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Article

Characterization of kerogen using combined pyrolysis- GC-MS and FT-IR in weathered and unweathered coals and coaly shales from the Central Myanmar Basin, Myanmar

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Abstract

Kerogens from weathered and unweathered coals and coaly shales from Myanmar have been characterized based on Rock-Eval pyrolysis, pyrolysis-gas chromatography–mass spectrometry (Py-GC-MS), CNS elemental composition, and Fourier transform infrared (FT-IR) spectroscopy. Three moderately weathered coaly shales and five unweathered coals/coaly shales were collected from relatively thin carbonaceous layers (15 – 150 cm) in the Upper Eocene sediments in the western margin of the Central Myanmar Basin. Although organic maturities are similar for all samples (T_{max}417 – 426ºC; immature), kerogens from the weathered samples exhibit low Hydrogen Index (HI, 29 – 42 mgHC/gTOC) and high Oxygen Index (OI, 96 – 168 mgCO_2/gTOC), whereas those from unweathered samples have higher HI (86 – 250 mgHC/gTOC) and lower OI (25 – 115 mgCO_2/gTOC), respectively. Kerogens with high S_3 (pyrolysed at 300 – 400ºC) by Rock-Eval also had high S_3' (pyrolysed at 400 – 650ºC), and show strong correlation. The ratio of S_3' to S_3 is higher in the weathered samples, suggesting an increase in S_3' or a decrease in S_3 values. Py-GC-MS pyrolysates from the weathered kerogens showed that lighter *n*-alkanes/alkenes less than *n*-C_{10} and methyl/dimethyl phenols are decreased. FT-IR analysis of the weathered kerogen showed lower peaks of aliphatic bonding at 2925 – 2850 cm^{-1}, and relatively constant peaks of C=O and C-O bonds at 1716 cm^{-1} and 1000 – 1300 cm^{-1}, respectively. These trends in the FT-IR were more apparent in the bitumen samples. These results suggested that rapid decomposition of alkyl chain moieties in kerogen by weathering faster than that of organic oxygen group had caused an increase in OI.

1. Introduction

Simple pyrolysis techniques such as Rock-Eval and on-line pyrolysis combined with gas chromatography/mass spectrometry (Py-GC-MS) are widely used to determine hydrocarbon potential, kerogen type and organic source (Sentfle et al., 1986; Øygard et al., 1988; Takeda and Asakawa, 1988; Gracia-Vallès et al., 2000; Odden et al., 2000; Behar et al., 2001). Although both methods yield only the light fragments of organic compounds, they are convenient ways for looking at general characteristics of macromolecular organic matter (OM) such as kerogen and asphaltene. Rock-Eval has been known as a useful method for characterizing sedimentary OM (Disnar et al., 2003; Sanei et al., 2005). The Rock-Eval system simulates the generation of hydrocarbons and CO_2 by heating from 300 to 650ºC. HI (mgHC/gTOC) and OI (mgCO_2/gTOC) are now key tools to characterize organic matter on the van Krevelen type diagram to identify kerogen type. In addition, Rock-Eval can also be used to examine the oxidation of organic matter. The Py-GC-MS

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technique is also valuable to clarify chemical characteristics of OM in both coals and petroleum source rocks (Horsfield, 1989; Larter and Horsfield, 1993).

In the tropical Asian areas such as Myanmar, coals and coaly shales are often moderately weathered, even at depth, due to high temperature and high precipitation associated with the prevailing humid climate. Several seams of coal and coaly shale are well exposed in the Upper Eocene formations in the Central Myanmar (Burma) Basin (CMB) (Bender, 1983). Thin carbonaceous seams are correlated with coal deposits in the Eastern Highlands, the Inner-Myanmar Tertiary Basin and the Arakan Coastal Area. In this paper, we investigate both weathered and unweathered coals and coaly shales, based on Rock-Eval pyrolysis and Py-GC-MS, with the aim of characterizing them, and examining the effects of weathering on kerogens. CNS elemental and FT-IR analyses were also performed to support the characterization.

2. Geological setting and Stratigraphy

Myanmar can be subdivided into four major tectonic provinces: the Shan Plateau (Eastern Highlands), Central Cenozoic Belt, Western Fold Belt (including the Indo-Myanmar Ranges and Arakan Yoma) and the Arakan Coastal Plain, from east to west. In early Tertiary time, the Shan Plateau formed a landmass in the east, and the Arakan Yoma formed a narrow strip of lands in the west (Chhibber, 1934; Bender, 1983). The CMB developed as a subsiding sub-basin within the Inner-Myanmar Tertiary Basin (IMTB). The total stratigraphic thickness of the CMB succession is immense, up to ca. 19 km (Tun, 1968; Myint and Soe, 1977). The CMB is a complex fore-arc/back-arc basin situated between a major right-lateral strike slip fault in the east, and the obliquely converging India plate beneath the Myanmar (Burma) micro-plate to the west (Pivnik et al., 1998).

