

<https://doi.org/10.3176/oil.1998.3.01>

VARIETIES OF SULPHUR IN THE ALGINITE SEQUENCE OF KÖSSEN FACIES FROM THE BOREHOLE REZI Rzt-1 (W-HUNGARY)

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During the Upper Triassic, in the western part of Hungary the paleogeographical conditions were favourable for the deposition of formations with high organic matter content. The best reference section of the Kössen Formation can be found in the Keszthely Mountains at Rezi Village: the borehole Rzt-1.

The alginite sequence of the Kössen facies in the borehole Rzt-1 can be characterized by a moderate organic matter content (average $C_{org} = 6.1\%$), relatively high carbonate-rich mineral part (average $C_{carb} = 6.1\%$) and relatively low total sulphur content (average $S_t^a = 1.6\%$). On the basis of comparison of the previous average values derived from the qualitative analysis of the alginite-bearing samples from the borehole Rzt-1 with others for other Hungarian and Slovakian alginites, it can be established that the carbonate carbon and total sulphur content is higher in the Rezi samples, than in the other Hungarian and Slovakian ones.

Two changes can be observed in the distribution among the varieties of sulphur in the studied alginite samples from the borehole Rzt-1. Above the interval 181.9-199.6 m the following order:

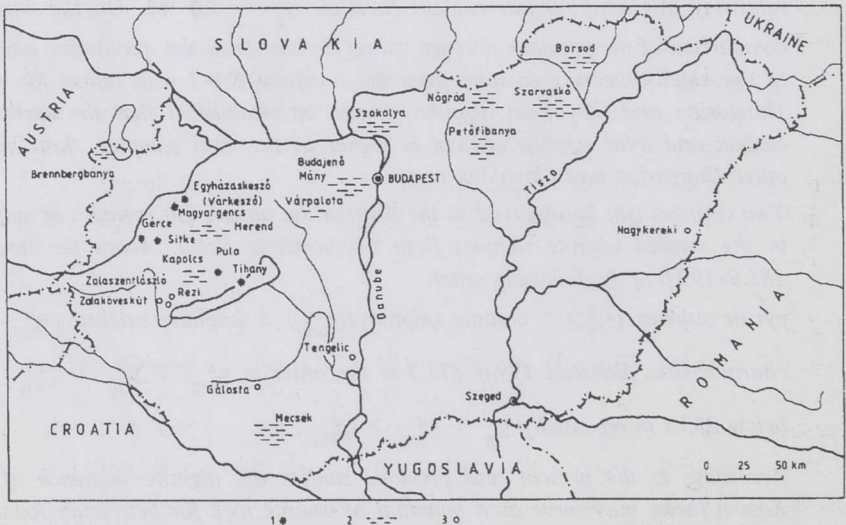
pyritic sulphur (S_p^a) > organic sulphur (S_{org}^a) > sulphate sulphur ($S_{SO_4}^a$) is characteristic. Between 199.6-233.3 m the order is $S_{org}^a > S_p^a > S_{SO_4}^a$ and below 233.3 m repeatedly $S_p^a > S_{org}^a > S_{SO_4}^a$.

According to the present and previous studies the alginite sequence of the Kössen facies may serve as a potential oil-source rock for petroleum found in the Zala basin. Presumably, the $\geq 20\%$ of the sulphur content belonging to the Nagylengyel-type petroleum might have originated in the Kössen Formation.

Introduction

As a result of the research work during the reambulation of geological mapping in Hungary, the first alginite (oil shale) deposits were discovered at Pula (Balaton Highlands) in 1973 and at Gércse (Vas county) in 1974 [1]. According to geological studies and results of the diatomological [2], palynological [3], petrological [4], and mineralogical [5] examinations, Jámbor and Solti got to the conclusion that these oil shales were deposited in the basalt crater lakes that had developed in the Pliocene time ($3.05\text{--}5.34 \pm 0.93$ m.y. [6]), when special conditions enabled the formation of this type of sedimentary facies. In both oil shales the organic matter is originated mainly from well-preserved colonies of *Botryococcus braunii* (a widespread, extant planktonic green alga, which can adapt to large salinity variations [7]), which is the main component of alginitic rocks all over the world.

