., 2015; Karta, 2016). In addition, countries like Turkey which has abundant lignite reserve (its 46% in Afsin-Elbistan region) makes research to find alternative oil production way for depletion of oil reserve in near future and to be self-contained country in respect to energy (Anonymous-a, 2016).

In the present study, the Adiyaman peat was mixed with the Elbistan lignite at the different weight ratios, and leached by tetralin solvent in an autoclave. The effect of the peat ratio on the properties of the products, which were obtained after the leaching process, and oil yield was investigated in detail. Adiyaman has a huge peat reserve which is just used for agricultural purposes. Peat is counted the first stage of coalification and composes of incomplete disintegration of dead plants (Karaca et al., 2016). Having a low ash content and cost than some other fossil solid fuels, the peat is put into a

catalogue of renewable natural energy sources by European Parliament (Tolonen, 2000). In the literature, there are some investigations related with the pyroloysis and gasification properties of the peat and the peat mixture with coal (Sutcu, 2007; Kim, 2011; Hayes, 2013; Huang et al., 2015). There are also a few papers about the direct peat liquefaction process, which investigate the liquefaction properties of peat in supercritical water with or without catalyst (Zhang et al., 2008; Li et al., 2011; Xu and Donald 2012). However, the effect of the peat in the mixture of the lignite in the autoclave leaching process on the properties of the products and oil yield has not been fully studied. In addition, using the lignite and the peat may not only increase the strength of competition of Adiyaman province in the future, but also contributes to be self-contained country in respect to energy.

2. Material and methods

Peat (P) and lignite (L) samples were collected from Adiyaman Cat Dam and Elbistan lignite basin (Turkey), respectively. The samples were dried in an oven at 50 °C for 24 h. In terms of our preliminary investigation, the starting samples were ground and sieved to obtain the product size of -1410 + 840 µm (80% weight of materials, controlled grinding). Tetrahydrofuran, tetralin (Merck), n-hexane, toluene (Riedel-de Haen), ethylene glycol (EG), acetone, and ethanol were used in the study. Leaching (liquefaction) tests were done using a mechanically stirred and electrically heated closed system: 500 cm^3 stainless-steel Parr reactor. For each experiment, 30 g of the samples (single and the mixture) were put into the Parr autoclave, 90 cm^3 of tetralin without any catalyst was added, the autoclave was sealed, and the pressure was adjusted to 20 bar by N$_2$. In the present study, the tetralin was chosen as a solvent due to its high hydrogen donating ability and thermal stability (Guo et al., 2011). The autoclave was heated until 400 °C at 60 min, and then cooled to the room temperature into an ice-water bath. The char, asphaltene, preasphaltene, and oil were separated from each other using a Soxhlet solvent extraction and evaporator system (Shui et al., 2011). The effect of the peat ratio (lignite:peat, 1:1, 2:1, 3:1, 1:2, and 1:3) on the properties of the liquefaction products and oil yield were determined using mass balance equations.

Characterization and chemical structure of the starting materials and the obtained products were subjected using Rigaku Miniflex 600 with Cu Kα (40 kV, 15 mA, λ=1.54050 Å) XRD, Perkin Elmer Spectrum One FTIR, CHNS Elemental analyzer device (Leco CHNS 932, LECO Corporation, St. Joseph, MI), Shimadzu TGA and DTA-50, IKA C-1 calorimeter, Agilent Technologies 6890 N Network GC System model gas chromatography, and Agilent Technologies 5973 inert Mass Selective Detector mass spectrometer (Agilent Technologies, Santa Clara, CA). In addition, the morphological study of the peat was also investigated by LeO EVO 40 scanning electron microscope.

3. Results and discussion

Elemental compositions of the Adiyaman peat and Elbistan Lignite are presented in Table 1. The elemental composition indicated that the peat was classified as low decompose peat type (C<53, 5.0<H<6.1) (Fuchsman, 1989), and it was expected to cause a synergistic effect in the direct liquefaction process depending on the high carbon and hydrogen content due to the hemi-cellulose, cellulose, and lignin (Chen et al., 2011; Karaca et al., 2016) in the structure. The calorific value of the peat was significantly higher than the lignite due to its high carbon content and the low ash content, obtained from proximate and ultimate analysis (Table 1).

