

# Molecular Organic Geochemistry of Mudstones from Koum Basin, Cameroon: Paleo-environmental, Age, Maturity and Genetic Implication

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**Abstract** Hydrocarbon resources are still invaluable to the economic growth and social development of most producing countries. Keeping in pace with studies of global source rock development for resourcing future generations, exposed organic-rich, dark-grey mudstones were collected from the Koum Basin and on the basis of their total organic carbon (TOC) content, noted to range between 5.48-6.91 wt.%, organic matter (OM) was extracted and characterized in order to determine the OM source input, paleo-depositional conditions, thermal maturity, age, by which the hydrocarbon potential was deduced using gas chromatography (GC), medium pressure liquid chromatography (MPLC) and gas chromatography-mass spectrometry (GC-MS) techniques. The extract yield and bitumen composition range between 4291–5116 ppm which depicts a fair to very good source rock generative potential, dominated by saturates (41.86-45.96%), aromatics (9.36-26.36%), and resin/asphaltene (31.78-44.68%) pointing to normal paraffinic generated hydrocarbons. Source-related molecular markers indicate a mixed aquatic algae and clastic-rich terrigenous OM input, preserved under sub-oxic to oxic conditions, typical of fluvial-deltaic lacustrine systems. Gammacerane/Hopane ratios (0.26-0.27), support the fact that the lacustrine source rocks were likely developed under somewhat restricted circulation and moderate/low salinity. From maturity ratios of  $Ts/(Ts + Tm)$ ,  $C_{32} 22S/(22S + 22R)$  homohopane, the  $20S/(20S + 20R)$  and  $\beta\beta/(\beta\beta + \alpha\alpha)$   $C_{29}$  and Methyl Phenanthrene Index with vitrinite reflectance value equivalent of 0.46-0.60%, it can give a conclusion that the analyzed extracts are from early thermal cracking of OM at the incipient oil window. Based on age-specific biomarkers, the extracts were probably derived from the Early Cretaceous. Accordingly, findings are comparable with some active Cretaceous lacustrine source rocks in some basins within the West Central African Rift system (WCARS) and therefore provide a better understanding of source rock development during the Cretaceous in a regional context. Nevertheless, the deeper unexposed organic-rich units of the Koum Basin may have a pod of active source rock.

**Keywords:** Source rocks, Biomarkers, Organic matter, Thermal maturity, Paleo-redox conditions, Cretaceous Koum Basin

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## 1. Introduction

Globally, hydrocarbons have traditionally been the driving force for most economies and will continue to play a vital role in the energy mix over the next decades. In accordance with the United Nations (UN) Sustainable

Development Goals (SDG) [1,2] access to affordable energy is essential for economic growth, social development and crucial to all 17 UN SDGs. Oil and gas have been used as affordable energy for the past century which has supported economic growth and has the potential to continue to contribute to all 17 SDGs. Furthermore, UNESCO, through the International Geoscience Program (IGCP) and in partnership with the

International Union of Geological Sciences (IUGS), has put in place a response system to this universal call to action, amongst which, the Resourcing Future generations (RFG) initiative which promotes sustainable development and corroborates the concept of SDGs [3]. In Cameroon, with the declining production from aging fields, it becomes important to find answers on how future hydrocarbon supply can keep pace with the increasing demand to maintain the standards of our modern life through accessible energy. Therefore, the search for new reserves is eminent.

The availability of these natural resources has diminished in the past decades while the demand progressively increases. To bridge the gap between this energy demand and supply, there is a pressing need for effective and efficient techniques that improve future petroleum exploration strategies for finding new hydrocarbon deposits. The search for source rocks and assessment of the potentiality of a particular unit is a significant task during oil exploration since every oil or gas play originates from the source rock [4]. According to [5] organic geochemistry articulates the source and fate of organic molecules and is a useful tool for identifying source rocks and grouping crude oils/bitumen into families both at the early and later stages in exploration for hydrocarbons [6,7]. Characterization of bitumen extracts is amongst the preliminary assessments for accessing paleo-preservation conditions, OM maturity, and the type of hydrocarbons generated [8,9] and this extract contains complex molecular compounds known as biomarkers. Biomarkers also known as molecular fossils, are molecules that retain sufficient biochemical structure to allow them to be linked to their original source organisms: their study places limits upon the geological processes that have subsequently affected their molecular structure [6,10]. Over the past few decades, molecular characterization of petroleum has drawn much attention and is used to solve issues related to exploration and production of oil and gas such as; (i) the paleo-depositional environment, organic facies, or types of organic input, paleo-preservation conditions [11-19]; (ii) the thermal maturity, one of the most important parameters in petroleum exploration and appraisal [11,20]; (iii) the characterization of charge risk [21,22] (iv) the estimated geological age of the source rock [23,24,25,26,27] and (v) to make oil-oil and oil-source rock correlations and diagenetic and catagenetic transformations in sediments [28].

Prolific petroleum-producing basins from lower Cretaceous source rocks are in WCARS, coupled with vigorous exploration activity in the upper Benue trough basins, with active source rocks has attracted the interest of various oil companies and researchers. Situating the Koum Basin within this prolific petroleum province, from a regional point of view, motivates detailed study on its source rock generative potential. The integration of such studies altogether would give a comprehensive knowledge of the Cretaceous units throughout the entire region. Although some preliminary studies had been undertaken on the hydrocarbon generation potential [29], significant gaps in our knowledge persist with respect to the

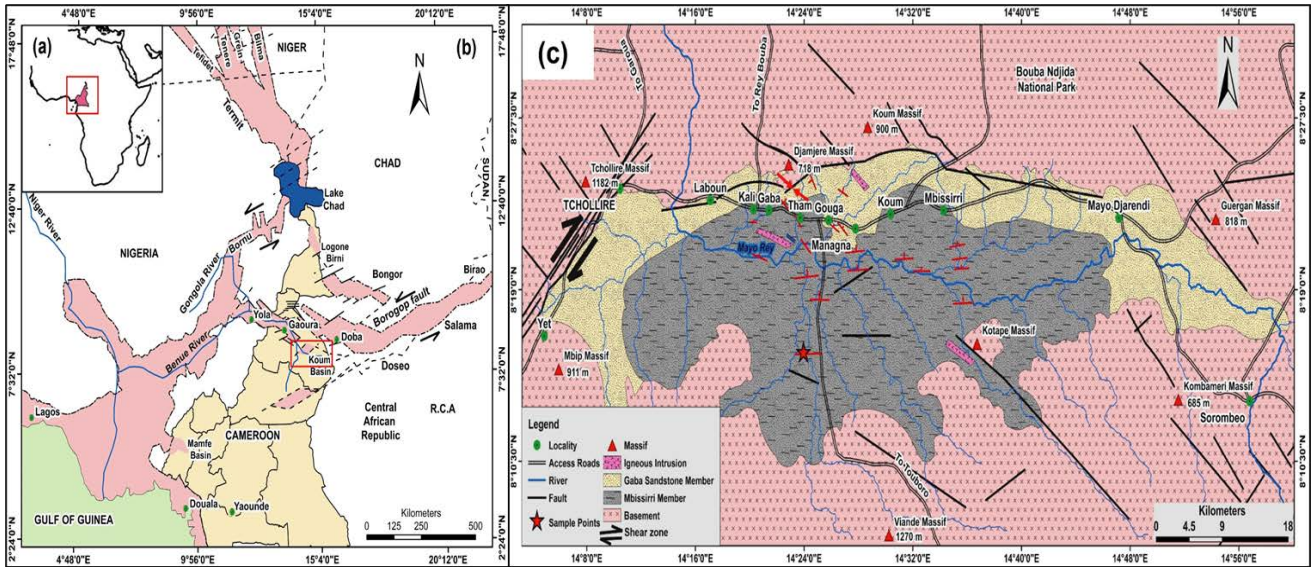
source input, maturity, and depositional environments of organic-rich sediments within the basin are lacking and may have been responsible for the unproductive exploration attempts.

To date, in the Koum Basin, there has been no systematic study of their organic facies character, i.e., the paleoenvironmental controls which influence the abundance, origin, composition, and source rock potential of their sedimentary OM content. Therefore, a significant gap in our knowledge persists with respect to the sedimentary facies, the relationship between the depositional environments, and the development of organic-rich sediments within the basin, and vital questions regarding the source rock remain unanswered. This may have been responsible for the unsuccessful exploration attempts.

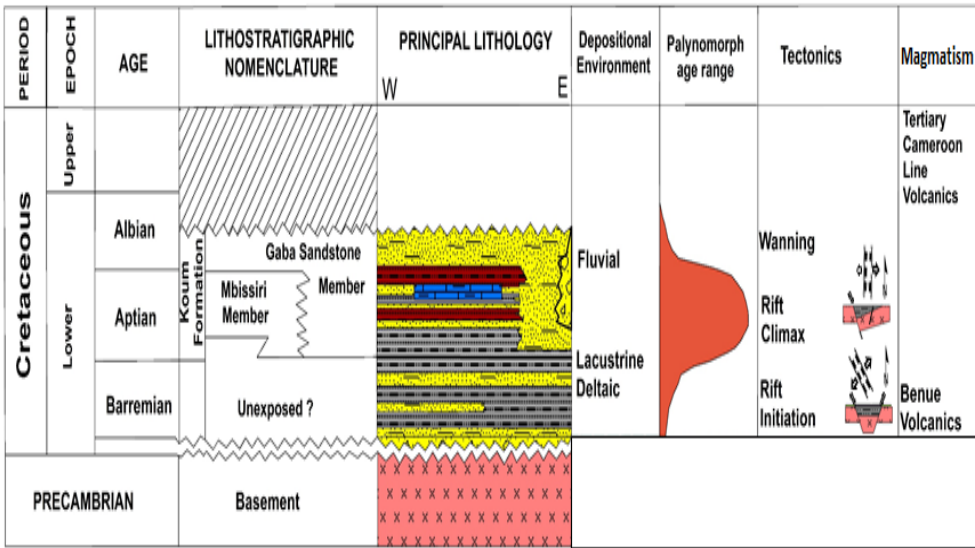
The objectives of this study are to understand the evolution of sedimentary OM at the molecular scale, identify biomarkers coded in the OM and use their parameters as proxies to deduce organic source input, type, paleo-redox conditions/ depositional environment during burial, thermal maturity, geologic age, and past ecosystems of the unexplored Koum Basin syn-rift sequence and possible correlations its WCARS counterparts. The outcome will improve our current knowledge and understanding of the basin for better prediction of source rock targets which would be guided further hydrocarbon exploration ventures in the basin.

## 2. Geological Setting

The Koum Basin is one in a series of intra-continental rift basins, associated with the Benue Trough Aulacogen, part of the West and Central African Rift Systems (WCARS), whose origin is generally associated with the progressive breakup of the Gondwana supercontinent and subsequent separation of South America from the African continent [30] (Figure 1). The basin's geological half-graben structure with an East-West orientation controlled by presumably listric normal, synsedimentary faults striking from N070°E to E-W that are associated with sinistral wrench faults trending in an NW-SE direction [35] govern sediment supply. Koum Basin is in the transition zone between the West African Rift System (WARS) and the Central African Rift System (CARS), developed by fault-controlled subsidence along with pre-existing structural trends in the basement during the separation of South America from the African continent. Its favorable situation, within the WCARS early Cretaceous tectonic province characterized by prolific hydrocarbon-producing basins, with thick sequences of lacustrine source rocks like Bongor Basin, Doba Basin, and proven source rock intervals in the Benue Trough makes it an exploration target. Some of these basins developed within and immediately adjacent to the Cretaceous shear zones and others formed near their endpoints at angles of 90°-120° to the shear movement like the Koum Basin and the Mbere rift basin with the Tchollire-Banyo Shear zone and the Central African Shear zones respectively.



**Figure 1.** Configuration of the WCARS showing the Koum Basin in relationship to other rift basins modified from [31,32] (c) Geological map of the Koum Basin showing the sample location used in this study (modified after [33,34])



**Figure 2.** Conceptual tectono-stratigraphy and gross depositional environments of the sedimentary fill of the Koum Basin (based on [33]).

The stratigraphy of this basin has not been studied in detail and little published information is available. Based on field observations and previous work by [33] the lithostratigraphic succession is summarized in (Figure 2). The pre-rift early Cretaceous sequence is unexposed in the Koum Basin and regionally characterized by an extreme peneplanation process and sandy fluvial-lacustrine deposits which directly overlies faulted Pan-African granito-gneiss basement prior to major rifting episodes.

The syn-rift succession is characterized by a classic tripartite cyclic sequence [36] of alluvial/fluvial-lacustrine lower section, deep lacustrine, and shallow ephemeral lacustrine middle section, and fluvial channel/overbank upper part. The upper section of the Mbissirri member consists of organic matter-rich shales and mudstones (distal lake deposits) and organic matter-poor bioturbated mudstones and siltstones (proximal lake deposits) deposited on a flooding surface with polygonal desiccation cracks on the flooding surfaces as evidence of exposure episodes. The lower part consists of a thick

sequence of dark grey-black organic-rich facies that may have source rock potential, probably deposited under humid climatic conditions.

The Early Cretaceous succession in the Koum Basin is correlative with the Bima Formation within the WCARS [37] and contains a Cretaceous lacustrine pod of source rocks. Furthermore, post-rift uplift of the Adamawa regions facilitates the erosion of ~3 km of syn-rift deposits in northern Cameroon and southern Lake Chad area [31], which is very evident in the Koum Basin.