In the Triassic, the paleogeographical conditions were favourable for the deposition of formations with high organic matter content. Thus, the Ladinian black shale sequences of the Mecsek Mountains, the Karnian marl series of the Transdanubian Central Mountains and the Kössen beds may be promising from this point of view. The Triassic deposits with the alginite layers were revealed by the boreholes Zalaszentlászló Zl-1 and Rezi Rzt-1 in 1983. The *Botryococcus*-bearing calcareous and argillaceous rocks were classified as alginite [8]. At Rezi village (the bottom of the borehole Rzt-1 is 270 m), the alginite sequence of the Kössen facies was formed in a contemporaneous lagoon. The organic geochemical studies of



Oil shale occurrences in Hungary [8, with some modifications and additions]:
 1 - maar-type volcanic deposits, 2 - lagoon-type oil shale indications and deposits associated with coal beds, 3 - oil shale indications in boreholes

the Rezi formation were carried out by Hetényi and Pápay [9]. By using Rock Eval pyrolysis technique, Miss Hetényi got to the conclusion that the organic matter is immature, has not yet reached the main oil generation zone and in terms of the Tissot's classification, it can be assigned to type II or, in a few cases, to type I.

As a result of subsequent Hungarian oil shale explorations performed since 1973, several oil shale deposits and indications have been discovered. With regard to the sedimentary environments, two main genetic groups can be distinguished [10] (Figure):

- Maar-type oil shale deposits formed in contemporaneous volcanic craters
- Lagoon-type oil shale deposits accumulated in a contemporaneous intramontane lagoon

The features of these two types of deposits have been summarised by Ravasz and Solti [11].

The characteristics of the Hungarian oil shales make them unsuitable for commercial hydrocarbon recovery or for being used as fuel in power plants, their average grade being low (Fischer assay oil yield 4-8 %). However, they are suitable for application first of all in agriculture as fertilizers and organic ameliorants [12]. The reports of the Alginite Meeting dealt with the possibilities of the agricultural utilization of oil shales [13] and analogous proposals can be found in other issues, too [14, 15].

The sulphur content of Hungarian alginites is interesting from both the agricultural (the available amount of the soluble sulphur for the plants) and the industrial (in researches of the coal briquetted with alginite [16]) point of view.

The sulphur in oil shale, as in coal, is commonly classified into inorganic and organic sulphur. The inorganic sulphur occurs mostly as iron disulphides, FeS_2 , with a small amount occurring as sulphates. Various organosulphur compounds including thiophenes and his derivatives [17], mercaptans, sulphides, disulphides [18] are also present in oil shales.

Pyrite is a common and widespread authigenic mineral in sedimentary rocks. Based on field observations and experimental studies, it is generally accepted that the iron disulphides form either via (1) replacement of FeS precursor or (2) FeS_2 nucleation. In many cases the first product is iron monosulphide phase and subsequent reaction of this phase with elemental sulphur or sulphur equivalent (depending on pH of polysulphides and thiosulfate [19]) will produce finally pyrite. However, in various sedimentary environments, especially in salt marshes [20-23] and in freshwater lakes [24], pyrite precipitates directly without any monosulphide intermediates.

Distribution of Sulphur Varieties in Alginite from the Borehole Rzt-1, Including Data for Moisture, Ash, Carbonate and Organic Carbon Content

Symbol	Interval, m	Kerogen*, type	H ^a , %	C _{carb} *, %	C _{org} *, %	S _p ^a , %	S _{SO₄} ^a , %	S _{org} ^a , % (diff.)	S _p ^{daf} , %	S _{SO₄} ^{daf} , %	S _{org} ^{daf} , % (diff.)
R-14	118.2-123.0	II	1.6	7.0	4.8	0.4	0.2	0.2	1.8	0.45	0.45
R-18	146.0-150.0	II	2.1	5.2	7.9	2.2	1.1	0.2	6.8	0.6	2.8
R-22	159.5-164.2	II	3.1	4.0	5.5	2.1	1.4	0.2	8.4	0.8	2.0
R-26	178.7-181.9	II	2.9	3.6	3.6	1.5	1.0	0.4	6.9	0.5	1.8
R-32	199.6-205.0	I-II	2.9	8.6	2.7	0.8	0.3	0.1	2.2	0.3	1.1
R-34	211.7-218.0	I	2.5	6.7	10.3	2.2	0.6	0.1	5.6	0.3	3.8
R-36	211.7-218.0	II	2.5	7.7	8.6	1.5	0.4	0.1	3.8	1.0	2.6
R-41	223.4-233.3	II	2.6	5.7	8.6	1.9	0.7	0.2	6.1	0.6	3.2
R-42	223.4-233.3	I-II	3.5	7.0	5.6	1.5	0.4	0.1	4.5	0.3	3.0
R-48	241.4-245.8	II	2.8	3.2	6.5	2.3	1.3	0.2	9.0	0.8	3.1
R-53	265.0-266.5	I	2.4	8.2	2.6	0.6	0.4	<0.1	1.6	-0.2	-0.3

H^a - analytical moisture wt%; A^d - ash (air dried sample) wt%; C_{carb} - inorganic or carbonate bond forms carbon content wt%; C_{org} - total organic carbon content (TOC) wt%.

