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STUDY OF ORGANIC MATTER IN BLACK SHALES OF THE MALÉ KARPATY MTS. CRYSTALLINE COMPLEXES

(Figs. 4, Tabs. 4)

Abstract: The authors summarise in this paper the results of a study of organic matter in black shales of the Malé Karpaty Mts. crystalline complexes, gained up to now. The highest contents of organic matter have been found in the Pezinok-Pernek crystalline complex, in the productive pyrite-bearing zones. There was ascertained a dependence of organic matter contents on the grain--size of the studied rocks — fine-grained varieties had an average content of 2.8 per cent C_{org}, medium-grained varieties only 1.1 per cent. From the total carbon balance the conclusion was made that the samples represent advancedly metamorphosed organic matter. According to their bitumen coefficient and their bitumen-carbon contents, we can classify the studied rocks as poor in bitumens; according to the results gained from the analysed samples, the rocks cannot be classified as potentialy oil-bearing. On the basis of the CPI indexes, the authors suggest two ways of origin (marine and terrigenous) of organic matter in black shales of the Malé Karpaty Mts. crystalline complexes.

Резюме: Авторы в статье подытоживают существующие результаты исследования органического вещества в черных сланцах кристалликума Малых Карпат. Самые высокие содержания органического вещества были установлены в пезинско-перненском кристалликуме в продуктивных пиритсодержающих зонах. Была определена зависимость содержаний органического вещества от размера частиц исследованных пород-среднее содержание C_{org} мелкозернистых разновидностей 2,8 %, среднезернистых только 1,1 %. На основе общего баланса углерода устанавливается, что образцы представлены высокометаморфизованным органическим веществом. По коэффициенту битуменозности и содержанию битуменозного углерода исследованные породы можно характеризовать как породы бедные битуменами и согласно анализированным пробам их нельзя характеризовать как потенциальные производители нефти. На основе CPI индексов было установлено двойное происхождение (морское и терригенное) органического вещества в черных сланцах кристалликума Малых Карпат.

Introduction

Because the dispersed organic matter as a rock component carries important information (facial conditions at the origin of the sediments, grade of diagenesis or lower grades of metamorphism, thermal history of the studied areas, morphology of the sedimentation base), a lot of attention is paid to

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Table 1

Average values of the complete analyses of organic matter in black shales of the Malé Karpaty Mts. crystalline complexes

Sample number	Organic matter as a whole %	Weigh % C in rock				Carbon balance		
		C_{org}	C_{rez}	C_{hum}	C_{bit}	% C_{rez}	% C_{hum}	% C_{bit}
Pezinok—Pernek crystalline area, productive zones								
K-16 B	6.27	4.94	4.93	0	0.01	99.90	0	0.1
7 OH A	5.51	4.34	4.31	0.004	0.0025	99.34	0.09	0.57
10 OH A	1.26	0.99	0.98	0.001	0.013	98.57	0.05	1.38
6 B	5.85	4.61	4.58	0.022	0.007	99.37	0.48	0.15
9 B	0.77	0.61	0.60	0.003	0.011	97.71	0.49	1.80
11 B	0.51	0.40	0.32	0.038	0.046	79.00	9.50	11.50
16 B	7.69	6.06	6.04	0.013	0.007	99.68	0.21	0.11
29 C	4.83	3.80	3.78	0.016	0.006	99.42	0.42	0.16
30 B	5.94	4.68	4.66	0.011	0.013	99.49	0.23	0.28
32 C	4.52	3.56	3.51	0.033	0.014	98.68	0.93	0.39
34 C	5.45	4.29	4.27	0.003	0.013	99.63	0.07	0.30
37 A	0.23	0.18	0.15	0.019	0.007	85.56	10.55	3.89
41 A	3.61	2.84	2.82	0.001	0.022	99.19	0.01	0.79
54 A	10.45	8.23	8.21	0.001	0.021	99.74	0.003	0.26
57 A	10.76	8.47	8.45	0.001	0.014	99.82	0.004	0.17
61 A	6.81	5.36	5.34	0.003	0.021	99.52	0.065	0.41
62 A	6.22	4.90	4.89	0.001	0.013	99.73	0.003	0.27
Pezinok — Pernek crystalline area, outside productive zones								
K-4 A	0.58	0.46	0.43	0	0.03	93.70	0.1	6.2
K-5 A	0.84	0.66	0.63	0	0.03	99.70	0	0.3
K-7 A	0.34	0.27	0.26	0	0.01	92.50	0	7.5
K-20 B	0.48	0.38	0.34	0.03	0.01	89.70	8.1	1.8
K-21 A	1.70	1.34	1.33	0	0.01	99.10	0	0.9
K-22 A	5.20	4.10	4.09	0	0.01	99.70	0	0.3
K-25 B	10.50	8.28	8.25	0.01	0.02	99.70	0	0.3
K-30 A	0.25	0.20	0.19	0.04	0.06	95.10	1.9	3.0
5 OH A	0.66	0.52	0.49	0.01	0.021	94.07	1.91	4.02
9 OH A	17.14	13.50	13.48	0.001	0.016	99.87	0.01	0.12