Tertiary coal seams are widely distributed in the CMB, and in other sub-basins of the IMTB. Coal seams in the CMB generally vary from 1.2 to 2.1 m in thickness (Bender, 1983). In our study area, seams of brownish to black coals with 15 – 150 cm thickness occur in the upper part of the Pondaung Formation. This formation is interpreted to have deposited in swampy brackish-water conditions and in near-shore fluvial environments, as evidenced by fossils and sedimentary facies (Myint and Soe, 1977). Black coal layers with ca. 1 m thickness occur in the lower part of the overlying upper Eocene Yaw Formation.

3. Samples

Three moderately weathered coaly shales and five unweathered coals/coaly shales were collected from relatively thin carbonaceous layers (15 – 150 cm: Fig. 1) in Upper Eocene outcrops in the western margin of the CMB. Four coal samples A1-92 (Lat-21˚34´54.6924; Long- 94˚20´11.04612), A1-113 (Lat-21˚34´57.89676; Long- 94˚20´12.03504), A1-114 (Lat-21˚34´58.34784; Long- 94˚20´10.0212), A2-55 (Lat-20˚05´9.56688; Long- 94˚31´16.14756) and four coaly shales of A2-54 (Lat-20˚05´24.477; Long- 94˚31´2.6238), A2-52 (Lat-20˚05´20.82876; Long- 94˚30´38.73996), A2-52A (Lat-20˚05´20.82876; Long- 94˚30´38.73996), A1-120 (Lat-21˚26´56.8716; Long- 94˚17´30.3612) were collected from the Upper Eocene Pondaung and Yaw Formations along the western margin of the CMB. Individual coal and coaly shale seams ranged from 15 to 150 cm in thickness. Based on microscopic observation, we detected signs of weathering in three coaly shale samples (A2-54, A2-52A and A1-120). Microscopic investigation showed many thin, irregular micro-fissures and dark oxidation rims on the edges of oxidized vitrinite particles in the three coaly shale samples of A2-54, A2-52A and A1-120, and the three samples were brownish color and more friable than the unweathered samples showing black/brownish-black color on the surface (Ei Mon Han et al., in press).

4. Methods

4.1 Kerogen and bitumen

Approximately 2 g of each powdered samples were treated with 6M HCl (100 ml, 24 h) and 46% HF

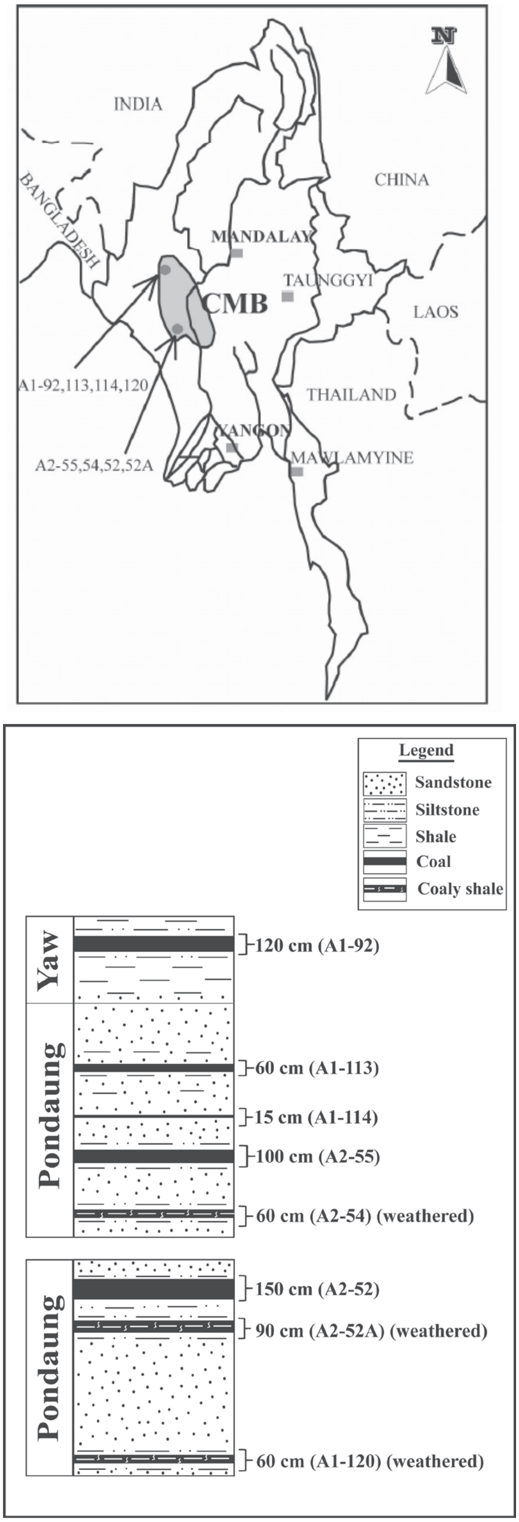

Fig. 1. Lithostratigraphic column for late Eocene coals and coaly shales from the Central Myanmar Basin, Myanmar.