### 3. Materials and Methods

The standard methods of geological or sedimentological field investigations were used during two-week intensive fieldwork in April 2021, on the exposed sediments of Mbissirri Member in the Koum Basin. Sedimentary features, color, textural, trace or body fossils, thicknesses of beds, their field relationships, formation boundaries,



sedimentary structures, stratigraphic sections were identified, measured, and dark grey-black samples were judged visually as mudstones were collected. Stratigraphic and composite sections at relevant localities were logged using standard procedures. Lithofacies and palynofacies investigations supported the determination of the paleodepositional environment and correlation. Fifteen (15) dark-colored samples were analyzed for TOC using LECO C230 instrument at the Stratochem Lab in Cairo after being decarbonized. Combustions were carried out using an induction furnace and carbon was measured by infrared absorption. Only samples with TOC (wt. %) exceeding [8] 0.5 wt. % were submitted for quantitative rock extraction and whole extract-GC (KB-KAL001, KB-KAL002, and KB-KAL003/4), all from the Mayo Gaba outcrop section. Based on GC results, two (2) extracted samples (KB-KAL001, and KB-KAL003/4) were further submitted for advanced analysis including MPLC and biomarker GC-MS for the saturated and aromatic fractions. All the laboratory analyses were performed at StratoChem Services (SCS) in Cairo, Egypt.

After cleaning and removing oxidized surfaces, the samples were pulverized through a 40-Mesh sieve, 5-10g was solvent-extracted with Dichloromethane using a Dionex Accelerated Solvent Extractor (ASE 150), for at least 24 hours. The extracted organic matter (EOM) was fractionated into aliphatic, aromatic, and polars (resins + asphaltene) by MPLC, following the standard procedures outlined by [38].

GC analyses were performed on the saturated fractions, using Hewlett Packard 6890N GC equipped with a dimethylpolysiloxane stationary phase capillary column (Column: J & W Scientific DB-1, 60m x 0.25mm i.d.; 0.25 $\mu$ m film thickness), with Helium as the carrier gas. The programmed temperature starts from 30°C is held for 5 minutes then ramped at 3°C/ minute to 320°C with a final hold time of 23.3 minutes, with Helium as the carrier gas. Finally, the concentration of the separated hydrocarbons (from C4 to C41) was detected by a flame ionization detector (FID). For quantification purposes, peak areas of all identifiable n-alkanes and the acyclic isoprenoid hydrocarbons were determined using an automatic integration tool from the GC data system. The compounds were identified through analyses of a known standard, the Norwegian Industry Guide to Organic Geochemical Analysis [39].

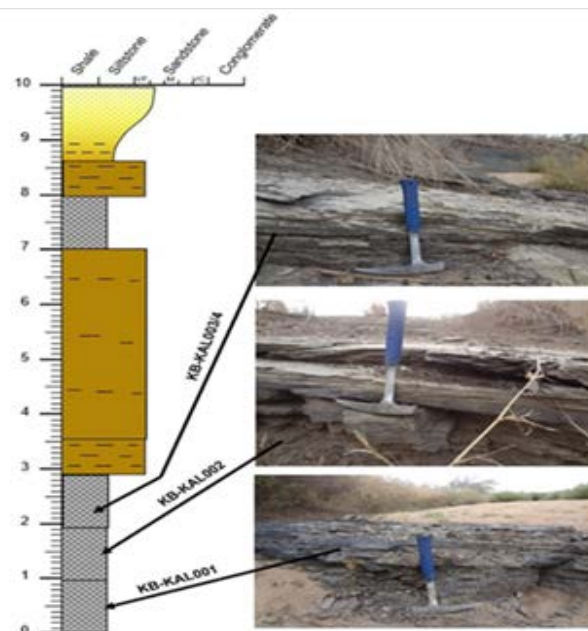
Furthermore, qualitative, and quantitative analysis via GC-MS was performed on both the aliphatics and aromatics GC-MS, using an Agilent 7890B GC split/splitless injector integrated with Agilent 5977A Mass Selective Detector (MSD). The GC/MS was fitted with a ZB-5, 60m x 0.25mm capillary column i.d.; 0.1 $\mu$ m film thickness, through which the vaporized hydrocarbons from the extract were passed by a stream of inert carrier gas (Helium). The temperature program initiates at 100°C and ramped at 20°C/ minute to 170°C then ramped at 1.5°C/ minute to 320°C followed by an isothermal period of 20 minutes. The mass spectrometer (ion source T=230°C, electron ionization at 70eV, quadrupole analysis: T=150°C) was operated in the Selected Ion Monitoring (SIM) mode. Data were processed with an Agilent Chemstation data system. Identification of individual compounds was based on retention time in total ion current (TIC) chromatograms and comparison of mass

spectra of a known standard, the Norwegian Oil Standard NGR NSO-1. Relative percentages of different compound groups in the saturated and aromatic hydrocarbon fractions were calculated using peak areas in relation to those of internal standards (Cholane and Orthoterphenyl, respectively). The analysis of the saturated fractions involved two important hydrocarbon classes: n-Alkanes and Acyclic Isoprenoids were identified from fragmentograms: terpanes from m/z 191 and steranes from m/z 217 and m/z 218. The aromatic hydrocarbon fractions were detected in their key mass chromatograms (m/z 253, 231, and 245) based on the relative retention times.

## 4. Results

### 4.1. Sedimentology

A total of five (05) dark grey mudrocks shale were collected within the Mayo Gaba outcrop section. Three of the samples were organic-rich composed of massive to fissile black shales and show coarsening upward sequence with the basal part comprising of the shale, whereas the upper part is made up of purple mudstones intercalated with siltstones. The shale layers reach a maximum thickness of about 75 m. The shales show strong evidence of weathering on the exposed surfaces as seen by a color change from black to whitish-grey.

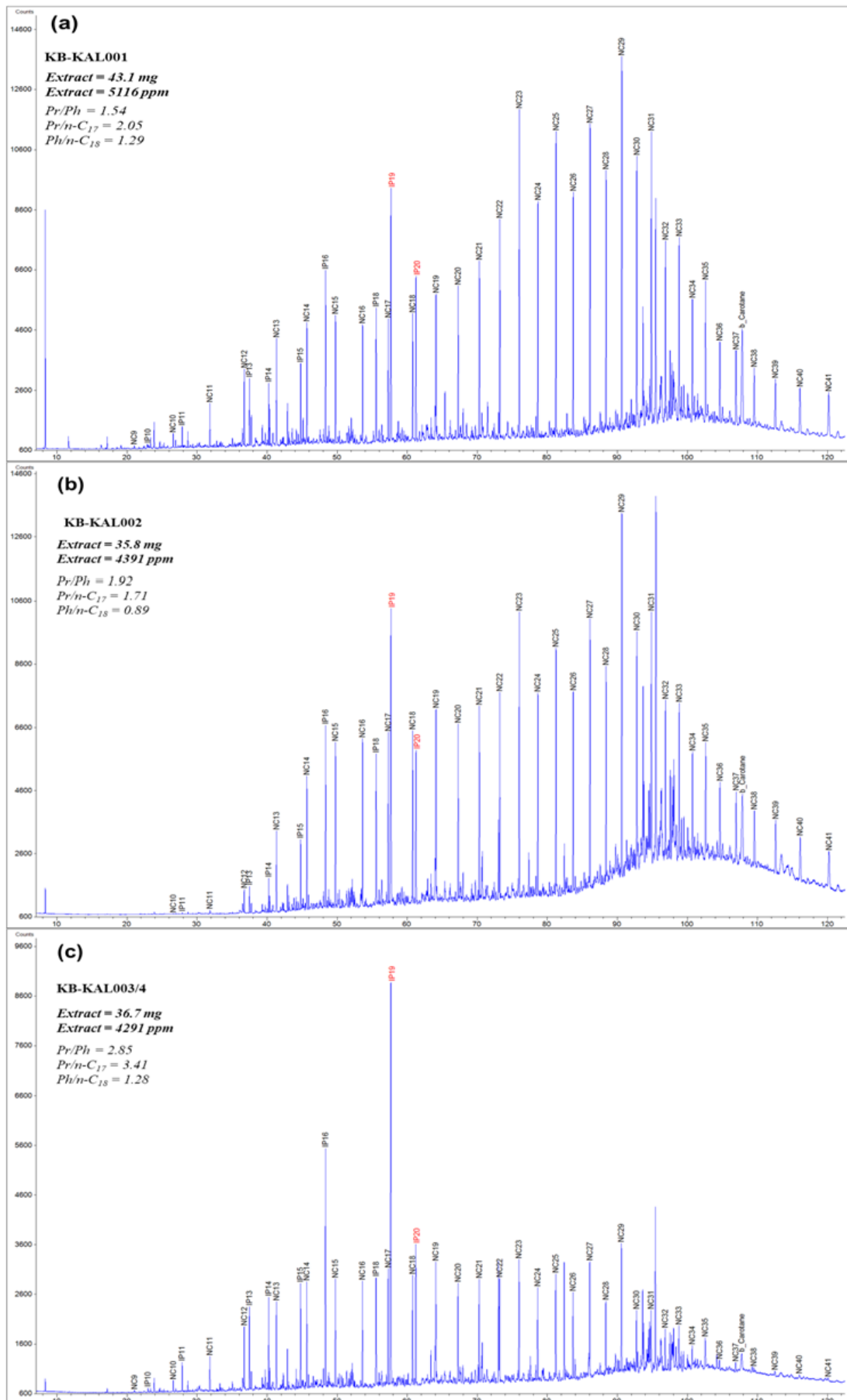


**Figure 3.** Representative outcrop and sedimentary log of the Gaba section, Koum Formation

### 4.2. Total Organic Carbon Contents (TOC wt. %)

Most of the samples (~90%) from the sampled litho-stratigraphic intervals are organically lean and ~10% organic-rich with a TOC content of 2.81-6.91wt.%. The Mayo Gaba section (Figure 3) shows an organic-rich episode with TOC values ranging from 5.92 wt. % (KB-KAL001) 5.48 wt. % (KB-KAL002) and 6.91 wt. % (KB-KAL003/4) (Table 1). However, these high TOC values

are not a guarantee that these are source rocks but rather one of the proxies for assessing source rock potentiality.



**Figure 4.** Gas Chromatograms for saturated fractions of the extracted Bitumen, Koum. IP13 to IP20 = Isoprenoid (IP19 Pristane; IP20 Phytane), n-C7-n-C20 = Low to medium molecular weight n-alkane, n-C21-n-C41 = High molecular weight n-alkane, b-Carotane= β-Carotane

**Table 1. n-Alkanes and isoprenoid ratios of the studied samples**

Sample ID	Whole Extract GC Ratios		
	KB-KAL001	KB-KAL002	KB-KAL003/4
TOC	5,92	5,48	6,91
Pr/Ph	1,54	1,92	2,85
Pr/nC17	2,05	1,71	3,41
Ph/nC18	1,29	0,89	1,28
CPI	1,35	1,40	1,39
OEP	1,31	1,40	1,44
TAR	2,94	1,98	1,00
Wax Index	4,27	2,83	1,77

NB: Pr pristane, Ph phytane, Pr/Ph pristane/phytane, Pr/n-C17 pristane/n-C17, Pr/n-C18 pristane/n-C18,  $TAR = \sum C27+29+31 / \sum C15+17+19$ ,  $OEP = C25+6 \times (C27+C29) / 4 \times (C26+C28)$  Waxiness index =  $\sum (n-C21-n-C31) / \sum (n-C15-n-C20)$ .

### 4.3. Gas Chromatography Results

The saturated hydrocarbon extracted from mudstones have been analyzed by GC-FID from C10 to about C41 and samples show a unimodal distribution of n-alkanes with an apex in the range n-C22 to n-C32, elevated C15-C20 isoprenoids in which pristane (pr) and phytane (ph) are dominant and relatively high concentration of the high-molecular-weight n-alkanes. Odd-even predominance (OEP), carbon preference index (CPI), terrigenous/aquatic ratio (TAR), and waxiness index were calculated and presented in Table 1. The distribution of saturated isoprenoid hydrocarbons is defined by the Pr/Ph ratios, and the proportion of these compounds to the n-alkanes given by the pristane/n-C17 (Pr/C17) and phytane/n-C18 (Ph/C18) ratios is presented in Table 1. Figure 4 a-c presents the mass chromatograms of saturated hydrocarbon fractions of the studied samples.

