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ORIGINAL PAPER

Organic geochemical characterization of crude oil from the Cretaceous reservoir rocks of the Khabbaz Oil Field, Kirkuk Area, Northern Iraq

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Abstract
Six crude oil samples from six wells in Khabbaz Oil Field were analyzed using gas chromatography/mass spectroscopy. One of the samples was from Lower Qamchuqa reservoir. The rest were taken from the Upper Qamchuqa reservoir. Biomarker distribution and characteristics are used to provide information on source of organic matter input, depositional conditions, maturation level, and possible source rock. The *n*-alkanes, terpanes, steranes, and aromatic compounds have been monitored through using specified mass/charge ratios. The crude oils from Khabbaz Oil Field are not affected by biodegradation as it is revealed from the gas chromatogram shapes of the studied samples and the high ratio of saturated and aromatic hydrocarbons to the NSO components. All samples reveal the dominant of short chain *n*-alkanes between C_{15}-C_{18}, with isoprenoids pristane and phytane. The unimodal envelope chromatograms, maximum peak carbon *n*-C_{15}-*n*-C_{20}, and low CPI values (0.89–0.93) indicating a high contribution of aquatic algae organic matter and with minor input from terrestrial plants. The high presence of C_{27} regular steranes also support this conclusion. In the studied oil samples of Khabbaz Oil Field, phytane has relatively higher concentrations than pristane with low Pr/Ph ratios in range 0.60–0.73. This indicates that these oils were derived from organic matter deposited in a marine environment under reducing conditions. In most samples, the abundance of C_{32} homohopanes is moderate and the 22S/22R + 22S epimerization ratio is around 0.55 to 0.61. In addition, gammacerane is recorded in the analyzed samples and the gammacerane index (gammacerane/C_{30} hopane) are indicating that the oils were generated from carbonate source rocks in anoxic and high salinity conditions. The relative C_{30} hopane abundance is less than C_{29} norhopane in all samples with high C_{29}/C_{30} 17α (H) hopane ratios in the range 1.33–1.62 which indicates that such crude oils are derived from carbonate-rich source rock. 20S/(20S+20R) and ββ/(ββ+αα) C_{29} sterane ratios are relatively consistent for all the analyzed samples, ranging between 0.43–0.46 and 0.58–0.63, respectively. This ratio (DBT/Phe) is also used to draw with Pr/Ph ratio as cross-plot, which is clearly indicates that the oils were generated from marine carbonate source rock. The Khabbaz crude oils have a 20S/ (20S +20R) and ββ/(ββ+αα) C_{29} sterane ratios in the range 0.43 to 0.46 and 0.58–0.63, respectively, which indicate thermal maturity (equivalent to peak oil generation stage). The Lower Qamchuqa reservoir is higher in pressure and temperature than the Upper Qamchuqa, as a result the oil of Lower Qamchuqa has higher maturity and API gravity. The studied area is underlain by several rock units which could make potential source rocks for the Khabbaz oils. The biomarkers distribution, and the $\delta^{13}C_{org}$ indicate that the ~~to~~ Chia Gara Formation considered as the main source rock of the oils within Khabbaz Oil Field.

Keywords Qamchuqa · Reservoir · Khabbaz Oil Field · Kirkuk · Hopane · Sterane · Carbon isotope

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Introduction

Kirkuk area is one of the most prolific areas in Middle East in containing petroleum. This area is including several oilfields, such as Kirkuk Oil Field, Jambour Oil Field, Khabbaz Oil Field, etc. (Fig. 1). Many recent studies have been done on these fields, mainly focusing on source rocks and reservoir characteristics, for examples: Mohialdeen and Al-Beyati2007; Al-Qayim et al. 2010; Mohialdeen et al. 2013, Mohialdeen et al. 2015; Kus et al. 2016; Qader and Al-Qayim

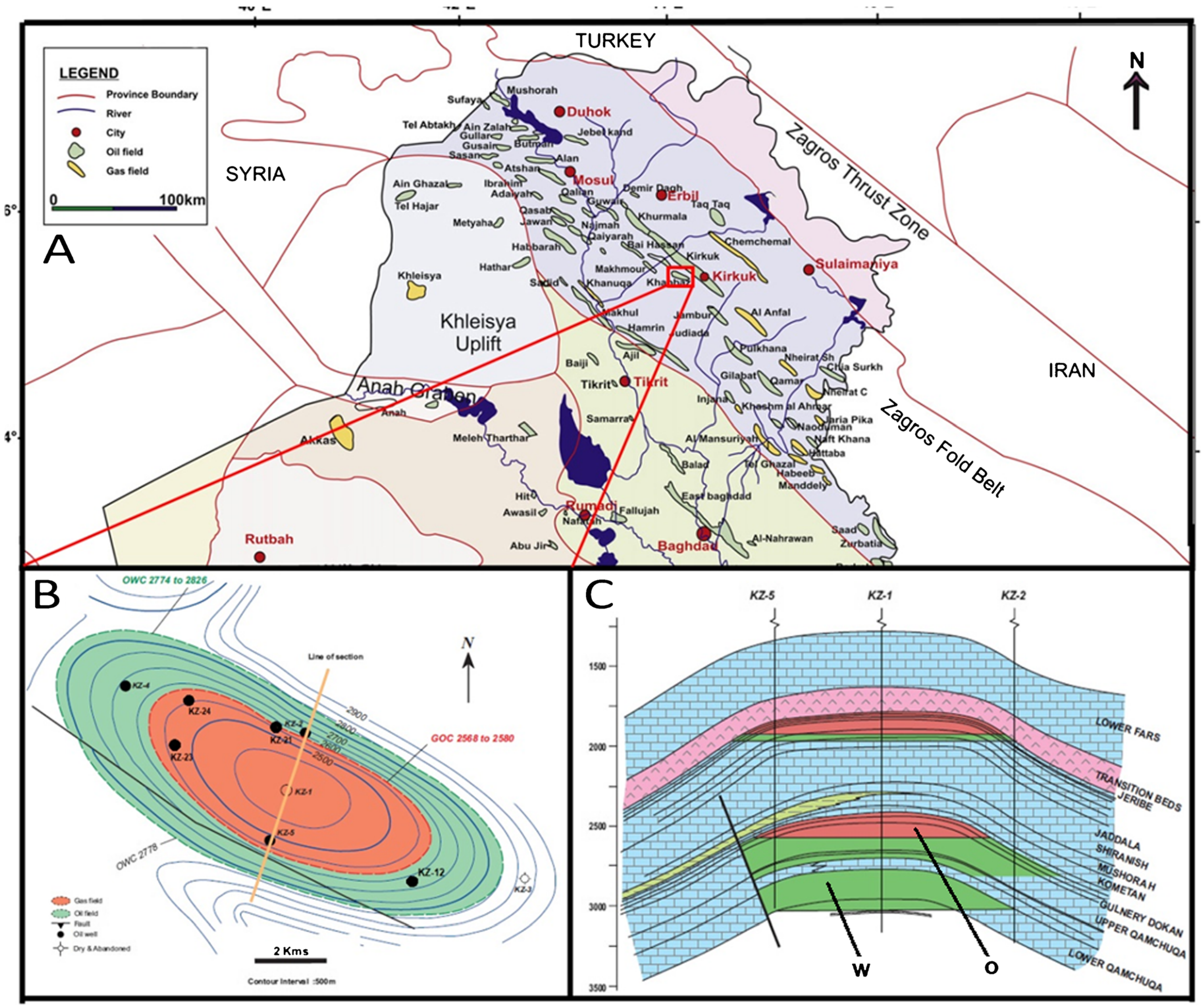

Fig. 1 (A) Regional map of northern Iraq showing the location of the Khabbaz Oil Field (after Al-Ameri and Zumberge 2012). (B) Structure contour map of the top of the Upper Qamchuqa Formation BSL in Khabbaz Oil Field with location of studied wells. (C) Schematic structural cross section along the Khabbaz Field showing the major reservoir zones (after Iraq Development Potential 2003)

2016; Rashid et al. 2020, etc.). One of these oil fields in this area is the Khabbaz Oil field. This field is located about 23 km northwest of Kirkuk City (Fig. 1).

Molecular geochemistry and especially the biomarker distribution in crude oil samples are of vital important in recognition and classification of crude oil sources (Wang *et al*., 1997; Al-Khafaji et al. 2021). These organic compounds often used for determining the different families of crude oils (Al-Ameri 2009; Saeed and Mohialdeen 2016; Al-Khafaji et al. 2018; Alizadeh et al. 2016). The chemical type and relative content of biomarkers in certain oil samples is considered as unique finger print (El-Sabagh et al. 2017). There are many different groups of biomarkers indicating the type of organic source input and thermal maturity of hydrocarbons (Peters et al. 2005; Killops and Killops 2005). Among the various types, hopanes and steranes are the most commonly used molecules (Mohialdeen et al. 2013; Mohialdeen et al. 2016; Mohialdeen et al. 2018).

The main purpose of this study is to investigate the geochemical characterization of the selected oil samples from the Cretaceous Qamchuqa Formation pay zones within the Khabbaz Oil Field using gas chromatography/mass spectrometry. In addition to characterization of the oils and the source rock genetic origin and depositional environments, attempt was made to appraise the thermal maturity level of the studied crude oils.

Geologic setting

Khabbaz Oil Filed (KOF) is located within the Zagros Folded Belt (ZFB) and specifically in the foothill zone (Hamrin-

Makhul Subzone) which is belongs to the Folded Zone of the Unstable Shelf (Buday and Jasim 1987). This zone is characterized by low amplitude of folds, generally rich in hydrocarbons (Al-Ameri and Zumberge 2012). The KOF represents a small subsurface asymmetrical anticline with around 20 Km length and 4 km width . Its northeastern limb steeper than the southwestern limb (Qader 2008). The KOF structure is located between Jambour and Bai Hassan structures (Fig. 1A). The axis of the structure runs in the same direction of most structures in the area with slight shifting from the axis of the adjacent Bai Hassan structure (Fig. 1B). Figure 1B shows the location of selected wells in this study which are Although a large number of wells were drilled in the field, about 40 wells were targeted the Tertiary reservoirs. However, more than half of them penetrating the Upper Qamchuqa reservoir, and only few wells reached the Lower Qamchuqa pay zone (Fig. 1C).

The studied wells are chosen for this study because of the availability of the examined crude oil samples and their fair distribution over the studied field (Fig. 1B). These wells are Kz-1, Kz-4, Kz-12, Kz-21, Kz-23, and Kz-24.

AThe stratigraphic sequence of the studied region is characterized by more than one petroleum systems (Fig. 2). The petroleum systems of KOF are generally associated with the Middle Jurassic to Late Tertiary rock units which are considered as the most important hydrocarbon regime in the region. Among the most potential source rocks for the field and the region are including Sargelu and Chia Gara formations (English et al. 2015). The KOF productions are mostly comes from three reservoirs; the Lower Qamchuqa, the Upper Qamchuqa and Tertiary formations (Figs. 1C and 2).

Samples and analytical procedures

Six crude oil samples from six different producing wells in Khabbaz Oil Field were chosen for analysis by gas chromatography-mass spectroscopy (GCMS). Five of them from the wells Kz-4, Kz-12, Kz-21, Kz-23, and Kz-24 from the Upper Qamchuqa (Mauddud) reservoir at different depths (Table 1). The temperature was 97.22 °C, and 4364 psi pressure were recorded at the depth of 2685m BSL in this reservoir. In addition to one sample from the Lower Qamchuqa (Shu'aiba) reservoir from well Kz-1 at depth of 2950 m BSL with 105.5°C temperature, and 4720 psi pressure. The latter sample was taken for the comparison purpose. Different physicochemical characteristics of the crude oils were determined, including; density, API gravity (ASTM D 1250), sulfur content using X-ray Sulfur Meter (Model RX-500S, Tanaka) according to ASTM D 4294, nickel and vanadium contents using microwave ash (Milestone-Pyro) for digestion then aspirated into flame atomic absorption spectrometer (ZEEnit, Analktikjena Co., TOTAL). The deasphalted crude oils were separated through column chromatography into saturated hydrocarbons, aromatic hydrocarbons, and polar compounds (NSO).

