METHODS FOR MEASURING THE MATURITY OF ORGANIC MATTER IN DIAGENESIS STAGE

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ABSTRACT

Several methods are used for measuring maturity of the organic matter. However, the degree of efficiency varies at different stages of the thermal maturity. During diagenesis and the beginning of catagenesis the fluorescence and O/C atomic ratio are considered to be the best indicators for characterizing the level of maturation. On the other hand, the techniques requiring long sample-preparation time can not be utilized as routine tools.

The aim of this paper is to propose a new applicability of a parameter measured by a generally accepted method. This parameter is the oxygen index determined by Rock Eval pyrolysis.

According to the results presented in this report the OI linearly decreases with the maturation level during the diagenesis and early catagenesis.

The correlation between oxygen index and maturity was experimentally demonstrated by laboratory thermal degradation of kerogen type I. Furthermore, the oxygen index has been proved to be an efficient indicator of coal rank from lignites to high vol. bitumenous coals. Applicability of the paramteres was checked on sedimentary organic materials of two boreholes.

The oxygen index decreased linearly in function of advanced diagenesis during both artificial and natural evolution. The correlation between the OI and the depth or between the OI and the temperature of thermal degradation is satisfactory ($r^2 = 0,81$—$0,99$). The connection seems to be valid independently of the type of organic matter. On the basis of our present results, however, the parameter can only be used in the case of coals and sedimentary organic matter of high organic carbon content. Preferred evolution interval of its application ranges from about $R_0 = 0,2$—$0,3$ per cent to $R_0 = 0,7$—$0,8$ per cent.

Keywords: coals, kerogen, maturity parameters, oxygen index, Rock Eval pyrolysis, early catagenesis.

INTRODUCTION

Several chemical and physical properties of the organic matter are used as indicators for its maturation. During the thermal evolution hydrocarbons, resins and asphaltenes have been formed with increase in depth and temperature. At the same time, the kerogen structure and as a consequence its properties also change. A great variety of analytical methods have been developed to determine the level of maturation. A lot of techniques are based on the properties of kerogen. Other parameters are proposed to measure the amount and quality of degradation products: bitumen, oil or gas.

Principal methods used for characterizing the rank of evolution can be summarized as follows: chemical indicators of maturity based on bitumen or crude oil, physicochemical methods based on kerogen, as well as optical microscopy and pyrolysis methods (TISSOT and WELTE, 1984).

Numerous procedures have been developed to measure the amount and the chemical composition of degradation products. The abundance of bitumen or hyd-

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rocarbons and the composition of hydrocarbons are well-known maturity indicators. The quantity and composition of light hydrocarbons (LEYTHAEUSER et al, 1979a, b; DURAND and ESPITALIÉ, 1972; PHILIPPI, 1975; SCHAEFER et al., 1983; THOMSON, 1979), as well as the Carbon Preference Index (BRAY and EVANS, 1961; TISSOT et al., 1977) and the different ratios of biological marker hydrocarbons (BRASSELL et al., 1983; MACKENZIE et al., 1980, 1981, 1982; SEIFERT and MOLDOWAN, 1979, 1981) are also useful as maturation indices.

Various chemical and physical parameters of the kerogen also give good informations about its maturity stage. Oxygen and hydrogen content of kerogen and its evolution path have to be considered one of the principal chemical methods used for characterization the type and maturity of the source rocks and coals, too (van KREVELEN, 1961; TISSOT et al., 1974). Some physical techniques measuring the rank of evolution are electron spin resonance (DURAND et al., 1977; BAKR et al., 1988), infrared spectrophotometry, thermal analysis etc.

Optical examination is widely accepted method for measuring the maturity of organic matter. Several scales of maturation based on color and structure alteration of palynomorphs have developed (STAPLIN, 1969). Fluorescence of liptinite macerals decreases during the diagenesis and partly during catagenesis (ALPERN et al., 1972; TEICHMÜLLER and DURAND, 1983). The classical maturation index is the vitrinite reflectance. It is the best and well known parameter characterizing both the coalification stage and the maturity of source rock (Dow, 1977; TEICHMÜLLER, 1982; TISSOT and WELTE, 1984).