Table 1. Proximate and ultimate analysis of the Elbistan lignite and Adiyaman peat

Sample	Proximate analysis. wt %				Elemental analysis (daf). wt %					Calorific Value kcal/kg
	M	A	V	FC	C	H	N	S	O*	
Elbistan lignite	4.99	38.08	35.09	21.84	28.08	3.483	1.035	3.552	63.85	2608
Peat	4.79	21.29	32.82	41.1	42.93	5.23	2.101	0.32	49.43	3920

* By difference

The XRD pattern of the lignite (not given in the text) indicated that the lignite had graphite (002) in the structure, and some peaks belonging to quartz, gypsum, pyrite, and calcium-magnesium silicate were detected, showing the heterogeneous coal structure (Guo et al., 2011). On the other hand, the main structure of the peat was determined as an amorphous, and the wide curve peaks (between 15° and 25° 2θ values) confirmed the high lignin composition (Ishikawat et al., 1998). SEM images of the lignite and the peat supported to the XRD results. The heterogeneous structure of the lignite was observed in Fig. 1a. The peat was composed of mostly lignin, hemi-cellulose, and lignocellulosic materials including a wood (W: roots and parts of trees), a leaf fragment with stomata (Lf), and spicules of fresh-water sponges (Spg) (Fig. 1b) (Rydelek 2006).
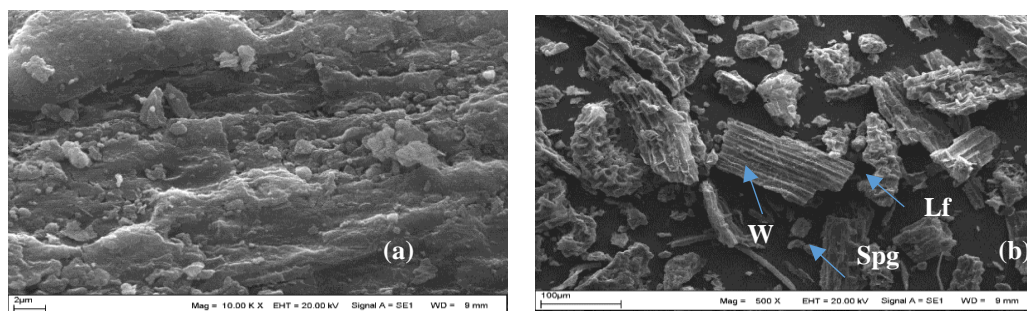

Fig. 1. SEM image of the lignite (a) and the peat (b) (W: wood, Lf: a leaf fragment with stomata, Spg: spicules of fresh-water sponges)

In the process, the most important part is the products yield. Fig. 2 shows the distribution of the yield of the co-liquefaction products depending on the blending ratio of the lignite and the peat. As seen from Fig. 2, the total conversion increased with the increasing the peat content in the mixture. The maximum ratio was calculated as 68.8%, which was 110% higher than the ratio belonging to the individual lignite feed, indicating that the liquefaction conditions (temperature, heating time, and gas pressure) were chosen properly (Akash et al., 1994; Stiller et al., 1996; Karaca and Bolat, 2000). The experimental conditions were selected in terms of our preliminary investigation. In addition, Speight (1994) declared that the young lignite exceedingly composes of high carboxyl and/or sulfur functional groups in the structure, and they are covalently bonded. These bonds are broken between 375 and 450 °C in the liquefaction process, and at this temperature, nitrogen and hydrogen removal takes place and the cyclic hydrocarbons in the open hydrocarbon chains decompose. Finally, the liquefaction process is complete. The literature survey also shows that the addition of biomasses as a co-liquefaction material in the lignite increases the total conversion ratio (Hua et al., 2011; Shui et al., 2013; Singh and Zondlo, 2017). The same result was also observed in the present study. The peat had high volatile content, and accelerated thermolysis of the lignite according to the DTA/TG analysis (not given in the text), and decreased the decomposition temperature of the lignite, showing the synergistic effect. Decomposition of the lignin, coming from the peat structure at low temperature causes the form of free radicals that break the aliphatic carbon-carbon bonds. Therefore, the total conversation ratio belonging to the mixture increases comparing to that of the individual feeds (Inaba and Okada, 1995). Theoretical efficiency of liquefaction increases with the increasing the amount of the peat in the mixture. Fig. 2 also indicated that the amount of gas increased as that of the residue decreased depending on the peat ratio in the mixture. In the liquefaction process, cellulose and hemicelluloses in the biomass or fuel structure decompose depending on heating process, as a result, low molecular activated cellulose occurs, and turns into volatiles (anhydrosugars) and gases. On the other hand, the decomposition of the lignin structure is very difficult, and it constitutes the main structure of the char (Younas et al., 2017). The content of cellulose and hemicelluloses in the peat structure were higher than that of the lignite, and the lignin ratio was exactly the opposite (Karaca et al., 2016). For this reason, the quantity of the char decreased while the amount of gas increased with the increasing the peat ratio in the mixture. On the other hand, the ratio of the asphaltene and preasphaltene were found close with each other due to the nearly same carbon and hydrogen content of the lignite and the peat. The similar conclusions were obtained in the previous investigations