Explanations: The total organic matter value represents the analytical results of C_{org} content multiplied by the conversion factor 1.27 determined for the Malé Karpaty Mts. black shales by Cambel — Šimánek — Kleinertová, (1975). C_{org} — organic carbon; C_{rez} — insoluble organic residue, residual carbon; C_{hum} — humin carbon; C_{bit} — bitumen carbon.

its study. The interest in this study is in the last years increasing. In Czechoslovakia, the research of organic matter in rocks is centralised in the Brno branch of the Geological Survey (Prague) and in the Moravian Oil Fields Company in Hodonín; the research is being started also in the Geological Institute of the Slovak Academy of Sciences in Bratislava.

Table 2

Sample number	Organic matter as a whole %	Weigh % C in rock				Carbon balance		
		C_{org}	C_{rez}	C_{hum}	C_{bit}	% C_{rez}	% C_{hum}	% C_{bit}
Harmónia Group								
K-1 A	0.14	0.11	0.05	0.04	0.02	46.90	34.8	18.3
K-6 A	0.27	0.21	0.20	0.005	0.005	95.00	2.5	2.5
K-9 A	4.85	3.82	3.41	0.37	0.04	89.80	9.6	0.6
K-10 A	3.58	2.82	2.80	0	0.02	99.70	0	0.3
K-12 A	6.26	4.93	4.92	0	0.01	99.90	0	0.1
K-13 A	0.81	0.64	0.63	0.01	0	99.50	0.5	0
K-24 A	0.24	0.19	0.17	0.01	0.01	86.80	3.1	10.1
K-26 A	1.24	0.98	0.97	0	0.01	98.70	0.7	0.8
K-31 A	0.30	0.24	0.23	0	0.01	96.10	1.0	2.9
K-32 A	0.38	0.30	0.28	0.04	0.13	94.30	1.3	4.4
1 OH A	1.48	1.17	1.15	0.006	0.016	98.05	0.56	1.4
6 OH B	0.99	0.78	0.77	0.001	0.01	98.59	0.12	1.29
8 OH A	17.68	13.92	13.90	0.005	0.01	99.89	0.04	0.07
Bratislava area								
K-3 A	0.59	0.47	0.47	0	0	99.60	0.1	0.3
K-17 B	0.04	0.03	0.02	0.01	0	99.60	0.25	0.15
K-18 A	1.80	1.42	1.41	0	0.01	99.10	0	0.9
K-19 A	0.35	0.28	0.27	0.01	0.004	94.50	4.1	1.4
K-27 A	0.09	0.07	0.06	0.002	0.008	85.40	3.3	11.2
K-28 A	0.04	0.03	0.01	0.01	0.008	27.00	44.4	28.9
K-29 A	0.09	0.07	0.05	0.01	0.01	72.40	11.3	16.3
2 OH A	9.02	7.10	7.07	0.013	0.012	99.64	0.19	0.17
Modra—Orešany area								
K-23 A	0.15	0.12	0.11	0.01	0	90.90	3.8	5.2
3 OH A	1.61	1.27	1.25	0.008	0.014	98.20	0.66	1.14
4 OH A	1.47	1.16	1.13	0.011	0.014	97.80	1.01	1.19

For *explanations* see Tab. 1.

