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# Preliminary results based on geochemical sedimentary constraints on the hydrocarbon potential and depositional environment of a Messinian sub- salt mixed siliciclastic- carbonate succession onshore Crete (Plouti section, eastern Mediterranean)

George Kontakiotis<sup>1</sup> · Vasileios Karakitsios<sup>1</sup> · Jean- Jacques Cornée<sup>2</sup> · Pierre Moissette<sup>1,3</sup> · Stergios D. Zarkogiannis<sup>1</sup> · Nikolaos Pasadakis<sup>4</sup> · Efterpi Koskeridou<sup>1</sup> · Emmanouil Manoutsoglou<sup>4</sup> · Hara Drinia<sup>1</sup> · Assimina Antonarakou<sup>1</sup>

## Abstract

This study details new geochemical analysis from an outcrop in Crete to improve understanding of the hydrocarbon potential of the southern margin of the Hellenic Arc along the continental convergent zone of the central Mediterranean Ridge. Seventeen samples were collected from the Late Miocene sub-salt sedimentary succession of Plouti section in central Crete and were studied in terms of their organic geochemical features using Rock–Eval VI pyrolysis. Results of this investigation revealed intervals with sufficient organic material of good enough quality and quantity to be considered as potential source rocks. The obtained data generally present poor to fair and/or good in some cases hydrocarbon generation potential. The TOC values range from 0.03 to 1.99%, with an average fair (2.1 mg HC/g rock) hydrocarbon potential. A Type III kerogen was identified, indicating a terrestrial origin organic matter.  $T_{max}$  and Production Index values suggest that the most promising parts of the section (organic-rich sediments) are immature with respect to oil generation and have not experienced high temperature during burial. Overall, the present study offers the opportunity to advance our understanding on the hydrocarbon potential onshore Crete and further investigate hydrocarbon prospectivity in the adjoining area, and particularly, the Greek part of the Mediterranean Ridge, a region with crucial economic and strategic importance.

**Keywords** Messinian Salinity Crisis (MSC) · Source-rocks · Sedimentary basin dynamics · Hydrocarbon potential · Kerogen type · Thermal maturity

## Introduction

The exploration of accretionary prisms around the world (e.g., Barbados, Makran, Irrawaddy-Andaman, West Timor Through) has led to the discovery of substantial petroleum reserves (Dolan et al. 2004; Wandrey 2006; Escalona et al. 2008; Persad 2008; Jones et al. 2011; Klein et al. 2011). In the eastern Mediterranean, a large number of accretionary prism complexes, characterized by the fastest rates of increase in the world (Kopf et al. 2003), are encountered within the Mediterranean Ridge (MR) (Mann et al. 2003; Foscolos et al. 2012). This structure results from

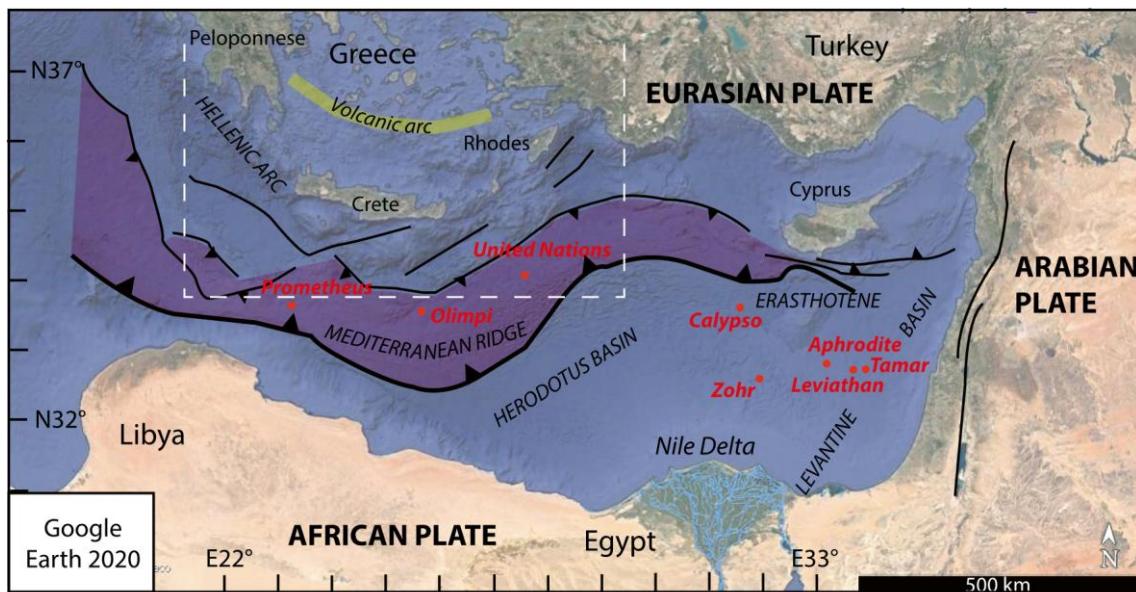
the subduction of the African plate beneath the European plate (Olivet et al. 1982; Reillinger et al. 1997; Kreemer and Chamot-Rooke 2004) (Fig. 1). Hydrocarbon potential in this region, particularly along the subduction zone and collisional

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margins, has been of long-standing academic and industrial interest (Zelilidis et al. 2003; Samuel et al. 2009; Bruneton et

al. 2011; Maravelis et al. 2012; Pantopoulos et al. 2013; Tsirambides 2015). However, recent discoveries have significantly altered the energy outlook in the region, with interest of the authorities and petroleum industry spurring additional exploration activities along the MR. The entire basin is increasingly becoming an exploration hotspot with both onshore and offshore frontier areas with potential for significant conventional and/or unconventional (shale gas and coal bed methane) hydrocarbon resources.

**Fig. 1** Geodynamic map of the Eastern Mediterranean region showing the position of the main structures (e.g., thrust front, subduction zones) and the location of the study area (white dashed inset corresponding to Fig. 2). Red dots correspond to hydrocarbon fields along the



- Hydrocarbon field
- ↘ Subduction front
- ↙ Thrust
- High angle dipping fault

Mediterranean Ridge (purple highlighted area) and offshore Herodotus and Levantine basins within the eastern Mediterranean Sea

Proven hydrocarbon reserves have been reported in the surrounding region (e.g., Levantine basin), while areas meriting further exploration (e.g., Herodotus basin, offshore Crete) have been also highlighted (Gardosh and Druckman 2006; Roberts and Peace 2007; Semb 2009; Foscolos et al. 2012; Elia et al. 2013; Maravelis et al. 2015a, b). The latter, particularly lies partly in the Greek Exclusive Economic Zone (EEZ) and appears to have significant oil/gas potential, including probable large stratigraphic traps and working petroleum systems (Bruneton et al. 2011; Maravelis and Zeligidis 2011; Maravelis et al. 2012). Direct hydrocarbon evidence of thermogenic origin, such as active mud volcanoes, pockmarks, and pipenecks, which emit methane (gas) hydrates, have been also documented (Montadert and Nikolaides 2007; Lykousis et al. 2009; Krois et al. 2010; Pape et al. 2010; Foscolos et al. 2012; Bohrmann et al. 2014; Bertoni et al. 2017; Tamborrino et al. 2019), thus enhancing the future prospectivity of the MR. Particularly, south of Crete, the existence of Olimpi, Prometheus, and United Nations mud volcano fields with many active mud volcanoes (Cita et al. 1981; Camerlenghi et al. 1992; Chaumillon et al. 1996; Limonov et al. 1996; Cronin et al. 1997; Huguen et al. 2004, 2006;

Loncke et al. 2004; Haese et al. 2006; Praeg et al. 2007, 2011; Maravelis et al. 2015b) highlights the extent of Mediterranean gas hydrate areas (Fig. 1). Moreover, several recent large natural gas field discoveries in the offshore Levantine and Herodotus basins (Needham et al. 2013; Ratner 2016; Grohmann et al. 2018, 2019) make the eastern Mediterranean one of the most promising frontier basins of the world. These new fields comprise mainly natural gas of biogenic origin and are located in either Miocene clastic turbidites (e.g., Tamar and Leviathan-Israel, Aphrodite-Cyprus; Montadert et al. 2014; Bou Daher et al. 2016) or in Mesozoic shallow marine carbonate buildups (Zohr-Egypt, Calypso-Cyprus; Schenk et al. 2010; Bertello et al. 2016; Esestime et al. 2016) (Fig. 1). However, in the offshore regions the characterization of potential source rocks is not a simple task, since almost no public well data and core samples are available. In this case, source rock properties and distribution have to be deduced from seismic interpretation and nearby onshore observations (e.g., Vafidis et al. 2012). High-resolution seismic data provide sufficient thickness of the sedimentary column, alternating sealing units, as well as direct hydrocarbon indicators (e.g., bright spots, gas chimneys; Roberts and Peace 2007; Montadert et al. 2014).