related with the co-liquefaction process of lignite and biomasses (Shui et al., 2011; Hua et al., 2011; Shui et al., 2013; Singh and Zondlo, 2017).
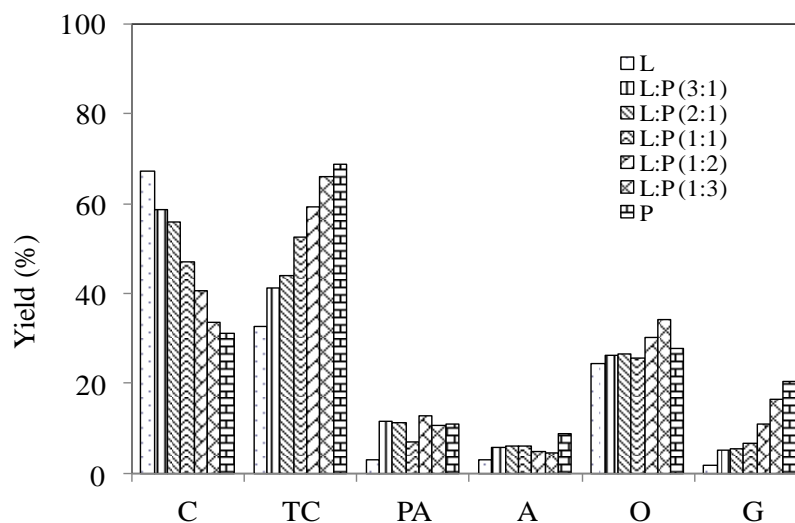

Fig. 2. Product distribution and comparison of the blending ratio of the product yields. C: char, TC: total conversion, PA: preasphaltene, A: asphaltene, O: oil and G: gas

One of the desirable characteristics of biomass in the process is a high lignin content, enhancing the liquefaction process and oil yield (Stiller et al., 1996; Singh and Zondlo, 2017). The direct liquefaction of the lignite and the peat produced 24.3% and 28% oils, respectively. On the other hand, Fig. 2 indicated that the oil yield increased from the leaching (co-liquefaction) process with the increasing the peat content in the mixture. The oil yield for co-liquefaction process was found as 34.3% to be higher than the average value of oil yields obtained from the individual feeds (lignite and peat). Due to the high lignin content in the peat structure, the highest oil yield was obtained from 1:3 blending ratio of the lignite:peat. Additionally, the amount of lignin in the biomass structure plays a very important role in increasing the oil yield (Stiller et al., 1996). Briefly, leaching the lignite with the peat increased the oil yield more than the direct lignite leaching process with tetralin due to the synergistic effects depending on the decomposition, and dissolution of the primary products which occurred as the results of the reaction between the free radicals, coming from the lignin structure, and hydrogen-donating materials (the tetralin and the peat) (Song et al., 2000; Trautman et al., 2014).

In the present study, the obtained products were also analyzed and evaluated in detail. The elemental compositions of the char, asphaltene, preasphaltene, and oil are given in Table 2. It appears that the carbon content decreased especially in the residues. The carbon structures in the lignite and peat were degraded, and the carbon passed to the asphaltene, preasphaltene, and oil structure in the process, which was supported by their carbon content (Table 2). As expected from the other side, the calorific values of the char, asphaltene, preasphaltene, and oil were found as 1200 (average), 9100 (average), and 5138.62 kcal/kg, respectively. On the other hand, the elemental carbon content in the char increased with the increasing the peat ratio. Aforementioned, the carbon content of the peat was higher than the lignite. In addition, the nearly same elemental composition for the asphaltene, preasphaltene, and oil were obtained from the direct lignite liquefaction, and co-liquefaction of the lignite, and peat process. This observation may be explained due to the nearly same carbon and hydrogen content of the starting materials.