Saturated fractions were analyzed using Agilent 7890 plus HP gas chromatograph equipped with FID using fused silica capillary column HP-5 of 30m in length, 0.32mm in internal diameter and 0.25μm of film thickness. The elution of the studied liquid was achieved with temperature programming from 80°C to 310°C at a rate of 3°C/min. Helium was used as a carrier gas flowing at a rate of 1m/min. The injector and detector temperatures were 320°C and 35°C, respectively. The data were calculated by integration of the area under the peaks using ChemStation software.

The following ions were monitored: m/z191 for tricyclic terpanes and hopanes, m/z 217 for steranes, and m/z 198 for aromatic compounds (Radke 1988; Peters et al. 2005; Al-Khafaji et al. 2021).

Stable carbon isotopes (**δ^{13}C ‰** relative to PDB standard with precision ± 0.02) analyses were conducted on saturated and aromatic fractions of KOF samples by using an elemental analyzer-isotope ratio mass spectrometer (EA–IRMS). The resulting CO_2 from high-temperature (1000 °C) combustion of carbon in the sample was later introduced into a chromatographic column held under isothermal conditions. The chromatographic peaks sequentially entering the IRMS module were separated according to their molecular mass and finally were measured by a Faraday cup universal collector array. The geochemical analyses were carried out by TOTAL Oil Company in their Fluid and Organic Geochemistry Department in France. The API gravity measurements for the oil samples were also carried out in the Research and Quality Control Department of North Oil Company, Kirkuk, northern Iraq.

Results and discussion

Bulk characteristics of crude oils

The bulk oil properties and composition for the studied samples are presented in Table 1. The gravity of the oil sample Kz-1 is 32.30 °API and of rest oil samples is ranged between 22.40 and 24.42 °API (Table 1). As mentioned before, sample Kz-1 is taken from the Lower Qamchuqa reservoir (Fig. 1C) which is deeper than the Upper Qamchuqa reservoir with about 265m. It conceivably has higher temperature and pressure and consequently lighter hydrocarbons is expected (Selley and Sonnenberg 1985). This is also indicated by the sulfur content, the Kz-1 oil contains lowest percentage of sulfur among the studied samples (1.63wt. %). There is a clear relationship between increasing API° gravity and the decreasing of sulfur content in the crude oil samples of Khabbaz Oil Field (Table 1). This relation is interpreted as being due to the effects of increasing thermal maturity (Makeen et al. 2015).

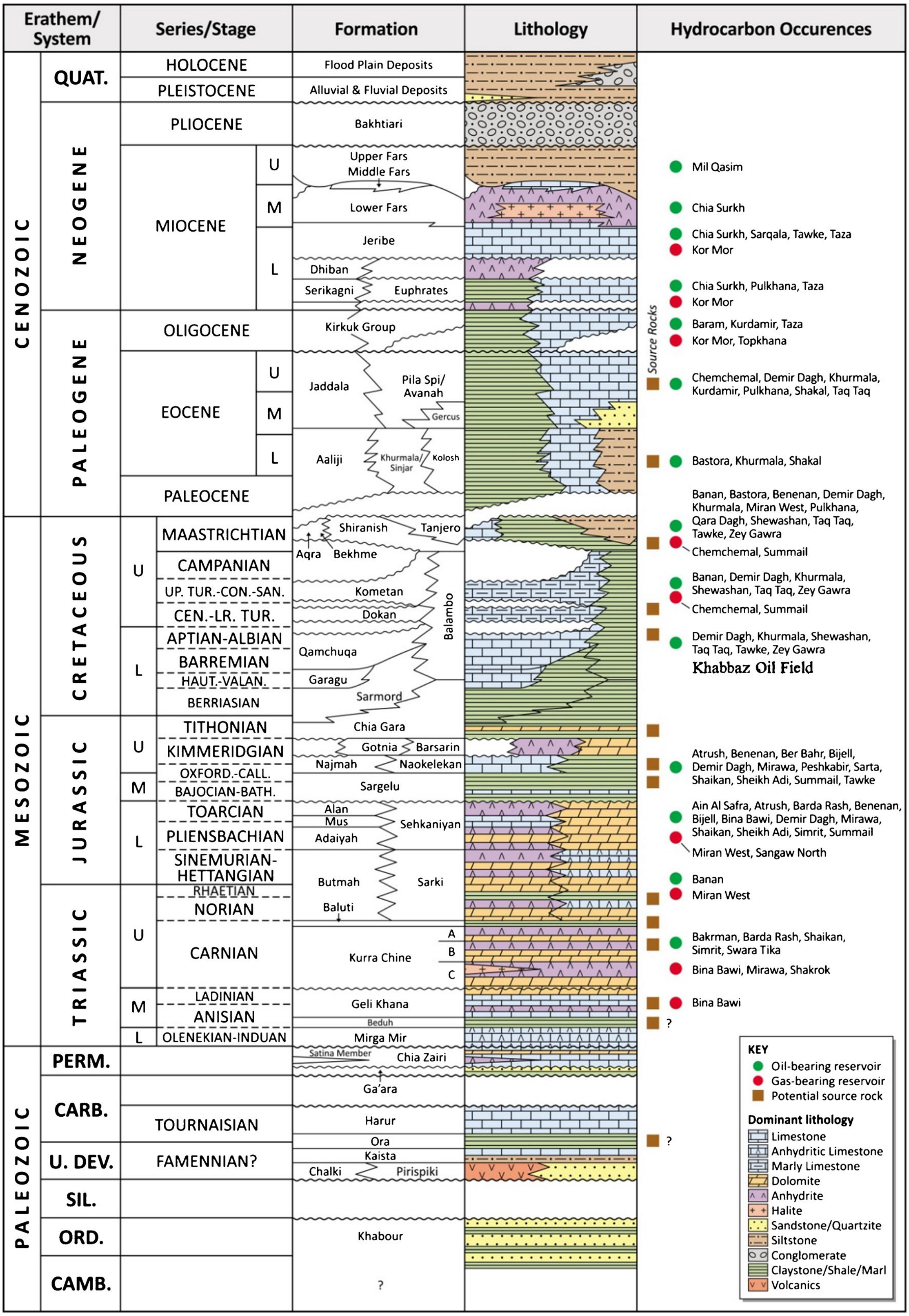

Fig. 2 Generaized stratigraphic column of the Phanerozoic rock units in the Kurdistan region of Iraq with source rocks and reservoirs (after English et al. 2015)

Table 1 Bulk inorganic geochemical analysis, the fraction percentages, and saturated, aromatic, and polar compounds percentages in the studied samples of Khabbaz crude oils

Oil Field	Wells	Depth BSL(m)	Reservoir	API	S	V	Ni	V/Ni	V/(V+Ni)	Fractions wt.%			Hydrocarbons %		
										Distillate	Residue	ASPH.	SAT	ARO	Polar
Khabbaz	Kz-1	2776-3072	Lower Qamchuqa	32.304	1.63	<5	<5			29.5	70.5	1.2	50.2	45.7	4.1
Khabbaz	Kz-4	2710-2751	Upper Qamchuqa	22.403	3.85	61	17	3.588	0.78205	20.3	79.7	11.2	30.5	53.9	15.6
Khabbaz	Kz-12	2666-2694	Upper Qamchuqa	24.422	2.4	33	13	2.538	0.71739	28.7	71.3	6.2	45	41.1	13.9
Khabbaz	Kz-21	2645-2705	Upper Qamchuqa	23.259	3.21	30	7	4.286	0.81081	21.7	78.3	6.6	34.5	52.5	13
Khabbaz	Kz-23	2640-2687	Upper Qamchuqa	23.27	3.22	26	6	4.333	0.8125	21.4	78.6	4.5	34.3	53.6	12.2
Khabbaz	Kz-24	2627-2683	Upper Qamchuqa	23.27	3.33	27	6	4.5	0.81818	19.3	80.7	4.6	36.3	54.2	9.4

BSL below sea level; *API* API gravity; *S* sulfur wt.%; *V* vanadium (ppm);*Ni* nickel (ppm);*SAT*:saturates; *ARO* aromatics; *ASPH* asphaltenes

On the other hand, crude oils that contain considerable quantities of sulfur compounds (>0.5%) are called sour crude oils, whereas those with less sulfur (<0.42%) are called sweet crude oils (Peters et al. 2005). Table 1 shows the sulfur content for the Khabbaz oil samples, ranging from 1.63 to 3.85% and thus indicates sour crude oils.

The crude oil fractions were measured and plotted on the ternary diagram of saturated-aromatic-polar compounds (NSO) (Fig. 3). Saturated compounds ranged between 30.5 and 50.2%, aromatics 41.1 and 53.9% and polar compounds 4.1 and 15.6% (Table 1). Two oil samples, Kz-1 and Kz-12, have relatively higher saturated compounds, 50.2% and 45% respectively. The Kz-1 oil sample belongs to the Lower Qamchuqa reservoir which contains the lowest ratio of polar compounds (4.1 %). The analyzed oil samples include more saturated and aromatic hydrocarbons than the NSO components (Fig. 3), which indicates a lack of biodegradation because this process increases NSO components (Peters et al. 2005).

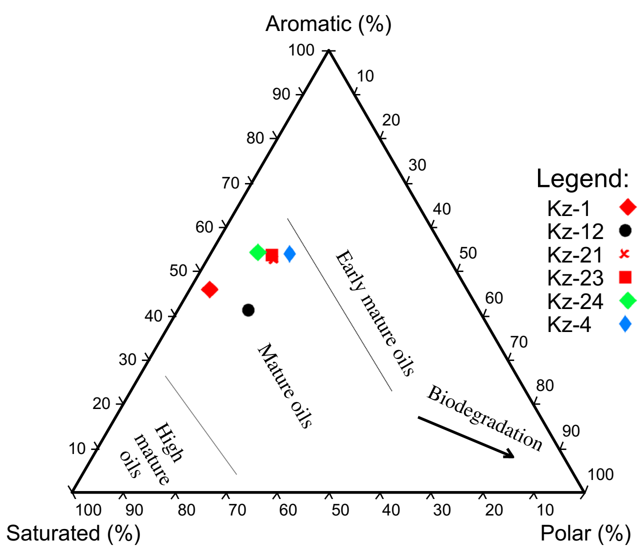

Fig. 3 Ternary diagram showing the gross composition (i.e,. saturated hydrocarbons, aromatic hydrocarbons, and NSO) of the analysed oil samples. The studied oils from Khabbaz Oil Field are mature and nonbiodegraded (Basic plot is after Peters et al. 2005)

From maturity point of view all samples are locating within the mature field (Fig. 3). In our opinion all samples may be related to one family of oil but with different grades of maturity!

