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Chemical and thermal characteristics of petrochemical industrial sludge

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ABSTRACT

Industrial sludges have different characteristics even in the same industrial sector. Therefore, the strategy development for the management of industrial sludges is a great challenge interim of sustainable and economic concern. In addition to the legislative restrictions on traditional recycling options including land application, agricultural usage etc., the recovery of energy and valuable materials from industrial sludges becomes progressively more important. Petrochemical industry produces a large amount of sludge having different pollutants, which are mostly hazardous. Recently, the treatment and disposal of petroleum and petro-chemical industry sludges have been an encountering problem regarding their huge amounts and complex structures. To build an appropriate and sustainable management strategy for these sludges, first important stage is the complete and correct characterization of them. However, the sludges are very complicated materials to be epitomized. This paper focused on the chemical and thermal properties of petrochemical industry sludge taken from a petro-chemical industry complex located in Izmir, Turkey to evaluate its suitability for the energy recovery purpose via pyrolysis and/or gasification processes. Experimental results showed that the sludge had high volatile solids content (VS) approx. 69% on dry basis and in accordance with this, had a very high low heating value of 26.30 MJ/kg. Thermal gravimetric analysis results also supported these findings showing the decomposition of major organic compounds available in the sludge. The paper discusses the experimental results on whether it could be a significant fuel for energy recovery purpose or not.

Keywords: Petrochemical industry; Sludge; Oily sludge; Energy recovery; Gasification; Renewable energy

1. Introduction

With rapid increase in the World's population, the use of petroleum has increased. As a result of this, the petroleum industry has drastically gained more importance. This intensification in the petroleum refining industries has also led to challenging environmental problems including the generation of great amounts of hazardous wastewaters and sludges, which is so-called petrochemical or oily sludge [1]. In petrochemical industries, huge amounts of the sludge have been produced at different stages of petroleum refining and wastewater treatment. It can mainly occur in crude oil tank bottoms, slop oil emulsions, oil/water separators, refinery product tanks, and wastewater treatment processes during crude oil exploration, production, transportation, storage, and refining processes [2–5]. Huang et al. [6] indicated that the petroleum sludge production was more than 6 million tons each year since 2010 in China. Hu et al. [2] has reported that each refinery in the United States produces an annual average of 30,000 tons of oily sludge according to a study carried out by US Environmental Protection Agency (EPA) [7]. Based on the recent global petroleum refining outputs, Hu et al. [2] has also pointed out that more than 60 million tons of oily sludge could be produced every year and more than 1 billion tons of oily sludge had been accumulated worldwide. Also, the rising demand on refined petroleum products guarantees the increased tendency of the total petrochemical

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industry sludge production. Beyond this, these sludges are currently either incinerated or disposed of in the landfill areas. Chen et al. [1] claimed that these methods may cause secondary air and groundwater pollution and also result in additional cost and energy burden. Economical and efficient treatment/disposal of the sludges is a great challenge for petroleum refining industries. However, energy or material recovery from these hazardous materials as a resource is a great interest.

The properties and amount of petrochemical sludge can vary from plant to plant depending on the wastewater characteristics and applied treatment processes [8]. This type of sludge is a complex mixture of oil-in-water, water-in-oil emulsion, and solid particles [9,10]. Although it seems as a simple two-phase system, it is mostly considered as a hazardous solid waste material due to its high concentration of toxic and hazardous constituents containing heavy metals, bacteria, organic pollutants such as polycyclic aromatic hydrocarbons, petroleum hydrocarbons (PHCs), and some chemical additives [3,6,9,11]. A safe and sustainable management of the petrochemical sludge is required because of the negative effects on the environment and potential risks to human health as a result of long term production and the use of different petroleum derivatives led to soil contamination around the plants [12,13]. Regarding the priority environmental pollutants categorized by the US-EPA, the sludge has been classified as a hazardous material due to its toxic, mutagenic, and carcinogenic properties [4,14]. Therefore, it needs special care to handling and disposal operations, which are very costly [15]. Hu et al. [4] have remarked that the energy recovery from these sludges and such oil-rich waste had recently received great interests, and a variety of methods have been developed for this purpose since the traditional methods for the sludge treatment like incineration and landfilling have been challenged by strict regulations [4,16].