XRD powder diffraction patterns of the chars are presented in Fig. 3a. By comparing the characteristic XRD patterns of the chars depending on the lignite:peat ratio showed that they had similar structure, meaning that the peat ratio in the mixture did not change the char structure. This result supported the results of elemental analysis. In addition, inorganic species in the lignite reacted with each other and also the species coming from the biomass, causing the formation of the calcium-based crystal (Naoko Ellis et al., 2015) on the XRD pattern. The peaks showing widespread between 10°C and 30° 2θ indicated the unreacted carbon which was close to Si and Al inorganic compounds. In

addition, quartz, calcite, and dolomitic structure depending on the lignite were detected in the XRD patterns (Sekine et al., 2006; Ellis et al., 2015). The XRD patterns of the asphaltene and preasphaltene for all the lignite:peat combination showed great similarity with each other, so just one patterns for both are given in Fig. 3b. As expected, the structures were amorphous, and no crystal structure was observed. Given the elemental analysis results, it was concluded that the wide spread peak was seen between 10 and 30° 2θ represented the amorphous carbon structure.

Table 2. Elemental analysis of direct liquefaction of lignite products and co-liquefaction of the lignite and peat products and lignin

Products	Sample Code	%C	%H	%N	%S	%O*
Char (C)	*Lignite-C*	*14.98*	*1.59*	*0.74*	*2.30*	*80.40*
	CLP(1/1)	17.78	1.77	0.94	2.44	77.07
	CLP (2/1)	15.60	1.58	0.84	2.51	79.47
	CLP (3/1)	15.23	1.55	0.81	2.75	79.66
	CLP (1/2)	17.23	1.65	0.91	2.23	77.98
	CLP (1/3)	18.66	1.76	0.93	2.10	76.56
Asphaltene (A)	*Lignite -A*	*79.13*	*7.06*	*1.71*	*2.01*	*10.09*
	ALP(1/1)	78.48	7.71	2.85	0.52	10.44
	ALP(2/1)	77.69	7.21	3.00	0.42	11.68
	ALP(3/1)	79.61	7.94	2.40	0.69	9.36
	ALP(1/2)	77.37	6.70	2.53	0.60	12.80
	ALP(1/3)	78.95	7.34	2.81	0.55	10.35
Preasphaltene (P)	*Lignite -P*	*72.67*	*5.78*	*2.50*	*1.84*	*17.20*
	PLP(1/1)	78.62	6.239	3.083	0.936	11.12
	PLP(2/1)	74.14	5.823	3.059	1.127	15.85
	PLP(3/1)	73.24	5.11	2.814	0.953	17.88
	PLP(1/2)	76.82	6.216	3.258	0.611	13.10
	PLP(1/3)	74.55	6.105	3.279	0.529	15.54
Oil (O)	*Lignite -O*	*87.21*	*8.81*	*0.86*	*0.33*	*2.79025*
	OLP	86.53	7.831	0.87	0.209	4.56