S_p^a, S_{SO₄}^a, S_{org}^a - total, pyrite (+ sulphide), sulphate, and organic (by difference) sulphur content in air dried sample.

S_p^{daf}, S_{SO₄}^{daf}, S_{org}^{daf} - total, pyrite (+ sulphide), sulphate, and organic (by difference) sulphur content in dry, ash-free basis.

* The data from the reference [9].

In the same sediment-porewater systems, many components (e.g. elemental sulphur, iron monosulphides, pyrite, dissolved polysulphides, ferrous ion, hydrogen sulphide) can co-exist, thereby obscuring the mechanism(s) by which pyrite is produced [21]. Studies on distribution of sulphur in freshwater sediments revealed that organic sulphur forms are dominant [25]. In some freshwater lakes, however, inorganic S forms predominate [26]. The formation mechanism of pyrite in freshwater systems is thought to be similar to that in marine sediments [19]. In addition, the hydrogen sulphide (and/or polysulphides) as well as elemental sulphur are important intermediates for pyrite and organic sulphur. Numerous investigators have reported the early diagenetic sulphur enrichments of macromolecular sedimentary organic matter [27-31 and others].

The purpose of the present paper is to determine the distribution of sulphur among the different bond forms in alginites from the borehole Rzt-1 to enrich the sulphur data basis of the Hungarian organic-rich sediments. I also attempt to compare some characteristics of alginites from Rezi with the other, previously examined Hungarian alginites from Gércé and Pula [32], as well as maar-type alginites (Pontian; 6-6.5 m.y.) from Pinciná (S-Slovakia) [33].

Stratigraphic Position of Studied Area

Significant changes took place in the evolution of the extremely extensive Upper Triassic carbonate platform at the western edge of the Tethys (Dachstein platform), in the middle part of the Norian. In the external zone of the platform, a large semirestricted back-platform basin developed: the Kössen Basin, where the multiphase basin evolution was initiated by the formation of restricted lagoon in the Rezi Dolomite area. Later on, as a consequence of the fine terrigenous input, the carbonate platform was drowned in the western part of the Transdanubian Range, and a restricted, oxygen-depleted basin developed giving the sedimentary environment of the Kössen Formation [34].

The early phase of the Dachstein platform development is represented by the Main Dolomite (Hauptdolomite) Formation. The Rezi Dolomite Formation rests on the Main Dolomite with transitional beds at its base, and passes continuously upwards into the dark-grey pelitic Kössen Formation. The boundary between the last two can be drawn where the calcareous, clayey marl becomes dominant. On the basis of conodonts, the base of the Rezi Dolomite Formation can be assigned to the upper part of the Middle Norian or to the lower part of the Upper Norian [35].

The main development of the Kössen Formation lies in the western part of the Transdanubian Range; it thins towards the east where it is

interbedded with the Dachstein Limestone. Westward in the basin areas of the Transdanubian Range structural unit (North Zala Basin) it is known from boreholes (e.g. Nagylengyel, Szilvágy, Nagytilaj). The best reference section of the Kössen Formation in the Keszthely Mountains at Rezi is the borehole Rzt-1, which penetrated the formation about 180 m. Based on foraminiferas the cored sequence can be assigned into the Upper Norian-Middle Rhaetien [36].

Samples and Analytical Methods

Eleven alginite-bearing samples have been examined from the borehole Rzt-1 from depths between 118 to 266 m. The air-dried samples were ground to grain size $d \approx 200 \mu\text{m}$ in an agate ball mill.

The total carbon content was measured in samples free of carbonate at 1000 °C under intense oxygen flow by combusting in a Carmhograph-8 (Wösthoff) equipment.

The CO₂ content of the samples was determined by gasvolumetric method. The C_{carb} data were calculated from the CO₂ content.

The total sulphur and sulphate sulphur contents were converted into BaSO₄ and weighed gravimetrically.

The pyritic sulphur content was determined by iodometry. The above-mentioned analytical methods are described in detail in a previously published study [32].

Results and Discussion

The results of the experiments are summarized in the Table.