The pyrolysis conducted under an inert atmosphere is a rapid and efficient method to determine maturity. GRANSCH and EISMA (1970) used the CR/CT ratio as an indicator of state of maturity. In 1977 ESPITALIÉ et al. developed a new standard pyrolysis method of source rock characterization and evaluation. The temperature (T_{max}) corresponding to the maximum of hydrocarbon generation increases progressively with the thermal evolution during the assay. There is a very good relationship between T_{max} and vitrinite reflectance (BERTRAND, 1984; ESPITALIÉ et al., 1985, 1986). Therefore this method is particularly valuable in the case of marine or lacustrine kerogen (types I and II) where vitrinite is often scarce or absent (TISSOT and WELTE, 1984). Nevertheless T_{max} can be used as a rank parameter for coals, too (BERTRAND, 1984; JOHNS et al., 1984; LEPLAT and PAULET, 1985; MONTHIOUX et al., 1985; PETERS, 1986; TEICHMÜLLER and DURAND, 1983; VERHEYEN et al., 1984).

The efficiency of methods mentioned above is very different. Almost all of them can be used for determining the boundaries between diagenesis, catagenesis and metagenesis. On the other hand, the sensitivity of various maturation indicators to individual evolution zone is not the same.

Some of them is particularly valuable in the catagenesis and metagenesis (for example vitrinite reflectance, aromatic compounds of oil and bitumen, T_{max} measured by Rock Eval pyrolysis etc.). Only a few parameters can be considered as a good index for evaluating maturity within the diagenesis stage (for example fluorescence of liptinites, ESR, elemental analysis of kerogen). Furthermore, many of these techniques are known more as research rather than routine tools due to the time and care needed in their application (TISSOT and WELTE, 1984). •

Concerning the immature coals the O/C atomic ratio was found to be the best qualification parameter (BERTRAND, 1984). However, the elemental analysis is not a routine method because the kerogen has to be isolated previously. On the other hand there is a good correlation between the O/C atomic ratio and the oxygen index measured by Rock Eval method.

In this work an attempt was made to utilize the oxygen index (OI) as an indicator for maturation. It is thought to be an efficient maturity indicator in the diagenesis zone, which is considered as "the zone of oxygen".

EXPERIMENTS

The total organic carbon content (TOC) was measured at 1000 °C under intense oxygen flow by combusting in a Carmbograph—8 equipment. Kerogen was isolated by physical method (HETÉNYI and VARSÁNYI, 1976). Experimental evolution was carried out from 300 °C to 500 °C under nitrogen atmosphere in a Heraeus-type temperature-programme furnace. Products were collected in cooled traps, residues were extracted by chloroform and by benzene-acetone-methanol mixture of 70:15:15 ratio. Coals, kerogens, as well as the residue of thermal degradation ("unconverted kerogen") were characterized by Rock Eval pyrolysis (ESPITALIÉ et al., 1977). The CR/CT ratio was measured on the basis of ASTM standard (CUMMINS et al., 1972).

RESULTS

1. *Change of maturity indices during the artificial evolution with special regard to diagenesis stage.*

Experimental assay of thermal evolution was carried out on an immature kerogen to study the efficiency of maturity indicators measured by Rock Eval pyrolysis. The kerogen type I reached the boundary of diagenesis and catagenesis at higher temperature than the other ones. Thus, kerogen type I seems to be the most suitable raw material to study in detail the diagenesis. The artificial evolution was performed on kerogen isolated from a maar-type oil shale (Pula, Hungary). This kerogen belongs to type I; its H/C atomic ratio is 1,7; CR/CT ratio is less than 0,1; hydrogen index is 890 mgHC/gTOC; oxygen index is 42 mgCO$_2$/gTOC.