(200 ml, 72 h). The supernatant was removed by careful decantation. The residue was washed several times with distilled water until pH reached 6. The residue was dried and extracted by an ultrasonic vibrator with 50 ml dichloromethane (DCM) / methanol (MeOH) (9/1 v/v) for 2 h. The residue 'kerogen' and extracts 'bitumen' were dried at room temperature for 1 day, and then at 70ºC for 2 h.

4.2 CHNS elemental analysis

The eight Upper Eocene kerogen samples were analysed using an EA1108 Elemental Analyzer (FISONS Co. Ltd) to determine total organic carbon (TOC), total nitrogen (TN), and total sulphur (TS) contents. The kerogen samples (ca. 2 mg) were placed in silver and tin capsules, and then sealed. BBOT standard [2,5-bis-(5-tert-gutyl-benzoxazol-2-yl)-thiophene] was used as calibration for this analysis. Ash contents of the coals were determined by heating in a muffle furnace at 850ºC for 2 hours. The ash contents of samples ranged from 0.59 to 24.47%, with highest values in the weathered coaly shales (Table 1).

4.3 Rock-Eval pyrolysis

We evaluated the amount of hydrocarbon yield (S_2 by FID) and CO_2 yield (S_3 by IR detector) of all eight samples to determine their potential hydrocarbon generation as source rocks and organic oxygen quantities, following Peters (1986) and Behar et al. (2001). The Rock-Eval analysis was performed using a Rock-Eval 6 instrumentat the JAPEX Research Center. Powdered samples weighing ca.5 – 9 mg were heated from 300ºC to 650ºC in a helium flow. The amounts of hydrocarbons released from organic matter were used to determine hydrogen index ($HI = S_2/TOC \times 100$), oxygen index ($OI = S_3/TOC \times 100$), and the maximum temperature of hydrocarbon generation (T_{max}).

4.4 Pyrolysis-GC-MS

Each sample (ca. 1 mg for kerogens and ca. 0.5 mg for bitumens) was analyzed at pyrolysis temperatures of 600ºC, using a double-shot pyrolyser (PY-2020D: small SUS cup slide-down system for 30 sec.) connected to a GC-17A gas chromatograph coupled with a mass spectrometer (*m/z* 50 – 850; Shimadzu GC-MS QP5050A) using electron impact ionization (70 eV). The GC was equipped with an automatic programmable-temperature system and a capillary column (30 m × 0.25 mm i.d.) coated with 5% phenyl-methylpolysiloxane (DB-5MS: Agilent Tec.). Helium was used as the carrier gas. The GC oven temperature was held at 40ºC for 2 minutes and stepped from 40 to 300ºC at 15ºC min^{-1} (held for 12 min at 300ºC). The interface was set at 280ºC. The identification of individual compounds was performed by ion chromatography, and comparison with published and NIST library data.

4.5 FT-IR spectroscopy

We identified functional groups of the kerogens and bitumens in the coals and coaly shale samples by means of FT-IR spectroscopy. FT-IR is well established as a useful technique for the identifying chemical characteristics of coal and kerogen (Fujii et al., 1970; Painter et al., 1978; Blob et al., 1988; Rochdi and Landais, 1991), and to determine the different structures of coals during oxidation (Mae et al., 2000).

The kerogens and bitumens were measured by FT-IR at Shimane University, using a JASCO FT/IR- 660 spectrometer. We measured the infrared spectra in the 400–4000 cm^{-1} wave number range. Extracted samples ca. 1 mg in weight were powdered in an agate mortar with 100 mg potassium bromide (KBr) for 2 min, and then pressed into pellets.

5. Results and Discussion

5.1. CNS composition and elemental Rock-Eval pyrolysis

TOC of the weathered kerogens (A2-54, A2-52A and A1-120) range from 43.8 to 51.8% (average 47.0). The average value is slightly less than that for unweathered kerogen, which ranges from 32.3 to 70.3% (average 57.6). C/N ratio and ash content (wt.%) show~~s~~ clearer contrast between weathered and unweathered samples; average C/N ratios are 33.9 and 52.1 respectively, with ash content averaging 16.7 and 1.5% (Table 1).