Onshore studies have identified several candidate source rock deposits in the sedimentary succession of the eastern Mediterranean Sea (Karakitsios and Rigakis 1996, 2007; Karakitsios et al. 2001; Zelilidis et al. 2003, 2015, 2016; Karakitsios 2013; Moforis et al. 2013; Maravelis et al. 2014, 2015a, 2016, 2017; Zelilidis and Maravelis 2015; Tserolas et al. 2018, 2019; Bourli et al. 2019a, b; Grohmann et al. 2019; Leila et al. 2020) in terms of Mesozoic oil-prone and Cenozoic gas-prone source rocks. Oil production in the study area is generally present in the sub-salt pre-Messinian sequences, while post-Messinian deposits have shown prospects for biogenic gas and are relatively immature in respect to hydrocarbon generation. More explicitly, the Late Miocene hydrocarbon generation potential onshore Crete and surrounding region has been documented in central Crete (Panagopoulos et al. 2011; Pasadakis et al. 2012; Maravelis et al. 2016), Gavdos Island (Pylotis et al. 2013), and Levantine basin in the south-easternmost sector of the Mediterranean basin (Gardosh and Druckman 2006; Roberts and Peace 2007; Semb 2009; Grohmann et al. 2019).

A great opportunity to advance our understanding on the hydrocarbon potential onshore Crete is offered by the Late Miocene sedimentary rocks on the Heraklion inland frontier basin, central Crete. In particular, the deposition of sub-salt sediments with source rock and/or reservoir potential is crucial, since the evaporites may potentially serve as seals and therefore hold large hydrocarbon columns. The main target of the present study is focused on the Messinian sub-salt sedimentary succession of the Plouti section. These rocks were investigated with respect to the quantity and quality of sedimentary organic matter, their Total Organic Carbon (TOC) content and source rock potential, thermal maturity, and the corresponding depositional environment. All this information is also used to assess the possible mechanisms of source rock deposition and petroleum systems onshore Crete, which may provide important prospect for the Neogene petroleum concern of the broader area of the eastern Mediterranean Sea.

## **Geological setting**

### **Tectonostratigraphic framework**

The eastern Mediterranean basin is a relic of the Neo-Tethys Ocean and evolved from successive prototype basins that developed as a result of extension-convergence tectonic cycles, which was controlled by the interplay of the African, Arabian and Eurasian plates (Robertson 1998; Gardosh et al. 2010; Robertson et al. 2012; Montadert et al. 2014; Gao et al. 2019). It covers a wide geographical region, with its southern part bounded by the Cyprus Island and Hellenic arcs, and its northern part comprising the forearc accretionary zone associated with the Alpine orogeny (Fig. 1).

The evolution of the eastern Mediterranean Sea began in the Late Permian, associated with the breakup of Pangaea into several large continental fragments (Gardosh et al. 2010; Robertson et al. 2012). It further developed during Jurassic-Triassic rifting phases which caused the disintegration of Gondwana and the evolution of the Neo-Tethys Ocean (Segev and Rybakov 2010; Hawie et al. 2013; Ghalayini et al. 2014) as well as the separation of distinct subbasins (e.g., Herodotus, Levant and Eratosthenes continental block; Ben-Avraham et al. 2006; Montadert et al. 2014). During the Late Cretaceous, the tectonic regime changed from extensional to compressional, following the collision of the African-Arabian with the Eurasian plate (Hawie et al. 2013; Montadert et al. 2014). Subsidence in the Oligocene created the

deep northern Levant basin, which was filled with thick sequences of coarse clastic sediments supplied by the Nile Delta (Steinberg et al. 2011; Macgregor 2012). Since the Middle Eocene, the shrinking Neo-Tethys Ocean became separated from the Indian Ocean in the east, with the final isolation of the eastern Mediterranean Sea in the early-middle Miocene (Robertson et al. 2012).

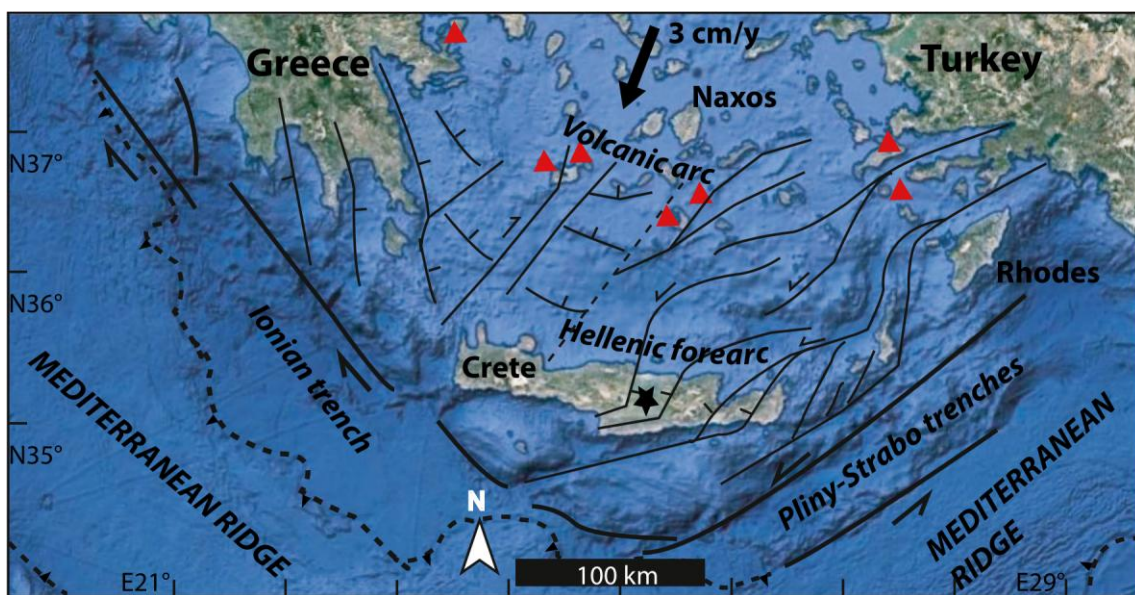
During the latest Miocene, the Mediterranean basin was cut off from the Atlantic Ocean resulting in the Messinian Salinity Crisis (MSC; Hsü et al. 1973; Krijgsman et al. 1999; Roveri et al. 2014). Throughout the entire basin, thick evaporites were deposited, while its eastern sector was exposed above sea-level and eroded (Robertson 1998). The re-establishment of open marine conditions took place during the basal Pliocene through a flooding event (Garcia-Castellanos et al. 2009) which resulted in the deposition of biogenic limestones (Trubi Formation; Langereis and Hilgen 1991; Hilgen and Krijgsman 1999; Hüsing et al. 2009; Kontakiotis et al. 2016) in the depocenters and/or clastic sediments in the shelf (e.g., Nile Delta) and slope areas (e.g., Herodotus and Levant basins; Gardosh and Druckman 2006; Frey-Martinez et al. 2007). At present, the region is dominated by the collision and continued convergence of the African and Eurasian plates, and related subduction, with the MR representing its accretionary wedge (Camerlenghi and Cita 1987; Reillinger et al. 1997; Robertson and Kopf 1998; Huguen et al. 2001; Kreemer and Chamot-Rooke 2004; Van Hinsbergen et al. 2005) (Fig. 2).

### Neogene sedimentary environments of Heraklion basin

Today, Crete, as part of the Aegean micro-plate, is connected with the European continent (Meulenkamp et al. 1994). The island of Crete represents a prominent horst structure formed in the recent fore-arc of the still ongoing Hellenic subduction. The structure of Crete consists of a pile of nappes that contain rock units from various paleogeographic zones (Papanikolaou and Vassilakis 2010; Zachariasse et al. 2011). Particularly, its Neogene basins formed in response to extensional geodynamic processes in the Aegean region during the Middle Miocene (Fassoulas 2001) and show a high degree of similarity with regard to lithofacies and history of subsidence and inversion (Meulenkamp 1979). Subsidence occurred in complex half-graben systems in a variety of larger and smaller basins being separated by pronounced uplifted blocks composed of pre-Neogene sediments (e.g., limestones, sandstones, phyllites-quartzites, ophiolites; ten Veen and Postma 1999).