The organic matter was thermally degraded at 8 different temperatures during 5 hours under nitrogen atmosphere (HETÉNYI, 1983). After extracting the bitumen the residue (so called "unconverted kerogen") was characterized by Rock Eval pyrolysis (Table 1).

According to the total organic carbon contents, the hydrogen indices and the PC/TOC ratios the boundary of the diagenesis and catagenesis is between 375—400 °C. Comparing the results of thermal degradations performed at these two temperatures the change of the TOC is 21 per cent, the change of HI is 39 per cent and the change of PC/TOC is 36 per cent. At lower temperature these parameters are nearly constant. For example the hydrogen index of the unheated kerogen is 890 mgHC/gTOC and the residue after thermal degradation at 375 °C is 868 mgHC/gTOC, e.g. the change is only 2 per cent (Table 1).

The T$_{max}$ is considered to be the most reliable indicator — among those measured by pyrolysis — for characterizing thermal evolution. However, the T$_{max}$ is influenced by the type of organic matter during the diagenetic stage and at the beginning of catagenesis (TISSOT and WELTE, 1984). As we can see in Table 1, the T$_{max}$ values are unchanged during the zone of evolution mentioned above, therefore it is not proved to be a properly sensitive index to characterize the maturity stage of an immature kerogen type I.

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TABLE 1

Thermal degradation of the kerogen type I.
Characterization of the unconverted kerogen by Rock Eval pyrolysis
(degradation period = 5 hours)

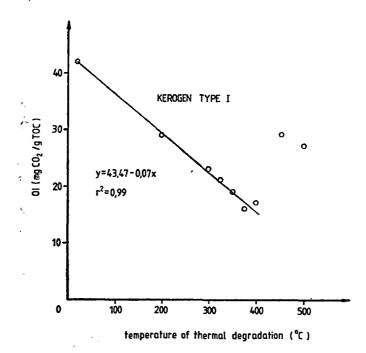| Temperature °C	TOC %	PC/TOC %	HI	OI	T$_{max}$ °C	S2/S3
Unheated kerogen	74.0	77	890	42	443	25.70
200	72.8	74	890	29	443	31.28
300	73.6	74	885	23	445	38.16
325	73.8	74	888	21	447	41.79
350	75.0	73	873	19	447	44.04
375	75.5	72	868	16	451	54.72
400	59.8	44	521	17	447	26.20
450	37.4	10	120	29	454	4.08
500	34.2	4	33	27	454	0.77

HI = mgHC/gTOC
OI = mgCO$_2$/gTOC

Fig. 1. Change of oxygen index in function of temperature of thermal degradation.

At the same time the oxygen index decreased progressively in function of the increasing degradation temperature *(Fig. 1.)* The excellent correlation ($r^2 = 0.99$) between the oxygen index and degradation temperature suggests that the oxygen index is a valuable indicator for characterizing the maturity of organic matter within the first zone of the evolution process.

2. *Oxygen index as a possible indicator of coal rank*

The vitrinite reflectance is to be considered the best tool for determining the coal rank. According to TEICHMÜLLER *et al.* (1982), however, the efficiency of this parameter depends on the maturation level. Concerning to its applicability there are three sections:

a) $R_0 > 1.1$: efficiency is good

b) $0.5 < R_0 < 1.1$: efficiency is low

c) $R_0 < 0.5$: the indicator is not properly sensitive.

TABLE 2

Rank parameters of a series of Hungarian coals measured by ROCK EVAL pyrolysis and their vitrinite reflectance

Samples	R_0 %	PC/TOC %	HI mgHC/gTOC	OI mgCO$_2$/gTOC	T_{max} $_0$C	S2/S3
Lignite	0.30	5.7	59	50	402	1.16
Lignite	0.34	5.7	64	45	407	1.41
Sub-Bit. C	0.42	3.3	38	40	417	0.94
Sub-Bit. C	0.43	7.4	85	38	421	2.23
Sub-Bit. A	0.48	7.1	81	37	428	2.16
High vol. Bit. A	0.82	6.9	76	5	446	12.77
High vol. Bit. A	0.87	8.9	105	7	448	13.77
High vol. Bit. A	0.95	10.2	120	5	453	20.28

Some Hungarian coals of low vitrinite reflectance were examined by Rock Eval pyrolysis. The value of R_0 ranges from 0.30 per cent to 0.95 per cent. Within the mentioned part of the coalification scale vitrinite reflectance is only weakly effective. It would be useful to develop another method to characterize the evolution level of lignites and subbituminous coals. Therefore T_{max} values and oxygen indices were studied as maturity indicator (Table 2).