Black shales of the Malé Karpaty Mts. crystalline complexes have been studied from the aspect of their organic matter contents by the authors Cambel — Šimánek — Kleinertová (1975) and Havlík (1977). It has been stated that the litofacial character of the studied rocks (grain-size of the rock and the grade of dispersion of organic particles) is the basic and primary decisive factor influencing the quantitative and qualitative composition of organic matter. The presented results are a supplement to the above-quoted studies and they were presented in thesis of Khun (1983).

Table 3

Average values of the analytical results of organic matter in black shales and bitumen coefficients

	Organic matter as a whole %	Weigh % of C in rock				Carbon balance			K_{bit}	n
		C_{org}	C_{rez}	C_{hum}	C_{bit}	% C_{rez}	% C_{hum}	% C_{bit}		
Productive zones	5.10	4.01	3.99	0.01	0.010	97.31	1.36	1.33	0.0540	17
Outside productive zones	3.77	2.97	2.95	0.09	0.011	96.31	1.21	2.48	0.0755	10
Harmónia Group	2.94	2.31	2.27	0.037	0.012	92.57	4.17	3.26	0.0778	13
Bratislava region	1.50	1.18	1.17	0.006	0.005	84.65	7.95	7.40	0.0943	8
Modra—Orešany	1.08	0.85	0.83	0.009	0.010	95.63	1.82	2.55	0.0222	3
Total	3.49	2.75	2.72	0.016	0.010	93.82	3.11	3.07	0.0870	51

Explanations: K_{bit} — bitumen coefficient; n —number of samples. For other explanation see Tab. 1.

For the study of dispersed organic matter in black shales were available 51 complete analyses. The results are in Tabs. 1 and 2. The samples were analysed by Kleinertová, Havlík and Khun; the analyses of separate C_{org} were carried out by J. Čáslavský from Geological Survey in Brno. A description of the analytical methods can be found in the papers of Šmeral (1966) and Kleinertová (1977).

Distribution of organic matter in rocks

As it follows from Tab. 3 of averange values calculated from the analytical results, the contents of organic matter in the studied areas vary within a relatively wide interval of values. The highest contents are in the Pezinok-Pernek crystalline complex, in the productive zones as well as in areas outside the productive zones of this complex; lower contents are in the Harmónia Group and in the rocks from the Bratislava region. This trend is confirmed also by calculation of the average C_{org} contents in the above-mentioned areas with the addition of other analytical data on C_{org} (results of the analyses are in the paper Cambel — Khun, 1983, where also the geological conditions in the area and the localisation of the samples are mentioned). When explaining this trend we have to take into consideration the fact that production and preservation of organic matter are dependent on the geological age; from the Paleozoic rocks, the richest in organic matter are the clay shales of Ordovician and Carboniferous (Ronov, 1958). This is in accordance with the latest studies of the ages of the rocks of the Malé Karpaty crystalline complexes (Bagdasarjan et al., 1983). The Harmónia Group and the shales of the Bratislava region are younger (Čorná, 1968, 1969;

Cambel — Čorná, 1974). Higher contents of organic matter in the Pezinok-Pernek crystalline complex could be connected also with basic volcanism of this area at the time of sedimentation of the studied black shales, when apparently a larger number of organisms perished.

