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## Oil-oil and oil-source correlation by application of Compound Specific Isotopic Analysis (CSIA): A frontier tool applied to Kali-Kuthalam area in Cauvery Basin.

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### Summary

The compound specific stable carbon isotopic characterization (CSIA) is a state-of-the-art geochemical technique to refine oil-oil and oil-source correlations. Using CSIA, the isotopic values of individual n-alkanes have been measured in the range of C<sub>17</sub> to C<sub>30</sub> for oil/condensates and also in bitumen of probable source sequences in Andimadam Formation in Kali-Kuthalam area, Cauvery Basin. Further the isotopic profiles of these n-alkanes in combination with the biomarker parameters have been applied to study organic matter input, depositional environment, and maturity of the source organics.

CSIA profiles of most of the studied oils/condensates shows similar trend with small differences in their n-alkane carbon isotopic values. Most of the studied source sequences show poor correlation with studied oils/condensates due to the differences in their thermal maturities and organic facies variations. Some source sequences are correlatable but many oils exhibit higher maturity compared with these source sequences. Thus some deeper sediment can be major source.

**Keywords:** Compound specific isotopic analysis (CSIA)

### Introduction

Cauvery Basin, an intracratonic basin is situated along the southeastern part of Indian Peninsula. Commercial hydrocarbon accumulations have been confirmed in the Cretaceous and Tertiary sediments. Hydrocarbons have been discovered in almost all the sub-basins of Cauvery Basin. Kali-Kuthalam area, located along the north western rising flank of Tranquebar sub-basin, is one of the thrust area in Cauvery Basin put on fast track exploration. Commercial amounts of oil/gas/condensates have been discovered in multiple stacked sandstone reservoirs in Bhuvanagiri and Andimadam formations. Most of the hydrocarbon finds in this area are gas/condensates. So far fifteen pay sands are identified in Kuthalam area of which nine is gas bearing. The extensive source rock evaluation of all the sub-basins of Cauvery Basin has established that the Andimadam Formation of Lower Cretaceous sediments are the major source rocks in Cauvery Basin and organic matter is confined to two stratigraphic units, namely, Lower Cretaceous and Early Oligocene to Late Eocene (Banerjee et al., 2003 and Vyas et al., 2001). Of

these, only the Lower Cretaceous sediments are matured enough to generate and expel hydrocarbons. The entire sedimentary sequences are recipient of good quantity of organic matter (upto 12% TOC). The hydrogen Indices are fair to very good in the depressional part (up to 300 mg HC/gm) of TOC. The organic matter is an admixture of Type-II & III, capable of generating both gaseous and liquid hydrocarbons (VR<sub>o</sub> 0.55-0.60, T<sub>max</sub> 435-440°C) (Vyas et al., 2001). The Geochemical data from deep wells indicated that the syn-rift Andimadam Formation is possibly the main source rock. This formation has been penetrated mainly in the flanks of depocentres where it was found to be dominantly sandstone with subordinate shales. It may become more shaly towards the sub-basin depocentres.

The objective of the study is to evaluate oils covering all pay sands and probable source rocks in Kali-Kuthalam area through Compound Specific Isotopic analysis (CSIA) in combination with biomarkers to infer specific source organics and depositional environment.

The oils/condensates from twenty two wells are selected in Kali-Kuthalam area from Andimadam Formation of Lower Cretaceous age in Cauvery Basin. Source rock samples from eight wells of Andimadam Formation are also taken for oil-source correlation studies.

### Geological setting and stratigraphy

The Cauvery Basin covers an area of about 25,000 sq. km. on land in the coastal plains of Tamil Nadu and Pondicherry and about 22,500 sq. km. offshore, upto bathymetry of 200 m in Bay of Bengal. The basin has evolved during L Jurassic-E Cretaceous period and is characterized by a horst-graben tectonic configuration. These tectonic elements are aligned in a NE-SW trend, parallel to the Pre-Cambrian Eastern Ghats. These grabens (sub-basins) are designated as Ariyalur-Pondicherry, Tranquebar, Nagapattinam, Tanjore and Ramnad-Palk bay from north to south. The horsts adjoining these sub-basins are likewise named as the Kumbakonam-Madanam, Karaikal, Vedaranniyam, Pattukottai and the Mandapam-Delft horsts. During Mesozoic era, sedimentation was mainly confined to the paleo lows. The detailed stratigraphy of Cauvery Basin has been discussed by Venkatarengan et, al., (1993). The prospect map of Cauvery Basin showing study area is shown in Fig.1 and generalized stratigraphy of Cauvery Basin is shown in Fig. 2.