As we can see in *Figs. 2* and *3* both parameters are linearly correlated to coal rank. But there is an essential difference between the change of OI and T_{max}. Oxygen index decreases linearly from an immature lignite ($R_0 = 0.30\%$) to high vol. bituminous coals (R_0 = about 0.80%) reaching finally a minimum value. As from high vol. bit. coals oxygen indices are so low that their change is also insignificant. Consequently the OI proved to be an excellent maturity indicator but only in the diagenesis and at the beginning of the catagenesis. At the same time T_{max} changes linearly during the diagenesis and catagenesis, too. The rises of the curves are very different in these two zones as a consequence of various chemical processes.

On the basis of the present results the data measured by Rock Eval pyrolysis — both the T_{max} and OI — seem to be useful supplements to other parameters for
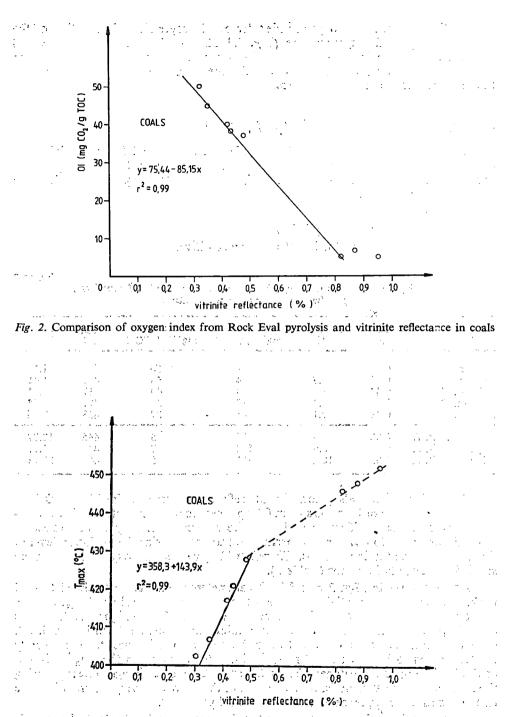
Fig. 2. Comparison of oxygen index from Rock Eval pyrolysis and vitrinite reflectance in coals

Fig. 3. Comparison of T_{max} from Rock Eval pyrolysis and vitrinite reflectance in coals (in diagenesis zone: full line, in catagenesis zone: dashed line).

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characterizing coal rank. One of the most valuable methods to determine the evolution level of lignites and subbituminous coals would be the simultaneous use of these parameters. Particularly, because they can be measured by a single Rock Eval pyrolysis.

3. Change of oxygen index in function of depth

Applicability of maturation parameters studied in this paper was checked on sedimentary organic materials. Core samples from Tpk-I borehole (Hungary) and cuttings from Makó-3 borehole (Hungary) were investigated by Rock Eval pyrolysis.