The present-day Heraklion basin is formed by a series of N-S trending half-grabens filled by clastics, carbonates

**Fig. 2** Present-day geodynamic framework of the Hellenic Arc and approximate relative motion vector of the south Aegean in respect to stable Eurasia (Le Pichon et al. 1995; Mantovani et al. 2000; Sakellariou and Tsampouraki-Kraounaki 2019). The dashed black line corresponds to the Mediterranean Ridge accretionary complex. The circular arrows represent total rotation at the two ends of the Hellenic arc since 15 Ma, consistent with paleomagnetic observations (Kissel and Laj 1988). The red triangles show the Pleistocene to recent volcanism along the Hellenic volcanic arc, and the black star the regional location of the Plouti section in central Crete, within the eastern Mediterranean Sea

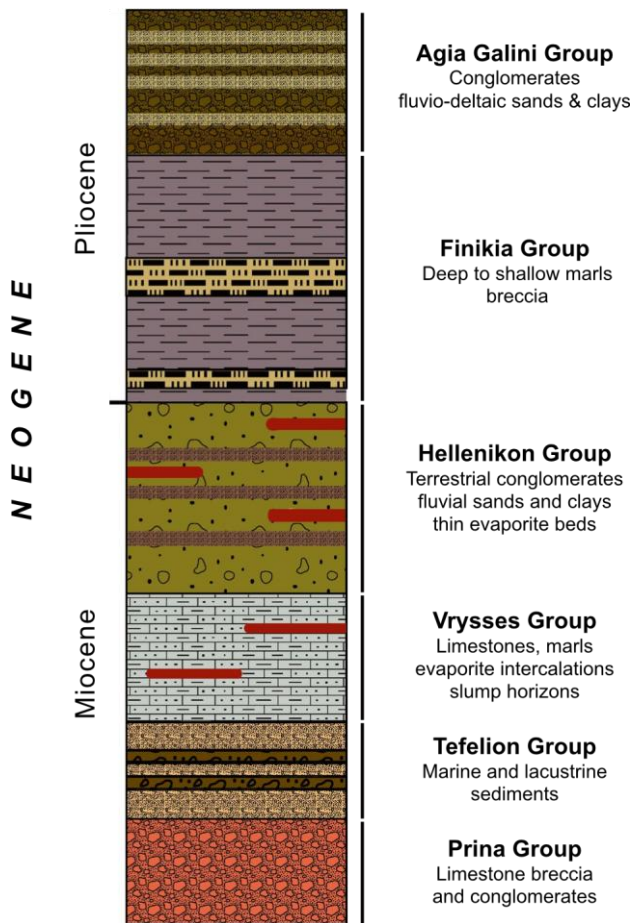


and evaporites of middle Miocene to Pliocene age (Meulenkamp 1979; Zachariasse et al. 2008, 2011). During this time interval, a sequence of different sedimentary environments from brackish lagoons to marginal and offshore marine settings have been documented in Crete (ten Veen and Postma 1999; Reuter et al. 2006; Drinia et al. 2008; Zachariasse et al. 2011; Moissette et al. 2018; Kontakiotis et al. 2019). Subsequent to a phase of non-marine and marginally marine sedimentation at the end of the Serravallian, the basin became a marine environment during the early Tortonian. Extensional tectonics during the late Tortonian triggered rotational uplift of blocks in positions distal to the basin margin and gave rise to pure offshore carbonate environments. Around the Tortonian-Messinian Transition, most shallow-water environments drowned during a pulse of relative basin subsidence and hinterland uplift. During the late Miocene, these blocks formed islands undergoing erosion and supplying the basin with siliciclastic fluvial, brackish, and marine sediments (Kontopoulos et al. 1996; Reuter et al. 2006; Zachariasse et al. 2008, 2011; Moissette et al. 2018).

### **Lithostratigraphy of the Plouti section**

The Plouti Sect. (35° 05' 44.1" N, 24° 56' 53.5" E) corresponds to the uppermost part of the Messinian Vrysses Group (Fig. 3) and to the upper Messinian Lower Evaporites unit which is 50- to 60-m thick (Zachariasse et al. 2008). The uppermost part of the Vrysses Group in the Plouti area comprises 10-m thick alternations of silty marls, limestones and sandstones, topped by an erosional unconformity below the first gypsum beds (Fig. 4a,c), that are related to the Primary Lower Gypsum (PLG) and reflect the first phase of the MSC (p-ev1 at 5.97–5.60 Ma; CIESM 2008; Roveri et al. 2008; Manzi et al. 2012, 2013). The interval studied here concerns the last 10 m of sediments below the first gypsum bed. This sedimentary sequence mostly consists of fossiliferous silty clays and sandy limestones with some intercalations of diatomaceous marls enriched in tree leaves and siliceous sponge spicules (Fig. 4). The siliciclastic character of the above sediments further confirms their deposition during stressed (e.g., reduction of ocean connections, salinity fluctuations; Esteban 1979; Martín and Braga 1994; Moissette et al. 2010; Leila et al. 2020) conditions of the Terminal Carbonate Complex (TCC; Roveri et al. 2009; Bourillot et al. 2010) and also records the onset of the MSC (Cornée et al. 2004). Along the section, abundant traces of sulfur are displayed (Fig. 4b), indicating that these sediments were deposited in lagoonal restricted paleoenvironments. The occurrence of dominant sponges, associated with poorly diversified and scarce benthic fauna, also reveal oxygen depleted conditions. However, the presence throughout, but mostly at the base of the section (black shales), even if scarce, of planktonic and benthic foraminifera, bryozoans, and echinoids, points to marine influences. At the top of the studied interval, there is





**Fig. 3** General stratigraphy of Neogene sedimentary sequence in Crete Island with the six lithostratigraphic groups of Meulenkamp (1979) (modified after Zelilidis et al. 2016)

evidence of breccias composed of gypsum- and non-fossiliferous limestone clasts floating in a silty to arenitic calcareous matrix (Fig. 4). Further up-sequence, an upward exposed alternation of non-fossiliferous clayey to silty marls with laminated gypsum beds, interpreted as shallow-water gypsum turbidites (Zachariasse et al. 2008), and fine-to coarsegrained terrigenous clastics forms a thick sedimentary package, equivalent to the Resedimented Lower Gypsum unit (RLG; Manzi et al. 2011) of the second phase of the MSC (p-ev2 at 5.60–5.55 Ma; Hilgen et al. 2007; CIESM 2008; Roveri et al. 2014).

## Materials and methods

### Sample collection and preparation

Seventeen rock samples, which were suspected to be rich in organic matter, were collected from the study outcrop on the island of Crete for geochemical analysis (Rock-Eval and detailed characterization of the contained organic matter). The selected samples were collected from the pre-evaporitic sedimentary succession of the Plouti section. Weathering is always a factor of concern for organic geochemical studies of outcrop samples and all analyzed samples were thus taken from at least 30 cm below the outcrop surface to minimize these effects. The fresh samples were immediately placed into plastic sample bags, secured with a zip tie, labelled with a unique sample number (PLO1-PLO17) and were taken to the laboratory for analyses. The experimental analytical work was carried out in the “Hydrocarbons Chemistry and