Diversity in chemical characteristics of the sludges as mentioned above even in the same plant strictly affects the thermal properties of them. However, these sludges have commonly high oil and organic content and high heating value (HHV). Therefore, they can be used as a potential energy source to improve the energy balance of oil refineries and can be converted into usable energy with different methods including anaerobic digestion and thermal methods such as pyrolysis and gasification [17].

Although some efforts have been made to investigate the petrochemical sludge characteristics, there are limited studies especially focusing on the thermal properties of sludge for energy recovery purpose. Therefore, objectives of the study are:

- to investigate the chemical and thermal properties of petrochemical industry sludge from the largest petrochemical complex located in Izmir, Turkey.
- to evaluate the sludge characteristics whether the sludge could be used as an energy source.

The findings from the work would provide valuable information to enhance a more regardful and sustainable sludge management strategy for petrochemical industries from point view of energy recovery.

2. Materials and methods

2.1. Sludge sampling

Sludge cake samples following the centrifuges used for mechanical dewatering purpose were taken from the wastewater treatment plant of the largest petrochemical industry located in Izmir, Turkey. The sludge cake having 26.46% of dried solids content (DS) were shaped as in pelletized form and dried at 37°C ± 2°C. Even the sludge cake samples were pelletized in 2–4 cm sizes, they become as granulated form after drying. The dried sludge samples had 96.41% ± 1.12% DS. Some photographs of the sludge samples are given in Fig. 1.

2.2. Sludge characterization

Sludge characterization studies were done to determine chemical and thermal properties of the dried sludge samples based on the parameters: pH, salinity, electrical conductivity (EC), DS, volatile solid content (VS), low heating value (LHV), heavy metals (Ba, Cd, Cr, Ni, Pb, Zn, Cu, Mo, As, Sb, Se, Hg), total organic carbon (TOC), total nitrogen (TN), total phosphorus (TP), thermal gravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR) and X-ray powder diffraction (XRD), proximate and ultimate (elemental) analyses, scanning electron microscopy-energy-dispersive X-ray spectroscopy (SEM-EDS), and differential scanning calorimetry (DSC). The analyses were done according to Standard Methods [18] and the most accepted protocols used in the scientific studies.

Fig. 1. Photographs of the dried petrochemical sludge.

Some of the analytical devices used in experimental studies are summarized in Table 1. HHV was measured by using an IKA C200 adiabatic calorimeter (Germany) and LHV was calculated based on the hydrogen and ash content on dry basis as explained in elsewhere [19]. DSC was done under the temperature range of –2°C–400°C. TGA was carried out at 10 mg sample, 100 mL/min Nitrogen gas flow with 10°C/min heating rate. Most of the measurements in this work were done in triplicate and the confidence intervals are at the 95% significance level.

3. Results and discussion

DS and VS values of the dried petrochemical sludge were determined as 96.41% ± 1.12% and 68.93% ± 1.57%, respectively. TOC value was measured as 657 gDS/kg, which is consistent with VS of the sample. The high VS and TOC values showed that the sludge might have a good fuel property in terms of energy recovery. Ayol et al. [19] have highlighted that volatile content of the sludges mainly contained hydrogen, oxygen, and others was very significant since they were converted to the tar gases during the pyrolysis stage and further gasification process. The PHCs and other organic compounds in oily sludge are regarded as four different groups as aliphatics, aromatics, nitrogen sulphur oxygen containing compounds and asphaltenes [2,20]. Among them, aliphatics and aromatics are the main components for up to 75% of PHCs in oily sludge [21].

pH of the sample was found as 8.1. Hu et al. [2] has reported that pH value of oily sludges were usually in a range between 6.5 and 7.5 and its chemical composition varies over a wide range, depending on crude oil source, processing scheme, and equipment and reagents used in refining process. EC value was measured as 0.13 mSi/cm, which is quite low when compared with typical domestic and municipal sludges. TN and TP contents of the sample were found about 0.35% and 0.41%, respectively. Kriipsalu et al. [22] mentioned that TN contents of oily or petro-chemical sludges were less than 3% since most of them were contained in the distillate residue.