## 4.4. Bulk Molecular Composition of Aliphatic Hydrocarbons

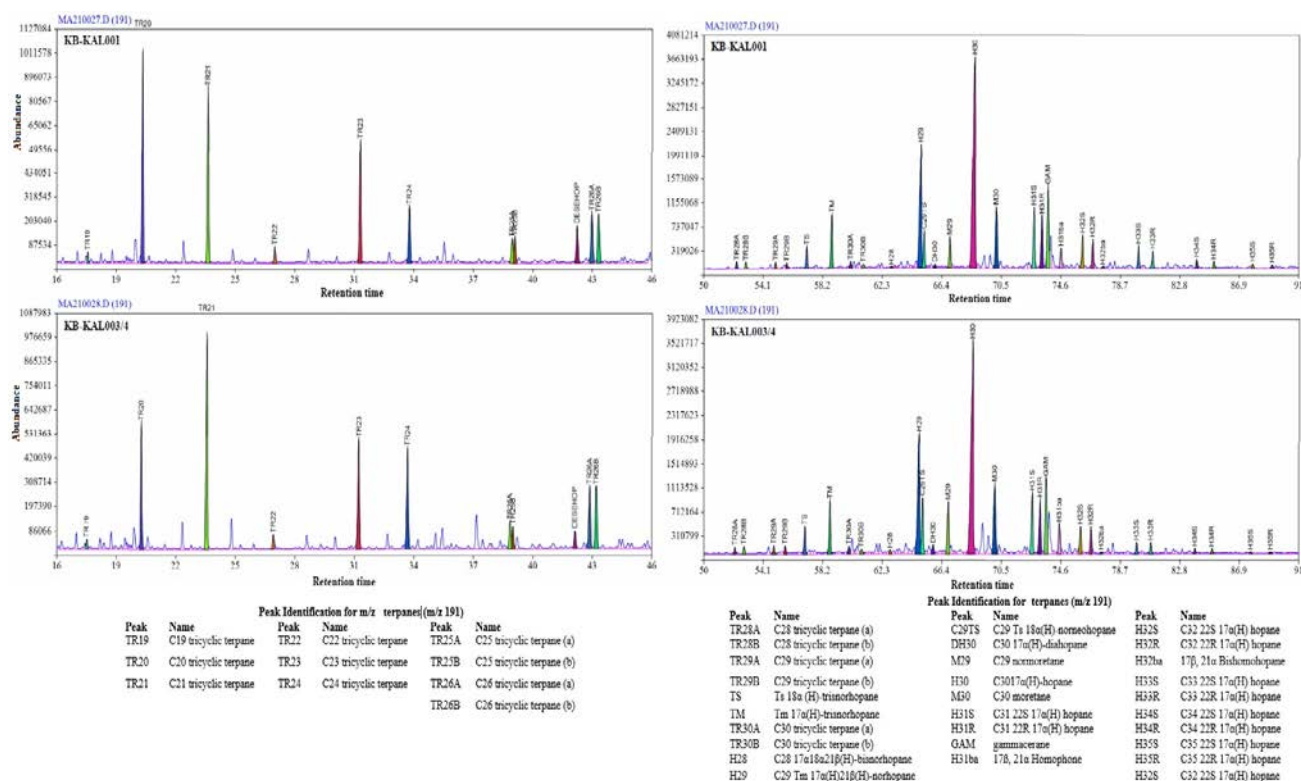
The bulk molecular composition of aliphatic hydrocarbons, derived from MPLC and biomarker GC-MS indicate that the samples are dominated by saturates (41.86-45.96%), with moderate amounts of aromatics (9.36-26.36%) and high 31.78-44.68% NSO+Asphaltene content Table 2.

**Table 2. Bulk composition Data**

Sample Description	Liquid chromatography Analysis	
	KB-KAL001	KB-KAL003
Saturate wt. %	45,96	41,86
Aromatic wt. %	9,36	26,36
Saturate / Aromatic	4,91	1,59
NSO wt. %	33,62	28,68
Asphaltene wt. %	11,06	3,10
NSO + Asph wt. %	44,68	31,78

### 4.5. Gas Chromatography-Mass Spectrometry (GC-MS)

The source rock extracts in the current study have been monitored through several masses by using Selected Ion Monitoring (SIM) mode for fragmented ions, among them: m/z 191 (terpanes (pentacyclic triterpanes (hopanes), tricyclic and tetracyclic terpanes), m/z 217 (steranes (14 $\alpha$ (H)17 $\alpha$ (H) regular sterane configuration) and m/z 218 (steranes (14 $\beta$ (H)17 $\beta$ (H) isosterane configuration) mass chromatograms for aliphatic hydrocarbons of the EOM are presented in Figure 5 a-c and their respective molecular ratios calculated using the peak areas are summarized in Table 3, as well as definitions and measurement procedures for the saturated fractions are summarized in Table 5.



**Figure 5a.** Representative fragmentograms of saturated hydrocarbon fractions; terpanes (m/z = 191) obtained by GC-MS analysis of the EOM from KB-KAL001 and KB-KAL003/4

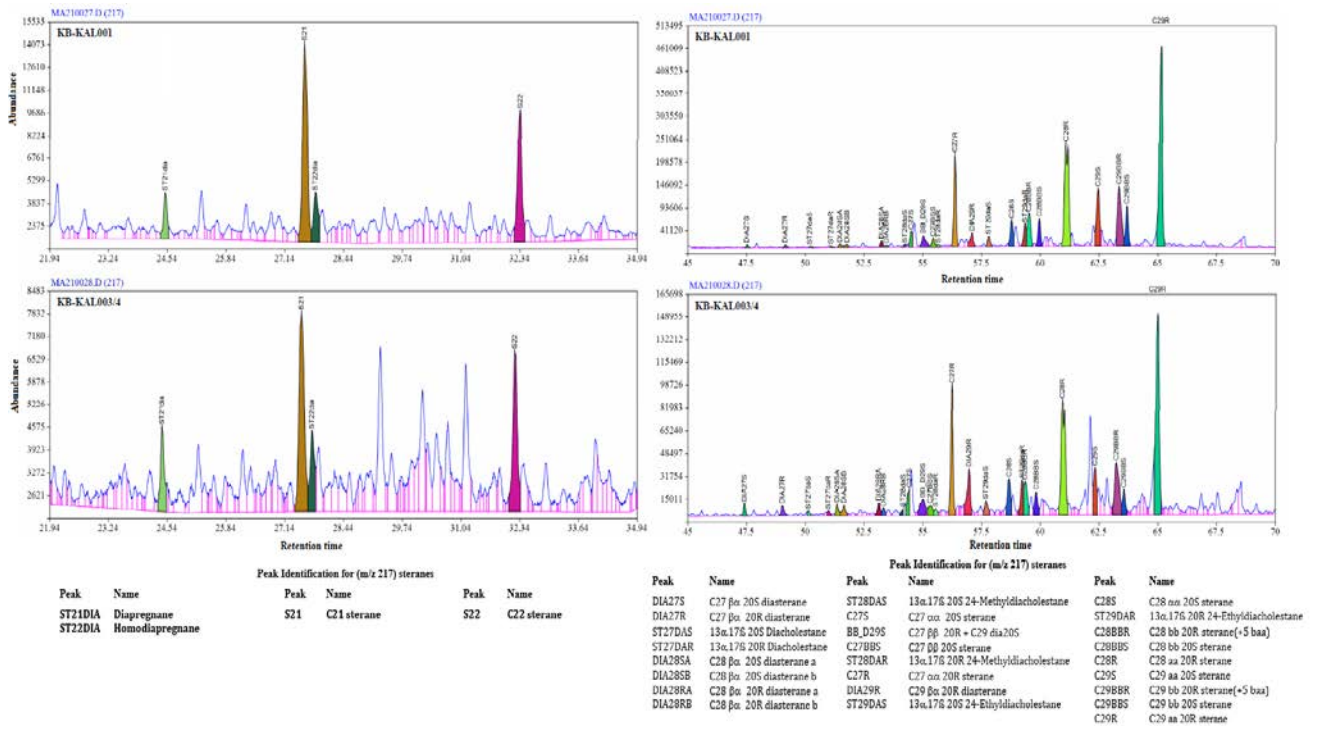


Figure 5b. Representative fragmentograms of saturated hydrocarbon fractions; steranes m/z = 217 obtained by GC-MS analysis of the EOM from t from KB-KAL001 and KB-KAL003/4

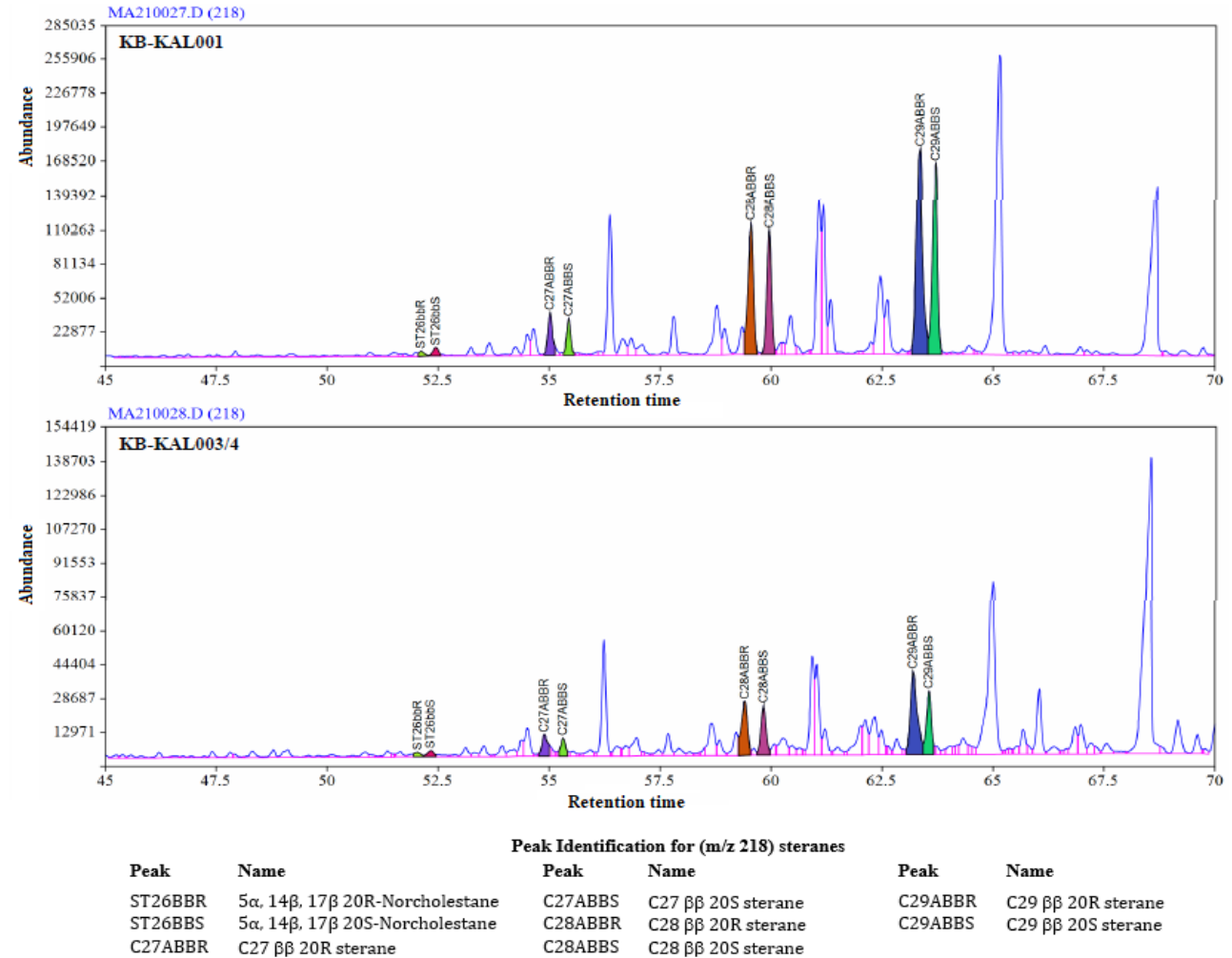
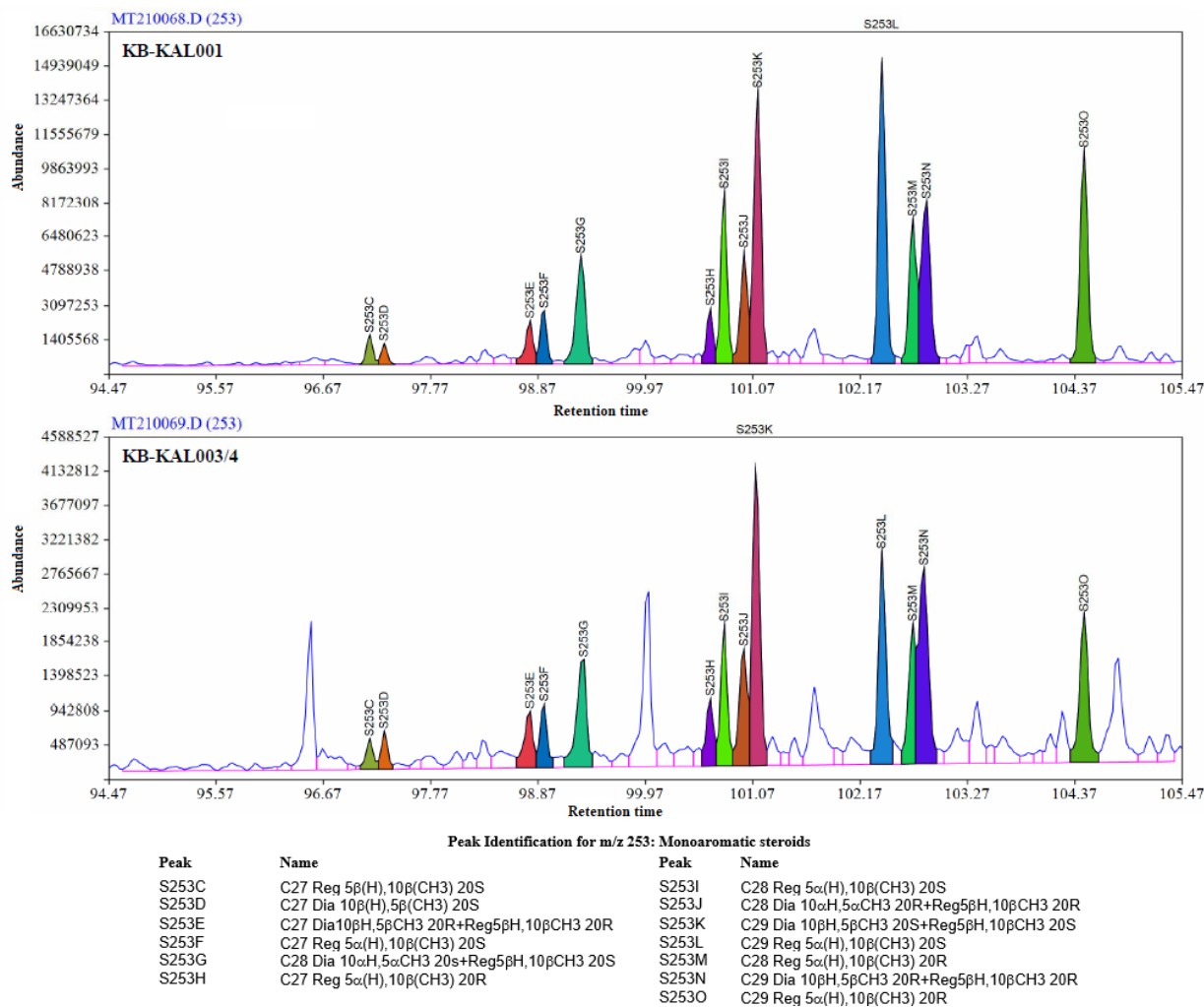