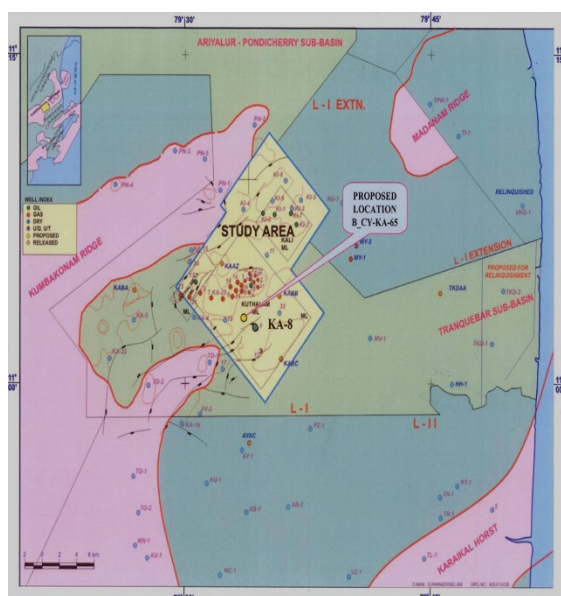


Figure 1

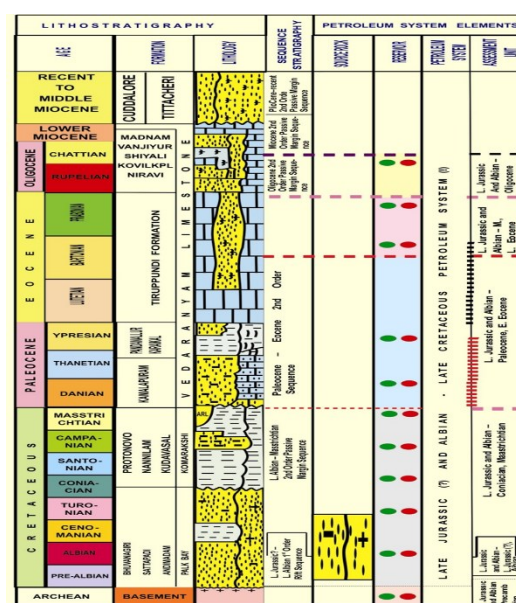


Figure 2

### Experimental methods

The bulk analysis API, Pour Point, asphaltene and wax content were carried out using standard ASTM procedures. Vanadium and Nickel concentration in oils/condensates were determined in ICP-Spectrometer in organic phase. The sulphur content was analyzed on Analytic Jena multi EA 3000 total sulphur & nitrogen analyzer. The powdered sediment samples were Soxhleted with dichloromethane and methanol (93:7) for 8 hrs. to obtain bitumen (EOM). The EOM and deasphalted oil samples were fractionated into saturate and aromatic fractions using activated silica gel and alumina dual column by successive elutions with petroleum ether and benzene respectively. The eluted solvent thus contain saturates and aromatic fractions respectively. The solvents are removed by evaporation and fractions subjected to gas chromatographic, carbon isotopic and biomarker analysis. NSOs were determined by difference. The saturate fractions were analyzed for normal and isoprenoid alkanes distribution on GC Varian CP-3800 using fused silica WCOT 5 CB column (60 m x 0.32 mm id x 0.25 µm) with oven temperature programming from 80 to 300 °C @ 3°C per min with hold time of 30 min. Nitrogen was used as the carrier gas. The branched / cycloalkane fractions of oil isolated from saturate fraction by urea adduction were analyzed on Perkin Elmer Clarus 500 system using fused silica capillary column MS-1 (30m x 0.25 mm x 0.25 µm). Helium was used as carrier gas. Column temperature was raised from 80 to 300°C @ 3°/min with final hold time of 20 minutes. Detector temperature was kept at 310°C. The



spectra were recorded in total scan mode in mass range of 50-550 amu. The oils were analysed for hopane and sterane distribution.