Another striking indication of bonds of organic matter in black shales of the Malé Karpaty Mts. crystalline complexes was the dependence of the C_{org} contents on grain- size of the rocks. The group of fine-graind shales had a C_{org} content of about 2.8 per cent, medium-grained shales only 1.1 per cent (Cambel — Khun, 1983). This is a confirmation of the generaly known fact about the bonds of organic matter in sediments (e. g. Ronov, 1958; Gehman, 1962; Borddovskij, 1965; Davis, 1970; Obr — Burdová, 1982). Such an enrichment of fine-grained sediments with organic matter is apparent in the littoral zones of the sea, where the hydrodynamic activity of waves and currents is low.

Metamorphism of organic matter

a) Carbon balance

The carbon balance suggests (Tab. 3) that the samples represent highly to very highly metamorphosed organic matter; in most samples (about 70 per cent) the criteria of an advanced grade of metamorphism of organic matter in rocks of the Malé Karpaty Mts. determined by Kleinertová (1974), i. e. 95 per cent of C_{rez}, are exceeded. This value is not reached in the samples from the region of the Harmónia Group and from the Bratislava region. In this regions, the C_{hum} content is higher. On the other hand, in the Pezinok-Pernek crystalline complex the absence of humine-type matter points to a very advanced stage of carbonification of the organic substance. This process is characterised by removal of heteroatoms, condensation reactions and by formation of kerogene.

b) Microscopy in reflected light

The grade of carbonification can be measured by a microphotometric determination of vitrinite reflectance. This study of dispersed organic matter by microscopy in reflected light has been introduced in the last 10—15 years. In its application have merit especially Teichmüller (1971, 1974, 1979), Amosov — Gorshkov (1971), Bostick (1974) and others. In Czechoslovakia, this problems are studied by Malán (1976), Müller (1983) and Holubář (1975). The results are used when solving the problems of diagenesis, lower grades of metamorphism of sediments, thermal history of an area as well as problems connected with the prognoses of occurences of oil and gas deposits (Vassoyevich et al., 1969; Bartenstein — Teichmüller, 1974). The measurements themselves are carried out by a microscope equiped with a photometer. The measured objects have to meet the following criteria:

— highest possible homogenity
— the smallest admissible grain-size of organic matter is 2 μm
— the measured particles have to be present in the rock in sufficient quantity.

For the determination of reflectance 26 polished specimens have been chosen from the set of samples of the studied black shales. The measurements were carried out in the Central Laboratory of Organic Chemistry of Geological Survey in Brno. After a detailed study it was determined that in these samples the size and quantity of organic matter particles were not sufficient for the measurement of reflectance. Organic matter was in a too finely dispersed state and the criterion of the least grain size was not complied with Franců (1980).

Oil geochemistry of the studied rocks

When we evaluate the properties of the studied black shales related to oil--bearing and collecting we can notice that the contents of C_{org} in the majority of samples vary within the interval of values given by Šimánek (1965) for oil-bearing rocks, i.e. 0.6—3 per cent. Some authors present different C_{org} contents as criteria for oil-bearing rocks, e. g. Ronov (1958) 0.4—1.4 p. c.; Schrayer — Zarella (1963, 1966) 1—3 p c.; Hunt (1979) 1—1.5 per cent C_{org}. According to the contents of organic carbon it would be possible to classify the studied rocks as potentially oil-bearing. Though, it is necessary to realise that organic matter in black shales of the Malé Karpaty Mts. crystalline complexes was carbonified, and organic matter metamorphosed in such way is not able to produce oil carbohydrates. The amount of organic carbon which is necessary for the production of oil carbohydrates depends also on the quality of organic matter in the sediment. If the organic carbon is largely in the form of carbonified substance, which is the case of the studied rocks, it is no possible to consider it autochtonous, i.e. originating in situ. Baker (1962, 1972) explains on the example of the Cherokee formation why there is different production of oil from different rocks with the same contents of organic matter. The clay rocks of the Cherokee formation contain frequently a substantial amount of redeposited C_{org} of terrigenous origin with very low production of oil carbohydrates. On the other hand, carbonate rocks contain only amorphous organic substance which originated from algae, represented by kerogene which produces the most carbohydrates. Even though pyrolyses have not been carried out up to now, most probably it is possible to class the kerogene of black shales of the Malé Karpaty Mts. crystalline complexes weith the type III, according to the interpretation of Tissot et al. (1974), i.e. kerogene with a higher amount of oxygen and aromatic carbohydrates and with limited production of oil and gas, which could originate from the detritus of higher plants.