Figure 5c. Representative fragmentograms of saturated hydrocarbon fractions; βsteranes m/z = 218 obtained by GC-MS analysis of the EOM from t from KB-KAL001 and KB-KAL003/4





**Figure 6a.** Mass chromatograms ( $m/z$  253) showing the distribution of Mono- (MAS) and Triaromatic Steroids (TAS) obtained by GC-MS analysis of the EOM from the analyzed samples

**Table 3.** Calculated GC-MS ratios (steranes ( $m/z$  217; 218) and terpanes ( $m/z$  191), based on peak areas (see Table 5)

		Steranes ( $m/z$ 217; 218)	
		KB-KAL001	KB-KAL003/4
Source input and paleodepositional conditions	%C <sub>27</sub> $\alpha\beta\beta$ S (218)	9.1	12.4
	%C <sub>28</sub> $\alpha\beta\beta$ S (218)	34.7	39.3
	%C <sub>29</sub> $\alpha\beta\beta$ S (218)	56.2	48.3
	%C <sub>27</sub> $\alpha\alpha\alpha$ R (217)	16.3	19.7
	%C <sub>28</sub> $\alpha\alpha\alpha$ R (217)	34.2	33.4
	%C <sub>29</sub> $\alpha\alpha\alpha$ R (217)	49.5	46.8
	C <sub>27</sub> /C <sub>29</sub> ( $\alpha\beta\beta$ S) (218)	0.16	0.26
	C <sub>28</sub> /C <sub>29</sub> ( $\alpha\beta\beta$ S) (218)	0.62	0.81
	Diaster/ $\alpha\alpha\alpha$ Ster (C <sub>27</sub> ) (217)	0.06	0.18
	Steranes/Hopanes	0.14	0.06
	C30 abbS Sterane Index (218)	0	0
Thermal maturity	S/(S+R) (C <sub>29</sub> $\alpha\alpha\alpha$ ) (217)	0.22	0.17
	$\beta\beta$ /( $\beta\beta$ + $\alpha\alpha$ ) (C <sub>29</sub> ) (217)	0.30	0.28
<b>Terpanes (<math>m/z</math> 191)</b>			
Source input and paleodepositional conditions	Oleanane/Hopane		
	Gammacerane/Hopane	0.27	0.26
	Norhopane/Hopane	0.46	0.48
	Diahopane/Hopane	0.01	0.04
	H35/H34 Homohopanes	0.54	0.48
	C24 Tetracyclic/Hopane	0.03	0.01
	C24 Tetracyclic/C26 Tricyclics	0.34	0.14
	C23/C24 Tricyclic terpanes	1.93	1.09
	C19/C23 Tricyclic terpanes	0.07	0.07
	C26/C25 Tricyclic terpanes	2.14	2.49
Thermal maturity	Ts/(Ts+Tm) trisnorhopanes	0.29	0.35
	C29Ts/C29 Hopane	0.23	0.37
	H32 S/(R+S) Homohopanes	0.54	0.51
	Tricyclic terpanes/Hopanes	0.22	0.24
	Tricyclic terpanes/Steranes	1.55	4.07



**Table 4. Calculated GC-MS ratios (m/z 253 MONO-(MAS) and m/z 231 triaromatic steroids (TAS), m/z 245 triaromatic methylsteroids, phenanthrenes, naphthalenes, and dibenzothiophenes, based on peak areas (see Table 6)**

Mono-(Mas) and Triaromatic Steroids (TAS) (m/z 253, m/z 231)			
	KB-KAL001	KB-KAL003/4	
Source input and paleo depositional conditions	%26 TAS	8.4	8.3
	%27 TAS	25.8	25.3
	%28 TAS	65.8	66.3
	%26 MAS	8.4	8.3
	%27 MAS	11.0	14.9
	%28 MAS	31.3	30.2
	%29 MAS	57.6	54.9
Thermal maturity	(C20+C21)/□ TAS	0.01	0.02
	TAS #1 20/20+27	0.06	0.11
	TAS #2 21/21+28	0.02	0.03
	(C21+C22)/S MAS	0.01	0.04
	TAS/(MAS+TAS)	0.78	0.81
	TA28/(TA28+MA29)	0.81	0.84
<b>Triaromatic Methylsteroids (m/z 245)</b>			
Age	Dinosteroid Index	0.33	0.37
	C4/C3+C4 Mester	0.42	0.60
<b>Phenanthrenes, Naphthalenes and Dibenzothiophenes</b>			
Thermal maturity	MPI-1	0.35	0.16
	Rc(a) if Ro < 1.3 (Ro%)	0.58	0.46
	Rc(b) if Ro > 1.3 (Ro%)	2.09	2.21
Source input and paleo depositional conditions	DBT/Phenanthrene	0.08	0.12

**Table 5. Definitions and measurement procedures used in the literature for saturated fraction**

Parameter	Formula
<b>Steranes (m/z 217; 218)</b>	
%C <sub>27</sub> αβS (218)	100*C27ABBS/(C27ABBS+C28ABBS+C29ABBS)
%C <sub>28</sub> αβS (218)	100*C28ABBS/(C27ABBS+C28ABBS+C29ABBS)
%C <sub>29</sub> αβS (218)	100*C29ABBS/(C27ABBS+C28ABBS+C29ABBS)
%C <sub>27</sub> aaaR (217)	100*C27R/(C27R+C28R+C29R)
%C <sub>28</sub> aaaR (217)	100*C28R/(C27R+C28R+C29R)
%C <sub>29</sub> aaaR (217)	100*C29R/(C27R+C28R+C29R)
S/(S+R) (C <sub>29</sub> aaa) (217)	C29S/(C29S+C29R)
ββ/(ααR+ββS) (C <sub>29</sub> ) (217)	(C29BBS+C29BBS)/(C29R+C29BBS+C29BBS+C29R)
ββ/(αα+ββ) (C <sub>29</sub> ) (217)	(C29BBR+C29BBS)/(C29S+C29BBR+C29BBS+C29R)
Diaster/aaa Ster (C <sub>27</sub> ) (217)	(DIA27S+DIA27R)/(C27S+C27R)
C30 αβS Sterane Index (218)	100*(C30ABBS)/(C27ABBS+C28ABBS+C29ABBS+C30ABBS)
<b>Terpanes (m/z 191)</b>	
Oleanane/Hopane	OL/H30
Gammacerane/Hopane	GAM/H30
Norhopane/Hopane	H29/H30
Diahopane/Hopane	DH30/H30
Ts/Tm trisnorhopanes	TS/TM
Ts/(Ts+Tm) trisnorhopanes	TS/(TS+TM)
C29Ts/C29 Hopane	C29TS/H29
H35/H34 Homohopanes	(H35R+H35S)/(H34R+H34S)
C24 Tetracyclic/Hopane	(Desehop)/H30
C24 Tetracyclic/C26 Tricyclics	(Desehop)/(TR26A+TR26B)
C23/C24 Tricyclic terpanes	Tr23/Tr24
C19/C23 Tricyclic terpanes	Tr19/Tr23
C26/C25 Tricyclic terpanes	(TR26A+TR26B)/(TR25A+TR25B)
(C28+C29 Tricyclics)/Ts	(Tr28A+Tr28B+Tr29A+Tr29B)/TS
<b>Various (m/z 191; 217)</b>	
[Steranes]/[Hopanes]	(DIA27S+DIA27R+DIA28SA+DIA28SB+DIA28RA+DIA28RB+C27S+BB_D29S+C27BBS+C27R+DIA29R+C28S+C28BBR+C28BBS+C28R+C29S+C29BBR+C29BBS+C29R)/(TS+TM+H28+H29+C29TS+DH30+H30+H31R+H31S+H32R+H32S+H33R+H33S+H34R+H34S+H35R+H35S)
[Tricyclic terpanes]/[Hopanes]	(DIA27S+DIA27R+DIA28SA+DIA28SB+DIA28RA+DIA28RB+C27S+BB_D29S+C27BBS+C27R+DIA29R+C28S+C28BBR+C28BBS+C28R+C29S+C29BBR+C29BBS+C29R)/(TS+TM+H28+H29+C29TS+DH30+H30+H31R+H31S+H32R+H32S+H33R+H33S+H34R+H34S+H35R+H35S)
[Tricyclic terpanes]/[Steranes]	(TR19+TR20+TR21+TR22+TR23+TR24+TR25A+TR25B+TR26A+TR26B+TR28A+TR28B+TR29A+TR29B+TR30A+TR30B)/(TS+TM+H28+H29+C29TS+DH30+H30+H31R+H31S+H32R+H32S+H33R+H33S+H34R+H34S+H35R+H35S)
	(TR19+TR20+TR21+TR22+TR23+TR24+TR25A+TR25B+TR26A+TR26B+TR28A+TR28B+TR29A+TR29B+TR30A+TR30B)/(DIA27S+DIA27R+DIA28SA+DIA28SB+DIA28RA+DIA28RB+C27S+BB_D29S+C27BBS+C27R+DIA29R+C28S+C28BBR+C28BBS+C28R+C29S+C29BBR+C29BBS+C29R)

Table 6. Definitions and measurement procedures used in the literature for the aromatic fractions

Mono- (MAS) and Triaromatic Steroids (TAS)	
(C20+C21)/S TAS	$(231A20+231B21)/(231A20+231B21+231C26+231D26+231E28+231F27+231G28+C29TA3)$
TAS #1 20/20+27	$(231A20)/(231A20+231F27)$
TAS #2 21/21+28	$(231B21)/(231B21+231G28)$
%26 TAS	$100*(231C26)/(231C26+231F27+231G28+C29TA3)$
%27 TAS	$100*(231F27)/(231C26+231F27+231G28+C29TA3)$
%28 TAS	$100*(231G28)/(231C26+231F27+231G28+C29TA3)$
%29 TAS	$100*(C29TA3)/(231C26+231F27+231G28+C29TA3)$
C28/C26 20S TAS	$(231E28)/(231C26)$
C28/C27 20R TAS	$(231G28)/(231F27)$
Dia/Regular C27 MAS	$(S253D)/(S253C)$
%27 MAS	$100*(S253C+S253D+S253E+S253F+S253H)/(S253C+S253D+S253E+S253F+S253G+S253H+S253I+S253J+S253K+S253L+S253M+S253N+S253O)$
%28 MAS	$100*(S253G+S253I+S253J+S253M)/(S253C+S253D+S253E+S253F+S253G+S253H+S253I+S253J+S253K+S253L+S253M+S253N+S253O)$
%29 MAS	$100*(S253K+S253L+S253N+S253O)/(S253C+S253D+S253E+S253F+S253G+S253H+S253I+S253J+S253K+S253L+S253M+S253N+S253O)$
(C21+C22)/S MAS	$(S253A+S253B)/(S253A+S253B+S253C+S253D+S253E+S253F+S253G+S253H+S253I+S253J+S253K+S253L+S253M+S253N+S253O)$
TAS/(MAS+TAS)	$(231A20+231B21+231C26+231D26+231E28+231F27+231G28+C29TA3)/((231A20+231B21+231C26+231D26+231E28+231F27+231G28+C29TA3)+(S253A+S253B+S253C+S253D+S253E+S253F+S253G+S253H+S253I+S253J+S253K+S253L+S253M+S253N+S253O))$
TA28/(TA28+MA29)	$(231E28+231G28)/(231E28+231G28+S253K+S253L+S253N+S253O)$
Triaromatic Methylsteroids	
Dinosteroid Index	$(DA+DB+DC+DD+DE+DF)/(C3S+C4S+E2S+E3SC3R+E4SC4R+S2S+DA+S3S+DB+S4SE2R+E3R+E4R+DC+DD+S2R+S3R+DE+S4R+DF)$
C4/C3+C4 Mester	$(C4S+E4R+S4R)/(C3S+C4S+E3R+E4R+S3R+S4R)$
Phenanthrenes, Naphthalenes, and Dibenzothiophenes	
MPI-1	$(1.5*(3MP+2MP))/(PHEN+9MP+1MP)$
Rc(a) if Ro < 1.3 (Ro%)	$((.6)*(1.5*(2MP+3MP))/(PHEN+9MP+1MP))+.37)$
Rc(b) if Ro > 1.3 (Ro%)	$((.6)*(1.5*(2MP+3MP))/(PHEN+9MP+1MP))+2.3)$
Rm (Ro%)	$(0.40+0.30*(4MDBT/1MDBT)-0.094*(4MDBT/1MDBT)*(4MDBT/1MDBT)+0.011*(4MDBT/1MDBT)*(4MDBT/1MDBT)*(4MDBT/1MDBT))$
MDR1	$(1MDBT)/(DBT)$
DBT/Phenanthrene	$(DBT)/(PHEN)$