Two of the transition metals (Vanadium and Nickel) ratios in crude oils are useful in the determination of source rock type, depositional environment and maturation because they remain unchanged irrespective of diagenetic and in reservoir alteration effects (Mohialdeen and Raza 2013; Makeen et al. 2015; El-Sabagh et al. 2017). The absolute concentration of both elements in crude oil samples can be used to classify and correlate oils. Both vanadium and nickel are the major metals in petroleum (Cooper 1990; Udo et al. 1992). Vanadium is generally enriched in comparison with Ni in anoxic marine environments (Peters and Moldowan 1993). Lewan (1984) indicted that a high V/Ni ratio reflects reducing conditions. Based on Galarraga et al. (2008) a V/Ni ratio >3 indicates that the source rock was deposited in a reducing environment, while V/Ni ratio ranging from 1.9 to 3 indicate deposition under suboxic conditions with precursor organic matter of mixed origin that is continental and predominantly marine. The studied samples show that V concentrations are more than concentration of Ni (Table 1; Fig. 4). The V/Ni ratios are high (2.54–4.50), thus based on Galarraga et al. (2008) reveal that the oils are generated from rocks deposited in suboxic to reducing marine conditions.

Biomarker characteristics and crude oil biodegradation

Biomarker analysis of saturated fractions focused on three groups of compounds, *n*-alkanes, steranes, and pentacyclic triterpanes (hopanes) and their derivatives. The results of the

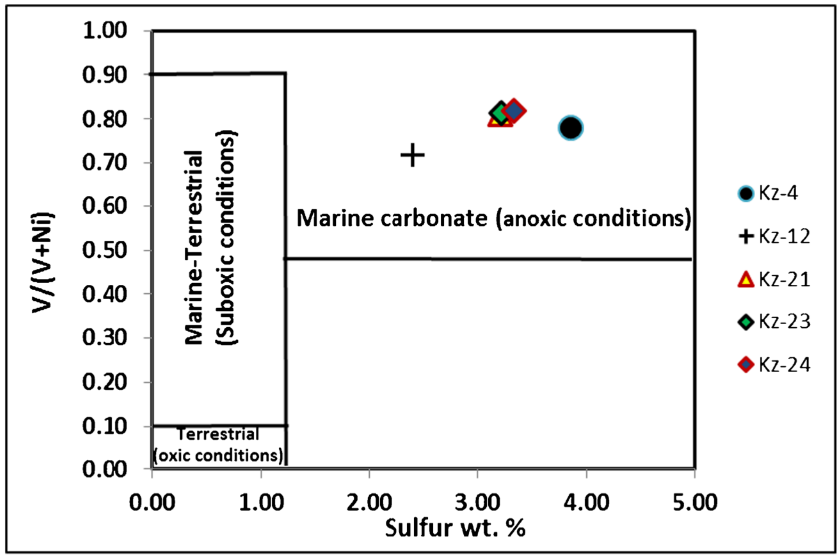

Fig. 4 The relationship between sulfur wt. % and V/(V+Ni) and the depositional environments of the studied crude oils from Khabbaz Oil Field, indicating the marine carbonate with anoxic condition for the source rocks (Basic diagram after Galarraga et al. 2008)

analysis based on integration of peaks in various ion chromatograms are summarized in Table 2.

One of the important points to evaluate the crude oil samples is the effect of biodegradation, which dramatically affect the hydrocarbon fluid properties (Miiller et al. 1987). The degree of biodegradation can be determined through the distribution of compounds in the sample. Losing of *n*-alkanes followed by loss of a cyclic isprenoids indicate to early stage of biodegradation (Peters and Moldowan 1993; Peters et al. 2005). Other compounds such as cyclic saturated hydrocarbons and aromatic compounds are more resistant to biodegradation (Larter et al. 2005). The crude oils from Khabbaz Oil Field are not affected by biodegradation as it is revealed from the gas chromatogram patterns of the studied samples (i.e., unimodal envelope, dominant light *n*-alkanes, and flat base line) (Fig. 5). Another point support this conclusion is the high ratio of saturated and aromatic hydrocarbons than the NSO components (Fig. 3; Table 1), because this process increases the NSO components (Peters et al. 2005). Another indicator is the absence or very rare presence of demethylated hopanes in the m/z 177 fragmentograms for the studied samples (Fig. 6) which also indicates that the oils were not biodegraded (Volkman et al. 1983). The characteristic signals for 25-norhopane (demethylated hopanes) were not appeared in m/z 177 fragmentograms before the major peak of 17α (H)-30-norhopane (Fig. 6).

n-alkane and isoprenoids

The *n*-alkanes and isoprenoid hydrocarbons were identified from their mass spectra in the total ion chromatogram (TIC) and the m/z 85 mass chromatogram, which was also used for peak integration (Fig. 5). The chromatograms of saturated hydrocarbons of all studied oil samples from Khabbaz Oil Field show similar *n*-alkane distributions, suggesting only one source rock (Mohialdeen et al. 2013). All samples reveal the dominance of short chain *n*-alkanes between C_{15}-C_{18}, with isoprenoids pristane (Pr) and phytane (Ph). The unimodal

Table 2 Biomarker indicators of the source organic matter and depositional environment for saturated fraction in the crude oils of Khabbaz oil samples

								Triterpanes and Terpanes m/z 191				
Oil Field	Wells	Depth BSL(m)	Reservoir	Pr/ Ph	Pr/n-C_{17}	Ph/n-C_{18}	CPI	C_{29}/ C_{30}	Ts/ Tm	G/ C_{30}	31H/ 30H	34H/ 35H
Khabbaz	Kz-1	2776-3072	Lower Qamchuqa	0.60	0.13	0.23	1.00	1.33	1.31	0.09	1.03	0.74
Khabbaz	Kz-4	2710-2751	Upper Qamchuqa	0.65	0.16	0.27	0.99	1.53	0.21	0.12	0.89	0.71
Khabbaz	Kz-12	2666-2694	Upper Qamchuqa	0.73	0.23	0.33	0.99	1.33	0.31	0.10	0.88	0.77
Khabbaz	Kz-21	2645-2705	Upper Qamchuqa	0.68	0.17	0.28	0.99	1.62	0.19	0.11	0.90	0.70
Khabbaz	Kz-23	2640-2687	Upper Qamchuqa	0.71	0.17	0.26	0.98	1.60	0.22	0.01	0.94	0.70
Khabbaz	Kz-24	2627-2683	Upper Qamchuqa	0.65	0.17	0.28	0.98	1.60	0.21	0.11	0.91	0.70

BSL below sea level; *Pr* pristane ; *Ph* phytane; *CPI* carbon preference index: $\{2(C_{23} + C_{25} + C_{27} + C_{29})/(C_{22} + 2[C_{24} + C_{26} + C_{28}] + C_{30})\}$; C_{29}/C_{30}: C_{29} norhopane/C_{30} hopane; Ts: C_{27} 18α(H)-22,29,30-trisnorneohopane; Tm: C_{27} 17α(H)-22,29,30-trisnorhopane; 31H/30H: C_{31R}/C_{30}: C_{31} regular homohopane/C_{30} hopane; 34H/35H: C_{34} homohopane / C_{35} homohopane

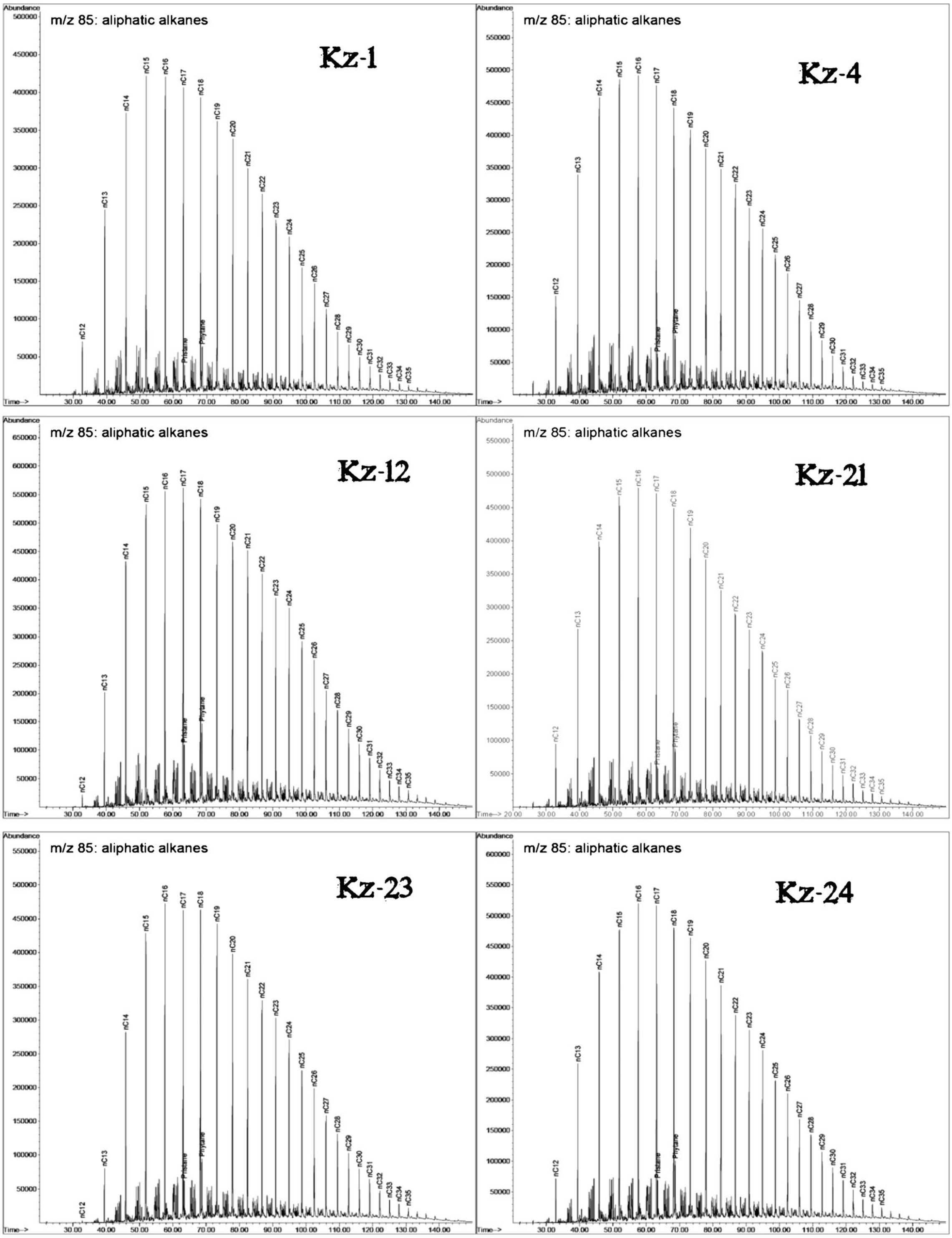

Fig. 5 Mass chromatograms (m/z 85) of the studied crude oil samples of Khabbaz Oil Field, northern Iraq

envelope chromatograms, maximum peak carbon n-C_{15}-n-C_{20}, and the low carbon preference index (CPI) values (0.89-0.93) (Fig. 7) indicate that organisms derived from phytoplankton, zooplankton and benthic bacteria with no terrestrial plants (Brassell et al. 1978). Therefore, it can be deduced that the studied oils were generated from source rock contained planktonic and bacterial organisms with minor contribution from terrigenous organic matter and marine depositional condition (Tissot et al. 1978; Murray and Boreham 1992; Lai et al. 2018).