The aromatic fractions of oils were analyzed for the distribution of phenanthrene, methylated isomers of phenanthrene and dibenzothiophenes on Perkin Elmer Clarus 500 system using fused silica capillary column MS-5 (30m x 0.25 mm x 0.25  $\mu$ m). Helium was used as carrier gas. Initial oven temperature was kept at 80°C for 1 min. and increased @ 2.5° C/min. upto 260°C and @ 3.5°C/min. upto 300°C with final hold time of 20 minutes. Detector temperature was kept at 300°C. Phenanthrene (P) and methyl phenanthrene (MP) were identified from retention indices. MPI was calculated as follows

$$\text{MPI 1} = 1.5x(2\text{MP}) + (3\text{MP}) / \text{P} + (1\text{MP}) + (9\text{MP})$$
$$\text{VRc} = 0.4 + (0.6x \text{MPI 1}) \quad \text{Radke et al. (1983)}$$

CSIA studies were carried out on VG IsoPrime Continuous Flow-Isotope Ratio Mass Spectrometer interfaced with Agilent 6890N GC, equipped with a high resolution GC column, HP-5 (30m x 0.32mm x 0.25  $\mu$ m). 2  $\mu$ l sample of saturate fraction diluted with HPLC grade n-hexane (1:4) was introduced into the GC through Agilent auto sampler. The effluents from the GC were passed through a cupric oxide combustion oven maintained at 850°C using helium as a carrier gas. The water vapors were removed through an online nafion membrane and finally the purified CO<sub>2</sub> introduced into the source of mass spectrometer. A CO<sub>2</sub> gas was employed as reference to calibrate the isotope ratios of samples measured. The mass spectrometer is calibrated using laboratory Indiana University CSIA standard alkane mixtures A3 and B2.

The results are reported as  $\delta^{13}\text{C}$  ‰ with respect to PDB. The isotopic ratio is expressed in the usual delta ‰ notation as follows:

$$\delta^{13}\text{C} (\text{‰}) = \left( \frac{^{13}\text{C}/^{12}\text{C}_{\text{sample}}}{^{13}\text{C}/^{12}\text{C}_{\text{standard}}} - 1 \right) \times 1000$$

## Results and discussion

The API gravity for oils varies from 41.6 to 45.7° while in condensates it varies from 51.7 to 59.4°. The saturate/aromatic ratio (3.8 to 31.3) indicates that these oils/condensates are predominantly aliphatic in nature. These oils/condensates have very low asphaltene contents

(0.04 to 0.22%) and low to medium wax content (5.28 to 8.48%). Pour Points of oils range from 6° to 27° C whereas for condensates it is < 0° C. Sulphur concentration in oils/condensates is in the range of 6.1 to 300.2 ppm.

These oils/condensates have very low Nickel (0.499-1.138 ppm) and Vanadium concentration (0-0.500 ppm). Nickel concentration is found to be more than Vanadium in oils and the ratio V/V+Ni is from 0.17 to 0.43 which shows oxic depositional environment for studied oils/condensates (Lewan, 1984). The individual concentrations of V & Ni are high in marine organic sediments, moderate in lacustrine sediments and low in terrestrial derived sediments. A high (>0.9) ratio of V/V+Ni indicates anoxic paleoenvironment of deposition. Values ranging from 0.6 to 0.9 indicate sub-oxic environment and values below 0.6 suggest oxic environment of deposition (Abubakr et al., 2010). In Kali-Kuthalam area, these values are in the range of 0.17 to 0.43 indicating the oxic environment of deposition for these oils/condensates.

The ternary diagram showing the relative abundance of saturate, aromatic and NSO fractions indicates that these oils are normal oils (Fig. 3). Normal alkane and isoprenoids are considered as important biological markers. N-alkane distribution gives valuable information on the source input. The isoprenoid Pristane (Pr) and Phytane (Ph) are most abundant and their relative amounts show source organic input, environment of deposition (Powell and Mckirdy; Lijbach, 1975; Phillipi, 1974). Low Pristane to Phytane ratio (<1) indicates anoxic environment of deposition and predominantly marine input whereas high value (>3) indicates oxic environment of deposition and predominantly terrestrial input.