The properties of the collector strata related to oil-bearing can be judged according to the bitumen coefficient (Šimánek, 1965).

$$K_{bit} = \frac{C_{bit}}{C_{rez} + C_{hum}} \times C_{org}$$

Šimánek put forward the K_{bit} values 0.5—4.0 for the productive zones of the rocks of the Danube plain, 0.1 for sediments next to them and for rocks only partially enriched by immigrated bitumens values less than 0.05.

When we evaluated the relation of C_{bit} and C_{org}, there was confirmed a hyperbolical dependence (V a s s o y e v i c h — K a r c e v a, 1971; Fig. 1). According to the quoted authors, from this relation it is possible to deduce that the majority of bitumens are geneticaly homogenous and migration did not cause any substantial heightening of the C_{bit} level. At the same time, the hyperbolical dependence proves that the matter included in C_{bit} is largely

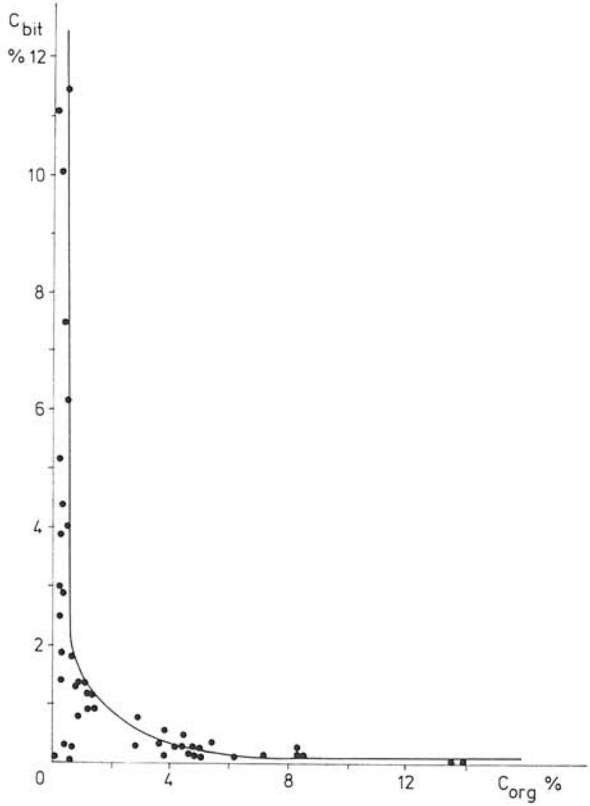

Fig. 1. Hyperbolical relation of C_{bit} and C_{org} in the studied samples of black shales.

"remanent", i.e. it resisted the differentiation and migration processes. Highest bitumen coefficients have the samples from the Bratislava region (Tab. 1), that is 0.0943. This value is approaching the data of Š i m á n e k (1965) for sediments directly next to productive horizons of the Danube plain (0.1). This can be applied also to the studied samples of the Malé Karpaty Mts. region, from NW of Bratislava (the region Lamač, Devín, Záhorská Bystrica, Borinka), which are the ones nearest to the productive zones of the Záhorská plain. Generally, the studied black shales of the Malé Karpaty Mts. crystalline complexes can be, according to the values of C_{bit} and K_{bit}, classified as poor in bitumens; with regard to the analysed samples, this rocks cannot be considered as potential source-rocks of oil.

Table 4

CPI indexes and other n-alkanes and n-monocarboxyle acids (n-MCA) ratios.