The Pr and Ph distributions considered as the most important acyclic isoprenoids hydrocarbons in terms of

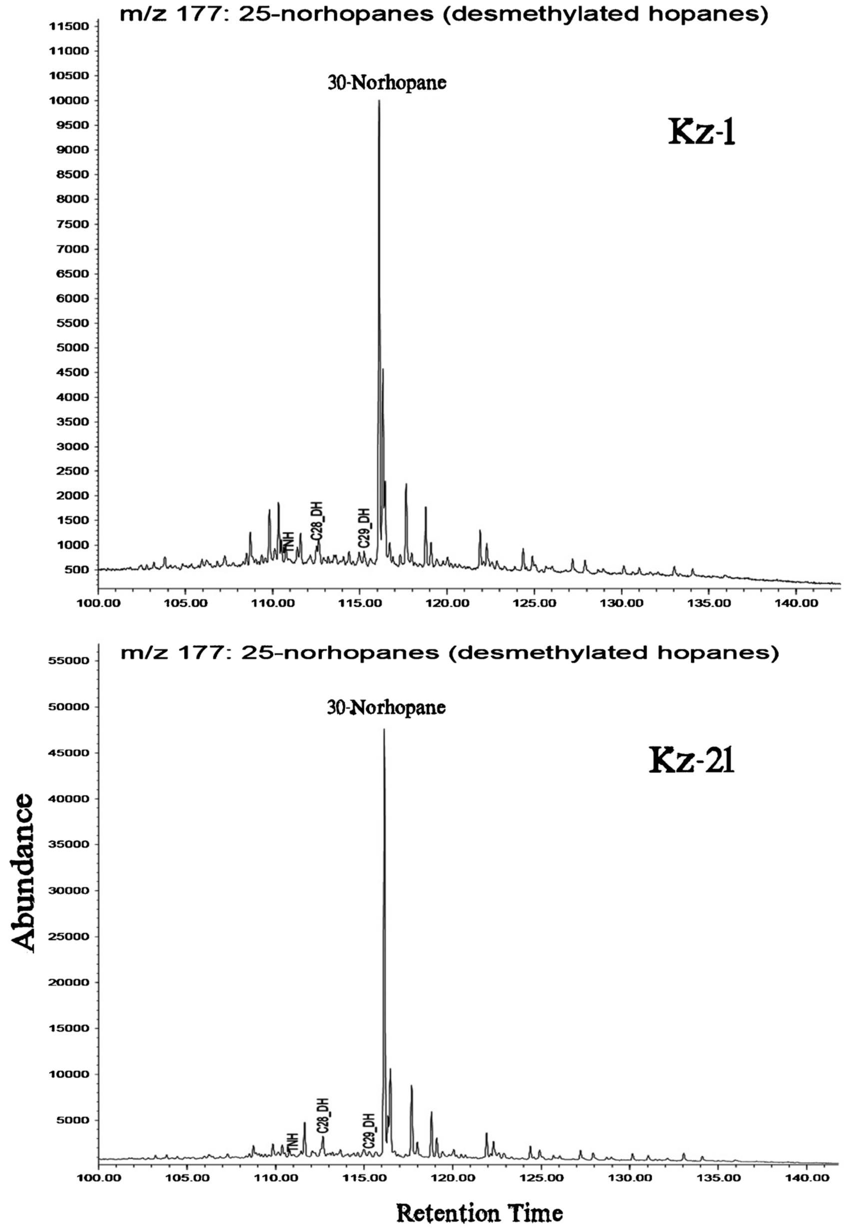

Fig. 6 Representative m/z 177ion fragmentograms of 25-norhopanes (demethylated hopanes). The dominance is 30-Norhopane, while C_{29}-demethylated hopanes are rare

concentration (Powell and McKirdy 1973), which reflect the paleodepositional conditions of source rocks. It is also considered as potential indicators of the redox conditions during sedimentation and diagenesis (Escobar et al. 2011). In the studied oil samples of KOF, phytane has relatively higher concentrations than pristane (Table 2) with low Pr/Ph ratios in range 0.6-0.73. This indicates that these oils were generated from organic matter deposited in a marine environment under reducing conditions (Tissot and Welte 1984; Hakimi and Abdulla 2013; Alizadeh et al. 2017). The relatively high Pr/Ph ratios of some of the oils indicate their high maturation levels (Onojake et al. 2015). The gross composition of the examined oil samples shown in Fig. 3. also support the high maturation stage of the Khabbaz oil samples. The graphical presentation between Pr/n-C_{17} and Ph/n-C_{18} is clearly indicates the marine organic matter which deposited under reducing environment (Fig. 8).

Terpanes

Hopanes found in the aliphatic fraction are pentacyclic triterpenoids derived from cell membranes of prokaryotes (i.e. heterotrophic bacteria and phototrophic cyanobacteria) (e.g., Ourisson et al. 1982; Ourisson and Rohmer 1992). This biomarker group is, like the steranes, characterized by numerous maturity-sensitive stereoisomers (Seifert and Moldowan 1980). It is possible to identify the majority of compounds in the m/z 191 fragmentograms (Brooks and Smith 1969; Peters et al. 2005).

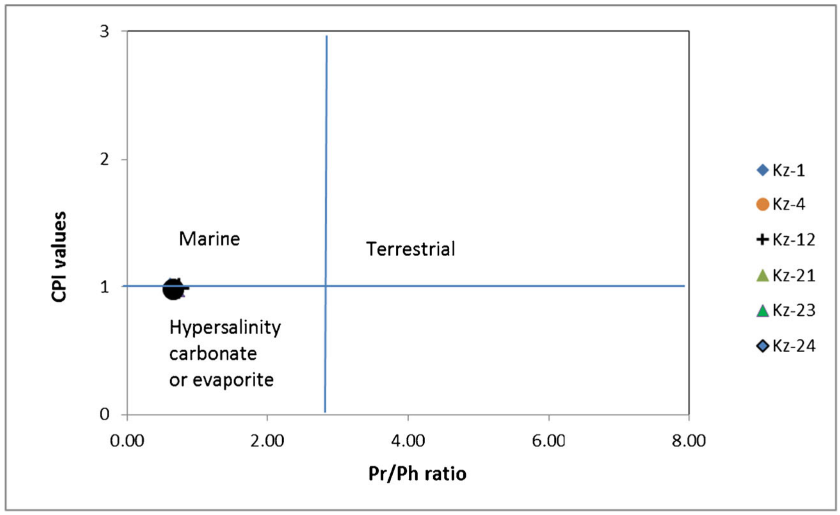

Fig. 7 Cross plot between Pr/Ph ratio and CPI values of the studied samples, indicating carbonate marine environment. The hypersalinity of environment may be present to some degree as the samples located on the border to evaporate division

The distribution and relative abundances of terpanes (tricyclic, tetracyclic, pentacyclic and hexacyclic terpanes) as calculated from m/z 191 chromatograms are shown in Fig. 9, and their parameters are given in Table 2. The analyzed oil samples are characterized by high proportion of hopanes relative to tricyclic terpanes (Fig. 9). Hopane biomarkers are dominated by the presence of C_{30}-hopane and C_{29}-norhpane with significant 17α(H)-trisnorhopane (Tm) and C_{31}–C_{35} homohopanes (Fig. 9). C_{29}-norhpane is higher than C_{30}-hopane in all of the analyzed oil samples and C_{29}/C_{30} ratios are >1 (Table 2).

Source rock and maturity level are affecting the Ts/(Ts + Tm) ratio, and thus it makes the most reliable indicator as a maturity for oils from the same organofacies (Moldowan et al. 1986). Ratios of Ts / (Ts + Tm) are sensitive to clay-catalyzed reactions, and oil from carbonate source rock generally has lower Ts/(Ts +Tm)ratios (<0.25) than that from shale (Peters et al. 2005; Peters et al. 2019). The KOF oils have low 18α (H)-trisnorhopane (Ts) relative to 17α (H)-trisnorhopane (Tm), except sample Kz-1 from the Lower Qamchuqa reservoir which has Ts/Tm =1.31 (Fig. 9; Table 2). This is possibly due to difference in maturity level, rather than source rock types. Homohopane distributions are dominated by C_{31} homohopane (Fig. 9) and ratios based on the homohopane distribution such as the C_{31}-22R-hopane/C_{30}-hopane and C_{34}/C_{35} ratios were determined (Table 2). In most samples, the abundance of C_{32} homohopanes is moderate and the 22S/22R + 22S epimerization ratio is around 0.55 to 0.61 (Table 2). The similar distribution patterns of m/z191 mass chromatograms show that the oils are all related to one family. The relative C_{30} hopane abundance is less than C_{29} norhopane in all samples (Fig. 9) with high C_{29}/C_{30} 17α (H) hopane ratios in the range of 1.33 -1.62 (Table 2), which indicates that such crude oils are derived from carbonate-rich source rock (Connan et al. 1986; Waples and Machihara 1991; Mohialdeen et al. 2013; El Nady et al. 2014; Onojake et al. 2015). The studied oils have low tricyclic terpane (C_{19} and C_{20}) that originated from higher plants (Fig. 9).

High C_{35} homohopanes are believed to indicate a highly reducing marine environments, while low C_{35} homohopanes

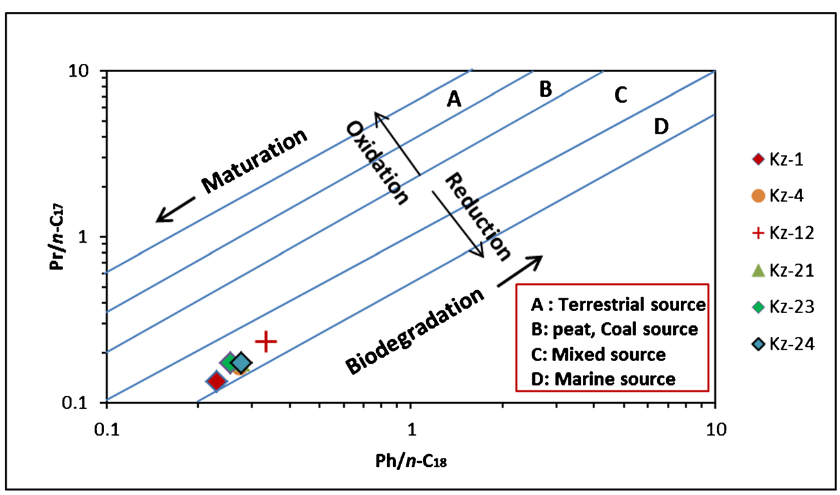

Fig. 8 Cross-plot of Pr/n-C_{17} versus Ph/n-C_{18} for the analyzed oil samples from Khabbaz Oil Field; the close correlations suggest that the analyzed oils were generated from similar source rocks