* By difference

Like the XRD patterns, IR spectrum of the chars (Fig. 3c) showed the lignite:peat ratio independent character. When the IR spectra of the chars were examined, the bands showing the C=C and C=O in cycloolefin structure of the characteristic carbon bands of the originally lignite recorded at about 2895 and 1600 cm[-1] were not observed (Jiang et al., 2012; Jingchong et al., 2014). This indicated that many C-C bonds were broken, and disintegrated during the co-liquefaction process. The other bands were at 1050 cm[-1] associated with v(C-O), 775, 665 and 453 cm[-1] associated with Si and inorganic materials in the structure (Figueroa et al., 2011; Jiuling et al., 2016). The IR spectra of asphaltene and preasphaltene are shown in Fig. 3d. It was seen that the asphaltene and preasphaltene had similar absorbance, showing to the lignite:peat mixture ratio independent character. The main bands of the asphaltene and preasphaltene represented to C-C and C-O bonds in the aromatic structure and aromatic carboxylates, respectively. The results are consistent with the elemental analysis and also literature data (Wang et al., 2014; Li et al., 2015). The IR spectra of the oil is also represented in Fig. 3d. The adsorption peaks are at 750 cm[-1] associated with alkyl C-H groups, 1595 cm[-1] associated with C=C stretching vibrations and 2918 cm[-1] associated with aliphatic hydrogen, and 3025 cm[-1] band ascribed to C-H vibration, and hydrogen in the aromatic structure (Benavente and Fullana, 2015). These bands indicated more aliphatic and aromatic hydrogen content in the oil than the asphaltene and the preasphaltene. Additionally, the broad band recorded between 3120 and 3570 cm[-1] represented that the preasphaltene had highest hydroxyl. The result coincides with the literature (Teng et al., 1992; Singh and Zondlo 2017).
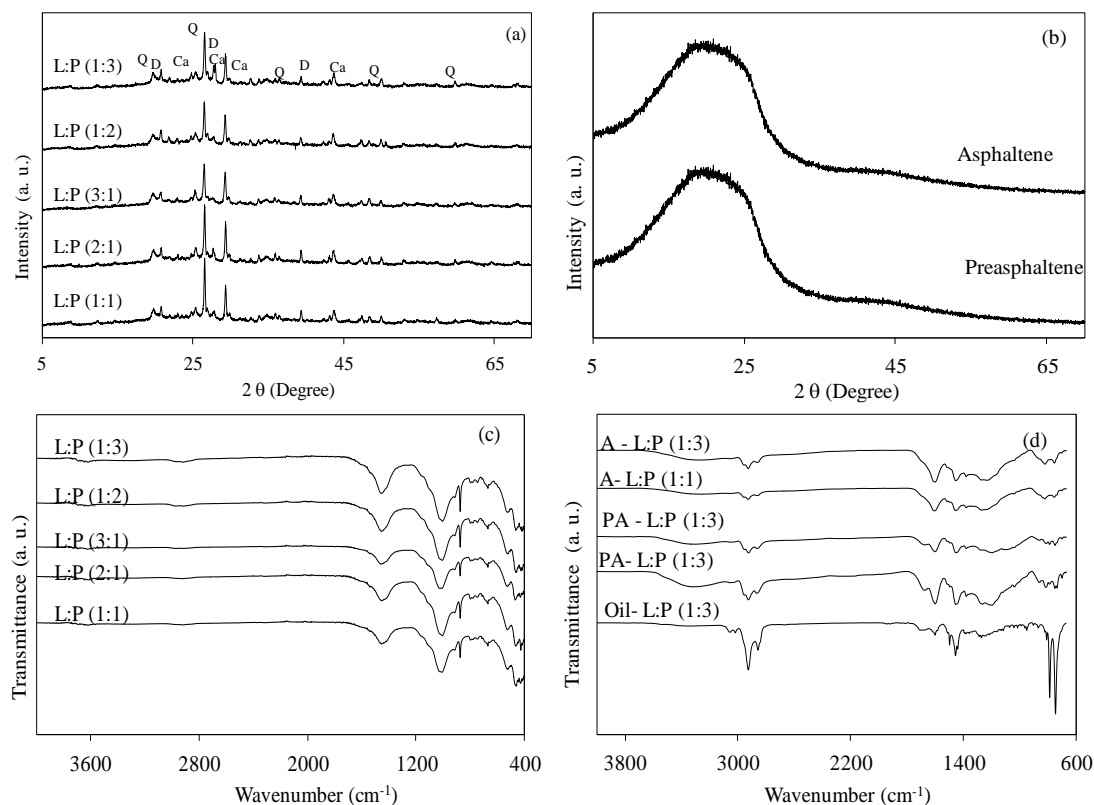
Fig. 3. Characterization of the leaching products. (a) and (b) XRD patterns (c) and (d) IR spectra of the chars and asphaltene (a, c) and preasphaltene (b, d)