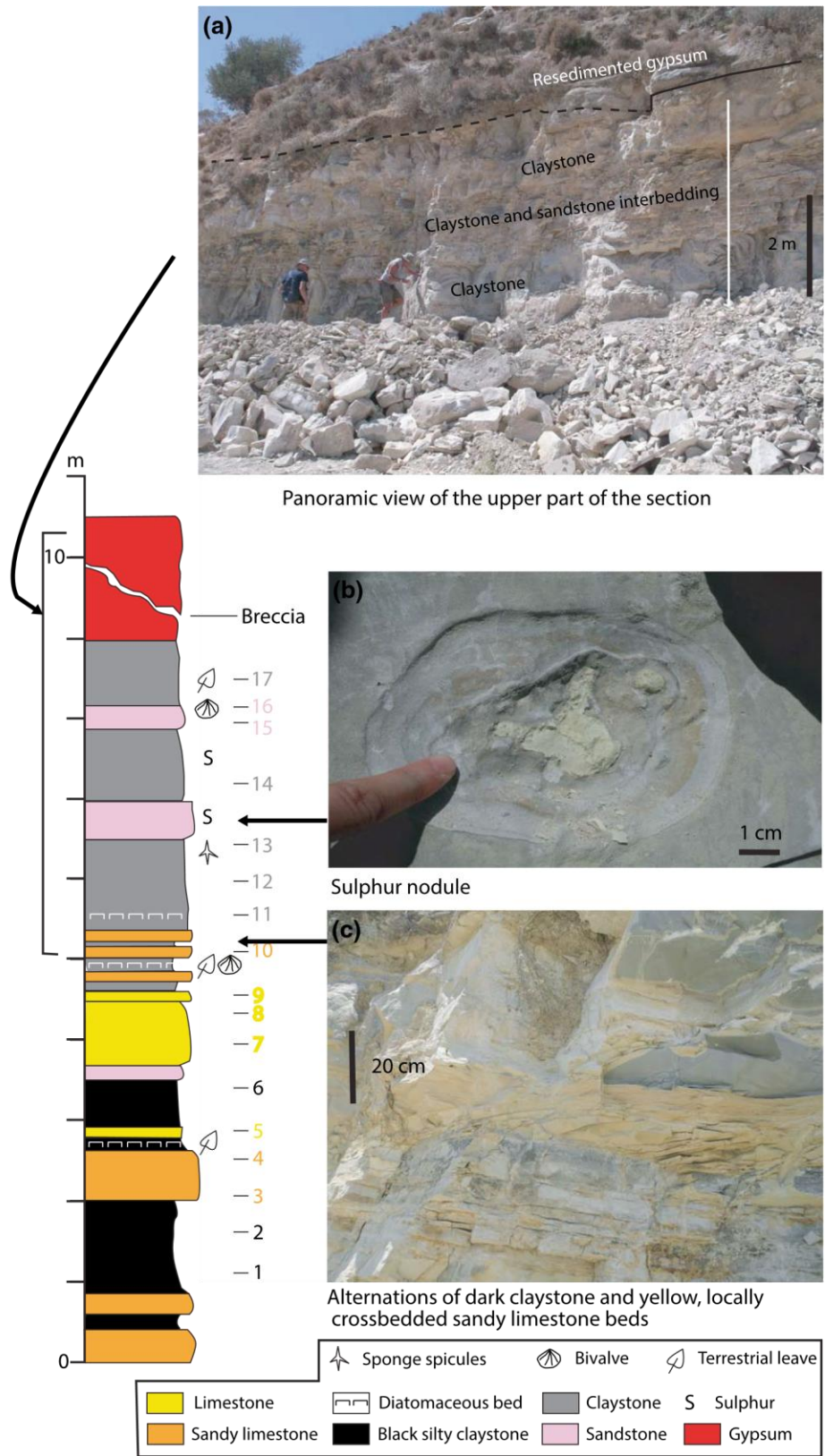
Technology Laboratory” of the Technical University of Crete. The samples were powdered and sieved through a 60-mesh (250  $\mu\text{m}$ ) sieve and analyzed using Rock–Eval (RE) pyrolysis technique with a Rock–Eval VI (Delsi Inc.) analyzer under standard conditions (e.g., programmed heating of  $\sim 100$  mg of pulverized rock samples in an helium atmosphere in a special oven, and the hydrocarbons generated are monitored as a function of temperature; Espitalié et al. 1977, 1985; Peters 1986; Lafargue et al. 1998; Behar et al. 2001).

### **RE pyrolysis- derived parameters and interpretive guidelines**

Source rock potential has been evaluated on the basis of three fundamental attributes: quantity, quality and maturity of the organic matter. Through this process, several important Rock–Eval derived parameters that are generated from the analysis are:  $S_1$  (free hydrocarbons present within the sample released during the pyrolysis stage; mg HC/g rock),  $S_2$  (heavier hydrocarbons released due to pyrolytic breakdown of kerogen released during the pyrolysis stage; mg HC/g rock),  $T_{\text{max}}$  (temperature at which maximum amount of pyrolyzate is cracked under the  $S_2$  peak of Rock–Eval;  $^{\circ}\text{C}$ ),  $S_3$  ( $\text{CO}_2$  released from oxygenated compounds during the pyrolysis stage; mg C  $\text{O}_2$ /g rock), pyrolyzable carbon (PC; calculated from  $S_1$ ,  $S_2$ , and  $S_3$  components),  $S_4$  (residual carbon (RC) content of the sample; mg carbon/g rock), hydrocarbon potential (HP equal to  $S_1 + S_2$ , mg HC/g rock), and TOC (sum of PC and RC; wt%), from which  $S_1/\text{TOC}$  ratio can be calculated. The free hydrocarbons liberated under the  $S_1$  peak could be indigenous (free or adsorbed gas/oil) and/or non-indigenous (migrated or contaminated) in nature (Hunt 1996). The heavier hydrocarbons generated under the  $S_2$  peak indicates the remaining hydrocarbon generation capacity of the rock (Tyson 1995), and is used to infer the Van Krevelen Types of organic matter input by calculating hydrogen indices (e.g., HI;  $S_2$  normalized to TOC, mg HC/g TOC). Similarly, the  $S_3$  peak is used to calculate oxygen indices (e.g., OI;  $S_3$  normalized to TOC, mg C  $\text{O}_2$ /g TOC; Espitalié et al. 1977). Both the HI ( $100 * S_2 / \text{TOC}$  mg HC/g Corg) and OI ( $100 * S_3 / \text{TOC}$  mg  $\text{CO}_2$ /g Corg) have been used to define the kerogen type present in the rocks (Tissot and



**Fig. 4** Lithostratigraphic log and sedimentary facies characteristics of the Plouti section. To the right of the log are indicated the positions of the studied samples along with those of sponge spicules, bivalves, and terrestrial leaves identified in the different stratigraphic levels of the section. **a** Panoramic view of the upper part of the study section, **b** sulphur nodule, and **c** alternations of dark claystone and yellow, locally cross-bedded sandy limestone beds



Welte 1984; Tyson 1995; Hunt 1996) through the kerogen classifying diagram (Espitalié et al. 1977). Particularly, HI reflects the quality and quantity of pyrolyzable organic compounds, from  $S_2$  relative to the TOC (mg HC/g TOC), while OI is related to the quantity of terrestrial organic matter.  $T_{max}$  is used as a maturity parameter for fossil organic matter

(Tissot and Welte 1984). Production Index (PI;  $S_1/S_1 + S_2$ ) reveals the total amount of hydrocarbons ( $S_1 + S_2$ ) that may be produced (Jones 1984). For the interpretation of the RE pyrolysis data, we refer to the specific guidelines of Tissot and Welte (1984), Peters (1986), Peters and Cassa (1994), Burwood et al. (1995), and Dymann et al. (1996). Geochemical parameters (e.g., HI,  $S_2/S_3$ ) describing the type of hydrocarbons generated were also used as initially introduced by Peters (1986).

## Results

### Screening Plouti data

Bulk organic geochemical characteristics of the Messinian depositional sequence of Plouti section are summarized in Table 1, while the correlation of the RE results with lithology is further illustrated in Figs. 5, 6, and 7. TOC values range from 0.03 to 1.99%, with an average of 0.65%. Most of the analyzed samples show very low (< 0.5%) TOC contents; however, 6 samples (4 claystones and 2 limestones) present TOC values between 1 and 2% that can be classified as “Good Source Rock”.  $S_1$  values reflecting the present-day hydrocarbon generative capacity of kerogen in the rocks range from 0.01 to 0.70 HC/g rock. This may reflect the oxidation of the organic matter as is usually the case in late Miocene outcrop samples. The consistent low  $S_1$  values further lead to relatively low production index values, ranging between  $0.07 < PI < 0.21$ .  $S_2$  values are quite variable presenting either low (< 0.10 HC/g rock) or extremely high (> 3.0 HC/g rock) values, and this distribution is also evident for the hydrocarbon potential of the analyzed samples with the relevant HP values ranging between 0.03 and 7.57 mg HC/g rock. All studied samples reach a very narrow range of  $T_{max}$  values from 400 to 417 °C, indicative of their thermally immature character. HI and OI values vary between 57.53 and 417.56 HC/g TOC, and 61.45 and 1020.39 mg CO<sub>2</sub>/g Corg, respectively.