Characterization of core samples from Tpk-I borehole (Hungary) by
Rock Eval *pyrolysis*

Depth (m)	TOC %	HI mgHC/gTOC	OI mgCO$_2$/gTOC	T$_{max}$ °C	S2/S3	Hydrocarbon potential kgHC/ton of rock
294.6	43.39	167	80	397	2.09	90.95
361.0	47.38	159	76	387	2.08	95.74
391.0	50.51	142	70	382	2.01	88.68
429.3	43.57	116	75	391	1.55	58.58
684.9	45.08	131	63	402	2.09	64.71
714.4	46.39	95	65	404	1.45	47.73
865.8	48.99	92	61	503	1.51	47.89
919.0	43.51	129	56	403	2.29	61.85
1212.2	49.48	129	38	408	3.37	67.99
1328.2	54.36	138	29	407	4.75	78.88
1466.0	62.94	156	19	409	8.05	1C˙32
1575,3	22.30	175	21	416	8.22	4˙22
1586.0	62.56	188	18	411	10.35	123.56

13 core samples containing high quantity of organic carbon were investigated from Tpk borehole. The total organic carbon content changes from 22.3 per cent to 62.9 per cent, average value is 47.7 per cent (Table 3). These samples are considered to be lignites or coals. In *Figs. 4* and *5* T$_{max}$ and oxygen can be seen plotting against depth. Both parameters change linearly in function of depth. Relationship, however between the oxygen index and depth seems to be better (r^2=0.97) than that between the T$_{max}$ and depth (r^2=0.78).

Large number of samples from Makó-3 borehole were analysed. From 1010 m to 2090 m cuttings in every 20 m were measured by Rock Eval pyrolysis (Table 4). The quantity of organic carbon is very changeable. It ranges from 7 per cent to 56 per cent. Some of the cuttings are coals and the others contain kerogen type III (HI = = 100—170 mgHC/gTOC). The high value of hydrocarbon potential (8—56 kgHC/ton of rock) is due to the significant organic carbon content. The T$_{max}$ seems to be an unreliable maturity parameter in these samples. Values change between 402 °C and 422 °C independently of depth (Table 4). At the same time oxygen index linearly decreases with depth *(Fig. 6)*. There is a fair correlation (r^2=0.81) between the OI and depth.
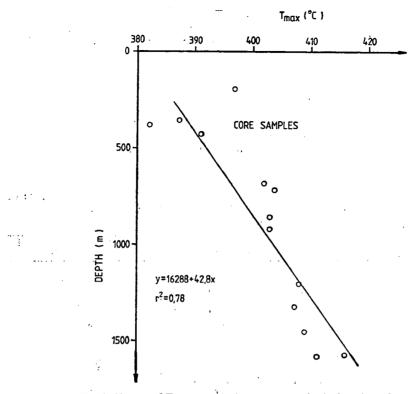
Fig. 4. Change of T_{max} measured on core samples in function of depth.

CONCLUSION

1. On the basis of results presented in this paper the oxygen index seems to be a useful supplement to other parameters for characterizing the maturity of different organic materials.

2. The oxygen index is a particularly effective maturation indicator during the diagenesis and at the beginning of the catagenesis.

3. Its applicability was checked among both artificial and natural conditions of the diagenesis. The OI decreased linearly in function of advanced evolution. A good relationship was found between the OI and the depth, as well as between the OI and the degradation temperature of experimental evolution. The correlation coefficient was excellent in the case of thermal degradation of a kerogen type I and in the case of core samples ($r^2 = 0.97$—0.99). A fair correlation coefficient ($r^2 = 0.81$) was determined by Rock Eval pyrolysis of cuttings.

4. In according to our results this parameter can only be used to measure the maturity of coals and sedimentary organic matter of high organic carbon content. Further examinations are necessary to apply this indicator for samples containing a small quantity of organic carbon.

5. Simultaneous use of OI and T_{max} has proved to be a valuable method to determine the coal rank from lignites to high vol. bituminous coals.
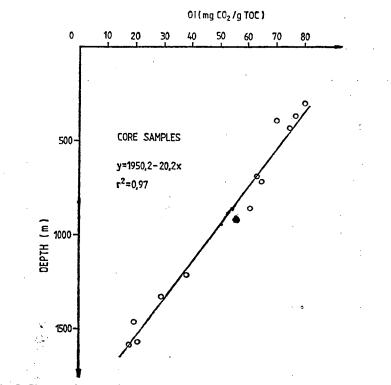
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OI (mg CO₂ /g TOC)

CORE SAMPLES

$y = 1950,2 - 20,2x$

$r^2 = 0,97$

DEPTH (m)

Fig. 5. Change of oxygen index measured on core samples in function of depth.