Sample number	n-alkanes				n — MCA	
	CPI	$\frac{C_{15}}{C_{17}}$	$\frac{C_{21}+C_{22}}{C_{28}+C_{29}}$	$\frac{C_{23}-C_{30}}{C_{14}-C_{30}}$	CPI	$\frac{C_{16}+C_{18}}{2\,C_{17}}$
1-OH A	0.95	—	0.3	89.6	5.6	9.8
2-OH A	0.95	1.06	11.9	40.3	2.1	0.78
3-OH A	0.95	0.19	26.9	45.6	2.0	4.6
4-OH A	0.99	0.17	3.8	54.2	3.3	5.3
5-OH A	0.97	0.14	1.9	60.9	—	—
6-OH B	0.92	0.42	6.5	57.5	—	—
7-OH A	1.04	0.13	14.4	24.4	0.7	3.0
8-OH A	1.01	0.33	10.9	14.6	2.6	6.2
9-OH A	0.99	0.32	7.8	23.6	2.7	14.1
10-OH A	0.93	0.31	2.8	41.7	—	—
41 A	0.96	0.18	6.2	30.9	—	—
54 A	0.98	0.07	1.0	57.7	5.6	11.4
57 A	0.93	0.78	2.6	28.5	—	—
61 A	0.86	0.71	50.0	13.3	—	—
62 A	—	—	—	—	4.2	15.5
37 A	0.86	—	7.6	35.1	1.3	7.9
29 C	—	—	—	—	3.7	44.0
32 C	—	—	—	—	1.9	43.0
K-1 A	3.14	—	0.2	93.8	—	—
K-9 A	1.01	0.12	13.6	89.8	—	

Explanations: The calculation of CPI indexes see in Hunt (1979). Modified according to Havlík (1977).

Source organic matter; paleogeography of the sedimentation environment

a) n-alkanes

The original organic matter has been subjected to many influences and has gone through a complex of changes from the time of sedimentation to the recent state. The effort to explain this processes leads to the study of absolute contents of individual components and their distribution (i.e. CPI index — that is Carbon Preference Index, the ratio of the number of odd members to even members in the molecules of n-alkanes).

In the source organic material, odd n-alkanes prevail over evene n-alkanes. The first CPI coefficient, which characterised the advance of changes ("maturity"), was derived by Stevens et al. (1956). For plants, the maximum value is C_{23} to C_{36}, while C_{15}, C_{17} and C_{19} are the maximum values of the sapropel carbohydrates, i.e. marine source material (Clark — Blumer, 1967). As a rule, littoral marine sediments always contain mixed marine and terrigenous organic matter, as it is indicated by the presence of both groups of carbohydrates, i.e. C_{23-26}, and C_{15}, C_{17} and C_{19}. E.g. in the sediments from Baffin bay was determined bimodal type of distribution of n-alkanes with two maximums which correspond with carbohydrates with a chain length characteristical for compounds produced by marine as well as terrigenous organisms (S e v e r, 1970 in H u n t, 1979).

For the study of n-alkans from black shales of the Malé Karpaty Mts. crystalline complexes were available 17 determinations of the compounds by the gas-chromatography method carried out by M. H a v l í k; the results are