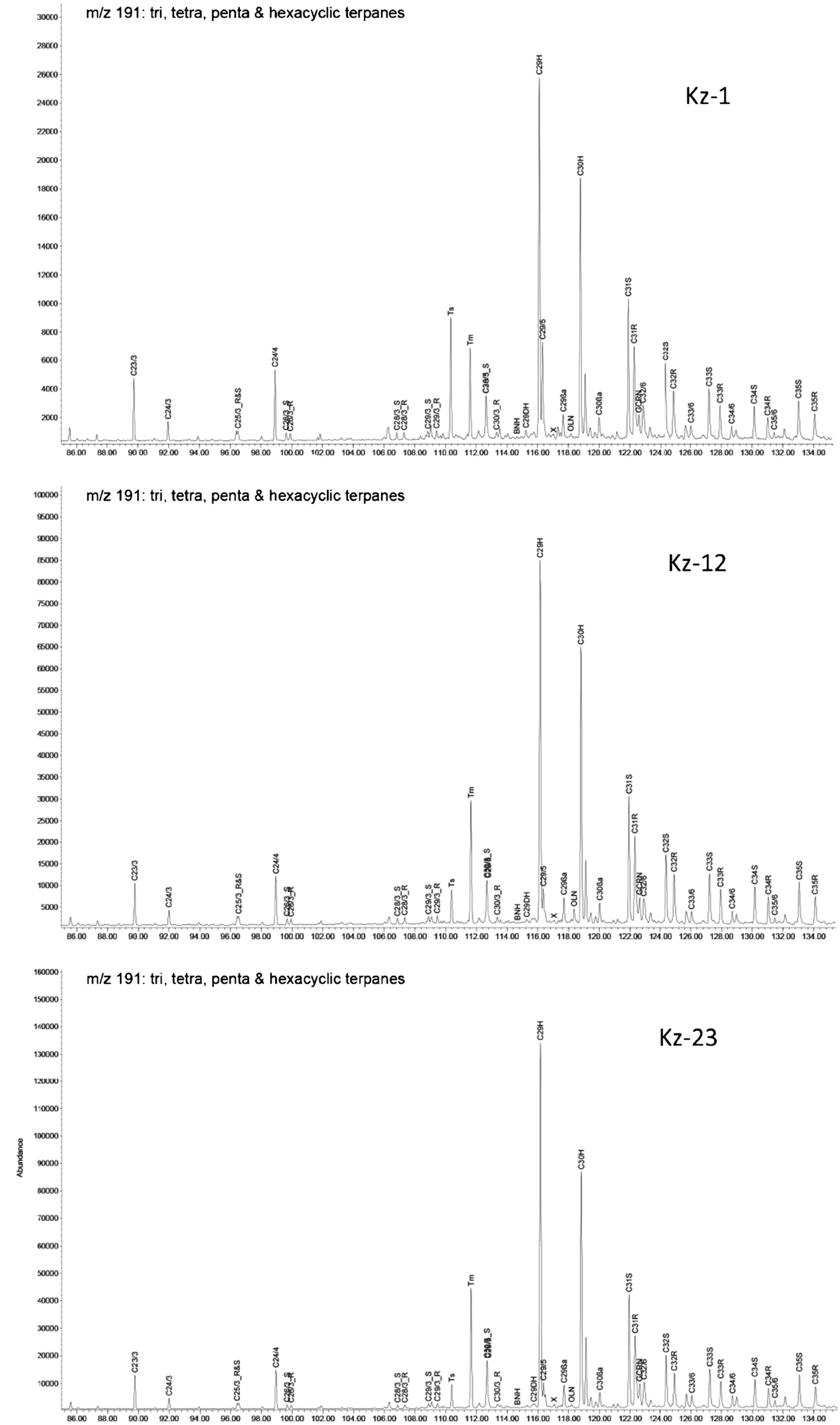

Fig. 9 Representative mass chromatograms (m/z 191) showing terpanes biomarkers distribution in the studied crude oil samples of Khabbaz Oil Field, Kirkuk, northern Iraq

are typically shown in oxidizing water conditions (Peters and Moldowan 1991). The distribution of homohopanes within the studied samples of KOF is clearly show the relatively high C_{34} and C_{35} homohopanes concentrations (Table 2; Fig. 9) which is used as an indicator to highly reducing marine environment. In addition, the abundance of C_{23} tricyclic terpane and C_{24} tetracyclic terpane in the m/z191 mass chromatograms (Fig. 9) indicate the contribution of algae-rich organic matter (Zumberge 1987; Hanson et al. 2000; Alizadeh et al. 2017).

Gammacerane is also identified in small amounts in all the studied oils (Fig. 9). The gammacerane index (Gammacerane/ C_{30}hopane) ranged from 0.01 to 0.12 (Table 2). The presence of gammacerane is used as an indicator for highly salinity-stratified water column during deposition of source rocks (Sinninghe et al. 1995; ten Haven et al. 1989). Although it does not occur in high amount, gammacerane is an indicator of anoxic/hypersalinity conditions by which its occurrence is often coeval with the increased abundance of C_{35} hopanes (Schaeffer et al. 1995). The molecular geochemistry of Chia Gara Formation from Kirkuk Oil Field, is also used to indicate the presence of nearly same amount of gammacerane (Mohialdeen and Hakimi 2016). In addition, the relatively low Ts/Tm ratios (0.19 to 0.31 except sample Kz-1 with 1.31) also suggesting that the oils are generated from marine carbonate source rocks (Table 2; Fig. 9). It can be deduced that the high percentage of Ts/Tm in sample Kz-1 is returned to the higher maturity level as compared to the other samples and possibly not to different source rock type.

Steranes

Steranes are derived from the sterols of cell membranes of eukaryotes, mainly algae and higher plants (Volkman 1986; Schwark and Empt 2006). The distributions of diasterane and sterane biomarkers are explained by the m/z 217 ion chromatograms (Fig. 10). Huang and Meinschein (1979) concluded that the dominance of C_{27} sterane indicates that it is primarily derived from algae, while the C_{29} steranes are typically associated with land plants. The study oils are characterized by high abundances of C_{27} regular steranes (43.7–47.0%) compared with C_{28} and C_{29} homologues (Table 3; Fig. 11). This reflects the high contribution of aquatic algal organic matter and carbonate marine of the source rocks (Huang and Meinschein 1979; Peters and Moldowan 1993; El-Sabagh et al. 2017). The 20S/ (20S+20R) and $\beta\beta/(\beta\beta+\alpha\alpha)$ C_{29} sterane ratios were relatively consistent for all the analyzed samples, ranging between 0.43–0.46 and 0.58–0.63, respectively (Table 4).

Another important biomarker ratio is the diasterane/ sterane, which is commonly used to distinguish carbonate from clay-rich source rocks. The low value of this ratio indicates anoxic clay-poor source rocks, while high values reveal oxygenic and clay-rich depositional environment of the organic matter (Wang et al. 2015; Peters et al. 2017). The disterane/sterane ratio for the KOF oils ranges from 0.19 to 0.34 (Table 3), this indicates that these crude oils were generated by source rocks lean in clay minerals and mostly carbonate (Hegazi and El-Gayer 2009; Mohialdeen et al. 2013).

Aromatics

Aromatic biomarkers can provide valuable information on organic matter input, biodegradation, maturity, etc. (Peters et al. 2017). Among the aromatic biomarkers nephthalens, phenanthrenes, dibenzothiophene, and methyldibenzothiophenes are used to deduce the source rock types of crude oils (e.g. Budzinski et al. 1995; Hughes et al. 1995; Mohialdeen et al. 2015; Peters et al. 2019). The studied samples from KOF have dibenzotheophene/ phenanthrene (DBT/Phe) ratio in the range 2.27 to 4.49 (Table 4; Fig. 12). Such values pointing to a carbonate source rocks of these oils (Hughes et al. 1995; Zhang and Huang 2005; Mohialdeen et al. 2013). This ratio (DBT/Phe) is also used with Pr/Ph ratio as cross-plot (Table 4; Fig. 13) which is clearly indicate that the oils were generated from marine carbonate source rock.

The methyldibenzotheophene isomers (MDBT) are also characterized in mass chromatograms m/z198 for the studied samples (Fig. 12). The isomers have the following order 4 >2+ 3< 1 (Fig. 12) which indicates the carbonate source rocks (Hughes et al. 1995; Mohialdeen et al. 2015; Mohialdeen et al. 2018).

Carbon isotope composition ($\delta^{13}C_{org}$)

The application of stable carbon isotope of organic matter ($\delta^{13}C_{org}$) has become an important tool in oil-oil and oil-source rock correlations, and in assessing biodegradation of crude oil (Mohialdeen and Hakimi 2016). The $\delta^{13}C_{org}$ has been used to evaluate the source of organic matter and to evaluate the depositional environments (Sofer 1984; Jasper and Gagosian 1990; Bird et al. 1994; Mason et al. 1995; Collister and Wavrek 1996; Yuones and Philp 2005). High and moderate $\delta^{13}C_{org}$ values are inferred to have originated from marine algae and microorganisms, while low $\delta^{13}C_{org}$ values indicate a predominantly terrigenous origin (Lini et al. 1992; Morgans-Bell et al. 2001; Nunn et al. 2009; Hammer et al. 2012). The stable carbon isotope compositions of the saturated fraction ($\delta^{13}C_{sat.}$) range between -26.6‰ and -27.5‰, while the range for aromatic fraction

Table 3 Sterane biomarkers (from m/z 217 fragmentograms) and carbon isotope occurrence in the studied oils from Khabbaz crude oils

Oil Field	Wells	Depth BSL(m)	Reservoir	C_{27}/C_{29}reg.ster	Regular Steranes %			Dis./Ster	$\delta^{13}C$ (‰ PDB)	
					C_{27}	C_{28}	C_{29}		Sat.	Aro.
Khabbaz	Kz-1	2776-3072	Lower Qamchuqa	1.20	45.00	17.50	37.50	0.19	-27.5	-27.3
Khabbaz	Kz-4	2710-2751	Upper Qamchuqa	1.24	44.50	19.40	36.00	0.22	-27	-27
Khabbaz	Kz-12	2666-2694	Upper Qamchuqa	1.31	45.90	10.00	35.10	0.34	-26.6	-26.5
Khabbaz	Kz-21	2645-2705	Upper Qamchuqa	1.23	43.70	20.80	35.40	0.31	-26.9	-26.9
Khabbaz	Kz-23	2640-2687	Upper Qamchuqa	1.30	45.90	18.80	35.30	0.28	-26.9	-27
Khabbaz	Kz-24	2627-2683	Upper Qamchuqa	1.36	47.00	18.40	34.60	0.29	-26.9	-26.9

BSL below sea level; C_{27}/C_{29} reg.ster: Dis./Ster: Diasteranes/Steranes;*Sat* saturated; *Aro* aromatics

($\delta^{13}C_{aro.}$) are -26.5‰ to -27.3‰ (Table 3). This indicates an almost marine origin of the Qamchuqa reservoir oils in Khabbaz Oil Field (Fig. 14).

The results of $\delta^{13}C_{org}$ for crude oils from the Upper Qamchuqa reservoir are close to each other, as well as show a well matching with the sample from the well Kz-1 which is producing from Lower Qamchuqa reservoir (Qader 2008). These results indicate that the oils of the two reservoirs belong to the same source rock and they belong to the same oil family.

Similarly, Mohialdeen and Hakimi (2016) studied the Chia Gara Formation from Kirkuk Oil Field, they had recorded the $\delta^{13}C_{org}$ values ranging from -29.99‰ to -26.93‰, suggesting that the Chia Gara rocks contain organic matter originating from a marine input (Tables 3 and 5). This supports our suggestion that the oils from KOF may be generated from the Late Jurassic-Early Cretaceous Chia Gara Formation.