The alginite sequence of the Kössen facies in the borehole Rzt-1 can be characterized by moderate organic matter content (average $C_{org} = 6.1 \%$), relatively high carbonate-rich mineral part (average $C_{carb} = 6.1 \%$) and relatively low total sulphur content (average $S_t^a = 1.6 \%$).

On the basis of comparison of the average values derived from the qualitative analysis of the alginite-bearing samples from the borehole Rzt-1 with others for the other Hungarian alginites (Gérce and Pula) as well as with Slovakian alginite from Pinciná (see below), it can be established that there are differences in the carbonate carbon and total sulphur content.

Alginite	C_{org}	C_{carb}	S_t^a
Gérce [32]	3.4	2.3	0.5
Pula [32]	21.1* ¹ -27.4* ²	2.6* ¹ -2.8* ²	0.4* ² -0.5* ¹
Pinciná [33]	8* ³ -9* ⁴	0.3	0.5* ³ -0.6* ⁴

* Average samples from the mine*¹, borehole Put-7*², boreholes VPA-1, 3-5*³, and borehole VPA-7*⁴.

The results indicate that the Rezi alginite contains more C_{carb} and S_7^a than the Hungarian and Slovakian ones. It is due to the different sedimentological conditions, namely the maar-type alginites (Gérce, Pula, Pinciná) accumulated in a freshwater system in contrast with the lagoon-type oil shale (Rezi) which are marine sediments. In general, the common characteristic of freshwater sediments is their low total sulphur content. In freshwater systems, the low concentrations of sulphate limit bacterial sulphate reduction.

The distribution of sulphur among different bond forms in the alginite sequence from the borehole Rzt-1 is the following in order:

- In four samples (R-14, -18, -22, -26):

Pyritic sulphur (S_p^a) > organic sulphur (S_{org}^a) > sulphate sulphur ($S_{SO_4}^a$)

- In five samples (R-32, -34, -36, -41, -42):

$S_{org}^a > S_p^a > S_{SO_4}^a$

- In two samples repeatedly:

$S_p^a > S_{org}^a > S_{SO_4}^a$

The first change in the distribution among varieties of sulphur is connected to the Norian/Rhaetian boundary at the interval 181.9-199.6 m. According to palynological studies, the Norian/Rhaetian boundary can be drawn within the interval 186.4-197.2 m [37]. Presumably, the second change in the sulphuric distribution is associated with the Rezi Dolomite F.-Kössen Formation transition, below 233.3 m.

It is known that the Kössen Formation reaches a significant thickness (200-500 m) in the Keszthely Mountains and westward in the Northern Zala Basin. More and more evidences reinforce the hypothesis that the Kössen Formation may be the oil-source rock of petroleum found in the Zala basin. The oil-source rock correlations based on carbon isotope ratios makes it likely that the bulk of the known oil reserves, belonging to the Nagylengyel-type (paraffinic) was originated in Upper Triassic (Rhaetian) source rocks during the Neogene burial [38].

The results of the present study are not in contrast with the above-mentioned hypothesis. The transfer of sulphur from the original shale to the produced oil depends mainly on the share of organic sulphur in its total sulphur [39]. The total sulphur content of the studied alginite samples is within a range of 0.6-2.3 %, and the content of organic sulphur varies between -0.1-1.5 %. In the Rezi samples the share of organic sulphur in the total sulphur amount is within a range of 16.7-68.2 %. In the five samples (R-32, -34, -36, -41, -42), the share of organic sulphur is 50-70 %. Considering the results connected with the experiences in Fischer retorts presumably, ≥ 20 % of sulphur from the original alginite might have transferred into the petroleum found in the Zala basin.

Conclusions

The total sulphur content (S_T^a) of the lagoon-type alginite sequence of the Kössen facies in the borehole Rzt-1 is relatively low, being on an average 1.6 %. At the same time, the content of S_T^a is higher compared with the shales of the other Hungarian and Slovakian maar-type oil shale deposits.

Two changes can be observed in the distribution among varieties of sulphur in the studied alginite samples from the borehole Rzt-1, that are connected to the Norian/Rhaetian boundary and the Rezi Dolomite F.-Kössen Formation transition.

According to the present study, the alginite sequence of the Kössen facies may serve as a potential oil-source rock of petroleum found in the Zala basin. Presumably, the ≥ 20 % of the sulphur content belonging to the Nagylengyel-type petroleum might have originated in the Kössen Formation.

Acknowledgments

This work has been supported by the Scientific Research Fund (Hungary), Grant No. T 023050.

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Presented by J. Kann

Received May 17, 1998