Bulletin of Geosciences, Vol. 80, No. 1, 23–32, 2005
 ISSN 1214-1119

The distribution of sulphur forms in high-S coals of the Maritza West Basin, Bulgaria

Irena Kostova [1] – *Stefan Marinov* [2] – *Maya Stefanova* [2] – *Kalinka Markova* [1] – *Vladka Stamenova* [2]

[1] "St. Kliment Ohridski" University of Sofia, Department of Geology and Paleontology, Tsar Osvoboditel blvd, 1000 Sofia, Bulgaria.
E-mail: irenko@gea.uni-sofia.bg
[2] Institute of Organic Chemistry, Bulgarian Academy of Sciences, 1113 Sofia, Bulgaria. E-mail: stif@bas.bg

Abstract. The present article concerns the Upper Miocene coal of the Kipra seams from the Maritza West Basin. A number of petrological investigations were carried out, including lithotype, quantitative maceral and mineral analyses, proximate and ultimate analyses, X-ray, and optical and scanning electron microscopy (SEM). We also examined the mineral matter from above, below, and within the coal layers. The proportions of sulphur in total (S_t), pyritic (S_p), organic (S_o) and sulfate(S_s) forms were also determined. The investigated coals have high sulphur contents, commonly above 7wt%, and can range from about 4wt% to more than 11wt%. The average sulphur proportions are 7.5wt% (4.1–13.5wt%) total sulphur, 1.6wt% (0.6–3.2wt%) pyritic sulphur, 4.2wt% (1.8–6.0wt%) organic sulphur, and 1.7wt% (0.3–2.9wt%) sulphate sulphur. Pyrite and gypsum are the major sulphur-bearing minerals. The syngenetic pyrite occurs mainly as isolated or clustered framboidal bodies and well-shaped euhedral crystals along stratification bands among humodetrinite and humotelinite. Epigenetic massive and infilled cell lumens of pyrite have also been observed. Well shaped prismatic crystals of gypsum were found as lenses, fine crusts, or fillings in cross bedding marks and cracks in the coal. Total sulphur slightly decreases from the base to the top of the coal bed. The highest content of total, sulphate, and pyritic sulphur near clay coal layers has been established. The highest content of organic sulphur has been found within the middle part of the coal bed.

Key words: coal, sulphur in coal, Maritza West Basin, Bulgaria

Introduction

The Maritza West Basin is situated in the Thracian coal province of central Bulgaria. The age of the coal-bearing strata is Late Oligocene to Late Miocene. There are two productive coal-bearing layers of the basin, the Brod and Kipra (Fig. 1). The Brod coal is sub-bituminous in rank, while that of the Kipra level coal is lignite (Kamenov and Panov 1976).

The economically important coals are associated with the Kipra level, in which two lignite seams occur: the Kipra and Havuzki (Fig. 2). The coal-bearing sediments are slightly folded. There are a number of tectonic faults in the area of the Maritza West Basin. The coal seams are compositionally complex, and are comprised of coals and shale coals, with some clay and sand layers. The coals are characterized by high sulphur and ash contents; there are therefore environmental problems related to their combustion in power plants.

The coals are typical autochthonous. They were formed from a grass-frutex population of the types *Nelumbo-Salvinia-Stratiotes* and *Phragmites-Typha* (Siskov et al. 1982).