Oils have moderate Pr/Ph ratio (2.7-3.1), low Pr/nC<sub>17</sub> (0.30-0.37) and low Ph/nC<sub>18</sub> (0.10-0.15). The condensates have moderate to high Pr/Ph ratio (3.1-5.7), low to moderate Pr/nC<sub>17</sub> (0.32-0.62) and low Ph/nC<sub>18</sub> (0.10-0.14). A higher Pr/Ph ratio in condensates is attributed to migration fractionation (Dzou and Hughes, 1993). These parameters indicate predominantly terrestrial source input deposited in oxic environment with variation in organofacies (Peters and Moldowan, 2005). Ratio of Pr+nC<sub>17</sub>/Ph+nC<sub>18</sub> is quite similar in oils (1.18-1.30) indicating genetically correlatability of these oils. This ratio shows wide variation (1.26-2.51) in condensates which could be due to migration fractionation. Ratio of nC<sub>21</sub>+nC<sub>22</sub>/nC<sub>28</sub>+nC<sub>29</sub> is more than 1.5 in studied oils/condensates indicating presence of lighter end hydrocarbons. The Gas chromatograms of these oils are similar and are indicative of their correlation. The cross

plot of  $Pr/nC_{17}$  and  $Ph/nC_{18}$  ratios of studied oils/condensates also indicate that the source rock for these oils are rich in terrestrial organic matter and were deposited in oxic environment (Fig.4).

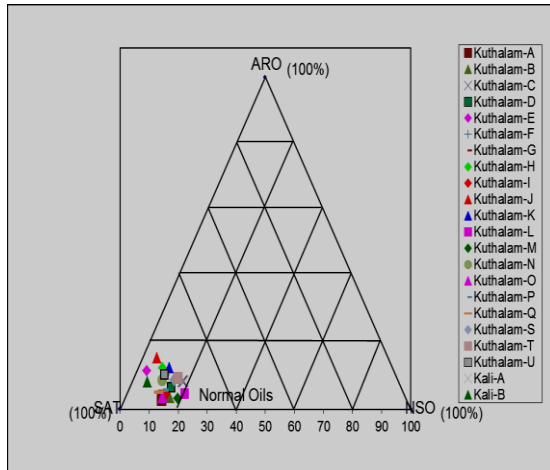


Figure.3

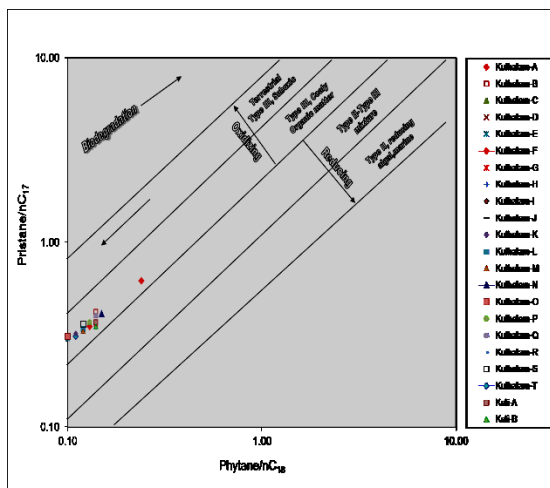


Figure.4

The  $m/z$  191 chromatograms of the oils/condensates of Kali-Kuthalam area are characterized by the presence of ubiquitous hopanes (Ourisson et al., 1984), low concentration of 28, 30-bisnorhopanes (3.5-9.8) and absence of oleanane. These oils/condensates show a predominance of  $C_{30}$  hopane over  $C_{29}$  hopane ( $C_{29}H/C_{30}H$  ratio; 0.40-0.63) suggesting clay rich source organics for studied oils/condensates which is further supported by the presence of dia steranes. Absence of oleanane in all studied samples suggests that the age of the source for oils and condensates are lower Cretaceous or older. Sterane fingerprints ( $m/z$  217) show slight dominance of

$C_{29}$ steranes over their  $C_{27}$  and  $C_{28}$  homologues. The ternary diagram (Fig.5) showing the relative abundance of  $C_{27}$ ,  $C_{28}$  and  $C_{29}$  regular steranes for these oils/condensates also suggest terrestrial source organic input (Huang, Wen-Yen and Meinschein W.G., 1979). The cross plots of  $Pr/Ph$  and  $C_{27}/C_{27}+C_{29}$  steranes ratio suggest predominantly terrestrial source input deposited in oxic conditions (Fig. 6).