## Discussion

### Definition of effective source rocks through TOC enrichment

There is a consensus that the TOC content, which is associated with free hydrocarbons and kerogen, can serve as a standard for evaluating the organic richness (quality) of source rocks. The lower threshold TOC limit for effective source rock is generally 0.5% (Hunt 1967, 1979, 1996; Tissot and Welte 1984). The Plouti TOC contents vary within the studied samples and range from 0.03% (PLO16) to 1.99% (PLO7). These values permit sample classification, in terms of their source rock potential, as mudstones with limited to good hydrocarbon generating potential. Samples PLO2, PLO6-PLO8, PLO12, PLO14, and PLO17 have merits for further consideration ( $0.5\% < TOC < 2.0\%$ ). According to Zhao et al. (2015), the value of TOC corresponding to the beginning of the  $S_1$ /TOC value reduction is the base limit of TOC values for high-quality hydrocarbon source rocks. However, the application of this method to our data is minimized due to the small number of analyzed samples. Based on the above criteria, we follow the classification of Tissot and Welte (1984), where the minimum TOC value required characterizing a sample as at least an immature source rock is 0.5 wt%. Rocks containing < 0.5% TOC are considered to have negligible hydrocarbon source potential, while between 0.5 and 1.0% TOC indicates marginal potential, and > 1.0% TOC has substantial source potential.

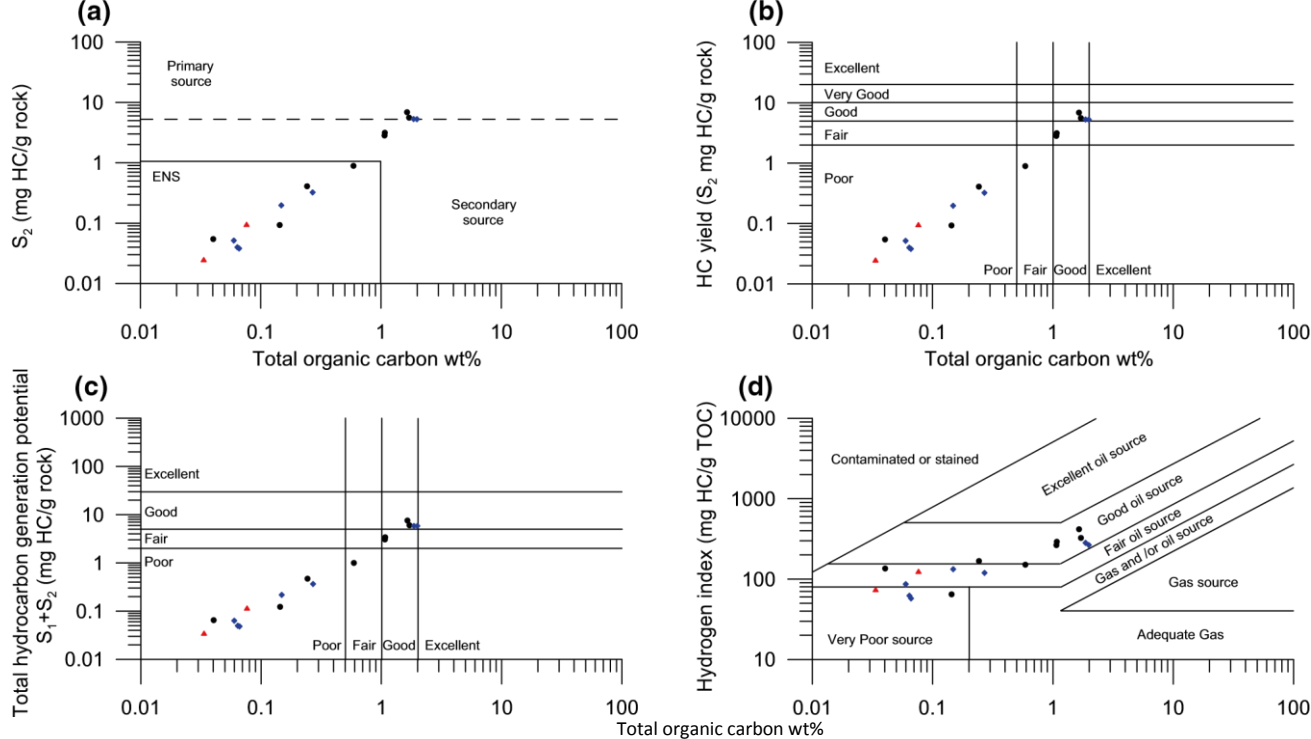
Overall, the Plouti succession is classified as variable, mostly “poor” (10 samples; 58.8%) to “fair” (1 sample; 5.9%) and “good” (6 samples; 35.3%), according to TOC content (Table 1). Seven samples reached the standard for effective source rocks (TOC > 0.5%), with 5 of them belonging to black shales/claystones (black circles in Figs. 5, 6 and 7) and the rest to be classified as limestones (blue diamonds, respectively). Both sandstone samples (red triangles) display very low TOC content (< 0.1%; Figs. 5 and 6). All of them were immature ( $T_{max} < 435$  °C), suggesting that these source rocks had not generated hydrocarbons in the study area. The average petroleum potential of 2.1 mg HC/g rock indicates a marginally “fair” source rock potential, which is in accordance with the average petroleum potential evaluated based on  $S_2$  values (1.84 mg HC/g rock; Table 1).

## **Source rock potential**

TOC alone is not a clear indicator of the hydrocarbon generation potential and thus, the carbon and hydrogen

**Table 1** Geochemical indices obtained from the Rock–Eval VI analysis of the studied rock samples of the Plouti section

Sample ID	Lithology	$T_{max}$ (°C)	$S_1$ (mg/g)	$S_2$ (mg/g)	$S_3$ (mg/g)	$S_4$ (mg/g)	TOC (%)	HI (mg HC/g TOC)	OI (mg CO <sub>2</sub> /g TOC)	PI	$S_2/S_3$	$S_1 + S_2$	$S_1/TOC$ (mg HC/g TOC)
PLO17	Claystone	405	0.23	2.83	1.02	8.18	1.07	265.05	95.36	0.07	2.78	3.06	0.21
PLO16	Sandstone		0.01	0.02	0.34	0.31	0.03	74.12	1020.39	0.29	0.07	0.03	0.30
PLO15	Sandstone		0.02	0.10	0.40	0.67	0.08	124.98	518.53	0.18	0.24	0.12	0.27
PLO14	Claystone	407	0.26	3.14	0.89	7.98	1.08	291.89	82.73	0.08	3.53	3.41	0.25
PLO13	Claystone		0.01	0.05	0.39	0.35	0.04	135.61	971.84	0.15	0.14	0.06	0.25
PLO12	Claystone	406	0.48	5.58	1.86	12.16	1.71	325.70	108.79	0.08	2.99	6.05	0.28
PLO11	Claystone	413	0.06	0.41	0.83	2.04	0.24	168.03	340.49	0.13	0.49	0.47	0.26
PLO10	Limestone		0.01	0.05	0.33	0.55	0.06	86.37	555.49	0.19	0.16	0.06	0.20
PLO9	Limestone		0.01	0.04	0.39	0.62	0.07	57.53	584.10	0.21	0.10	0.05	0.15
PLO8	Limestone	403	0.52	5.29	1.15	13.88	1.86	283.47	61.45	0.09	4.61	5.81	0.28
PLO7	Limestone	400	0.56	5.28	1.29	15.11	1.99	265.43	64.96	0.10	4.09	5.84	0.28
PLO6	Claystone	417	0.10	0.89	0.95	5.08	0.59	150.96	161.25	0.11	0.94	1.00	0.18
PLO5	Limestone	415	0.04	0.32	0.64	2.41	0.27	119.59	237.99	0.11	0.50	0.36	0.15
PLO4	Limestone	416	0.02	0.20	0.30	1.31	0.15	133.20	201.02	0.09	0.66	0.22	0.14
PLO3	Limestone		0.01	0.04	0.37	0.60	0.06	62.25	572.43	0.20	0.11	0.05	0.16
PLO2	Claystone	403	0.70	6.87	1.24	10.25	1.65	417.56	75.35	0.09	5.54	7.57	0.42
PLO1	Claystone		0.03	0.09	0.71	1.34	0.14	64.71	494.30	0.24	0.13	0.12	0.21



**Fig. 5** Source rock appraisal of the examined samples from Plouti section based on: **a, b** cross-plots of  $S_2$  versus total organic carbon, **c** total hydrocarbon generating potential versus total organic carbon