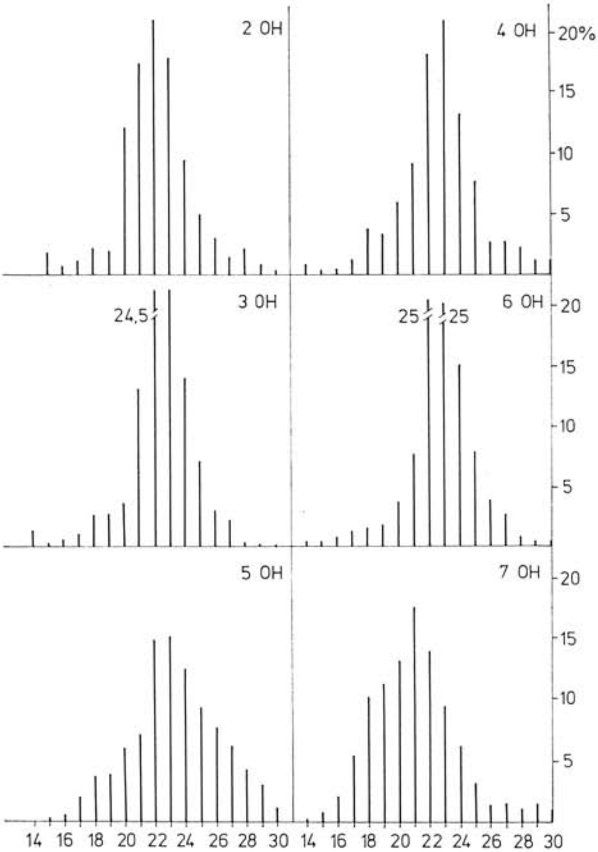

Fig. 2. Distribution of n-alkanes in the studied black shales. According to H a v l í k (1977).

in Tab. 4. It is apparent that in some samples the member C_{22} is clearly predominant (Figs. 2—4), in the rest of the samples it is always more or less prevalent and in no one of the samples is its content less than the average of other members. It is clear that the special position of C_{22} has certainly greater importance and it is necessary to evaluate it from many different aspects. The prevalence of C_{22} in organic matter of sediments was determined already by S c h e n c k (1968) together with the higher contents of C_{22} in the peripheral parts of the sea, where there are transported remnants of terrigenous plants. Similarly, prevalence of C_{22} was also mentioned by Powel — McKirdy (1973) and in brakic waters it was determined by Shimoyama — Ponampentura (1975).

In all studied samples the CPI index varies within a very narrow interval of values. While in recent sediments there is a marked prevalence of odd

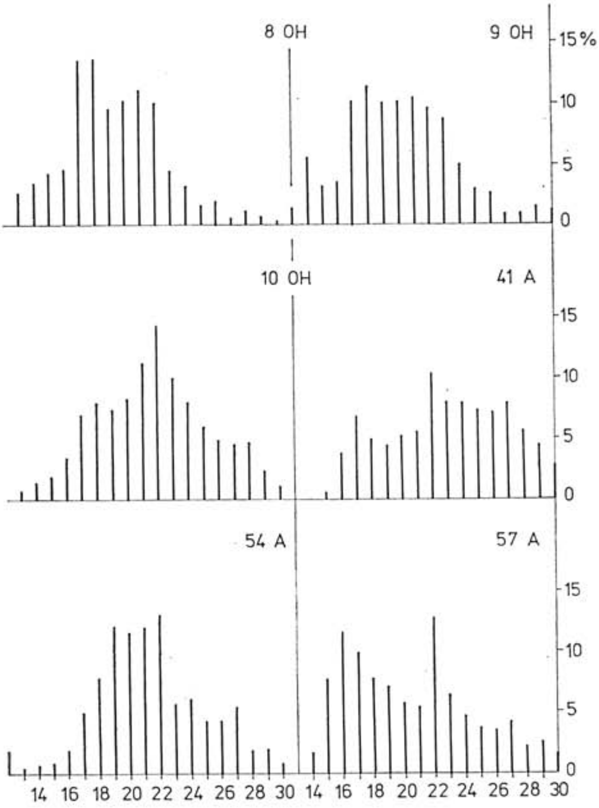

Fig. 3. Distribution of n-alkanes in the studied black shales. According to Havlík (1977).

members, in older sediments this difference of the contents of even and odd members disappears and in our case in the majority of samples the even members are prevalent (Tab. 2), what is true not only for samples with high contents of C_{22}. The origin of organic matter can be also deduced from the ratio C_{22-30}/C_{14-30} which varies within a wider interval of values. The limit of prevalence of terrigenous source matter over marine source is considered to be at the value of the ratio of approximately 50 per cent. On the basis of this ratio we can conclude that organic matter in the studied black shales had two sources.