Results of ultimate, proximate, and heating value analyses of the petrochemical sludge are shown in Table 2. The sludge had high volatile matter, moderate ash content, and low fixed carbon. Similar observations were obtained in the literature [23,24]. Ayol et al. [19] have stated that the fixed carbon content showed how much of the carbon in a biomass would persist as biochar following the pyrolysis. The volatile organic to fixed carbon ratio of the sample (68/5)

Table 1
Some analytical devices used in experimental studies

Parameter	Standard method	Analyzer
TOC	TS 12089 EN 13137	Hach Lange IL 550-Solid Module, Germany
TN	TS 8337 ISO 11261	VELP Scientifica DK series, Italy
TP	TS EN 13346	Perkin Elmer Lambda 25UV/Vis Spectrometer, USA
Sample preparation for heavy metals	EPA 3051, EPA 3052	Berghof Microwave (Speedwave-MWS-3), Germany
Heavy metals	SM 3120 B	ICP-OES/Perkin Elmer OPTIMA 7000 DV, USA
Heating value	TS 2678, TS 2390	IKA C200 Adiabatic Calorimeter, Germany
TGA	ASTM TGA Methods	Perkin Elmer TGA 4000, USA
DSC		Perkin Elmer – Diamond DSC, USA
Proximate analysis	ASTM D3173, D3174, D3175	
Ultimate analysis		Leco Truspec CHN-S Analyzer, USA
XRD		Rigaku - Rint 2200/PC (Ultima 3), Japan
FTIR		Perkin Elmer, Spectrum BX, USA

Table 2
Ultimate, proximate, and heating value analysis results of the petrochemical sludge

Proximate analysis, wt.% on dry basis				
Moisture (wt.%)	Volatile solids		Fixed carbon	Ash
3.59 ± 1.12	68.93 ± 1.57		5.15	25.93 ± 0.70
Ultimate (elemental) analysis, wt.% on dry basis				
Carbon, C	Hydrogen, H	Nitrogen, N	Sulphur, S	Oxygen*, O
64.23	6.02	0.8	0.96	27.99
High heating value (HHV), MJ/kg			Low heating value (LHV), MJ/kg	
27.18			26.30	

*Calculated by difference

indicated that the sludge could be processed via pyrolysis and gasification process. VS, TOC and C results were found as consistent with each other. The higher volatile content means the higher heating value. The LHV of petrochemical sludge sample was determined as 26.30 MJ/kg DS. LHV is proportional with C and H while it is in reverse with N and S [19,25].

The quantities of heavy metals coming from different sources at the petrochemical industry are given in Table 3. The most abundant heavy metals were determined as Zn, Cr, Cu, Ba, Ni, Mo, and Pb, respectively. American Petroleum Institute (API) [26] reported that different metal contents available in oily sludge taken from petroleum refineries were found in the range of 7–80 mg/kg for Zn, 0.001–0.12 mg/kg for Pb, 32–120 mg/kg for Cu, 17–25 mg/kg for Ni, and 27–80 mg/kg for Cr. In addition to this, more recent researches have reported higher heavy metal concentrations as 1,299 mg/kg for Zn, 60,200 mg/kg for Fe, 500 mg/kg for Cu, 480 mg/kg for Cr, 480 mg/kg for Ni, and 565 mg/kg for Pb, respectively [2].

Table 3
Heavy metal results of the dried petrochemical sludge sample as mg/kg

Ba	16.77
Cd	–
Cr	60.34
Cu	52.612
Mo	3.79
Ni	6.22
Pb	3.66
Zn	163.83
Hg	0.07
Se	0.28
As	0.88
Sb	0.10

When comparing the results with previous findings, most of the metals were almost the same; for example, Cd was not reported in the previous studies as in this work. However, the quantities of some heavy metals differ from the literature data like Pb and Cu. Fig. 2 presents the SEM-EDS results of the sludge sample. Also, EDS results are summarized in Table 4. According to the SEM-EDS analysis, C, O, Al, Si, and Ca were found as abundant elements in the sludge sample. N, P, Na, Mg, Fe, Cl, S, and K were the moderately and/or less available elements in the sample. SEM-EDS results were considered as consistent with the heavy metals, TN, TP, and elemental analysis results.