Total organic carbon wt%

classification diagram, **d** hydrogen index versus total organic carbon biplot. These plots indicate the presence of source rocks with poor to good hydrocarbon generating potential

contents of the organic matter are required (Dembicki 2009). The  $S_2$  value that reflects the quantification of the amount of hydrocarbons formed during the thermal decomposition of the kerogen, integrated with TOC values can add constraints in the hydrocarbon generating potential of the source rocks. The  $S_2$  values range from 0.02 mg HC/g rock (PLO16) to 6.87 mg HC/g rock (PLO2). The cross-plot of  $S_2$  versus TOC classifies the studied sediments principally as secondary effective non-source rocks (ENS;  $S_2 < 1$  mg HC/g rock), with the exception of 4 samples which are marginally classified as primary source rocks ( $S_2 > 5$  mg HC/g rock). This plot portrays the occurrence of relatively lean source rocks with poor to good hydrocarbon generating potential (Fig. 5a). This is further supported by the hydrocarbon yield  $S_2$  versus TOC crossplot of Burwood et al. (1995) which indicates that samples with higher  $S_2$  values correspond to higher quality source rocks (Fig. 5b). The cross-plot showing the relationship of total generative hydrocarbon potential HP versus TOC demonstrates a variable distribution of the source rock potential. Although most of the samples suggest insignificant (HP < 2 mg HC/g rock) oil (but some gas) potential, two samples (black shales) were classified as moderately rich source rocks with fair (2 mg HC/g rock < HP < 5 mg HC/g rock) oil potential, and four other samples (two black shales and two limestones) classified as good (HP > 5 mg HC/g rock) source rock potential (Tissot and Welte 1984; Dymann et al. 1996) (Fig. 5c). The different source rock potential estimations could be partly explained by inert organic carbon within the TOC. A better rating of source rock potential is given by the cross-plot of HI versus TOC (Jackson et al. 1985), which shows a clear oil-prone source rock potential, with the majority of the samples almost equally distributed within the fair and good oil-prone windows (Fig. 5d). However, the integration of parameters such as chemistry, maturation status, and description of organic matter, is essential before a rock can be regarded as oil-prone.

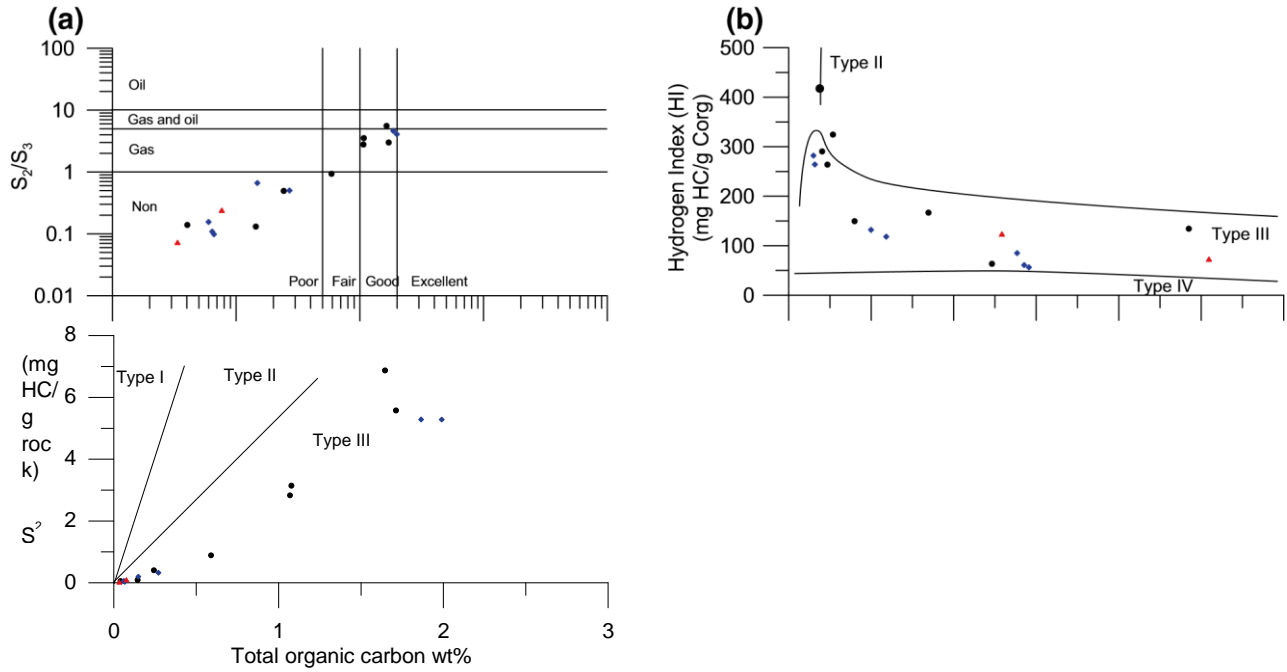
### Quality and type of organic matter

The type of organic matter contained in the source rocks was evaluated by the HI and the  $S_2/S_3$  ratio (Peters 1986). Both these parameters indicate mainly the occurrence of gas and/ or oil + gas prone organic matter (Fig. 6a,b). The reported

HI values below 150 mg HC/g TOC indicate a potential source for generating gas (mainly type III kerogen), while the relevant values ranging between 150 and 300 mg/g contain

0.01 0.1 1 10 100

Total organic carbon wt% (c)

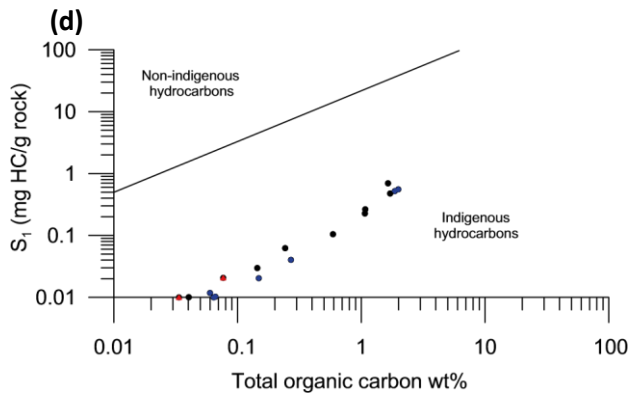


**Fig. 6** Results of Rock-Eval VI pyrolysis plotted on: **a**  $S_2/S_3$  versus total organic carbon, **b** hydrogen index versus oxygen index kerogen classification diagram, **c**  $S_2$  versus total organic carbon classification

0 200 400 600 800 1000 1200

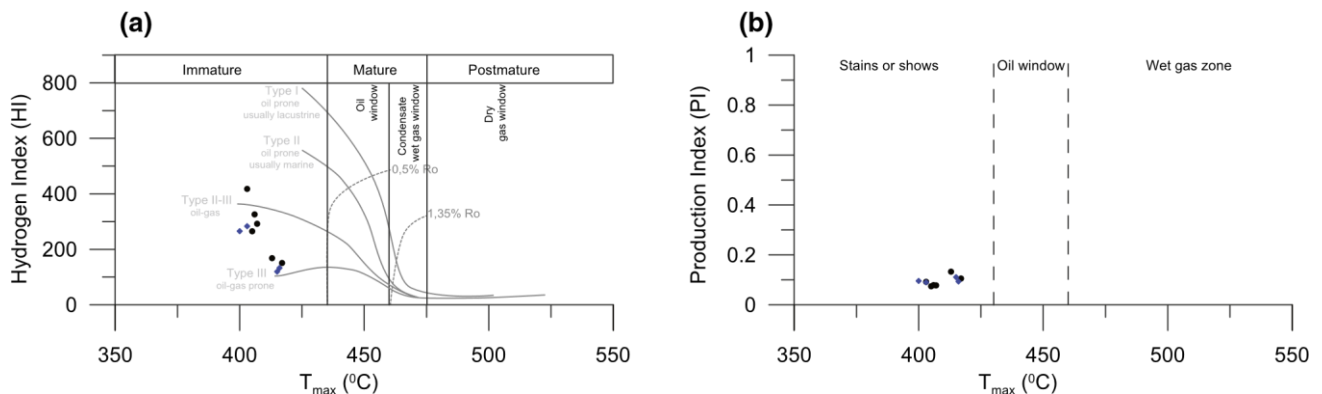
Oxygen Index (OI) (mgCO<sub>2</sub>/gCorg)





diagram, **d** plot of  $S_1$  versus total organic carbon. These plots indicate the presence of nonproductive to gas-prone source rocks capable of generating autochthonous hydrocarbons