In view of the above-mentioned facts we can make the conclusion that organic matter of black shales of the Malé Karpaty Mts. crystalline complexes sedimented in the conditions of the littoral zone of the sea, with a contribution of terrigenous plant matter, which mixed with the autochtonous marine matter. This does not contradict the results of palynological studies (Čorná, 1969; Cambel — Čorná, 1974). On the same localities of black shales as the

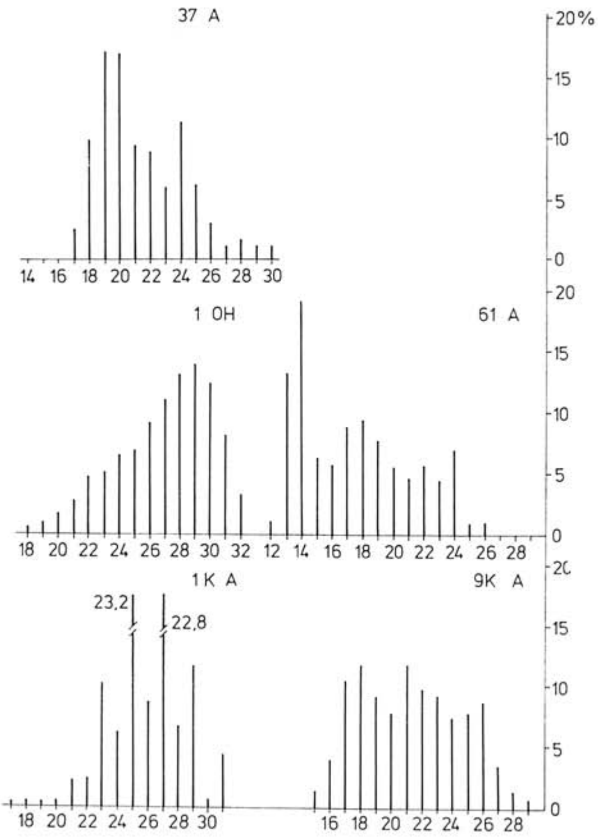

Fig. 4. Distribution of n-alkanes in the studied black shales. According to Havlík (1977).

ones studied by the authors of this paper were found remnants of vascular terrigenous plants *(Ptéridophytes, Lycopodiales, Cordaitales)* as well as remnants of bentonic *Scolecodontes.* The prevalence of the C_{22} member in the homological order of n-alkanes is according to. Shimoyama — Ponampentura (1975) caused by transport from continent, because with the depth in which the rock sedimented the ratio $2C_{22}/C_{21} + C_{23}$ decreases.

b) Monocarboxyle acids

Monocarboxyle acids (MCA) are because of their stability and presence in all living organisms as well as in the structure of plants (Bouška, 1977) geochemically very important compounds. They begin to appear in sediments from the time of the beginning of the existence of life on Earth (Han — Calvin, 1969). The role of MCA in organic matter cycle in sediments, although it has been the subject of very intensive studies especially in the last 10—15 years, has not been sufficiently explained. Changes caused by microorganisms especially in the early diagenetic stage as well as thermal changes are supposed to take place.

MCA in black shales of the Malé Karpaty Mts. crystalline complexes have been determined by Havlík. The distribution of n-MCA confirmed other studies in this area, except for one sample (7-OH-A), the CPI indexes are greater than 1 (note: for n-MCA, the CPI index is calculated as the ratio of even n-MCA to odd MCA in the whole range of measurements). The prevalence of even members over odd members in the composition of the homological order is in accordance with the data on natural material. The studied MCA had members in the range C_{10} to C_{28}. The contents of odd members were very low and practicaly uniform in the whole range of measurements. The member C_{16} prevails, the content of C_{18} is lower than expected. The presence of C_{22} indicates a terrigenous source of material in accordance with the distribution of n-alkanes.

Another applications of C_{org} contents in geochemical interpretation of the genesis of the Malé Karpaty Mts. black shales are presented in other papers in this review as well as in the paper Cambel — Khun (1983).

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