The thermogram (TG) of the dried petrochemical sludge sample is depicted in Fig. 3. DTG curve attained by the derivatization of TG curve showed that mass loss to with increasing temperature. DTG curve exhibited to three different regions having distinct mass loss as in the study done by Ayol et al. [19]. First region indicated the moisture loss region approximately until 200°C. The mass loss (3.25%) in TG curve depends on moisture at boiling point less than 200°C and the evaporation of light components available in the sludge at temperatures below 160°C–200°C [4,24,27]. The second

Table 4
SEM-EDS analysis results of the petrochemical sludge sample

Element	wt.%	at.%	Element	wt.%	at.%
C	60.81	73.06	S	1.16	0.52
N	0.94	0.96	Cl	1.33	0.54
O	18.73	16.90	K	0.09	0.03
F	0.17	0.13	Ca	2.91	1.05
Na	0.28	0.18	Ti	0.07	0.02
Mg	4.21	2.50	Mn	0.26	0.07
Al	2.74	1.46	Fe	2.32	0.60
Si	3.55	1.82	Zn	0.20	0.04
P	0.24	0.11			

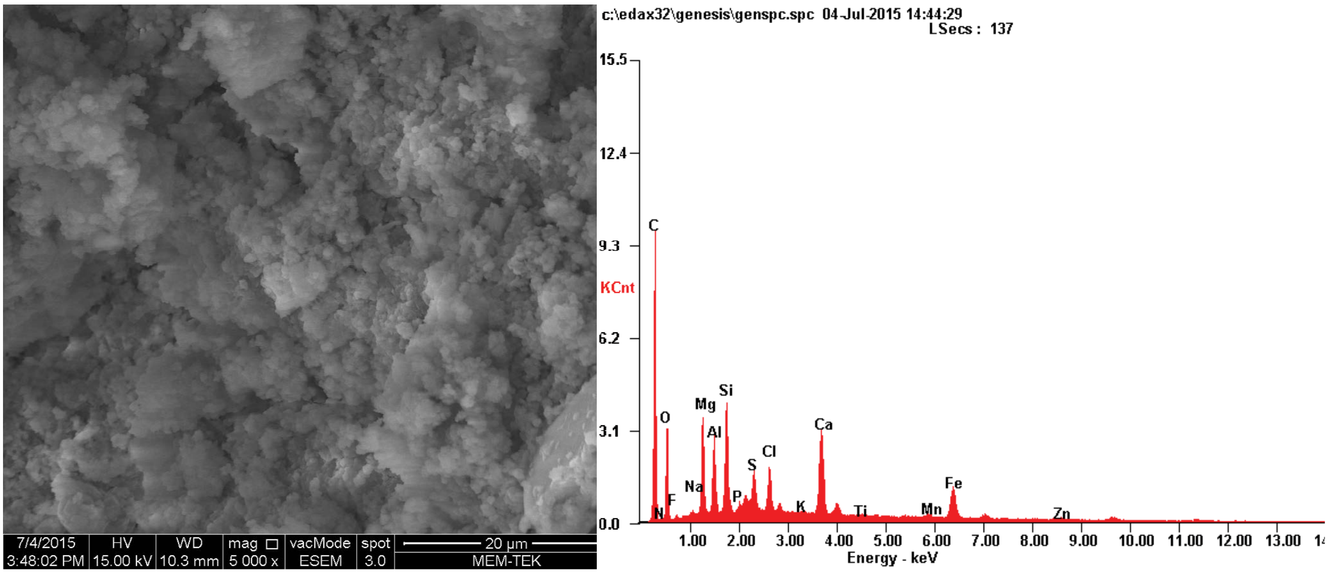

Fig. 2. SEM-EDS results of the petrochemical sludge sample.

region which was between the end of the first region and until 500°C, occurring to pass through gas phase from organics in the sludge, and the third region as burning of fixed carbon is between the end of the second region until 700°C [24,28]. Cheng et al. [27] remarked that the both regions represent the main pyrolysis reaction process, during which the light organic components in the sample were first vaporized, and then the rest of the heavy organic components were cracked into low-molecular-mass gas and oil products. Mass loss of volatile organic materials occurred very rapidly following the first region, after reaching the peak it was gradually slowed down. Mass loss of the sludge sample in the second region was determined as 58.16% at 502°C. The maximum combustion rate of volatile matters was mainly observed up to 500°C. Cheng et al. [27] detected a fourth region in only some samples. They concluded that this region was governed by the condensation reaction of the coke residue and the decomposition of inorganic minerals in the matrix, such as calcite. In this study, fourth region was also observed. The DSC curve and its derivative heat flow curve given in Fig. 4