The HI – OI is shown in Fig. 2, where we compare results for the equivalent whole-rock data for the same samples. The whole-rock data were acquired by the same method, but without acid treatment. Results for the whole-rocks (open symbols in Fig. 2: data of the whole-rocks were cited from Ei Mon Han et al., in press) differ somewhat from those of the kerogens. HI values of three of the unweathered rock samples are lower than those of their corresponding kerogens (A2-52, A2-55 and A1-92). In contrast, OI values of the weathered rocks A2-52A and A1-120 are greater than those of their corresponding unweathered kerogen. The unweathered kerogens plot in the area around type II-III (Fig. 2), and clearly differ from the weathered kerogens, which plot as type IV, with high OI values (90-170 mgCO$_2$/gTOC). This trend suggests a decrease in HI values and increase in OI values with weathering.

The original CO$_2$ measurement record of the kerogen

Table 1. Elemental and Rock-Eval pyrolysis data of kerogens from the late Eocene coals and coaly shales of the Central Myanmar Basin.

Sample No.	TOC (%)	TN (%)	TS (%)	C/N	Ash (%)	S_1 (300ºC) (mg/g)	S_2 (300-650ºC) (mg/g)	S_3 (300-400ºC) (mg/g)	S_3' (400-650ºC) (mg/g)	S_1+S_2 (mg/g)	S_2/S_3	HI (mg/gTOC)	OI (mg/gTOC)	T_{max} (ºC)
A1-92 kerogen	32.3 (26.3**)	0.70	0.00	46.4	1.1	1.9	57.5	37.1	34.0	59.4	1.5	178	115	420
A1-113 kerogen	67.4 (23.1**)	1.28	0.58	52.6	0.6	2.4	57.7	63.4	51.2	60.1	0.9	86	94	417
A1-114 kerogen	56.6 (57.6**)	1.00	0.19	56.8	1.6	1.9	54.0	20.9	23.5	55.9	2.6	96	37	420
A2-55 kerogen	61.4 (31.3**)	1.18	0.08	51.9	1.2	1.6	114.3	32.2	29.9	115.9	3.6	186	52	426
A2-54* kerogen	45.3 (7.4**)	1.35	0.14	33.5	2.1	0.9	16.3	76.3	80.4	17.2	0.2	36	168	426
A2-52 kerogen	70.3 (8.6**)	1.33	0.37	52.7	3.0	2.0	175.6	17.3	17.8	177.6	10.1	250	25	426
A2-52A* kerogen	43.8 (2.1**)	1.20	0.15	36.5	23.4	0.9	18.4	44.8	47.1	19.3	0.4	42	102	424
A1-120* kerogen	51.8 (7.2**)	1.62	0.13	31.9	24.5	0.8	15.2	49.7	58.8	16.0	0.3	29	96	425
unweathered average	57.6	1.1	0.2	52.1	1.5	2.0	91.8	34.2	31.3	93.8	3.7	159	65	422
weathered average	47.0	1.4	0.1	33.9	16.7	0.9	16.6	56.9	62.1	17.5	0.3	36	122	425

*: weathered sample **: rock data (cited from Ei Mon Han et al., in press)

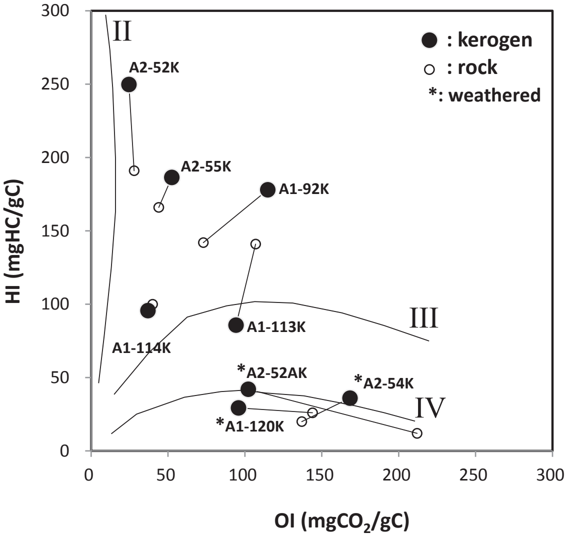

Fig. 2. Van-Krevelen diagram showing types of kerogen for rock and kerogen of coals and coaly shales. Tie lines join whole-rock and kerogen analyses from the same sample. Whole-rock data cited from Ei Mon Han et al. (2014, in press).

is shown in Fig. 3. This method is similar to the temperature-elevation Py-GC-MS with monitoring *m/z*=44 (e.g. Wang et al., 2013). The OI value is calculated from the ratio of S_3 (Fig. 3 and Table 1) between 300 and 400ºC and the TOC content. Two peaks are evident between 300 and 400ºC, suggesting the presence of both weak-bonding and strong-bonding CO_2 in the kerogen. The former is presumed to originate from carboxyl units and the latter from oxygen linkage units (Christiansen et al., 1995; Cervantes-Uc et al., 2006), according to their respective bonding energies. Both S_3 peaks are consistently higher in the weathered kerogens (A2-54, A1-120 and A2-52A) at 300-400ºC than in the unweathered group.