**Fig. 7** Classification of kerogens and maturation status of the examined samples based on: **a** Hydrogen index versus  $T_{max}$  diagram, and **b** cross-plot of production index versus  $T_{max}$ . These diagrams portray co-existence of samples with oil and gas generating potential. The majority of the Plouti samples are immature with respect to oil generation and have not experienced very high temperatures during burial



mostly type III kerogen, and therefore, are capable of generating mixed gas and oil (Fig. 6b). Only two samples (PLO2 and PLO12) present  $> 300$  mg/g value, along with their high TOC content (1.65 and 1.71 wt%, respectively), suggesting the presence of a significant amount of oil-generative lipid materials possibly derived from autochthonous organic matter inputs under a reducing depositional environment. Their gaseous generation potential is further indicated using the  $S_2/S_3$  scheme proposed by Peters (1986), which suggests the occurrence of gas-prone rocks ( $1 < S_2/S_3 < 5$ ) (Fig. 6a). Additional characterization of the organic matter quality was made based on HI versus OI cross-plot (Fig. 6b; Peters and Cassa 1994). The application of the above classification scheme on the studied samples indicates the occurrence of type III kerogen that originated from terrestrial plant debris and/or aquatic organic matter, and was deposited in an oxidizing shelf environment. Supporting evidence for the dominance of type III kerogen in the examined samples comes from the  $S_2$  versus TOC cross-plot (Fig. 6c; Tissot and Welte 1984). Although type III kerogen can be associated with aquatic organic matter in marine settings (Tissot et al. 1974), its prevalence in the studied sediments can be also related to transportation by fluvial processes (Vandenbroucke and Largeau 2007). Late Miocene runoff into the eastern

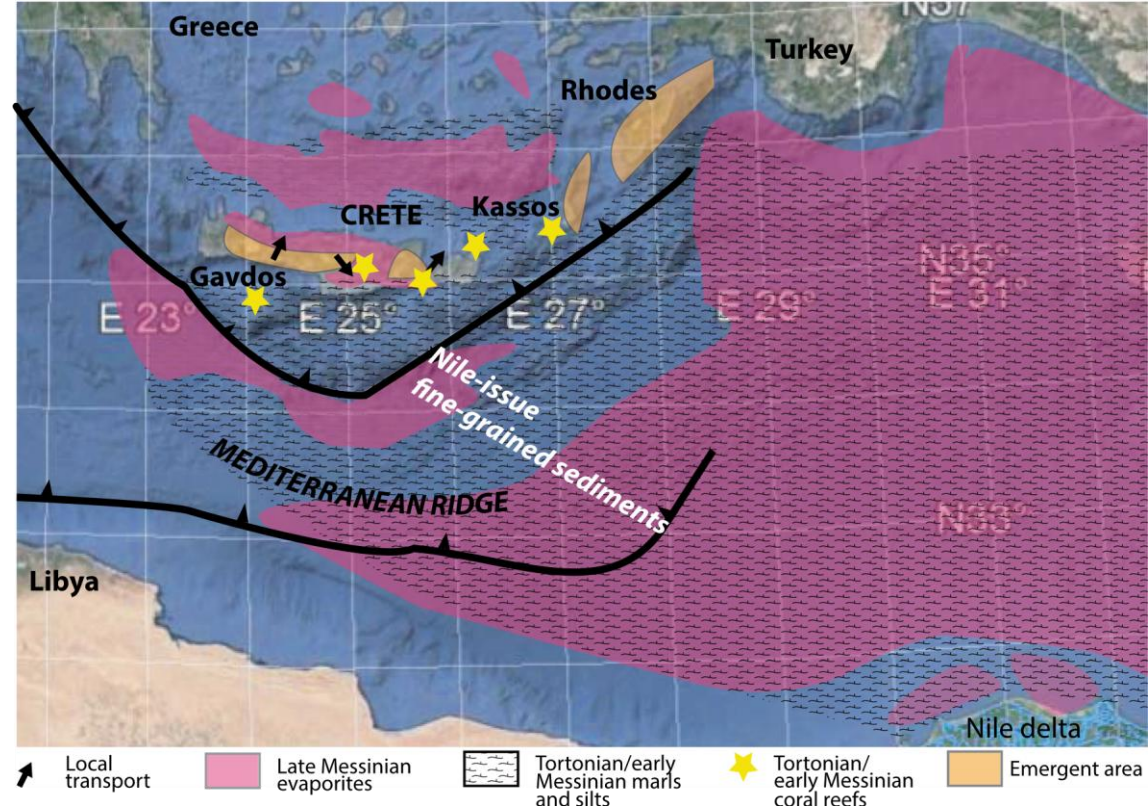
Mediterranean basin was at least three times greater than that of today (Gladstone et al. 2007), based on the cumulative effect of the African rivers in the south and the Paratethys freshwater supply in the north (Krijgsman et al. 2010; Moissette et al. 2010; Natalicchio et al. 2014; Karakitsios et al. 2013, 2017a, b; Van Baak et al. 2017; Vasiliev et al. 2019; Grothe et al. 2020). Therefore, the presence of type III kerogen could at least partly be associated with terrestrial inputs associated with the above two fluvial systems building out into the marine depositional environment. The  $S_1$ -TOC cross-plot (Hunt 1996) revealed autochthonous hydrocarbon production with gas-generating potential (indigenous hydrocarbons with most of the  $S_1$  values being low and ranging from 0.01 mg HC/g TOC to 0.1 mg HC/g TOC; Fig. 6d).

## Thermal maturity

Thermal maturity of sedimentary organic matter is another crucial parameter for the characterization of source rocks, since their hydrocarbon generation potential changes with thermal maturation and the related loss of functional groups (Peters et al. 2005; Zieger et al. 2018). In the present study, the thermal maturity is determined by Rock-Eval PI and  $T_{max}$  measurements. An initial assessment of the degree of thermal maturity reached by the studied sediments is provided by  $T_{max}$  values. To be reliable, we used  $T_{max}$  values in our plots merely for the samples with  $S_2$  values greater than 0.2 mg HC/g rock. Thus,  $T_{max}$  values from the samples with negligible organic richness and very low values for source rock potential were not taken into consideration, because such values cannot be considered as accurate (Peters and Cassa 1994). All reliable samples reach a relatively narrow range of  $T_{max}$  values from 400 to 417 °C (Table 1, Fig. 7a,b) indicating low thermal maturity and the presence of a large portion of unconverted original organic matter. Based on the HI- $T_{max}$  plot (Espitalié et al. 1985), this pattern is further reflected in the kerogen type determination with a dominant gas-prone, type III kerogen distribution within the immature oil-window stage (Fig. 7a). Moreover, the PI- $T_{max}$  cross-plot, which is also used to assess thermal maturity, clearly indicates that the examined upper Messinian sedimentary sequence is immature with respect to oil generation (Fig. 7b). Thus, it can be assumed that the results represent the initial quality of the organic matter. The determination of thermal maturity could be supported by additional geochemical analyses, such as vitrinite reflectance ( $R_o\%$ ) and/or Thermal Alteration Index (TAI) (Peters 1986). However, the studied profile corresponds to a continuous short sedimentary sequence, and therefore significant trends related to changes in regional thermal maturation level cannot be expected. Overall, these deposits have not experienced high temperature during burial and most likely contain very little charcoal or recycled material from older mature rocks. However, thermal maturity and hydrocarbon contribution to working petroleum systems might be enabled for Late Miocene intervals in deeper parts of the basin, where greater burial depths are reached.