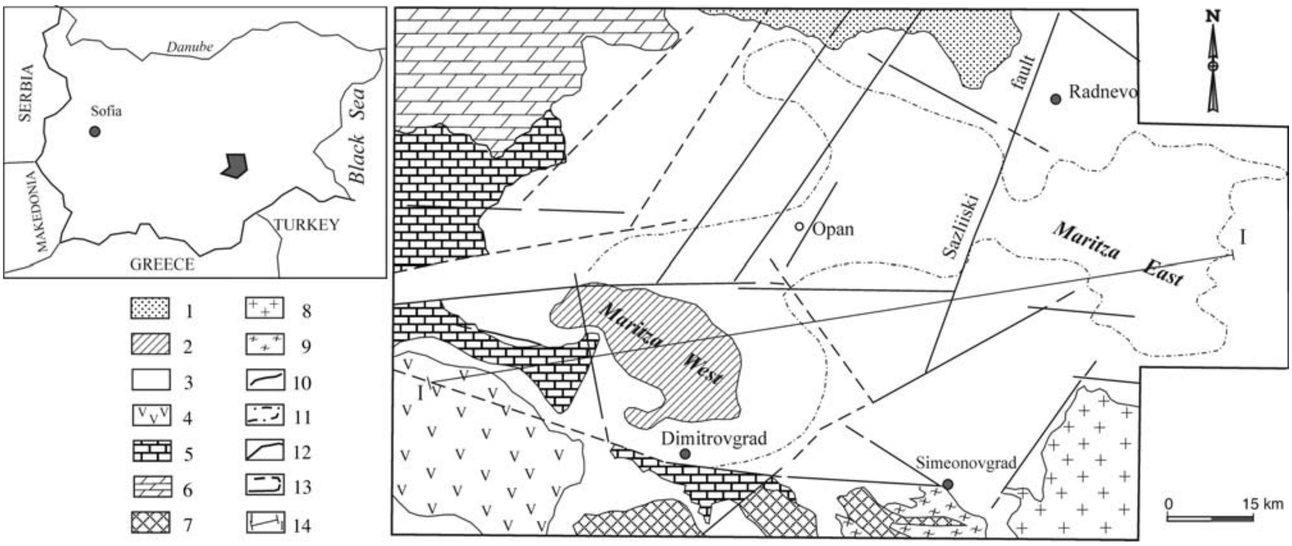


Figure 1. Geological map of the Maritza West Basin and its surrounding areas (modified after Kamenov and Panov 1976).
1 – Pleistocene, 2 – clays and sands, 3 – variegated clays and sands, 4 – Paleogene volcanics, 5 – Paleogene sediments, 6 – Upper Cretaceous sediments, 7 – Triassic sediments, 8 – Haramian granites, 9 – pre-Mesozoic crystalline shists and granite-gneisses, 10 – boundary of the Kipra coal-bearing Member, 11 – boundary of the Brod coal-bearing Member, 12 – pre-Pliocene faults with neotectonic activity, 13 – post-Pliocene faults, 14 – prospecting line.

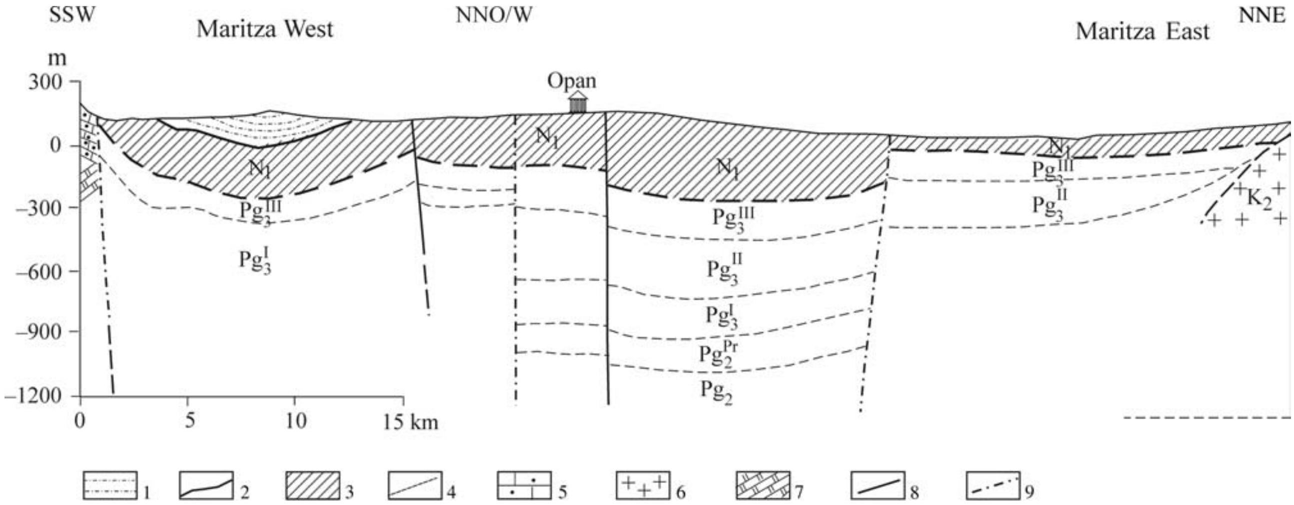


Figure 2. Cross section of the Zagora depression, showing the relation between the Maritza West and Maritza East coal basins, and the correlation between the Kipra and Brod (Lower Maritza) coal-bearing sediments (after Kamenov and Panov 1976).
1 – Miocene clays and sands, 2 – Kipra coal-bearing Member (includes Kipra and Havuzki coal seams), 3 – variagated clays and sands, 4 – Brod coal-bearing Member; 5 – pre-Miocene basement, 6 – Late Cretaceous granites, 7 – Triassic, 8 – post-Pliocene faults, 9 – pre-Pliocene faults.