The CO_2 releasing area expands into the high temperature area above 400ºC (Fig. 3). The range from 400–650ºC yields the S_3’ values (Table 1, Fig. 4). The S_3 value shows strong correlation with S_3’ (Fig. 4): $S_3'=0.976\ S_3+6.574$ ($R^2=0.957$) in the weathered samples and $S_3'=0.695\ S_3+7.512$ ($R^2=0.991$) in the unweathered samples. The S_3 and S_3' values and the ratio of S_3' to S_3 are higher in the weathered samples (Fig. 4). This can be speculated that strong-bonding oxygen compounds to kerogen are abundant to some extent in the weathered group more than unweathered samples suggesting an increase in S_3' or a decrease in S_3 values.

The variation of HI and OI for unweathered kerogen is mainly associated with the characters of kerogen (e.g. Katz, 1983). Low organic carbon contents of the weathered original rock samples (TOC** in Table 1) seems to give high OI.

5.2. Characterization of Py-GC-MS

The *n*-alkenes and *n*-alkanes doublets are clearly produced by pyrolysis, in both the weathered and unweathered kerogens (Fig. 5). The *n*-C_7 – *n*-C_{27} homologous series are clearly recognized in all samples. The unweathered kerogens contain abundant aromatics such as benzene, toluene, indane, and p-xylene and phenolic materials (Fig. 5). Light hydrocarbons (less than *n*-C_{10}) generated by the pyrolysis are relatively abundant in the unweathered kerogens. The cause is considered to be weathering, rather than maturity as the T_{max} is constant and immature (417–426ºC in Table 1). Phenol is relatively abundant in the weathered group (Peaks 3 in Fig. 5). Phenol is known to be generated from hydroxybenzoic acids (Kuder et al., 1998). On the other hand, methyl phenols (Peaks 4 and 6) and dimethyl phenols (Peaks 7 and 8) seem to decrease in the weathered kerogen. This indicates that methyl/dimethyl phenolic moiety in the kerogen has been decomposed by the weathering. Although oxidized kerogen has been reported to be abundant in oxygen bonds linking to benzene compounds (Christiansen et al., 1995), the above relationship between phenol and methyl/dimethyl phenols suggests that decomposition rate of the methylene groups is faster than that of forming organic oxygen structures by the weathering.

5.3. FT-IR spectra of kerogen and bitumen

The FT-IR spectra for kerogens and bitumens in all samples are illustrated in Figs 6a and 6b. The aliphatic (C-H) stretching bands for all studied samples occur at 2925 and 2850 cm^{-1}, CH_2 stretching at 1375 and 1383 cm^{-1}, and CH_3 at 1455 and 1457 cm^{-1} (Takeda and Asakawa, 1988; Ibarra et al., 1996; Faure et al., 1999).

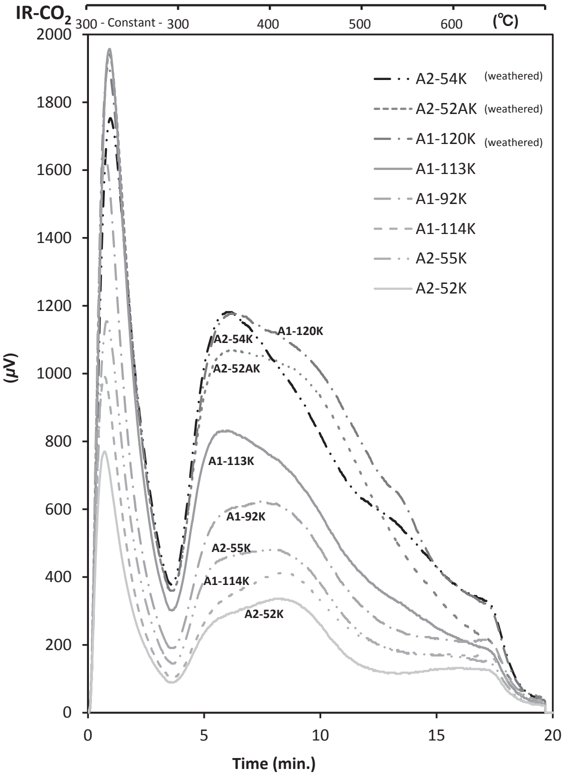

Fig. 3. Rock-Eval pyrograms (S_3 and S_3') for kerogen from the coals and coaly shales.