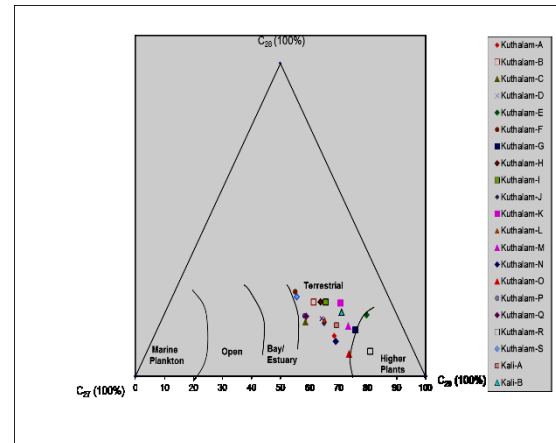


Figure.5

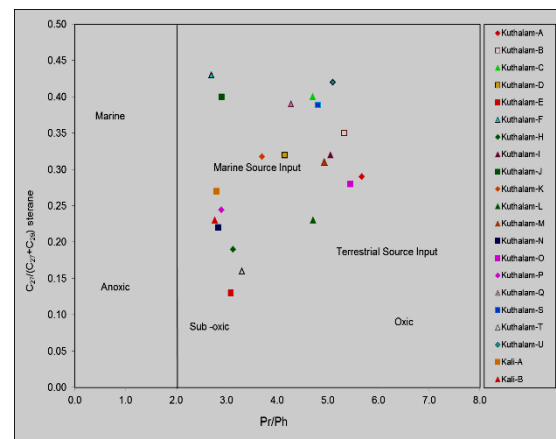


Figure.6

The maturity level of oils has been assessed from the extent of isomerisation of cycloalkane biomarkers of hopane and sterane class. The biomarker range hydrocarbons for maturity assessment are hopane isomerisation ratios  $C_{32}$  hopane, 22 S/S+R, Ts/Ts+Tm,  $C_{30}$  moretane/ $C_{30}$  hopane,  $C_{29}$  sterane S/S+R and  $C_{29}$  sterane  $\beta\beta/\beta\beta + \alpha\alpha$ . The hopane isomerisation ratios  $C_{32}$  S/S+R for most of the oils/condensates have attained or are close to equilibrium values (0.52-0.61) suggesting maturity more than 0.60 VRo (Schoell et al., 1983 and Zumbege 1987b). The  $C_{30}M/C_{30}H$  ratio in most of studies oils/condensates varies

from 0.11-0.19 which indicates moderate maturity (Seifert and Moldowan, 1980 and Mackenzie et al., 1980). The sterane isomerisation ratios  $C_{29}$  sterane S/S+R (0.33-0.57) and  $C_{29}$  sterane  $\beta\beta/\beta\beta + \alpha\alpha$  (0.18-0.53) for studied oils/condensates show low to moderate maturity (Seifert and Moldowan, 1986). A cross plot of  $C_{29}$  sterane S/S+R vs  $C_{29}$  sterane  $\beta\beta/\beta\beta + \alpha\alpha$  also shows that studied oils are low to moderately mature (Fig.7). The ratio  $Ts/Ts+Tm$  range between 0.37 to 0.57 suggests that all oils were generated from low to moderate maturity of source rock (Seifert and Moldowan, 1978). VRc values range from 0.48 to 0.83 suggesting low to moderate maturity level for the studied oils/condensates.

Compound specific stable carbon isotope analysis (CSIA) was performed on n-alkanes in the saturated hydrocarbon fractions of oils/condensates from Kali-Kuthalam area to predict the depositional environment and organic matter characteristics of their potential source rocks. A negatively sloping n-alkane profile is obtained for the studied oils/condensates having terrigenous biomarker characteristics. These oils/condensates show depletion in  $\delta^{13}C$  values for n-alkanes at  $C_{18}$  and after that it shows almost steady isotopic values with slight variations in  $\delta^{13}C$  values towards lighter values at higher n-alkanes. This negative slope fingerprint (Fig 8) has been described as characteristic of crude oils derived from deltaic and terrigenous organic matter (Bjorøy et al., 1991; Wilhelms et al., 1994; Xiong et al., 2005). Assuming that the primary carbon isotopic fractionation in sub-aerial plants occurs when  $CO_2$  enters the stomata of the leaf, the increased fractionation of the longer chain waxes (and precursor fatty acids and alcohols) suggests progressive fractionation within the synthesis pathway leading to these molecular groups.