Thermal maturity of the crude oils

In order to evaluate the thermal maturity of KOF crude oils, many of biomarker maturity indicators were used; such as pentacyclic triterpanes and sterane isomer ratios, methyl phenanthrene index (MPI), methyl dibenzothiophene ratio (MDR) and triaromatic steroid hydrocarbons (TA) (Table 4). The C_{32} 17α (H), 21β (H)-hopane 22S/(22R+22S) ratio is widely used as a biomarker maturity parameter (Ensminger 1977; Moldowan et al. 1985; Mohialdeen et al. 2015). The Khabbaz oils have C_{32} 22S/ (22R+22S) ratios in the range 0.54 to 0.58 (Table 4), which suggests that they reached equilibrium for oil window phase. The C_{29} sterane ratios increase with increasing thermal maturity (Seifert and Moldowan 1980). The KOF oils have a 20S/ (20S +20R) and ββ/(ββ+αα) C_{29} sterane ratios in the range 0.43 to 0.46 and 0.58–0.63, respectively,

Table 4 Biomarker maturity parameters for the saturated and aromatic compounds in the crude oils from Khabbaz oil field

Oil Field	Wells	Depth BSL(m)	Reservoir	DBT/ Phe	MDR	MPI	VRc%	Ts/ (Ts+Tm)	C_{29} 20S/ (20S+20R)	C_{29} ββ/ (ββ+αα)	C_{32} 22S/ (22S+22R)
Khabbaz	Kz-1	2776-3072	Lower Qamchuqa	4.49	2.80	0.91	0.95	0.56	0.46	0.62	0.61
Khabbaz	Kz-4	2710-2751	Upper Qamchuqa	3.15	2.69	0.84	0.90	0.14	0.45	0.63	0.55
Khabbaz	Kz-12	2666-2694	Upper Qamchuqa	2.27	3.54	0.94	0.96	0.13	0.43	0.58	0.58
Khabbaz	Kz-21	2645-2705	Upper Qamchuqa	3.02	2.82	0.85	0.91	0.21	0.46	0.63	0.60
Khabbaz	Kz-23	2640-2687	Upper Qamchuqa	3.15	2.86	0.85	0.91	0.20	0.46	0.63	0.59
Khabbaz	Kz-24	2627-2683	Upper Qamchuqa	3.21	2.82	0.86	0.92	0.15	0.44	0.63	0.59

Ts: (C27 18α(H)-22,29,30-trisnorneohopane; Tm: C27 17α(H)-22,29,30-trisnorhopane; MDR: MDR, 4-MDBT/1-MDBT

MPI methylphenanthrene Index; VRc (%):0.60 * MPI + 0.40; 20S/(20S+20R): C_{29}**20S/(20S+20R):** 5α,14α(H), 17α(H)-steranes 20S/ (5α,14α(H), 17α(H)-steranes 20S+ 5α,14α(H), 17α(H)-steranes 20R); **C_{29}ββ/(ββ+αα): C_{29} ββ/(ββ+αα):** 5α,14β(H), 17β(H)-steranes 20(R+S)/ (5α,14β(H), 17β(H)-steranes 20(R+S) +5α,14α(H), 17α(H)-steranes 20(R+S); **C_{32} 22S/(22S+22R): C_{32}**17α,21β(H)-homohopane (22S)/ (**C_{32}**17α,21β(H)-homohopane (22S) + **C_{32}**17α,21β(H)-homohopane (22R))

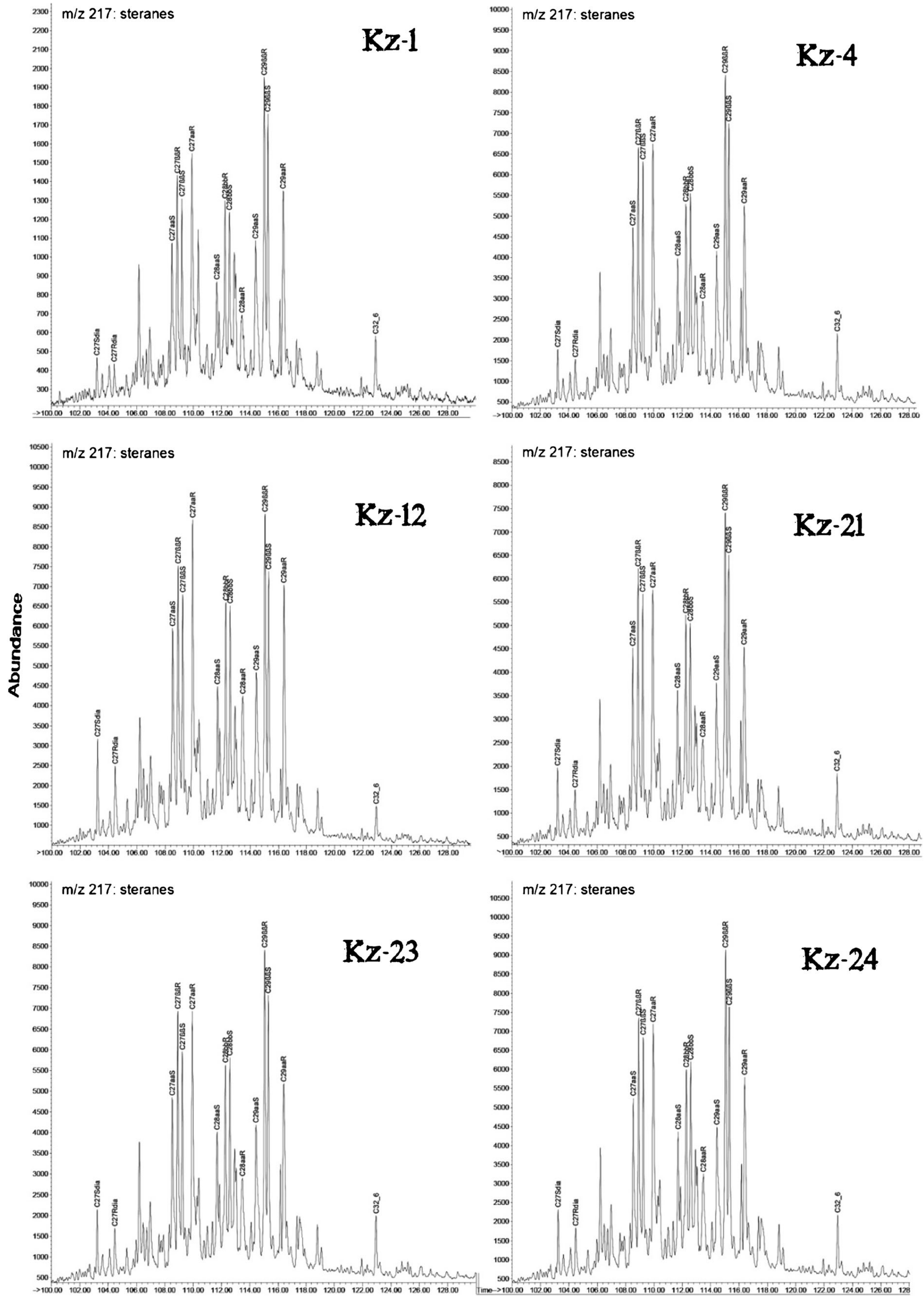

Fig. 10 Mass chromatograms (m/z 217) showing sterane biomarkers distribution in the studied crude oil samples of Khabbaz Oil Field, northern Iraq

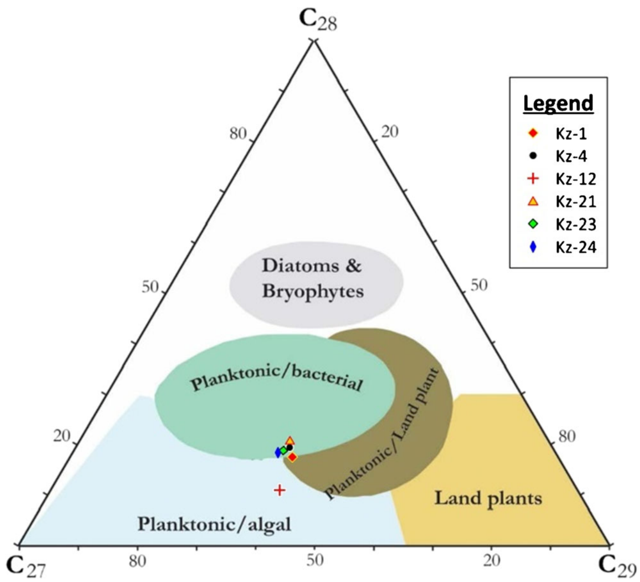

Fig. 11 Ternary diagram of regular steranes (C_{27}-C_{29}) of the studied crude oils indicating the relationship between sterane compositions in relation to organic matter input and depositional environments (Basic diagram after Huang and Meinschein 1979)

which indicate thermal maturity (equivalent to peak oil generation) (Peters and Moldowan 1993) (Fig. 15A). In addition, the maturity level deduced from C_{29} 20S/(20S + 20R) sterane versus the C_{32} 22S/(22R+22S) hopane ratios (Fig. 15B), also indicates that the studied oils are in peak oil-window (Peters and Moldowan 1993). Further support of such an interpretation is the relationship between the isoprenoid Pr/$_n$-C_{17} and Ph/$_n$-C_{18} ratios (Fig. 8; Table 2). Tissot and Welte (1984) observed that CPI values around or equal to one, is generally indicate a high maturity. Another parameter that can be deduced from mass fragmentograms of m/z 191 is Ts/(Ts+Tm) (Mustafa et al. 2015; Al-Khafaji et al. 2018). However; this ratio is controlled to some degree by the lithology and oxicity of the depositional environments (El-Sabagh et al. 2017). The Ts/ (Ts+Tm) ratio increases with increasing maturity (Peters and Moldowan 1993). This ratio for the studied samples ranged between 0.13 and 0.56 (Table 4) indicating the mature oils, with oil sample Kz-1 have more maturity than the other samples.

The aromatic biomarkers, the methyl phenanthrene index (MPI), and methyl dibenzothiophene ratio (MDR) can be used as thermal maturity indicators (Yessalina et al. 2006; Mohialdeen et al. 2018). The methyl phenanthrene index (MPI) (Radke and Welte 1983; Radke 1988) yields a calculated vitrinite reflectance (VRc %) (Table 4). The studied oils have vitrinite reflectance (VRc %) values in the range of 0.9–0.96, which indicates similar maturation level and mature oil window (Radke and Welte 1983; Mohialdeen et al. 2015).

Non-biomarker parameters; such as API gravity, sulfur and metal (e.g., V and Ni) contents are used in appraising the maturity level of crude oils (Peters et al. 2005; Mohialdeen and Raza 2013; El-Sabagh et al. 2017). The API values for all wells samples except sample Kz-1 ranged between 22.4° and 23.7°, while for sample Kz-1 is 32.3°. This indicating a higher maturity level for sample Kz-1, which is taken from the Lower Qamchuqa reservoir (Table 1). In any given field or geographical region, the level of oil maturity increases with reservoir age (Zumberge et al. 2017). This leads to a reservoir temperature differences reaches more than 8 °C. This difference in maturity is related to reservoirs depth differences as well as temperature and pressure. The depth differences between the two reservoirs reaches 265m. The concentrations of Ni and V, as described previously, also are consistent with the obtained results, i.e. sample Kz-1 contain low Ni and V as compared to the other crude oil samples (Table 1).

Possible source rocks (oil-source correlation)

The aim of this section is to investigate the genetic relation between the oils sources from the KOF in northern Iraq with the possible prolific source rock in the region, such as Sargelu, Naokelekan, Barsarin, Chia Gara formations. These rock units are from the Upper Jurassic-Early Cretaceous Megasequence of Iraq (Jassim and Goff 2006). The oil-source rock correlation of biomarker data from several source rocks were shown in (Table 5). It shows a number of biomarker ratios which are used for comparison of KOF crude oil samples with different source rocks. Overall, the KOF oil data seems strongly and closely match the Chia Gara source rock data; however, these Jurassic rocks generally have common biomarker compositions. The molecular geochemistry of Chia Gara Formation was studied by many researchers (Al-Beyati 1998; Mohialdeen and Al-Beyati2007; Mohialdeen et al. 2013, Mohialdeen et al. 2015; Hakimi *et al.,* 2016; etc.

The Chia Gara Formation has Pr/Ph ratio in the range 0.65 (average), which suggests marine organic matter input deposited under anoxic conditions. The pristane, phytane, and high *n*-alkane component levels confirms that such oils were derived from marine organic matter deposited under reducing conditions and relatively mature source rock extract (Figs. 8, 11, 13, and 14). The average of carbon preference index (CPI) value is 0.97, which also indicates a high contribution from marine organic matter with minor terrestrial organic matter input in the Chia Gara source rock (e.g., Mohialdeen et al. 2013).