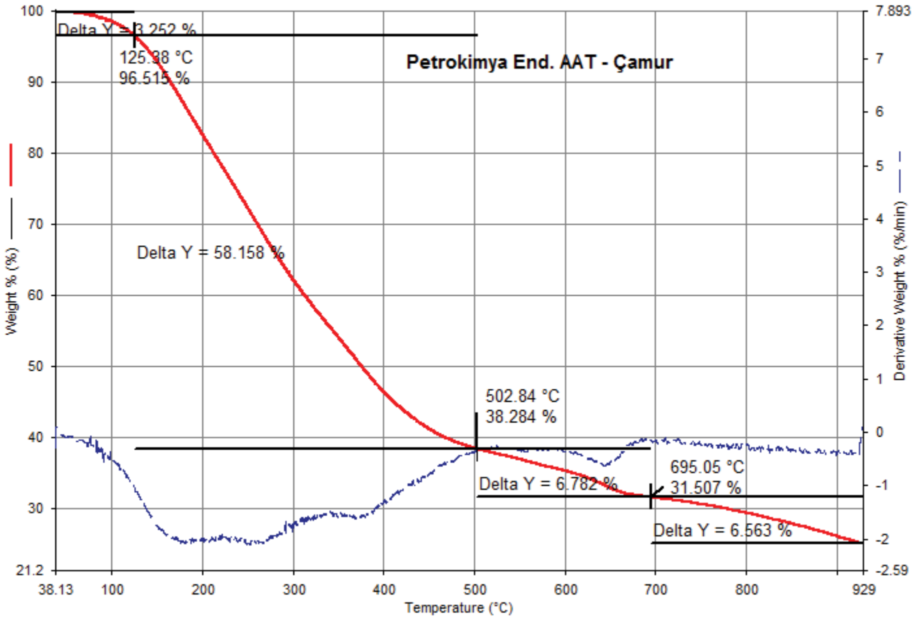

Fig. 3. Thermogram of the dried petrochemical sludge sample.

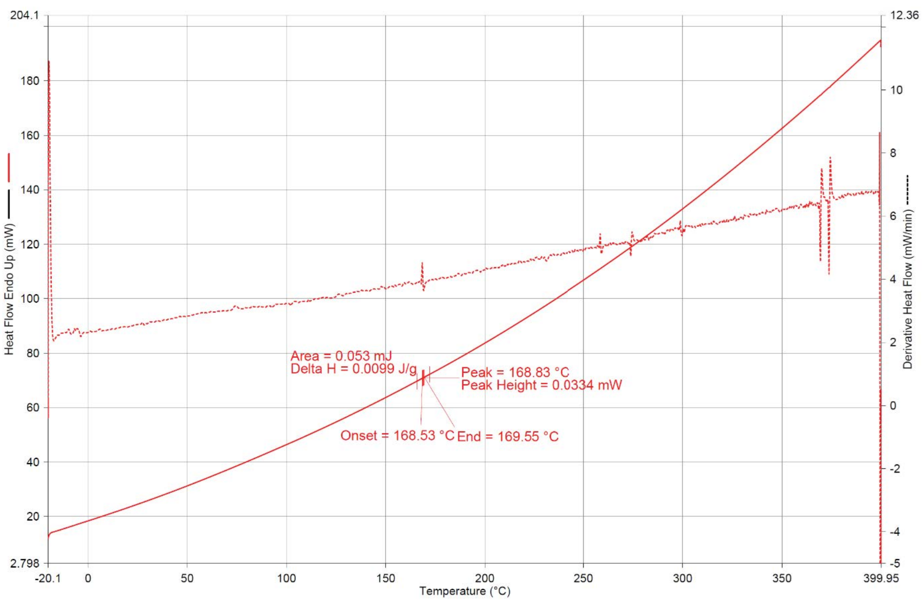

Fig. 4. DSC thermogram of the dried petrochemical sludge sample.

show that the most noticeable peaks were formed at 172°C, 261°C, 278°C, 300°C, 375°C and 379°C. The derivative curve revealing the changes like phase change, glassy transition or combustion did not show a single or certain thermal peak. No thermal changes were observed at high temperatures. Similar observations were also case in our previous studies for yeast industry sludge and municipal sludge [19,25].