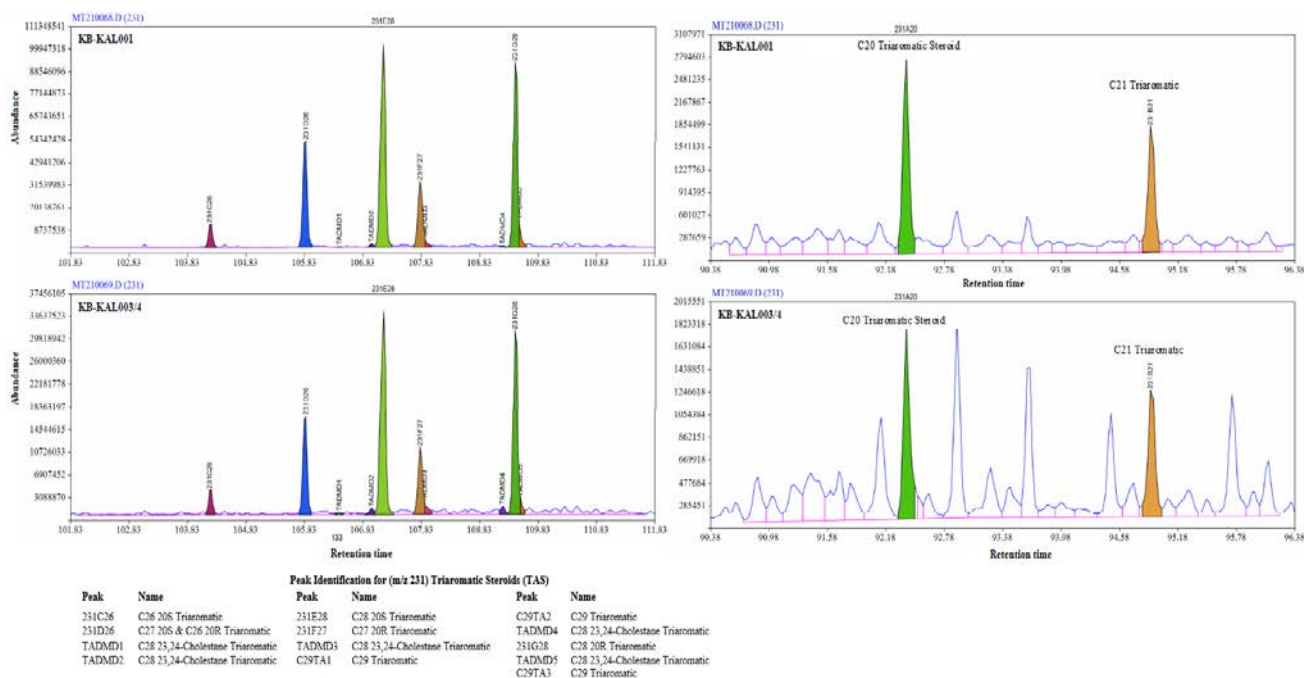
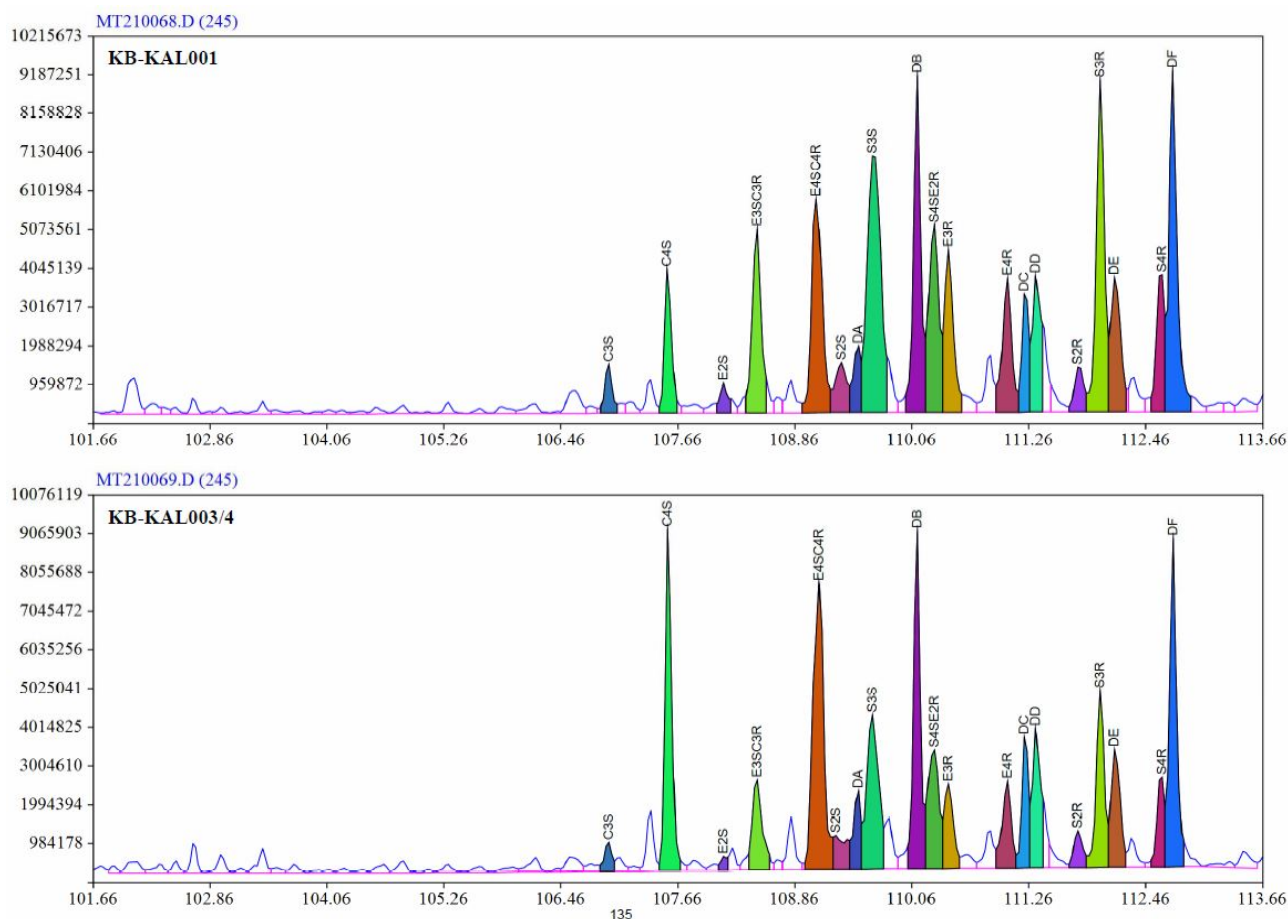


Figure 6b. Mass chromatograms m / z 231 show the distribution of triaromatic steranes by GC-MS analysis of the EOM from the samples KB-KAL001 and KB-KAL003/4



**Figure 6c.** Mass chromatograms  $m/z$  245, showing the distribution of Triaromatic Methylsteroids obtained by GC-MS analysis of the EOM from the samples KB-KAL001 and KB-KAL003/4

Mass chromatograms of the monoaromatic ( $m/z$  253) triaromatic ( $m/z$  231) and triaromatic methyl steroids ( $m/z$  245) in the aromatic hydrocarbon fractions of representative samples are shown in Figure 6a-c, and their respective molecular ratios calculated using the peak areas are summarized in Table 4. The definitions and measurement procedures for the saturated fractions are summarized in Table 6.

## 5. Discussion

The bulk composition of the two extract samples KB-KAL001 and KB-KAL003/4 indicate that the samples are dominated by saturates with moderate amounts of aromatics and high content (Table 2). Such compositions, according to [8] classification (Figure 7), suggest that the analyzed two extracts generally contain normal paraffinic generated hydrocarbons.

### 5.1. Proxies Related to Paleoredox and Salinity Conditions

Several biomarker proxies have been used to interpret redox conditions: homohopane index, Pr/Ph, and gammacerane ratio. Pr/Ph ratio of source extract has been widely used as an indicator of depositional conditions/paleoredox indicator [40] and the origin of organic matter

[41]. Pr/Ph ratios of  $< 0.8$  commonly indicate hypersaline and anoxic conditions, ratios of  $> 3$  indicate terrigenous organic matter input under oxic conditions, and ratios from 0.8 to 3 indicate suboxic to oxic conditions [6,42].

The Pr/n-C17 and Ph/n-C18 also provide valuable information on organic source input. For this study, moderate Pr/Ph (1.54-2.85), high Pr/n-C17 and Ph/n-C18 ratios (1.71-3.41 and 0.89-1.29) suggest a significant contribution of terrigenous organic matter with a small amount of aquatic organic matter input that was preserved under suboxic to relatively oxic conditions thermal maturity from a mixed source deposited under oxic to sub-oxic depositional conditions [42]. These ratios were plotted in a graphical form to infer source rock extract depositional environments [12], Figure 8.

Salinity conditions that prevailed during source rock deposition were evaluated using the gammacerane/hopane ratio [43]. C30 triterpane gammacerane and  $\beta$ -carotane are very specific for highly saline and stressed lake environments [11,44]. They are also usually associated with arid or semiarid lacustrine settings where the OM is primarily derived from algae and bacteria [45,46]. Gammacerane has also been linked to water-column stratification [47]. The gammacerane/C30 hopane ratios of the extract samples range from 0.26-0.27, which may reflect somewhat restricted circulation and moderate/low salinity of the depositional environment of the source rocks.

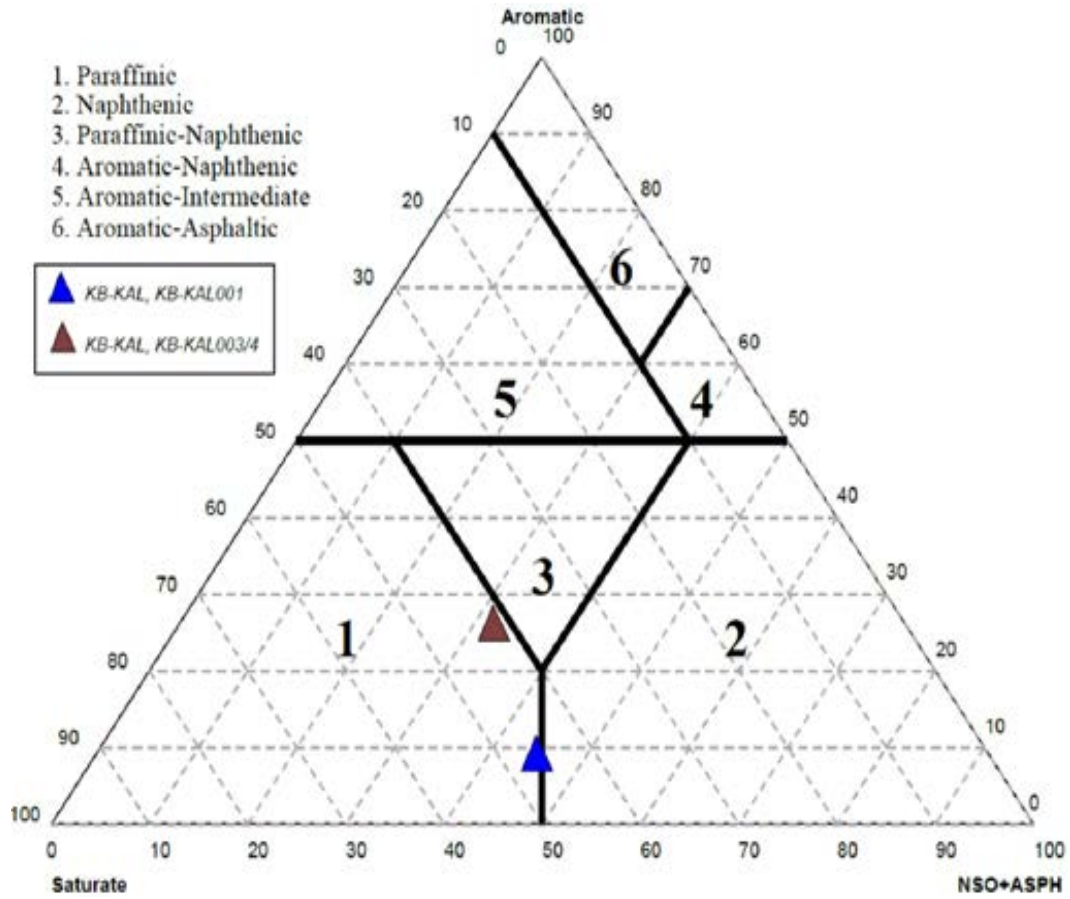


Figure 7. Bulk composition ternary diagram (Modified after [8])