The triterpane mass chromatograms ( $m/z$  191) of sediments of source rock show a predominance of  $Tm$  over  $Ts$ . Predominance of  $C_{30}$  hopane over  $C_{29}$  hopane ( $C_{29}H/C_{30}H$  ratio; 0.57-0.91) suggesting siliciclastic source rocks. Absence of oleanane in all studied samples suggests that the age of the source are Lower Cretaceous or older. 28, 30-bisnorhopanes is absent in most of the sediments except Kuthalam-X1, Kuthalam-X2 and Kuthalam-X4 where very low concentration (4.7-8.2) suggesting suboxic depositional environment. Sterane fingerprints ( $m/z$  217) show predominance of  $C_{29}$  steranes over their  $C_{27}$  and  $C_{28}$  homologues. These sediments have low to moderate abundance of all the  $C_{27}$  steranes (9.69-39%).

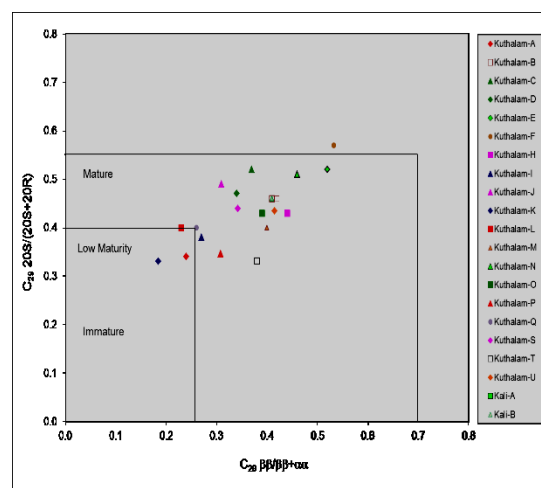


Figure 7

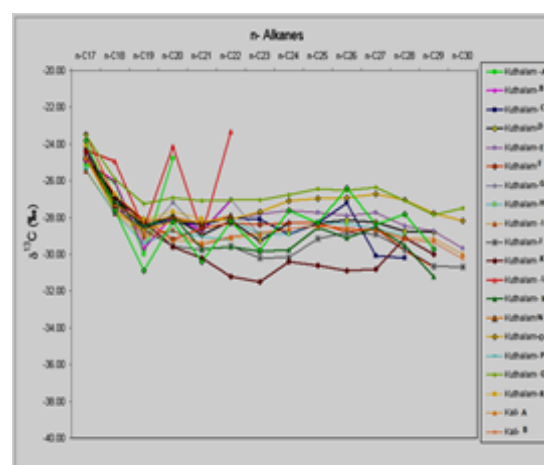


Figure 8

These parameters indicate predominantly terrestrial source input. Maturity level of sediments has been assessed from the extent of isomerisation of cycloalkane biomarkers of hopanes and steranes.  $C_{32}$  hopane S/S+R ratio (0.41-0.52) has not reached equilibrium values in all the studied sediment samples (except in Kuthalam-X5, where it is 0.58) indicating that the organic matter has not crossed diagenetic-catagenetic boundary.  $C_{29}$  sterane S/S+R (0.11-0.30) and  $\beta\beta/\beta\beta + \alpha\alpha$  ratios (0.14-0.40) are showing low values suggesting low maturity for these studied sediment samples. This is further supported by the high values of  $C_{30}M/C_{30}H$  ratios (0.15-0.49). VRc (0.47-0.68) calculated from phenanthrene and its methyl isomers also indicate low to moderate maturity for these sediments. On the basis of source specific biomarkers, the oils/condensates of Kali Kuthalam area are classified in mainly two groups. Group A oils comprise of Kuthalam-E, F, N, O, Kali-A and Kali-B. These oils/condensates are

characterized by moderate Ts/Ts+Tm (0.52-0.57), moderate C<sub>29</sub>H/C<sub>30</sub>H (0.40-0.52) and moderate Pr/Ph (2.7-3.3) ratios. Group B oils comprises of Kuthalam-C, I, L, M, R and T. These oils/condensates are characterized by lower Ts/Ts+Tm (0.39-0.48), moderate C<sub>29</sub>H/C<sub>30</sub>H (0.50-0.61) and high Pr/Ph (4.7-5.1) ratios. The other studied oils/condensates show scattered values for these ratios.