FTIR spectra of the petrochemical sludge are given in Fig. 5. The evaluations of FTIR spectra were based on the study done by Yang et al. [29]. The spectrum indicated a broad and strong absorption band between 3,800–3,200 cm^{-1} because of the O–H and NH groups stretching vibrations. The hydrophobic nature of organic phase available in the sample as the methyl and methylene aliphatic groups COO–H,.C–H, Csp$_3$–H was observed at 3,200–2,800 cm^{-1}. The peaks in the range of 1,800–1,400 cm^{-1} represented the carboxylic acid, ketones, aldehydes, phenyl and fluoride (C=N, C=C, phenyl, C–O, F). The peak at 1,000 cm^{-1} indicated the C–O bond (carbohydrates) while the peaks observed in the range of 800–650 cm^{-1} showed C–N and C–X bonds in the sample. The peak at 2,926 cm^{-1} occurred from C–H$_n$ stretching alkyl, aliphatic, aromatic compounds. The stretching vibration at 1,601 cm^{-1} was possibly by C=O in acids and aldehydes. IR absorbance at 1,453 cm^{-1} showed the OH bending acid. The peak at 1,010 cm^{-1} occurred from the C–O stretching and C–O deformation, C–OH (ethanol). The peaks were caused by aromatic rings in the range of 900–700 cm^{-1}. Similar findings were achieved for petrochemical industry sludge in different studies [8,5,30].

XRD patterns of the sludge sample are depicted in Fig. 6. XRD results of the sample showed that the sludge was mainly composed of calcite ($CaCO_3$) and quartz (SiO_2) as inorganic minerals. Ayol et al. [19] have found the comparable results for the municipal sludge.

4. Conclusions

The chemical and thermal characterization of a petrochemical sludge taken from the largest petrochemical complex located in Izmir, Turkey was investigated in detail to evaluate the potential for energy recovery from sludge. The main conclusions from the research were as follows:

- DS and VS values of the dried petrochemical sludge were measured as 96.41% ± 1.12% and 68.93% ± 1.57 %, respectively. The high VS and TOC values, which were consisted with ultimate analysis, showed that the sludge had a good fuel property in terms of energy recovery.
- The sludge had high volatile matter, moderate ash content, and low fixed carbon. The volatile organic to fixed carbon ratio of the sample was an important indicating that the sludge could be processed via pyrolysis and gasification process.
- LHV of the petrochemical sludge sample was determined as 26.30 MJ/kg DS. LHV is proportional with C and H while it is in reverse with N and S.
- The most abundant heavy metals were found as Zn, Cr, Cu, Ba, Ni, Mo, and Pb, respectively. The quantities of some heavy metals differ from the literature data.
- EDS analysis showed that C, O, Al, Si, and Ca were the most abundant elements in the sludge sample while N, P, Na, Mg, Fe, Cl, S, and K were the moderately and/or less available elements in the sample. SEM-EDS results were consistent with the heavy metals, TN, TP, and elemental analysis results.
- TG/DTG curve exhibited to three different regions having distinct mass loss as the moisture loss, passing through gas phase from organics in the sludge, and the burning of fixed carbon. However, a fourth region, which is not common in previous studies in the literature was also