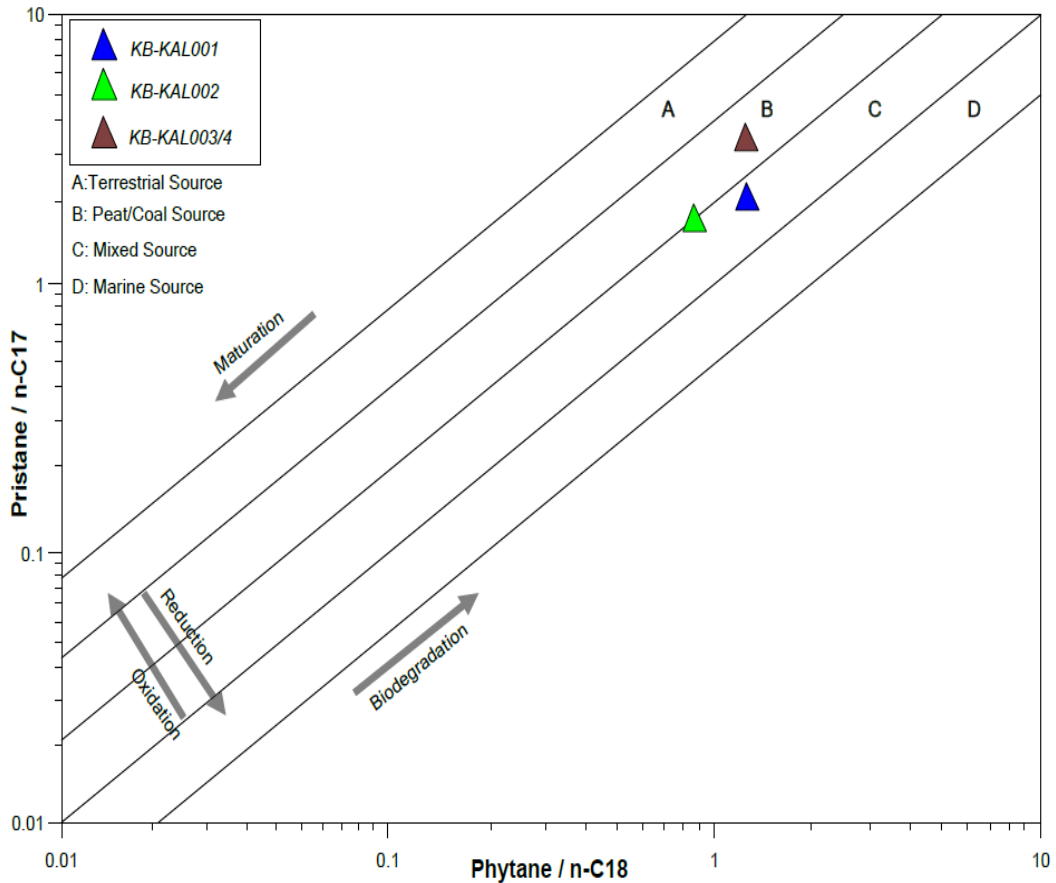


Figure 8. Crossplot of Pr/n-C17 versus Ph/n-C18 for extracts



## 5.2. Source and Depositional Environment-Related Proxies

The OM input of source rocks is fundamental to its quality, hydrocarbon potential, and generation kinetics. The distribution of n-alkanes is an indication of the source input of the precursor(s). For example, [7,42,48] reported that algal-sourced organic matter is rich in short-chain n-alkanes < C20, and land plant-derived organic matter is dominated by long-chain n-alkanes > C27 with odd-over-even carbon number predominance. In the studied Koum samples, the unimodal pattern of n-alkanes, the relatively high concentration of the high-molecular-weight n-alkanes (wax components) indicates input from algal/land plant waxes, characterized by a strong odd over even carbon number predominance, most clearly indicated between n-C23 and n-C35, supporting the derivation from lacustrine input of algal biomass with/or perhaps terrestrial organic matter (peat/coal source). The CPI value according to [5] is a numerical mean for representing the odd-over-even carbon predominance, in a particular range of n-alkane compounds. CPI values greater than 1.0 indicate land plant input, while CPI values less than 1.0 are a characteristic of algae and bacterial input and thereby, provide some insights into the source input and depositional conditions of the OM. The CPI values for the analyzed extracts range between 1.35 and 1.40 (average 1.38), (Table 1). This indicates a mixed terrigenous /aquatic input with relatively substantial contribution by terrestrial OM as also suggested by the OEP vary between 1.31 and 1.44 [6] Waxiness index may be used to determine the amount of land-derived organic materials in sediments relative to the aquatic/marine materials in the sediments. This ratio is utilized based on the assumption that the

presence of terrigenous materials will increase the contribution of high molecular weight components [6] as is the case of the analyzed samples (Table 1). This is corroborated by the terrigenous/aquatic ratios (TAR) which is a commonly used parameter to demonstrate the ratio of terrestrial origins (n-C27, C29, and C31) to aquatic sources (n-C15, C17, and C19). High molecular weight hydrocarbons n- C27 to n-C31 present in high abundance as indicated by terrigenous/aquatic ratio TAR being greater than one (1,0 to 2,94) suggests the relative contribution of land plant source organic input. It can be used as a qualitative proxy for the relative contributions of OM from both terrestrial areas and aquatic sources.

The ratio of dibenzothiophene (DBT) to phenanthrene (P) is used as an excellent indicator to infer source rock lithology and depositional environment [49]. Pristane/phytane (Pr/Ph) data for extracts/oils allow fluvio-deltaic source rocks to be distinguished from marine and lacustrine shales. Therefore, the cross-plot of these parameters in Figure 9 allows the depositional environment of an oil source rock to be deduced [12].

Carbonate source rocks yield Pr/Ph ratios < 1 and DBT/P > 1, while shale source rocks have Pr/Ph ratios of 1-3 and DBT/P < 1. Fluvio-deltaic source rocks have Pr/Ph ratios of >3 and DBT/P < 0.5. The analyzed extract samples have low DBT/P ratios (<1) and Pr/Ph ratios greater than 1.0 and less than 3.0. Therefore, the plot in the marine and/or lacustrine region of the graph. The distribution of C27-29 steranes, shown in Figures 10 and 11, is sensitive to source organic matter type (i.e., algal vs. higher plant organic matter in the source rock). The C27-29 steranes distribution suggests that the precursor of these extracts is mainly Deltaic-terrigenous (Figure 10) [15].

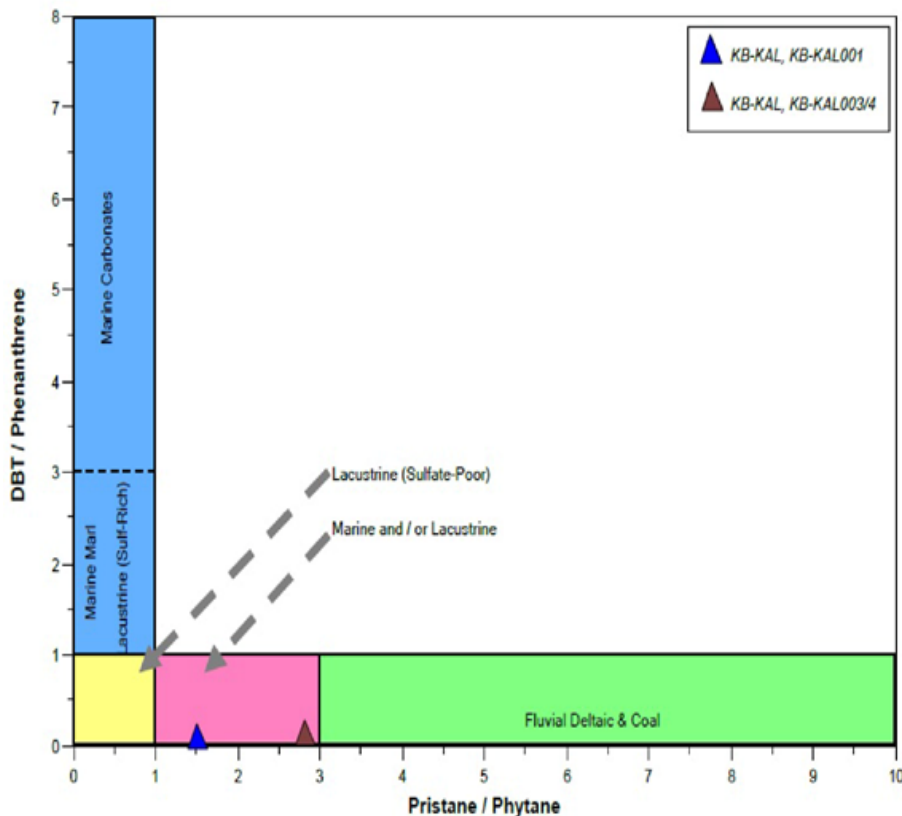


Figure 9. Pristane/Phytane vs. DBT/Phenanthrene cross-plot (Modified after [12])

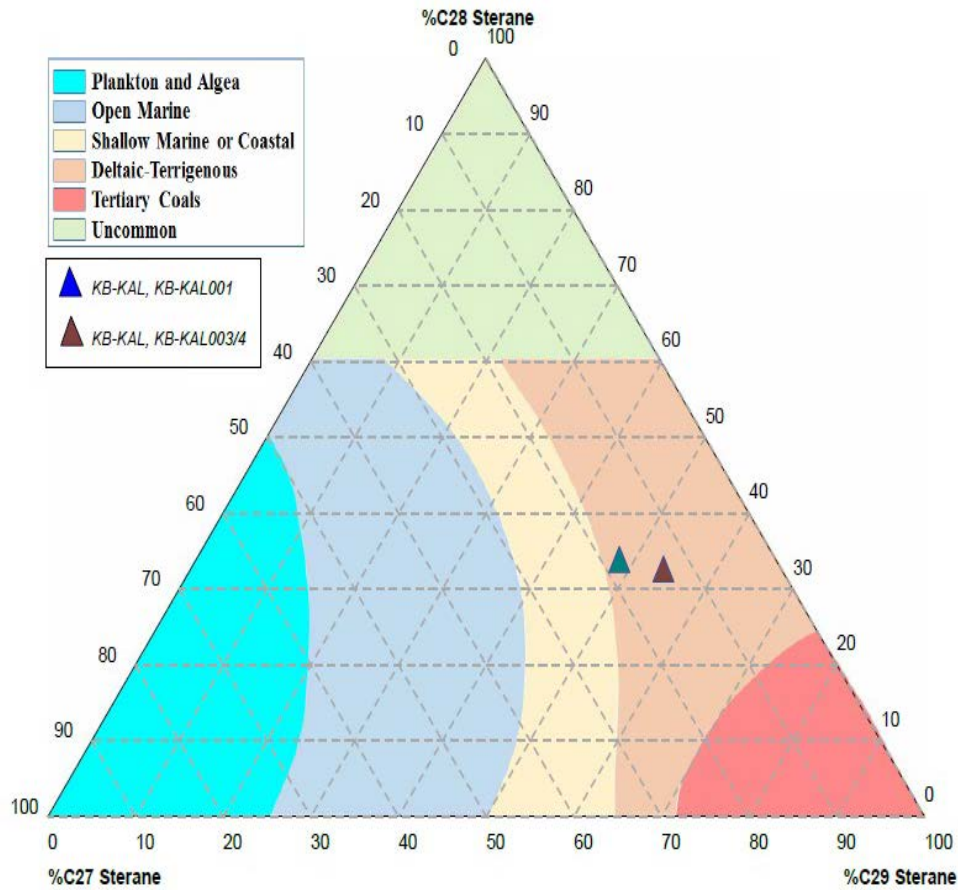


Figure 10. C27-C29 sterane ternary diagram (Modified after [15])

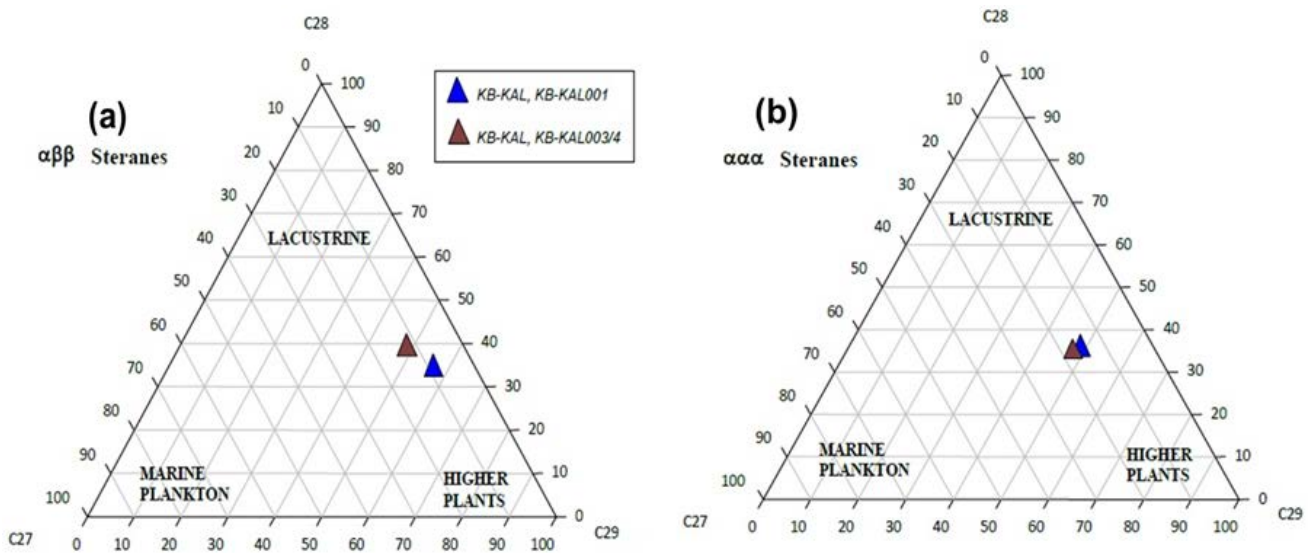


Figure 11. C27-C29  $\alpha\alpha$  and  $\alpha\beta$  sterane ternary diagrams [11]

The steranes (C27, C28, C29) distribution can be used as indicators of depositional environments [43]. The C27-29 (the 20S)  $\alpha\alpha$  and  $\alpha\beta$  steranes distributions further support that the precursor of these extracts is dominated by terrestrial organic matter as illustrated by a relatively high abundance of C29% over C28 and C27 sterane suggests a relatively high contribution from land plant to OM input for rock extracts (Figure 11) [11]. C31 22R homohopane/C30 hopane ratio has been proposed as an indicator for distinguishing lacustrine and marine depositional environments, and sediments having the ratio

less than 0.25 are interpreted as lacustrine deposits. The C 3122R/C 30 ratios of the Koum Basin samples are generally low (0.17 and 0.18). Moreover, the absence of C30 steranes suggests no or minimal marine input to the source rock [11].