In the present study, in addition to the elemental analysis, the calorific value and IR analysis of the oil products, GC-MS analyze was performed for all mixture ratio of L:P to accurately determine the compounds due to the multitude of chemical compounds in the oil structures. Generally, biomasses not only increase the oil yields but also effect and cause the difference in the chemical structure of the oil (Matsumura et al., 1999; Shui et al., 2013). On the contrary of the literature, comparing to GC-MS results obtained for all blending ratio of L:P showed that the peat did not change the chemical compositions of the obtained oils due to the high lignin content, and the nearly same elemental compositions which was supported by the XRD patterns and SEM images. In addition, the chemical composition of the produced oils from the individual feeds showed great similarity with each other. Therefore, one of the GC-MS graph (Fig. 4) and the chemical compounds of the oil (Table 3) are given in the text, as an example. The main compounds of the oil were determined as saturated n-alkenes, isoprenoids, branched alkenes, n-alkyl cyclohexane, terpene, and other aromatic structures, like the literature data (Speight 1994; Methakhup et al., 2007; Wang et al., 2014).
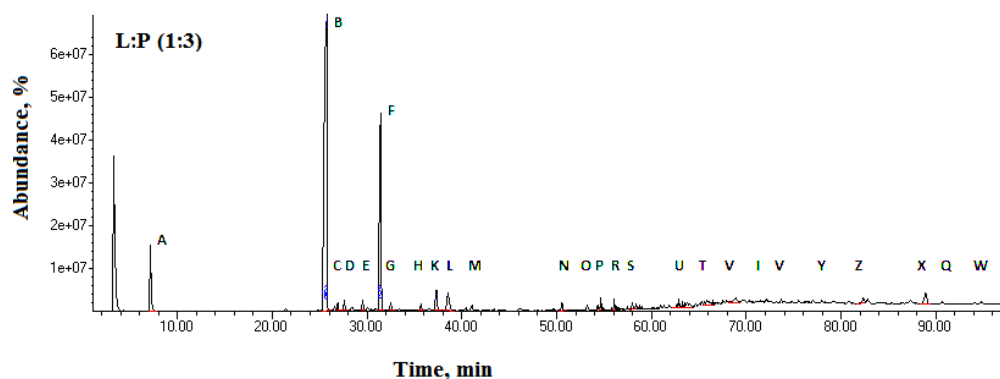

Fig. 4. GC-MS graph of the obtained oil

Table 3. GC-MS analysis of the oil obtained from co-liquefaction of L:P (1:3) mixture and the detected compounds in the oil structure

Peak No	Retention Time (min)	Probable Compound	Molecular Formula	Abundance, %
A	21.480	Benzen,1-etinil-3-Ethyl	C_9H_{12}	0.21
B	25.691	Naphthalene, 1,2,3,4-tetrahydro, Tetralin	$C_{10}H_{12}$	43.67
C	26.630	2-Ethyl-2,3-Dihidro-1H-indene	$C_{11}H_{14}$	0.35
D	27.608	Naphthalene,1,2-dihidro	$C_{10}H_{10}$	0.82
E	29.576	Naphthalene,1-Ethyl-1,2,3,4-tetrahydro	$C_{12}H_{16}$	0.94
F	31.419	Naphthalene	$C_{10}H_8$	18.82
G	32.546	Naphthalene, 1,2,3,4-tetrahydro-1-propyl	$C_{13}H_{18}$	0.80
H	35.722	Naphthalene, 1-mEthyl	$C_{11}H_{10}$	0.55
K	37.330	Naphthalene, 2-mEthyl	$C_{11}H_{10}$	1.88
L	38.583	Butile Hidroksiytoluen	$C_{15}H_{24}O$	2.74
M	41.095	Naphthalene, 1-Ethyl	$C_{12}H_{12}$	0.58
N	50.542	1 (2H)-Naftalenon, 3,4-dihidro	$C_{10}H_{10}O$	0.71

4. Conclusions

Product distribution and comparison of the blending ratio of the product yields showed that the total conversion increased with the increasing the peat content in the mixture, and higher than the ratio belonging to the individual lignite feed, indicating that the liquefaction conditions were chosen properly. In terms of the elemental analyzes and the characterization studies, the carbon structures in the lignite and peat were degraded, and the carbon passed to the asphaltene, preasphaltene, and oil structure in the co-liquefaction process. On the other hand, the chemical compositions and the structure of the products showed the lignite:peat ratio independent character. Co-liquefaction of the lignite with the peat increased the oil yield more than individual lignite liquefaction due to the synergistic effects depending on the decomposition and dissolution of the occurred primary products and hydrogen-donating solvent. The oil yield for co-liquefaction process was found as 34.3% to be higher than the average value of oil yields obtained from the individual feeds (24.3% for lignite and 28% for peat). The main organic compounds in the oil structure was found as saturated n-alkenes, isoprenoids, branched alkenes, n-alkyl cyclohexane, terpene, and other aromatic structures, and the obtained oil was defined paraffinic-low waxy oil with 5138.62 kcal/kg of calorific value and 0.94 g/cm^3 density. The overall results indicated that the application of peat, directly or indirectly in the liquefaction process, is a good candidate for production of oil in the future.

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