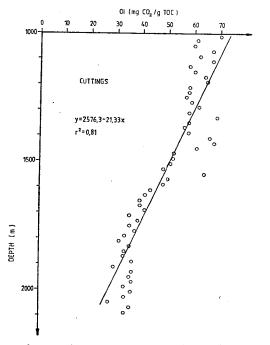OI (mg CO₂ /g TOC)

CUTTINGS

$y = 2576,3 - 21,33x$

$r^2 = 0,81$

DEPTH (m)

Fig. 6. Change of oxygen index measured on cuttings in function of depth.

TABLE 4

Characterization of cutting samples from Makó-3 borehole (Hungary) by
ROCK EVAL *pyrolysis*

Depth m	TOC %	HI mgHC/gTOC	OI mgCO₂/gTOC	T$_{max}$ °C	S2/S3	Hydrocarbon potential kgHC/ton of rock
1010	7.44	106	70	417	1.50	8.41
1030	14.23	112	61	415	1.83	16.95
1050	14.62	113	60	422	1.87	17.39
1070	12.55	114	47	414	1.70	15.28
1090	23.49	128	62	415	2.04	31.65
1110	22.92	133	67	413	1.96	32.07
1130	32.99	141	58	415	2.42	49.09
1150	18.08	126	60	419	2.10	24.21
1170	15.40	122	64	418	1.90	19.83
1190	28.04	120	65	415	1.82	35.42
1210	32.20	139	58	413	2.37	46.88
1230	38.35	136	58	413	2.31	54.77
1250	30.18	115	57	414	2.03	36.36
1270	22.71	119	59	416	1.98	28.21
1290	17.16	123	62	416	1.96	22.13
1310	9.81	108	58	418	1.85	11.10
1330	11.20	112	69	421	1.62	13.19
1350	18.59	130	58	414	2.22	25.26
1370	36.20	150	51	414	2.92	56.42
1390	13.89	• 114	58	417	1.97	16.64
1410	5.90	102	66	416	1.52	6.32
1430	13.93	127	68	414	1.86	18.69
1450	7.02	96	56	420	1.68	7.06
1470	18.17	124	47	413	2.62	23.50
1490	21.91	128	47	418	2.71	29.24
1510	14.26	132	51	421	2.17	19.79
1530	37.06	120	48	414	2.48	47.44
1550	9.89	123	64	418	1.90	12.82
1570	19.66	122	50	417	2.43	24.93
1590	14.28	139	48	418	2.87	20.76
1610	27.47	129	43	418	2.96	37.07
1630	24.94	121	41	421	2.93	31.64
1650	48.87	121	39	410	3.09	62.35
1670	22.51	136	39	416	3.46	32.54
1690	47.58	134	41	417	3.26	66.83
1710	40.41	141	35	414	4.02	59.69
1730	9.23	126	38	420	3.27	12.12
1750	55.99	155	35	414	4.37	92.46
1770	24.58	154	37	421	4.12	39.85
1790	21.37	162	33	421	4.90	35.82
1810	32.26	168	31	412	5.34	57.16
1830	55.67	133	35	409	3.79	78,53
1850	45.65	164	28	416	5.76	78.58
1870	33.14	162	28	413	5.66	56.27
1890	50.53	138	31	417	4.41	72.98
1910	28.87	128	29	418	4.43	38.43
1930	18.01	144	36	419	3.91	27.03
1950	11.38	148	35	419	4.12	17.40
1970	8.74	137	36	422	3.72	12.42
1990	8.68	108	33	417	3.27	9.85
2010	27.27	154	38	417	3.98	43.50
2030	12.39	157	33	421	4.67	19.94
2050	20.07	172	27	419	6.36	35.76
2070	14.65	156	35	417	4.41	23.83
2090	16.68	148	33	418	4.41	25.66

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