Figure 12 is a ternary plot of C27-29 monoaromatic steroids (MAS). In EOM, the distribution of C27-29 monoaromatic steroids is sensitive to source organic matter type (i.e., algal vs. higher plant organic matter in the source rock). In this figure, the distributions for the extracts are generally related and show the highest value

for the C29 MAS and further support that these extracts are dominated by terrestrial organic matter [11]. This is generally consistent with the previous steranes ternary diagrams. Figure 13 is a cross plot of two biomarker ratios, C22/C21 and C24/C23 tricyclic terpanes. Both are indicators of source rock lithology. High C24/C23 tricyclic ratio and low C22/C21 tricyclic ratio suggest

more shale input to the source rock. Generally, the studied samples plot close to the Shale/Lacustrine regions. The presence of high amounts of C30TP1 and C30TP2 compounds indicates lacustrine input to be the likely source rock(s). The lacustrine input is further supported by the very high C26/C25 Tricyclic Terpanes ratios of 2.14-2.49.

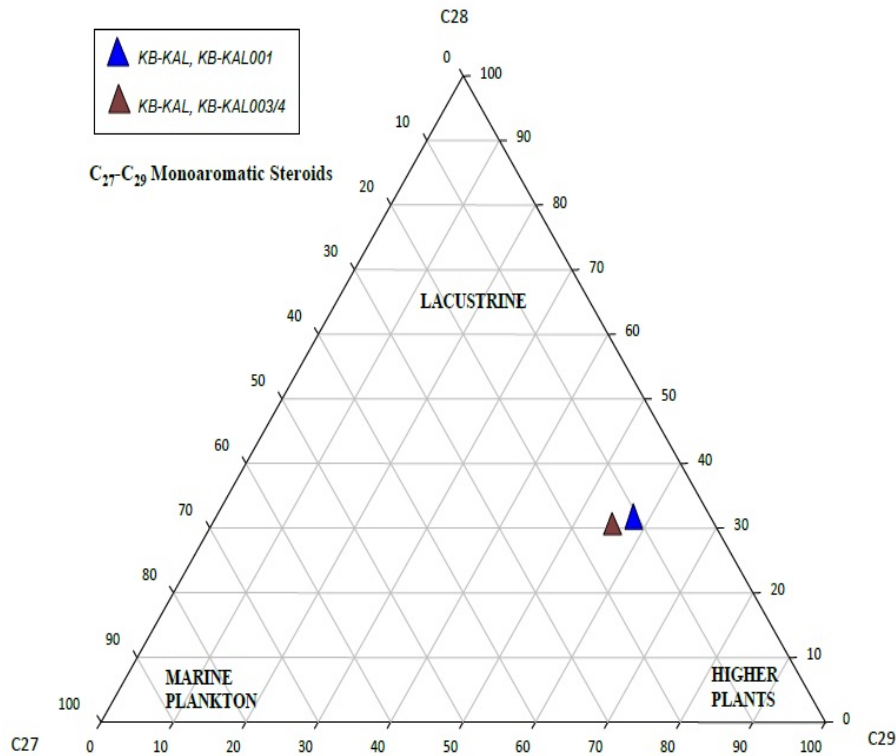


Figure 12. C27-C29 monoaromatic steroids (MAS) ternary diagram

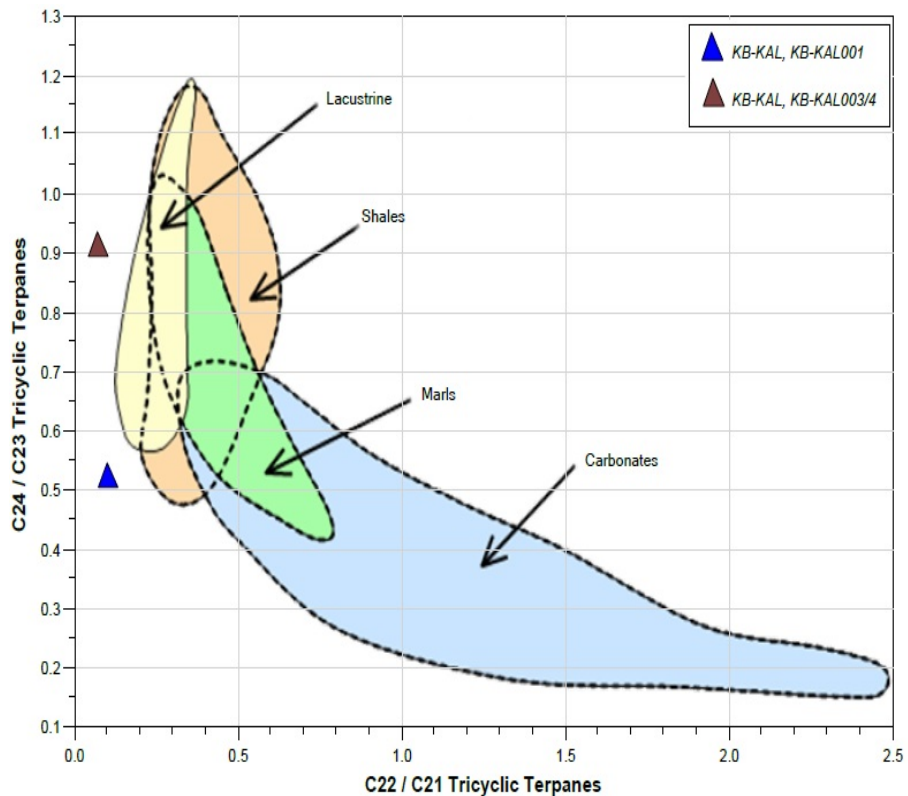


Figure 13. C22/C21 Tricyclic Terpanes vs. C24/C23 Tricyclic Terpanes cross-plot

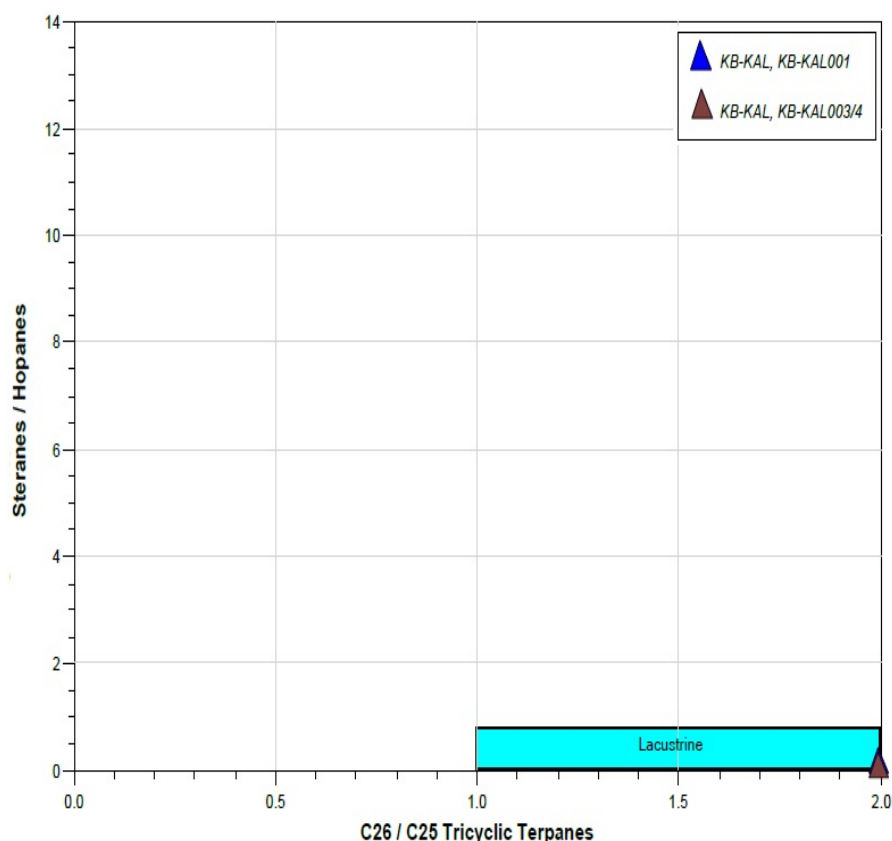


Figure 14. C26/C25 Tricyclic Terpanes versus Steranes/Hopanes ratio cross plot [50]

Figure 14 is a cross plot of C26/C25 tricyclic terpanes versus the Steranes/Hopanes ratios. Many lacustrine sourced oils have C26/C25 tricyclic terpane ratios  $> 1.0$  [50]. The extract samples have C26/C25 tricyclic terpane ratios  $> 1.0$  and Steranes/Hopanes  $< 1.0$ , suggesting dominant lacustrine components within clastic-rich source rocks. Acritarchs palynomorphs are also present in the Koum Basin Formation but their signature does not figure prominently in the n-alkane profile, probably because of their low abundance. In this case, the marine influence could not be confirmed by the molecular evidence since no GC-MS trace was obtained for any of the samples. However, the signal of contribution marine signature by the presence of dinosteroid suggests a brief marine episode. The calculated ratios from Steranes (m/z 217; 218), Terpanes (m/z 191), Mono- (m/z 253) and Triaromatic Steroids (m/z 231) are summarized in Table 3 and Table 4.

### 5.3. Maturity-Related Biomarkers Parameters

These are ratios of certain saturated and aromatic biomarker compounds that result from either cracking source rocks (including aromatizations) or isomerizations at certain asymmetric carbon atoms (Table 3 and Table 4).

The ratio of odd to even carbon numbered n-alkanes can be used to estimate the sediments' organic maturation level [6,11]. These measurements include the CPI; using the formula proposed by [11], CPI values  $> 1$  or  $< 1$  indicate thermal immaturity, while the OM that is thermally matured has values of CPI = 1 Table 1. As a result, the CPI for the analyzed samples ranging between

1.35 and 1.40 (average 1.38), suggests that the samples under investigation are thermally immature.

Application of biomarkers in types of OM and environmental condition requires consideration of the level of maturity, which has a potential influence on biomarkers [6] Maturity indicators such as Ts/(Ts+Tm) trisnorhopanes, C29 20S/(20S+20R) versus C29  $\alpha\beta\beta/(\alpha\alpha\alpha+\alpha\beta\beta)$  steranes, Methyl Phenanthrene Index (MPI-1) and C20+C21 TAS/Total TAS).

Figure 15 is a cross plot of the sterane maturity parameters, C29 20S/(20S+20R) versus C29  $\alpha\beta\beta/(\alpha\alpha\alpha+\alpha\beta\beta)$  steranes. These two maturity parameters are known to reach terminal values in the peak oil window and then decrease at very high maturity [11].

The two extracts are of early maturity based on both the C29  $\alpha\beta\beta/(\alpha\alpha\alpha+\alpha\beta\beta)$  steranes and the C29 20S/(20S+20R) steranes. [51] mentioned that it can be used MPI as vitrinite reflectance (VRo) to assess the maturity of source rocks. However, several factors affected MPI ratios, such as lithologic variation and OM type in source rocks. Figure 16 is a cross plot of two aromatic steroid maturity parameters, the Methyl Phenanthrene Index (MPI-1) and C20+C21 TAS/Total TAS, and further supports that the extracts are of early thermal maturity. The MPI-1 values of 0.16-0.35 correspond to vitrinite reflectance value equivalent of 0.46-0.58% and suggest that the two extracts were generated from early mature source rocks [51]. Furthermore, the C32 Homohopane 22S/(22S+22R) isomerization ratios (0.51- 0.54) are below the equilibrium value of 0.60, suggesting a maturity. of lower than 0.60% Ro [52] In addition, the Ts/(Ts+Tm) trisnorhopanes values of 0.29-0.35 are low and further support that the extracts were generated at early thermal maturity [11].