Weathered kerogens show a decreased aliphatic bond (Fig. 6a), indicating that some parts of alkyl chains in functional group might be likely reduced by weathering. The aliphatic oxygen-containing bond (C=O) is detected at 1716 cm^{-1} in the weathered samples, and in one unweathered sample (A1-113). C-O bands (1000–1300 cm^{-1}) show a higher frequency compared to C-H bands (Fig. 6a). This suggests that carboxylic and/or ester groups are predominant in the weathered samples. Although C=O/C-O bonds possibly indicate significant occurrence of esters or ether formation (e.g. Jackson et al., 1996; Faure et al., 1999) and aliphatic side chains are readily oxidized to form the aliphatic acid or ester C=O groups (e.g. Guo and Bustin, 1998), the weathered kerogen and bitumen in Fig. 6 also suggest significant and clear decrease in aliphatic groups.

The aromatic C=C stretching band at 1617 cm^{-1} is similarly high in all samples. The aromatic (C-H) stretching bands at 702 cm^{-1}, 751 cm^{-1}, and 819 cm^{-1} also seem to differ little between the samples.

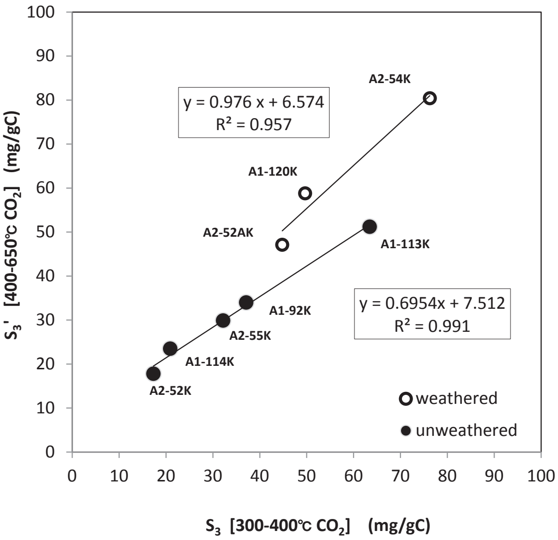

Fig. 4. Correlation of Rock-Eval pyrolysis data (S_3 and S_3’) for kerogen from the coals and coaly shales.

For the bitumens (Fig. 6b), functional groups such as aliphatic stretching groups (C-H, CH_2, CH_3), aromatic bonds (C-H, C=C) and aliphatic stretching C=O groups (e.g. COOH, ester, ketones) are more abundant than those in the kerogens.

5.4. Characterization of kerogen in weathered and unweathered samples

Based on the analytical results, noticeable alterations of the kerogens are summarized as follows. The weathered kerogens exhibit low HI (29–42 mgHC/gTOC) and high OI (96–168 mgCO_2/gTOC), whereas those from unweathered samples have higher HI (86–250 mgHC/gTOC) and lower OI (25–115 mgCO_2/gTOC), respectively (Fig. 2). S_3' (mgCO_2/g released during 400–650ºC: Fig. 3) is more abundant in the weathered kerogens. Lighter *n*-alkanes/alkenes less than *n*-C_{10} and methyl/dimethyl phenols in py-GC-MS results are relatively decreased in the weathered kerogens (Fig. 5). FT-IR analysis showed that the weathered kerogens have a decreased peak of C-H methylene group and relatively constant (or somewhat increased) C=O (1716 cm^{-1})/C-O (1000–1300 cm^{-1}) peaks from esters or ether groups (Fig. 6).