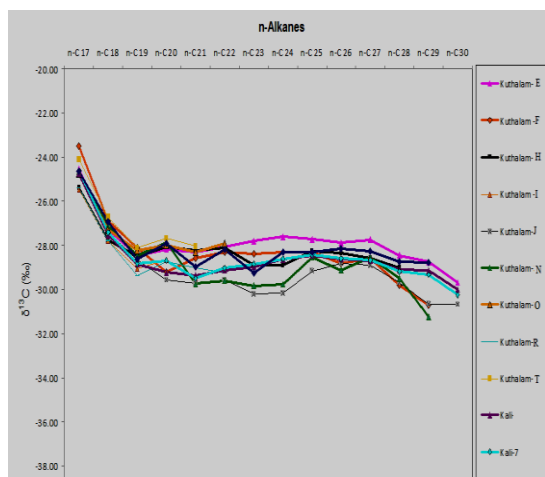


Figure 9

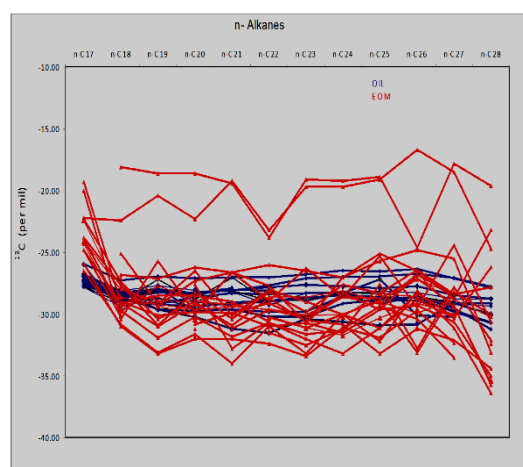


Figure 10

CSIA profile of studied oils/condensates (Kuthalam-D, E, F, H, I, J, N, O, R, S, T, Kali-A and Kali-B) shows similar trend with small differences in their n-alkane carbon isotopic values (Fig. 9). These oils/condensates are thus correlatable on the basis of their CSIA profiles. The other studied oils/condensates (Kuthalam-A, B, C, G, K, M) show scattered CSIA profiles. CSIA profile of n-alkanes for these source sequences and oils are shown in Fig.10.

The studied Andimadam source sequences have wide variation in their isotopic values and show almost flat profile in the range of nC<sub>18</sub> to nC<sub>29</sub>. Most of the studied source sequences show lighter carbon isotopic values as compared to oils. These differences in isotopic value may be due to the variation in organic input and level of maturity. The differences of carbon isotopic values of n-alkanes in oils and source sequences are more than 2 per mil and thus have poor correlations with the studied oils/condensates. However; some source sequences from Kuthalam have the similar CSIA profile. Still deeper sediments may be the major sources as many oils exhibit higher maturity than the studied sediments.

## Conclusions

- The oils/condensates from Kali Kuthalam field are normal oils with high API gravities. The saturate/aromatic ratio indicates that these oils are predominantly aliphatic in nature. The oils have low asphaltene and wax content.
- These oils are derived from terrestrially dominated organic matter source input deposited under oxic environment and are generated at low to moderate level of maturities.
- Studied source sequences are deposited in suboxic to anoxic environment and thermal maturities of these sediments is inferred to be immature to low mature.
- The oils/condensates show similar CSIA trend indicating that these are correlatable. A wide scattering in CSIA profile of source is observed which indicates diversity of organic matter.
- Most of the studied source sequences show poor correlation with studied oils/condensates due to the differences in their thermal maturities and organic facies variations. However, some source sequences are correlatable. Still deeper sediments may be the major sources as many oils exhibit higher maturity than the studied sediments

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