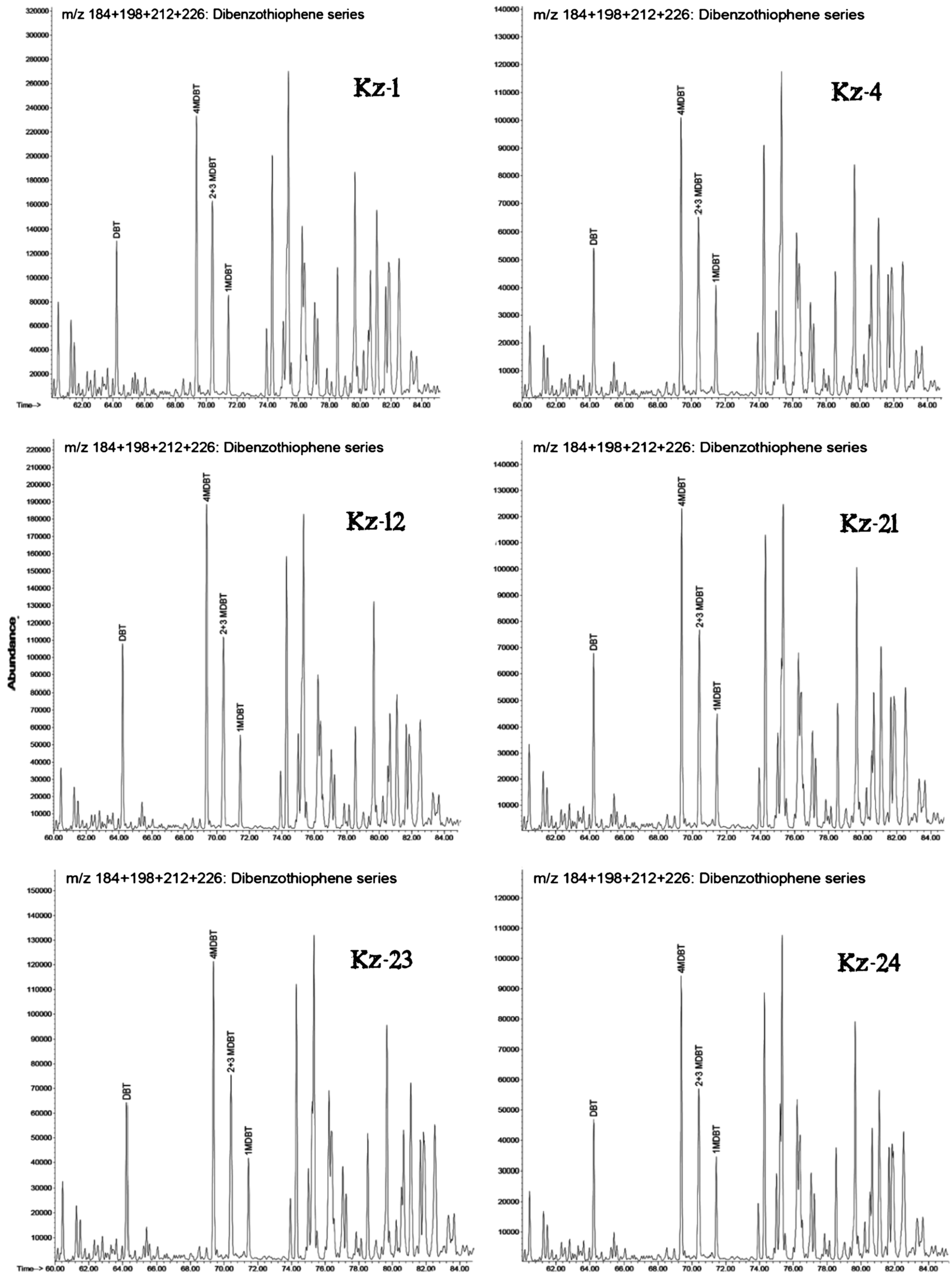

Fig. 12 Mass chromatograms (m/z 198) of aromatic compounds showing dibenzotheophene series in the studied crude oil samples of Khabbaz Oil Field, Kirkuk, northern Iraq

In the terpane distributions (m/z 191) from the Chia Gara source rock extract, the tricyclic terpanes, relatively high C_{29}-norhopane/C_{30}-hopane ratios, and high homohopanes index (Table 5), suggest a carbonate-rich marine source rock and that highly anoxic conditions prevailed during the deposition. In the sterane

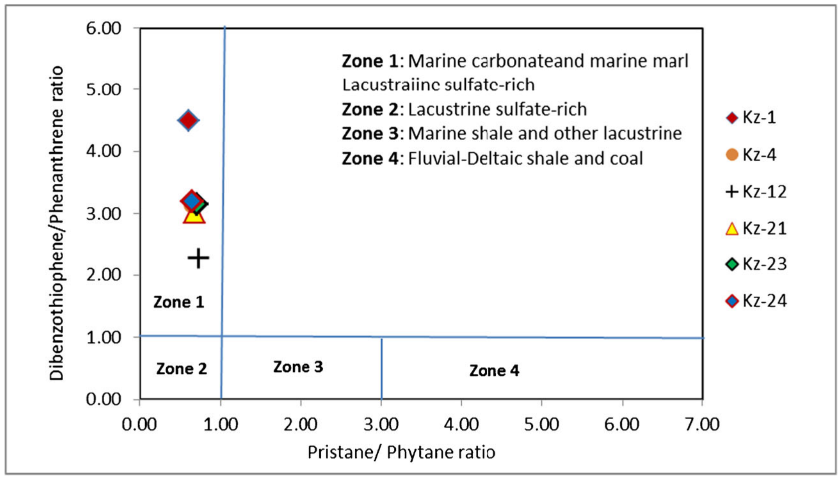

Fig. 13 Cross plot between DBT/Phe versus Pr/Ph provides a powerful way to infer source rock depositional environments and lithologies (after Hughes et al. 1995). All the studied oil samples from Khabbaz Oil Field, Kirkuk, Northern Iraq are located in marine carbonate environment

distribution (m/z 217), the C_{27} steranes strongly predominate with relatively high C_{27}/C_{29} regular steranes ratios (Table 5), which suggests predominantly plankton/algal and bacteria with minor input from terrigenous organic matter (Fig. 11; Huang and Meinschein 1979).

The ratios of C_{29} 20S/(20S +20R) sterane and C_{32} 22S/(22R+22 S) hopane of Chia Gara Formation are very close to those of KOF oils, and suggesting that the Chia Gara Formation is thermally mature and the oil window was reached (Table 5). The biomarker maturity ratios of KOF oils, such as; C_{32} 22S/(22S + 22R), C_{29} 20S/(20S+20R), C_{29} $\beta\beta/(\beta\beta+\alpha\alpha)$, MPI-1, and VRc% are also very similar to those of Chia Gara Formation (Table 5). The results of δ^{13}Corg for crude oils from KOF close to those of Chia Gara Formation from Kirkuk Oil Field (Mohialdeen and Hakimi 2016) (Table 5). Therefore, we strongly suggest the Late Jurassic-Early Cretaceous Chia Gara Formation as a main source rock for the Khabbaz Oil Field crude oils. However, the characteristics of the Sargelu Formation in the well Miran-3 (Table 5) is also close to the oils from KOF, which may form another source for the studied oils.

Conclusions

A detailed organic geochemical study of crude oil samples from Khabbaz Field, Kirkuk area reveals the following points:

1. The oils from Khabbaz Oil Field are not affected by biodegradation as it is revealed from the gas chromatogram patterns of the studied samples. Another point support this conclusion is the high ratio of saturated and aromatic hydrocarbons than the NSO components.
2. All samples reveal the dominance of short chain *n*-alkanes between C_{15}-C_{18}, with isoprenoids pristine (Pr) and

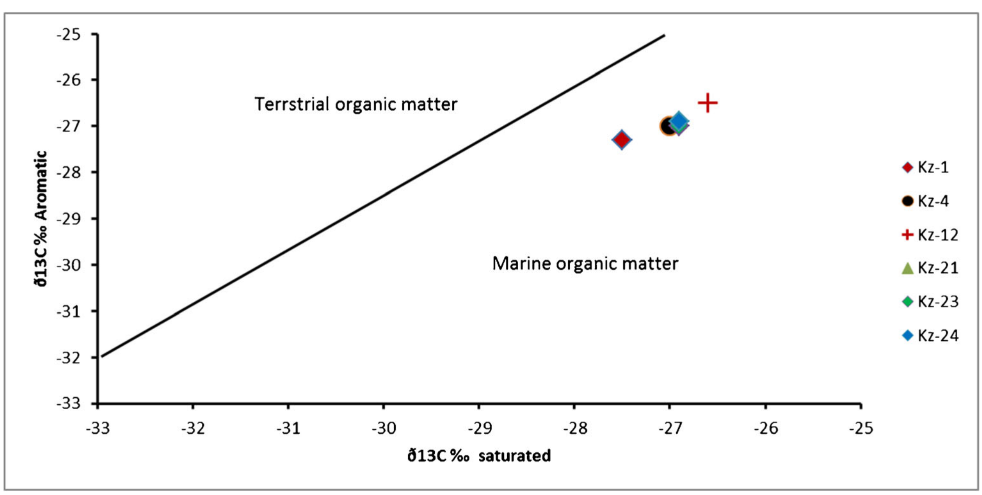

Fig. 14 The cross plot between $\delta^{13}C$‰ saturated and $\delta^{13}C$ ‰ aromatics clearly indicating the marine organic matter for the origin of crude oils. The line represents the best fit separation for waxy and non-waxy oils and is described by the equation $\delta^{13}C_{Aromatic} = 1.14\ \delta^{13}C_{saturated} + 5.46$ (after Sofer 1984)

Table 5 Biomarker ratios comparison of the average analyzed six crude oils from Khabbaz Oil Field, with possible source rocks common in the region

Samples ID (well)	Pr/Ph	Pr/C_{17}	Ph/C_{18}	CPI	C_{29}/C_{30}	G/C_{30}	HCR_{31}/HC_{30}	DBT/Phen	C_{27}/C_{29} Regular steranes	Regular steranes (%)			Dias/Ster	Ts/Tm	C_{32} 22S/(22S + 22R)	C_{29} 20S/(20S+20R)	C_{29} ββ/(ββ+αα)	MPI-1	VRc	$\delta^{13}C_{org}$ (‰ PDB)
										C_{27}	**C_{28}**	**C_{29}**								
Qamchuqa Reservoir (This study)	0.67	0.17	0.28	0.98	1.5	0.09	0.93	3.05	1.27	45.3	17.48	35.65	0.27	0.41	0.59	0.45	0.62	0.87	0.93	-26.6‰ to -27.5‰
Balambo[1] (Bn-1)	0.45	0.22	0.41	1.1	0.99				1.19	40.77	24.16	35.07		0.97	0.63	0.44	0.57			
Chia Gara[2] (K-109)	0.65	0.27	0.41	0.97	0.99	0.15	0.43	1.2	1.26	54.1	18.8	23.3	0.11	0.45	0.57	0.41		0.83	0.90	-29.99‰ to -26.93‰
Barsarin[3] (M-2)	0.37	0.24	0.24	0.75				1.96		69.04	11.38	19.60			0.56	0.55		0.46	0.67	
Naokelekan[4] (M-2)	0.48	0.17	0.18	0.6				3.3		57.65	15.48	26.87			0.57	0.57		1.21	1.13	
Sargelu[5] (M-3)	0.66	0.18	0.27	1.03	1.11	0.03		4.57	1.39	43.48	25.32	31.2				0.42	0.64	0.79	1.83	
Sargelu[6] (SwN-1)	0.6	0.24	0.36	0.99						38.27	30.81	30.92								
Sargelu [7] (Qc-2)	0.70	0.25	0.34	1.02	0.80					35.71	19.38	44.91		0.16						
Sargelu[8] (Shorish-1)	0.71	0.16	0.24			0.18		4.00								0.52		1.1	1.07	