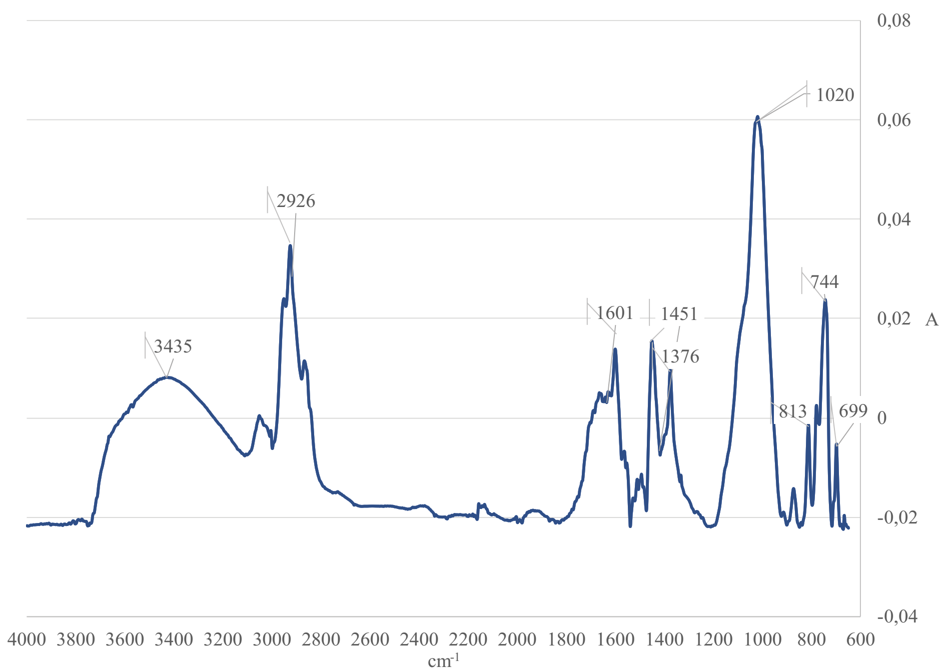

Fig. 5. FTIR spectra of the petrochemical sludge sample.

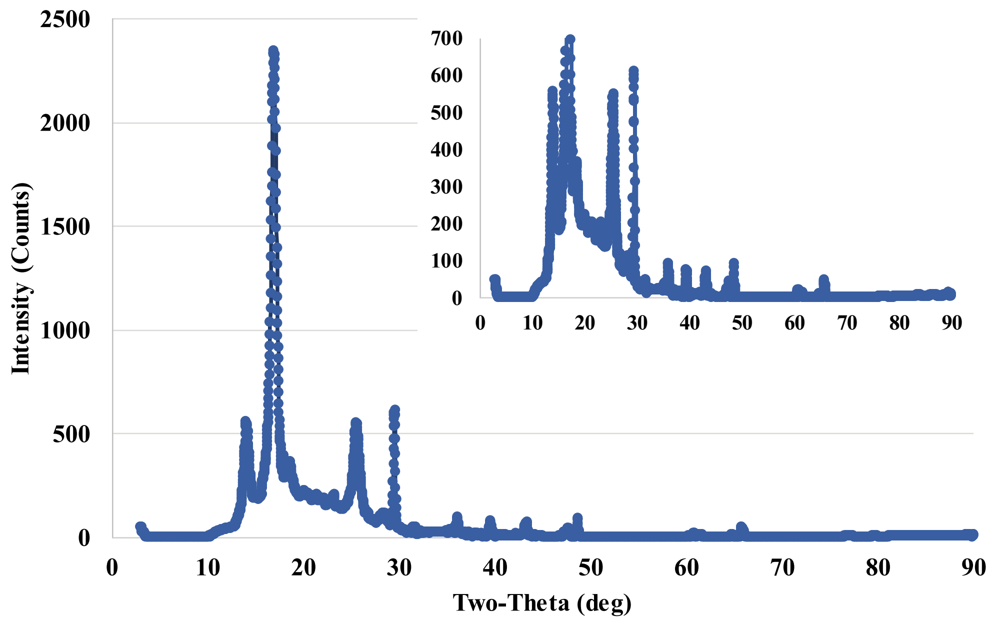

Fig. 6. XRD results of the petrochemical sludge sample (Inset displays the lower magnitude up to 700 counts).

observed. It was governed by the condensation reaction of the sludge sample and the decomposition of the inorganic minerals. Mass loss of the sludge sample in the second region was determined as 58.16% at 502°C.

- As in the TGA results, DSC curve indicated that the sludge had a specific structure and chemical nature supported by the FTIR spectra exhibiting various organic groups.
- XRD results of the sample showed that the sludge was mainly composed of calcite ($CaCO_3$) and quartz (SiO_2) as inorganic minerals.

This characterization study on the petrochemical sludge showed that the sludge having different organic groups, heavy metals and toxic compounds could be used as a significant fuel for energy recovery with the suitable thermal applications like pyrolysis and gasification.

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