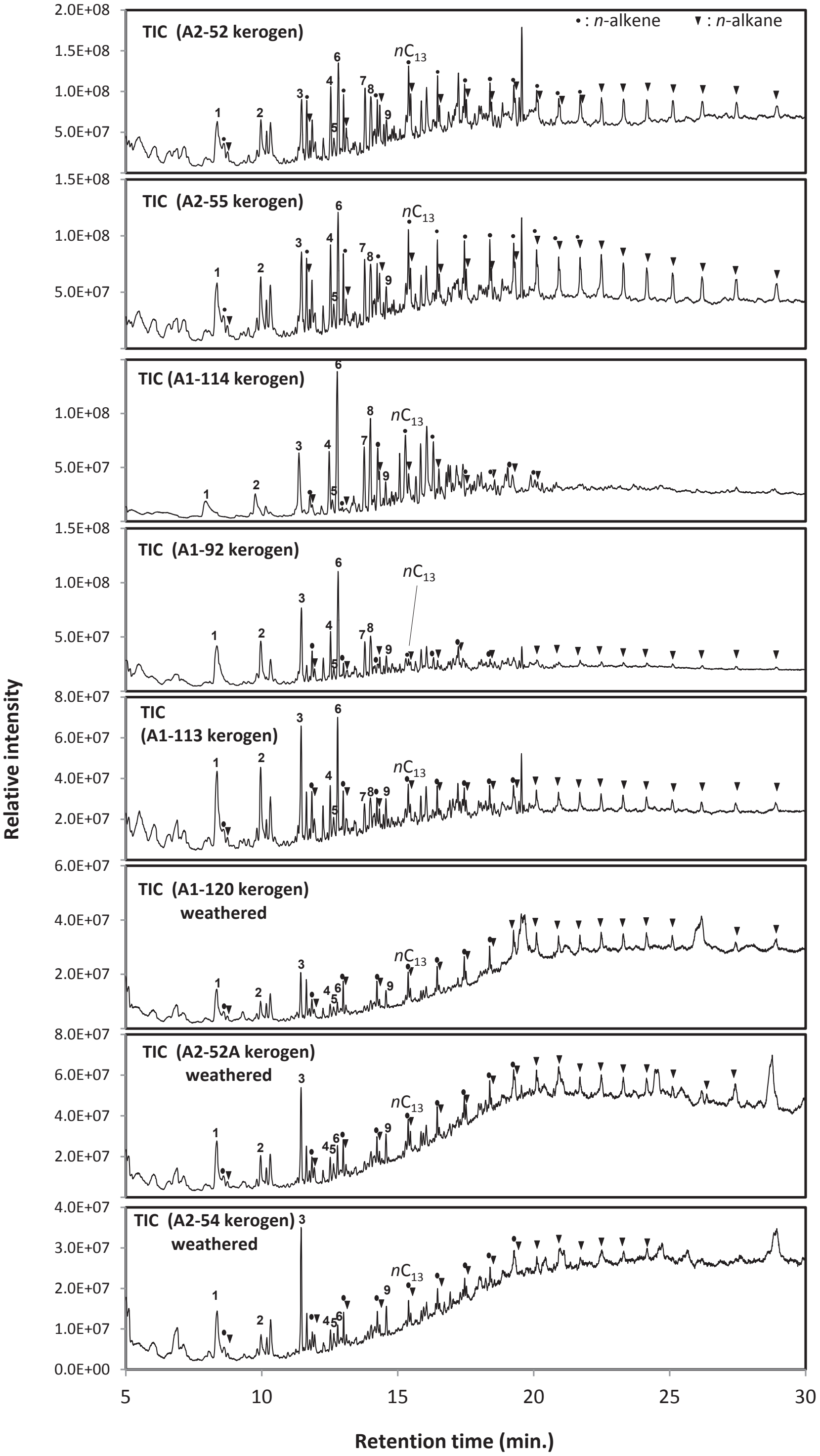

Fig. 5. Py-GC-MS data for kerogens from the coals and coaly shales.
1: toluene; 2: xylene; 3: phenol; 4 and 6: methyl phenol; 5: indene; 7 and 8: dimethyl phenol; 9: naphthalene.