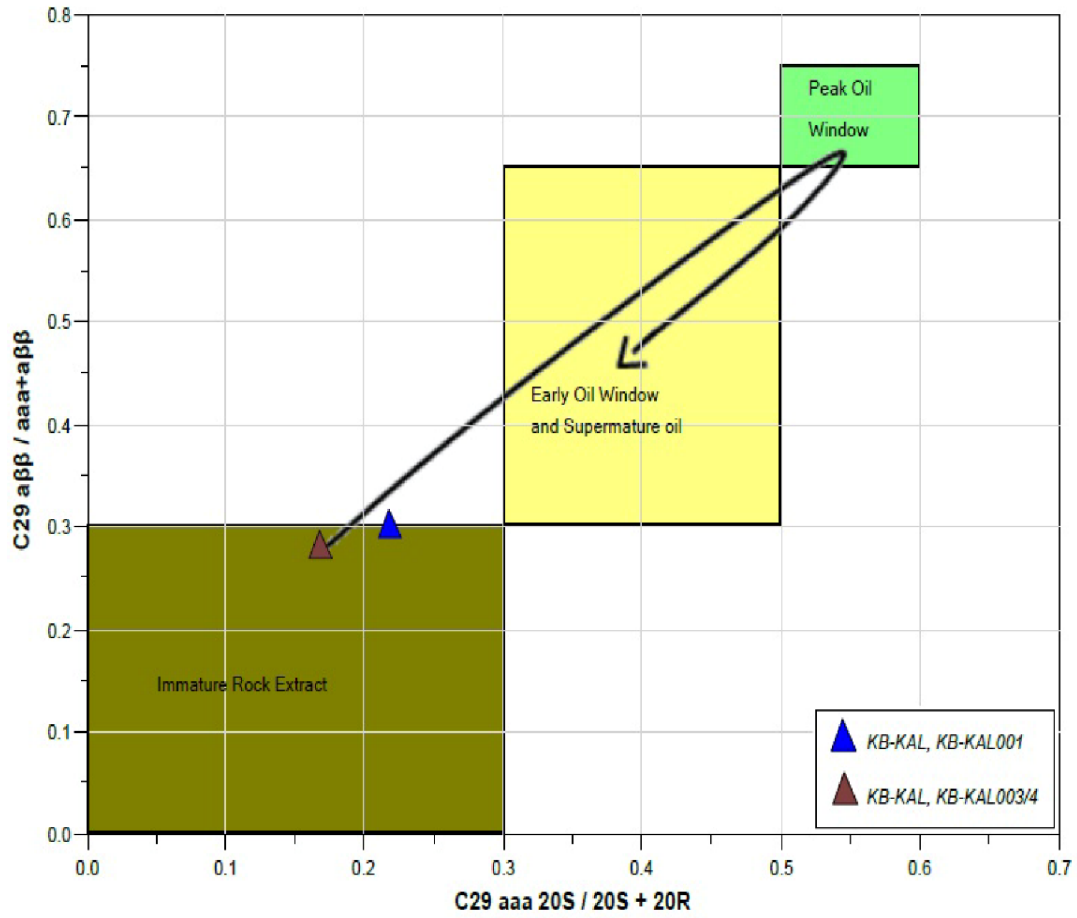


Figure 15. Sterane maturity parameters,  $C_{29} \alpha\alpha\alpha 20S / (20S + 20R)$  and  $C_{29} \alpha\beta\beta / (\alpha\alpha\alpha + \alpha\beta\beta)$  cross-plot [11]

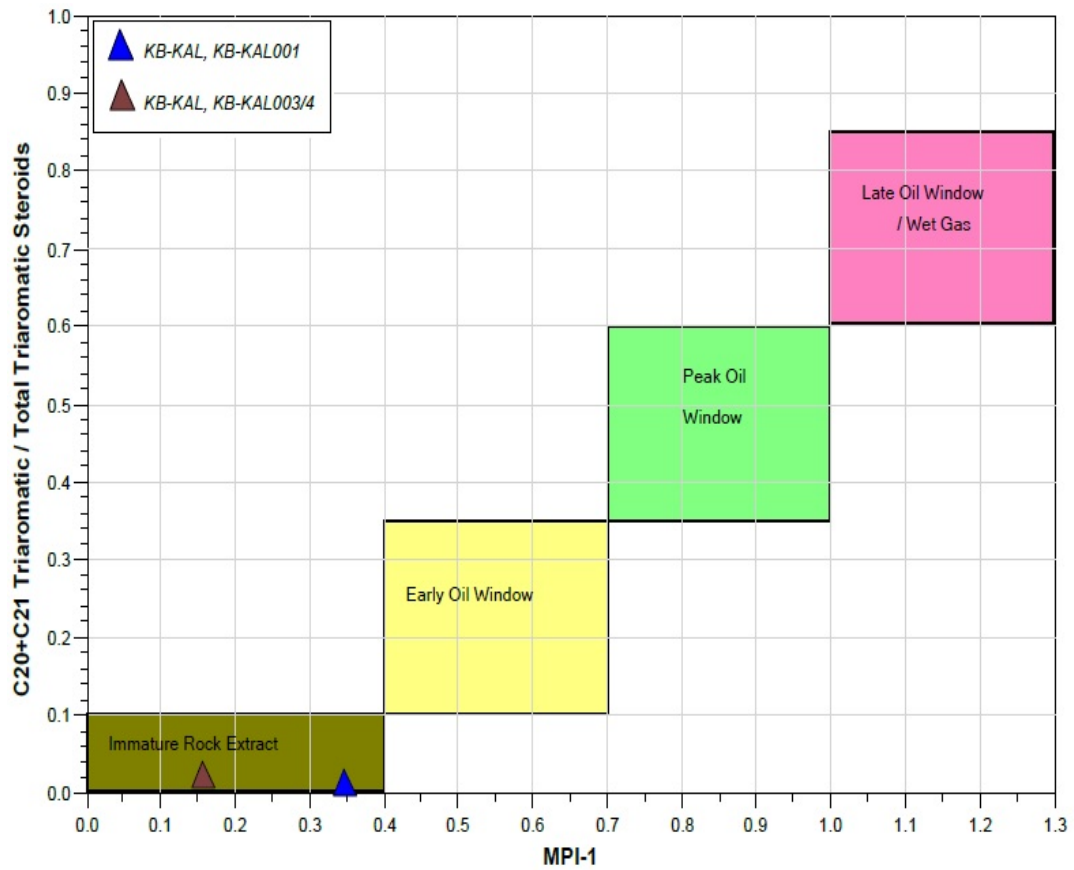


Figure 16. Plot of Aromatic maturity parameters, MPI vs.  $C_{20} + C_{21}$  triaromatic steroids (Modified after [49])

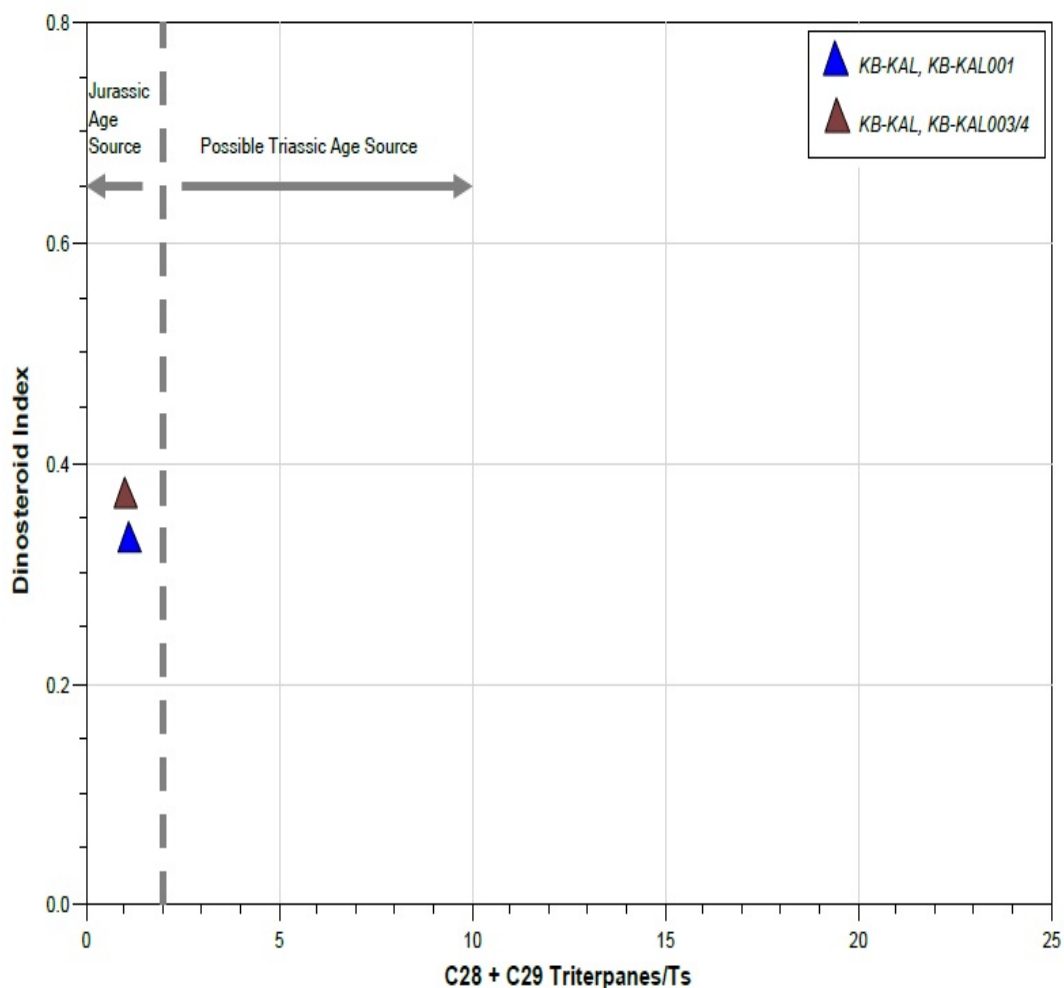


Figure 17. A plot of (C28+C29 Tricyclics)/TS vs. Dinosteroid Index. Both parameters are age-diagnostic biomarkers [53,54]

#### 5.4. Source Age Determinations from Biomarkers

There are a variety of GC-MS biomarker parameters that can be measured on oil to constrain the age of the source rock that generated the oil. The calculated ratios Triaromatic Methylsteroids are summarized in Table 4. Each of the various age-diagnostic biomarker parameters has utility within certain portions of the geologic record. For example, if an oil contains oleanane, it must have received input from source rock(s) of the Late Cretaceous age or younger [24]. The dinosteroids indicate input from dinoflagellates, which (most of the time) indicate post-Palaeozoic origin. The proportion of C4-methylsteranes versus C3-methylsteranes tends to be higher in younger samples compared to Palaeozoic samples [53].

The C28+C29 Tricyclics/Ts ratio, when  $>2.0$ , indicates Triassic input, while when  $<2.0$ , suggests a Jurassic source [52]. The dinosteroids (0.33-0.37) and methylsteranes (0.42-0.60) values suggest that the extracts are derived from a Mesozoic age or younger source rock(s). Moreover, the absence of oleanane is highly suggestive of generation from pre-Cretaceous age source(s) or a Cretaceous or younger source deposited where oleanane input was absent. The C28+C29 Tricyclics/Ts ratio indicates that the source(s) of extracts is of Jurassic or younger age

(Figure 17). Given that we are in Koum Basin, which is dominated by Cretaceous sediments it is unlikely that the source rock is older than Mesozoic and is most likely in the Early Cretaceous section.

#### 6. Petroleum System Implication

Based on the molecular fossil characteristics, the EOM from this study can be correlated with the identified main source rocks from the WCARS: early Cretaceous of Bima Formation of the Yola Basin, Benue [55,56] Bongor Basin [57], and Mamfe Basin [19] in terms of input of aquatic and terrestrial organic facies, the predominance of paraffin waxy hydrocarbon type and the age-related biomarkers. However, the maturity evolution of the source rock extracts is influenced by different local factors, amongst which the probable uplift of this source rock units above the hydrocarbon generation window is due to inversion. While most of the WCARS basins have good data and seismic control, allowing a good understanding of their basin development, history, and petroleum system, the Koum Basin remains largely unexplored. It is evident that mega lacustrine systems developed during the early Cretaceous within the WCARS basins with locally prevailing paleoclimates and vegetation cover.

## 7. Conclusion

Biomarker fingerprints proved to be a powerful tool for assessing and predicting source rock attributes within the Koum Basin and they represent a clear manifestation of the relationship between tectonics and sediment supply in a typical continental rift setting. The integration of all the source- age and maturity-related biomarker parameters show that the extracts belong to one main oil family, which can be characterized as generation from an aquatic-derived with significant terrigenous OM contribution, deposited under sub-oxic to oxic fluvial-deltaic-lacustrine depositional environment with tidal influence. The absence of oleanane and low C28+C29 Tricyclics/Ts ratios, together with the dinosteroids and Methyl Triaromatic Steroid distributions, suggest that the candidate source rock for these extracts is of Cretaceous or older age. The extracts from the Koum Basin are generated at early thermal maturity (maturity close to ~0.60% Ro<sub>eq</sub>). Finally, with active petroleum systems in the Benue trough and active lacustrine source in the Bongor and Doba petroleum systems, deeper unexposed sections of the Koum Basin may contain pods of mature source rock. However, the confident correlation of the results from this study with adjacent basins is severely hampered by the paucity of well-constrained stratigraphy available. The results add a special direction and the data now available may be regarded as valuable. With the world's aspirations being aligned with global energy needs, it becomes important to find answers on how future hydrocarbon supply can keep pace with the increasing demand. Significantly, the study provides information necessary to guide future exploration ventures and further investigate hydrocarbon prospectivity in Northern Cameroon, a region with crucial economic and strategic importance. Therefore, the Koum Basin, in general, contains organic facies classified as poor to very good potential source rock, with some limitations in maturity level.

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