## Sedimentary basin dynamics: implications for the onshore depositional environment and petroleum play assessment

The studied samples of Plouti section belong to a mixed siliciclastic-carbonate sequence, which contain gas-prone (Type III kerogen) organic material, but they have not attained adequate thermal burial maturity. These samples are mainly representative of thin, clay organic-rich layers interbedded with massive sandstone and limestone packages. However, even in some of the sandy limestones, terrestrial organic particles could be identified, leading to elevated concentrations of TOC in these intervals. Generally, productivity (Totman Parrish and Curtis 1982; Pedersen and Calvert 1990), preservation (Demaison and Moore 1980; Demaison 1991; Vasiliev et al. 2019), sedimentation rate (Ibach 1982; Creaney and Passey 1993), and salinity influence (Horsfield et al. 1994; Yang and Schulz 2019) are typically considered as the most important factors controlling the organic matter and TOC enrichment in petroleum source rocks (Littke et al. 1997). In Plouti section, organic matter is more abundant in the more siliciclastic parts of the section and less common in the more calcareous intervals. This is evident at the outcrop scale, where the organic matter- and silty clay-rich intervals are interbedded with organic matter-lean and more calcareous intervals. This observation indicates that nutrient supply and thus bio-productivity (Nixon et al. 1986; Van der Zwaan and Jorissen 1991) during that interval were correlated with the deposition of siliciclastic material leading to higher accumulation of organic matter in the sediment. In case of slightly oxygenated bottom waters were still present, increased clastic (i.e., clays) sedimentation would increase both



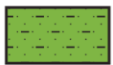
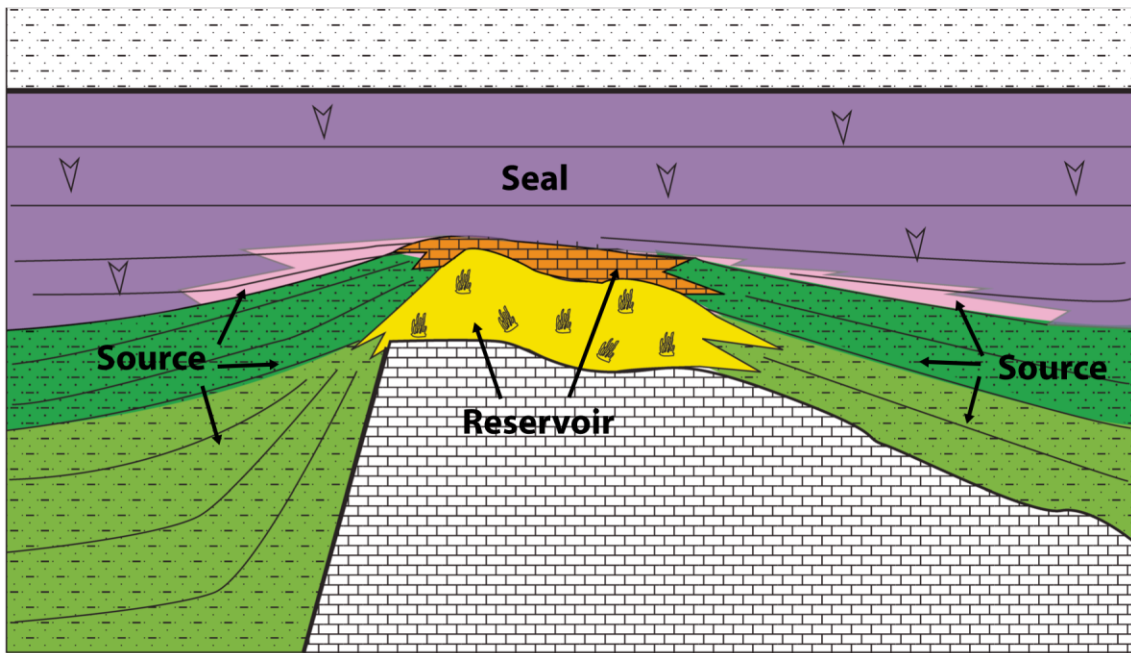
the burial efficiency and the potential of organic matter preservation by insulating the organic matter and shifting the oxic-anoxic boundary within the sediment closer to the sediment–water interface. The above interpretation leads to the

**Fig. 8** Simplified paleogeographic map of the Hellenic subduction zone during the late Miocene. Coral reef and sediment distribution data obtained from Tsaparas and Marcopoulou-Diacantoni (2005),

Brachert et al. (2006), Popov et al. (2006), Antonarakou et al. (2007), Brachert et al. (2007), and Perrin and Bosellini (2013)

lagoonal environmental concept, where a distinct influence of regional freshwater can be expected (e.g., Drinia et al. 2004, 2007) (Fig. 8). Particularly, the terrestrial inputs (e.g., plumes of fine sediments and nutrients) might originate from the northern African margin, which was the closest landmass during the Miocene (Robertson et al. 2012) and high amounts of clastic material were shed into the eastern Mediterranean basin through the Nile Delta (Sestini 1989; Hawie et al. 2013). This scenario is in accordance with the recent findings of Grohmann et al. (2018, 2019), where similar late Miocene clastic sediments derived from the Nile Delta and/ or the Levant margin, have been also characterized as gaseous source rocks onshore and offshore Cyprus.

Respecting the paleogeographic setting of Crete during the middle-late Miocene, characterized by local and regional factors (e.g., horst and graben structures, barrier effect and existence of restricted basins before the MSC, strong terrestrial influence with long distance transport of fine sediments and nutrients from the Nile, marine diagenesis; Fassoulas 2001; Antonarakou et al. 2007, 2019; Zachariasse et al. 2011; Zelilidis et al. 2016; Moissette et al. 2018; Kontakiotis et al. 2019), it can be assumed that similar constellations allowed the deposition of source rocks in several fore-arc basins between Crete and Mediterranean Ridge. However, strong lateral variations in the thickness of the salt layer in the upper deforming sequence (Late Miocene evaporites) between the western and the eastern foreland basins (Maravelis et al. 2015b), along with the local presence of mud diapirism (volcanoes), the diverse trap styles (stratigraphic and structural traps; Pantopoulos et al. 2013; Pantopoulos and Zelilidis, 2014), and the unusually great thickness and diverse character (various lithotypes) of the sedimentary infill differentiate the Mediterranean Ridge from most other accretionary complexes. All these very critical attributes can result to the recognition of several potential hydrocarbon plays in the eastern Mediterranean Sea. Particularly for the study area, where the four ingredients of a good petroleum system namely source, reservoir, trap and seal (Fig. 9) appear to occur (Maravelis et al. 2015b, 2016), it is recommended that the Greek authorities (Ministry of Environment, Energy and Climate Change) proceed with additional extensive onshore geological studies and detailed offshore seismic surveys to explore the existence of hydrocarbons around Crete Island, where the sedimentary succession has been more deeply buried, experienced a higher thermal evolution, and therefore might have matured sufficiently to enter the oil



Tortonian-early Messinian carbonate siliciclastics



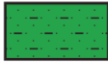
Late Messinian siliciclastics and organic-rich levels



Pliocene shales



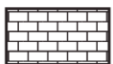
Tortonian-early Messinian carbonate buildups



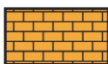
Late Messinian siliciclastics



Late Messinian evaporites



Pre-Miocene carbonates



Late Messinian limestones (TCC)

Fig. 9 Ge

his study,

highlighting the relationship between the stratigraphic development of sedimentary sequences and the southwestern margin of the eastern Mediterranean petroleum system. The Plouti section, which corresponds to the lower part of the gypsum-TCC association, is not to scale and, only provides a conceptual organisation of Late Miocene source to trap sediments generation zone. On this regard, remote sensing, seismic and wire-line log data, as well as cores and fluid samples from wells should be integrated to define the exact thickness and lateral extent of the possible seal, reservoir and source rocks, and the trap style.

## **Conclusions**

The siliciclastic sub-salt sedimentary succession of the Plouti section exposed in the Heraklion basin (central Crete, eastern Mediterranean) was analyzed in order to evaluate the origin, type and maturation level of organic matter, as well as to assess the depositional environment. The geochemical results establish a potential regional Messinian gaseous source rock and analysis details its characteristics in terms of the total content of organic matter, its type, quality, and maturity level, along with the analysis of their generative potential. The TOC content of several samples is more than 0.5 wt % indicating some hydrocarbon generating potential, with both poor to fair ( $0.5\% < \text{TOC} < 1.0\%$ ) and good ( $\text{TOC} > 1.0\%$ ) potential. Rock-Eval parameters reveal the occurrence of source rocks with poor to fair and/or good (organic-rich sediments) hydrocarbon generating potential, characterized by gas-prone kerogen, Type III. Although the investigated sedimentary rocks have sufficient TOC contents and HP values, and contain gas-prone organic material, they have not attained adequate thermal maturity at the study location, as the sedimentary overburden was not sufficient to reach the beginning of the oil window. The immature pre-evaporitic interval identified here could be considered as a potential source of biogenic gas, which is produced at shallow burial depths.

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