Pr pristane, Ts– (C_{27} 18α (H)-22, 29, 30-trisnorneohopane), *CPI* carbon preference index: $\{2(C_{23} + C_{25} + C_{27} + C_{29}) / (C_{22} + 2[C_{24} + C_{26} + C_{28}] + C_{30})\}$, *Ph* Phytane, Tm– ($C_{27}$ 17α (H)-22, 29, 30-trisnorhopane), HCR_{31}/HC_{30}: C_{31} regular homohopane/C_{30} hopane, C_{29}/C_{30}: C_{29}norhopane/ C_{30} hopane, G/HC_{30}= Gammacerane/C_{30} hopane, M_{30}/C_{30}= C_{30} moretane/C_{30} hopane, DB= Dibenzothiophene, Phen= Phenanthrene, MDR= 4-MDBT/1-MDBT, MPI-1: Methylphenanthrene Index = 1.5 × (2 − MP+ 3 − MP)/ (Phenanthrene + 1 – MP + 9 − MP), VRc (%) = 0.60 ∗ MPI-1 + 0.40 Sources of data: [1] Sarraj and Mohialdeen (2021), [2] Mohialdeen et al. (2013); Mohialdeen and Hakimi (2016), [3] Mohialdeen et al. (2018), [4] Mohialdeen et al. (2018), [5] Fatah (2014), [6] Saeed and Mohialdeen (2016), [7] Abdula (2015), [8] Sachsenhofer et al. (2015)

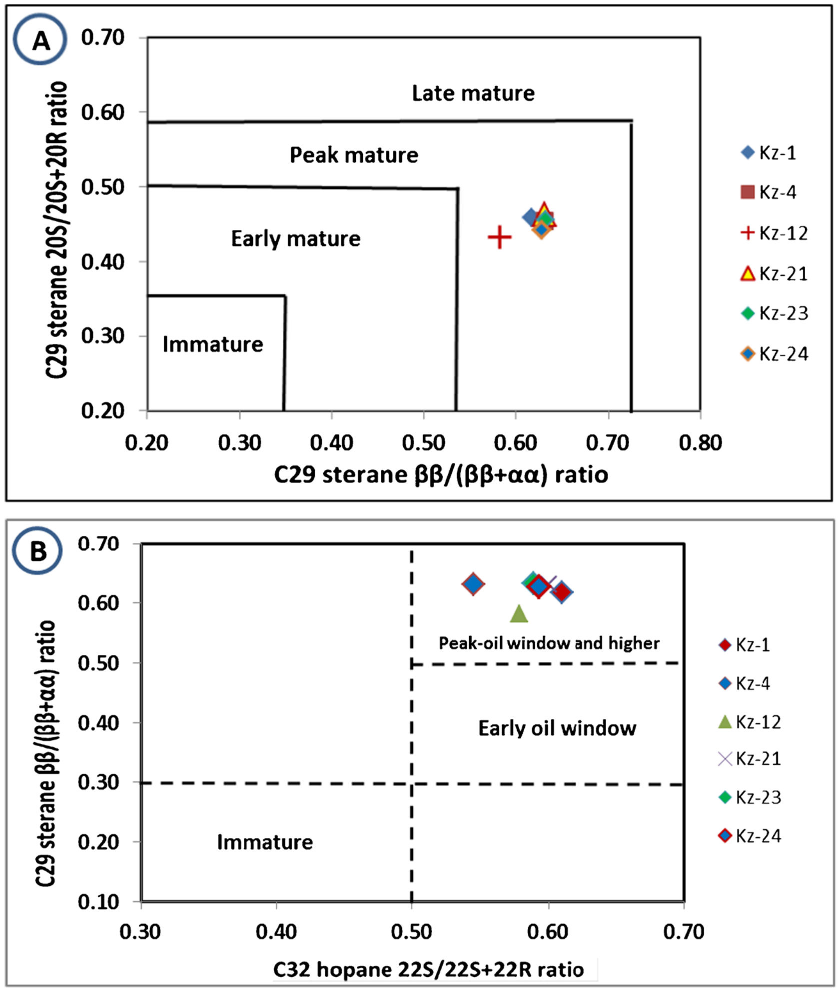

Fig. 15 **A**: Cross plot of 20S/(20S +20R) versus ββ/(ββ+αα) C_{29} sterane ratios indicating the peak mature of the selected oil samples (equivalent to peak oil generation) (basic diagram after Peters and Moldowan 1993) . **B**: The relation between C_{29} 20S/(20S + 20R) sterane and the C_{32} 22S/(22R+22S) hopane ratios, also showing that the studied oils are mature and within peak-oil window (basic diagram after Waples and Machihara 1991)

phytane(Ph). The unimodal envelope chromatograms, maximum peak carbon n-C_{15}-n-C_{20}, low CPI values (0.89–0.93) and high occurrence of C_{27} regular steranes, reflecting high contribution of aquatic algal organic matter with a minor input from terrigenous organic matter.

3. Khabbaz Oil Field samples revealed that phytane has relatively higher concentrations than pristane, with low Pr/Ph ratios in range 0.6–0.73. This indicates that these oils were generated from organic matter deposited in a marine environment under reducing conditions.
4. In most samples, the abundance of C_{32} homohopanes is moderate and the 22S/22R + 22S epimerization ratio is around 0.55 to 0.61. Gammacerane was identified in the analyzed samples and the gammacerane index (gammacerane/C_{30} hopane) was in the range of 0.01–0.12. The relative C_{30} hopane abundance is less than C_{29} norhopane in all samples, with high C_{29}/C_{30} 17α (H) hopane ratios in the range 1.33–1.62. This indicates that such crude oils are derived from carbonate-rich source rock.

20S/ (20S+20R) and ββ/(ββ+αα) C_{29} sterane ratios were relatively consistent for all the analyzed samples, ranging between 0.43–0.46 and 0.58–0.63, respectively. The DBT/Phe ratio versus Pr/Ph ratio as cross-plot, which is clearly indicates that the oils were generated from marine carbonate source rock.

5. The Khabbaz crude oils have a 20S/ (20S +20R) and ββ/(ββ+αα) C_{29} sterane ratios in the range 0.43 to 0.46 and 0.58–0.63, respectively, which indicate thermal maturity (equivalent to peak oil generation stage. The Lower Qamchuqa reservoir is higher in pressure and temperature than the Upper Qamchuqa, as a result the oils of Lower Qamchuqa has higher maturity level and API gravity.
6. The oils of the both reservoirs from KOF possibly have the same source rock. The biomarkers data strongly indicate that the Chia Gara Formation makes a potential source of these crude oils.

Appendix 1

Identified peaks in the m/z 177, m/z 191, m/z 217, and m/z 198 mass fragmentograms of Figures 6, 9, 10, and 12.

Peaks on m/z177		
TNH	Trisnorhopane	TNH
C_{28}DH	C_{28} Demethylated hopane	C_{28}DH
C_{29}DH	C_{29} Demethylated hopane	C_{29}DH
30-norhopane	30-Norhopane	30-norhopane
Peaks on m/z191		
$C_{23/3}$	C_{23} Tricyclic (Cheilanthane)	Tri C_{23}
$C_{24/3}$	C_{24} Tricyclic (Cheilanthane)	Tri C_{24}
$C_{25/3\ R\text{-}S}$	C_{25} Tetracyclic R and S	Tri $C_{25\ R\text{-}S}$
$C_{24/4}$	C_{24} Tetracyclic	Tet C_{24}
$C_{26/3}$ -S	C_{26} Tricyclic (Cheilanthane)	Tri C_{26}
$C_{26/3}$ -R	C_{26} Tricyclic (Cheilanthane)	Tri C_{26}
$C_{28/3}$ -S	C_{28} Tricyclic (Cheilanthane)	Tri C_{28}
$C_{28/3}$ -R	C_{28} Tricyclic (Cheilanthane)	Tri C_{28}
$C_{29/3}$ -S	C_{29} Tricyclic (Cheilanthane)	Tri C_{29}
$C_{29/3}$ -R	C_{29} Tricyclic (Cheilanthane)	Tri C_{29}
Ts	18α(H),22,29,30-trisnorneohopane	Ts
Tm	17α(H),22,29,30-trisnorhopane	Tm
BNH	Bisnorhopane 17a,21b(H)-Dinorhopane	BNH
C_{29}DH	C29 Demethylated hopane	C29DH
C_{29}	17α,21β(H)-nor-hopane	C_{29} hop
OLN	18α(H) and 18β(H)-oleanane	OLN
C_{30}H	17α,21β(H)-hopane	Hopane
C_{30}M	17 β,21α (H)-Moretane	$C_{30}\beta\alpha$
C_{31}S	17α,21β(H)-homohopane (22S)	C_{31}(22S)
C_{31}R	17α,21β(H)-homohopane (22R)	C_{31}(22R)
GCRN	C_{30} Gammacerane	GCRN
$C_{32/6}$	17α,21β(H)-homohopane	$C_{32/6}$
C_{32}S	17α,21β(H)-homohopane (22S)	C_{32}(22S)
C_{32}R	17α,21β(H)-homohopane (22R)	C_{32}(22R)
$C_{33/6}$	17α,21β(H)-homohopane	$C_{33/6}$
C_{33}S	17α,21β(H)-homohopane (22S)	C_{33}(22S)
C_{33}R	17α,21β(H)-homohopane (22R)	C_{33}(22R)
$C_{34/6}$	17α,21β(H)-homohopane	$C_{34/6}$
C_{34}S	17α,21β(H)-homohopane (22S)	C_{34}(22S)
C_{34}R	17α,21β(H)-homohopane (22R)	C_{34}(22R)
$C_{35/6}$	17α,21β(H)-homohopane	$C_{35/6}$
C_{35}S	17α,21β(H)-homohopane (22S)	C_{35}(22S)
C_{35}R	17α,21β(H)-homohopane (22R)	C_{35}(22R)
Peaks on m/z217		
C_{27} Sdia	13β,17α(H)-diasteranes 20S	Diasteranes
C_{27} Rdia	13β,17α(H)-diasteranes 20R	Diasteranes
C_{27} ααS	5α,14α(H), 17α(H)-steranes 20S	$C_{27}\alpha\alpha\alpha$20S
C_{27} ββR	5α,14β(H), 17β(H)-steranes 20R	$C_{27}\alpha\beta\beta$20R
C_{27} ββS	5α,14β(H), 17β(H)-steranes 20S	$C_{27}\alpha\beta\beta$20S

(continued)

Peaks on m/z177		
TNH	Trisnorhopane	TNH
C_{27} ααR	5α,14α(H), 17α(H)-steranes 20R	C_{27}ααα20R
C_{28} ααS	5α,14α(H), 17α(H)-steranes 20S	C_{28}ααα20S
C_{28} ββR	5α,14β(H), 17β(H)-steranes 20R	C_{28}αββ20R
C_{28} ββS	5α,14β(H), 17β(H)-steranes 20S	C_{28}αββ20S
C_{28} ααR	5α,14α(H), 17α(H)-steranes 20R	C_{28}ααα20R
C_{29} ααS	5α,14α(H), 17α(H)-steranes 20S	C_{29}ααα20S
C_{29} ββR	5α,14β(H), 17β(H)-steranes 20R	C_{29}αββ20R
C_{29} ββS	5α,14β(H), 17β(H)-steranes 20S	C_{29}αββ20S
C_{29} ααR	5α,14α(H), 17α(H)-steranes 20R	C_{29}ααα20R
Peaks on m/z 198		
DBT	Dibenzothiophene	DBT
4MDBT	4 methyldibenzothiophene	4MDBT
2+3 MDBT	2+3 methyldibenzothiophene	2+3 MDBT
1MDBT	1 methyldibenzothiophene	1MDBT

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