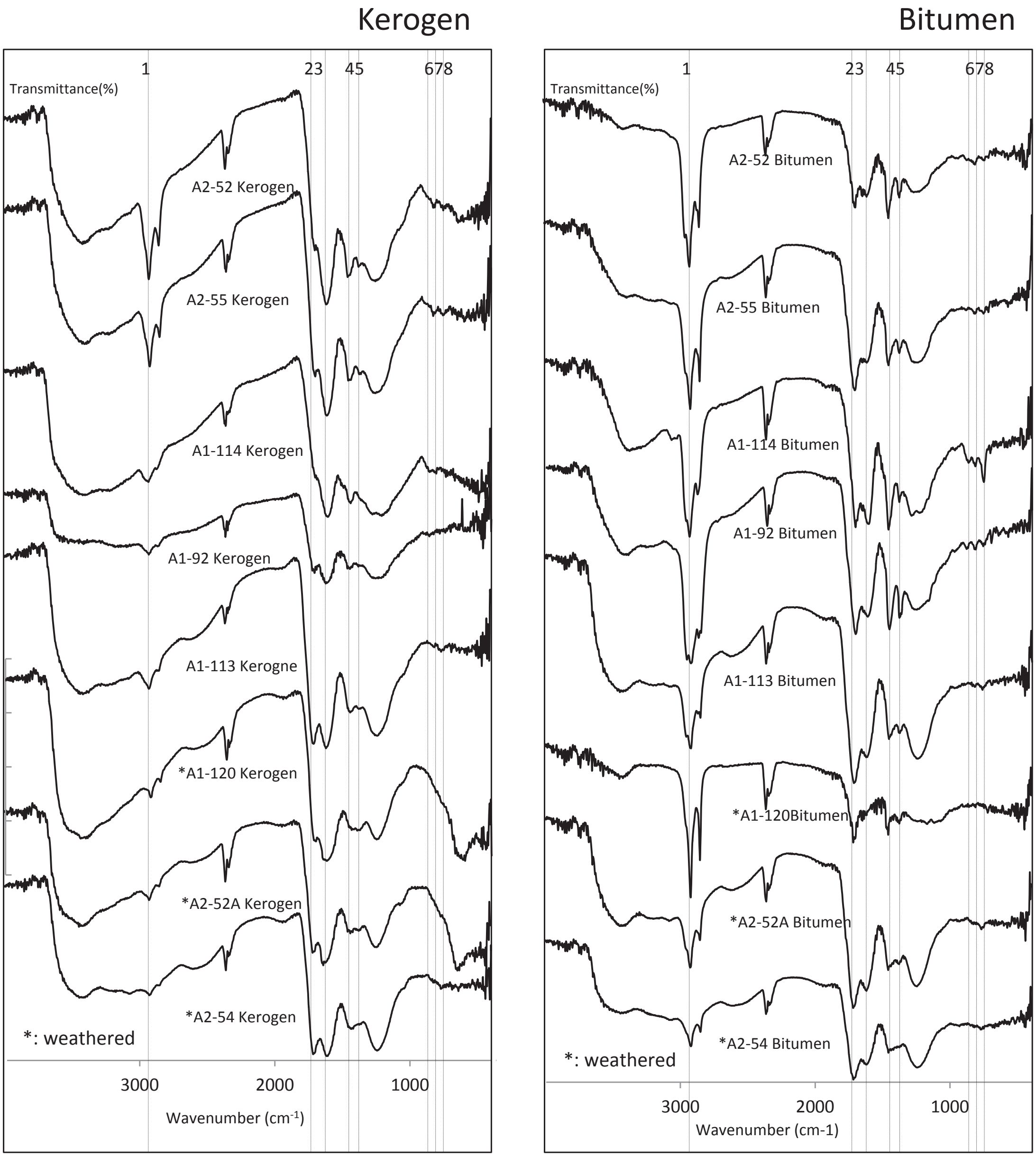

Fig. 6. (a) FT-IR measurements showing chemical characteristics of kerogen from the coals and coaly shales. 1: 2925 and 2850 cm^{-1} to C-H stretching (aliphatic methylene groups); 2: 1716 cm^{-1}to aliphatic C=O stretching group; 3: 1617 cm^{-1} to aromatic nucleus C=C stretching; 4-5: 1455 and 1457 cm^{-1} to aliphatic C-H deformation; 6-7-8: 702, 751 and 819 cm^{-1} to aromatic C-H out-of-plane bending, and 1000-1300 cm^{-1} to C-O bonds. (b) FT-IR data showing chemical structure of bitumen from the coals and coaly shales.

These results suggest that C-C methylene structures in the weathered kerogens have been cleaved and the alkyl chain moieties in the kerogen might be considerably released by weathering. Oxygen-containing moieties such as methyl/dimethyl phenolics in kerogen are also decreased by the weathering. In general, the oxygen-containing moiety in geomacromolecule is thought to be easily decomposed during oxidation and oxic biodegradation except peculiar cases as reported by Jenisch-Anton et al. (1999) and Guo and Bustin (1998). The oxygen-containing group in organic matter is commonly thought to be labile and difficult to be preserved, although such organic oxygen in geomacromolecule has been poorly understood. The results of present study could indicate that rapid decomposition of alkyl chain moieties in kerogen by weathering faster than that of organic oxygen group had caused an increase in OI, because OI is a relative value based on organic oxygen to organic carbon in kerogen.

6. Conclusions

The conclusions of the present study are:

(1) All samples have similar organic maturities ($T_{max}=417-426$ºC: immature), but the weathered kerogens show significantly low HI (29–42 mgHC/gTOC) and high OI (96–168 $mgCO_2$/gTOC). In contrast, the unweathered kerogen show relatively high HI (86–250 mgHC/gTOC) and low OI (25–115 $mgCO_2$/gTOC).

(2) The kerogens with high S_3 values at 300–400ºC by Rock-Eval also show high S_3' values at 400–650ºC. Although the S_3 values thus show a strong relationship with the S_3' values, the ratio of S_3' to S_3 is higher in the weathered samples, suggesting an increase in S_3' or a decrease in S_3 values.

(3) The Py-GC-MS pyrolysates from the weathered kerogens showed that lighter *n*-alkanes/alkenes less than n-C_{10} and methyl/dimethyl phenols relatively decreased.

(4) FT-IR analyses of the weathered kerogens exhibit low peaks of aliphatic bonding at 2925–2850 cm^{-1}, and relatively constant (or somewhat increased) peaks of C=O and C-O bonds at 1716 cm^{-1} and 1000–1300 cm^{-1}, respectively.

These results could indicate that rapid decomposition of alkyl chain moieties in kerogen by weathering faster than that of organic oxygen group had caused an increase in OI.

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