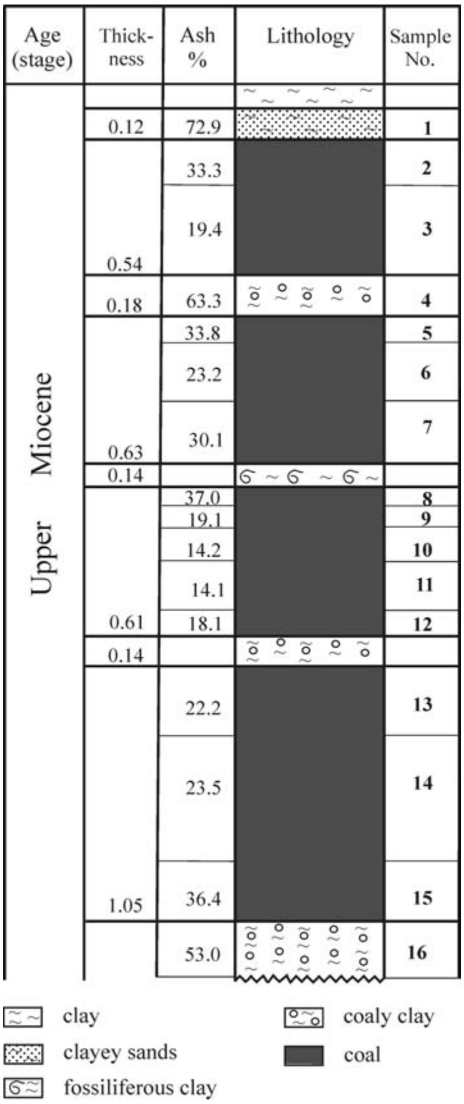


Figure 3. Generalised lithostratigraphic chart of the Kipra coal bed from the Maritza West Basin showing the sampling locations.

General information about the quality, mineral matter, maceral composition, and oxidation level of the lignite from the Maritza West Basin have been reported by Plachkov and Stoikova (1961), Kortenski and Dimitrov (1990), Vassilev and Vassileva (1998), and Markova and Kortenski (2001).

The purpose of the present study is the determination and detailed description of the content, mode of occurrence, and vertical distribution of the different types of sulphur in these coals (total, pyrite, sulfate, and organic), and the characterization of the major and minor sulphur-bearing minerals, including their morphology and relation to various coal macerals. Some genetic peculiarities concerning high total sulphur content, especially high sulfate sulphur, have also been noted.

Materials and methods

These investigations were carried out on sixteen samples taken from various layers of the whole Kipra coal seam (Fig. 3).

The amounts of total, sulphide, sulphate, and organic sulphur were defined by standard chemical analyses according to ISO 1462-86 and Bulgarian standards. The inorganic composition of the coal samples, including sulfide and sulfate minerals, was determined by crystallographic and optical studies, X-ray powder diffraction analyses, and by scanning electron microscope (SEM) equipped with EDAX X-ray microanalyser with Si(Li) detector. The quantitative maceral analysis was carried out using an Amplival POL-U microscope with an Eltinor automatic point counter, in reflected polarised light with oil immersion lenses. The proximate and ultimate analyses were done according to Bulgarian standards.

Table 1. Chemical characteristics and sulphur content of coal from Maritza West Basin, Bulgaria

Samples No.	Proximate analyses (wt%)			Ultimate analyses, daf (wt%)					Sulphur forms, daf (wt%)			
	A^d	V^{daf}	W^a	C	H	N	S	O	S_t	S_o	S_p	S_s
1	72.9	–	8	–	–	–	–	–	5.9	1.6	3.0	1.0
2	33.3	–	10.6	64.6	5.9	0.6	3.0	25.9	6.3	3.5	1.0	1.8
3	19.4	56.5	13.3	63.7	5.0	0.6	3.2	27.5	5.2	3.1	1.1	1.1
4	63.3	–	8.3	60.8	6.2	0.6	2.8	29.6	4.1	2.7	1.1	0.3
5	33.8	–	11.2	62.7	6.0	0.6	3.3	27.4	7.8	3.6	1.8	2.5
6	23.2	65.5	13.7	62.8	5.0	0.6	3.2	28.4	11.5	4.5	2.4	4.6
7	30.1	–	12.8	63.0	5.8	0.6	3.5	27.1	8.8	4.1	1.8	2.9
8	37.0	–	12.0	63.9	6.0	0.6	2.3	27.2	6.0	3.7	1.2	1.1
9	19.1	57.3	14.0	60.0	6.3	0.6	3.0	31.1	8.2	6.0	1.1	1.1
10	14.2	56.3	13.0	60.6	5.0	0.6	2.9	30.9	7.6	5.6	0.9	1.2
11	14.1	57.7	10.2	66.1	5.7	0.6	2.6	25.0	7.0	5.2	0.6	1.2
12	18.1	56.8	13.9	63.8	5.0	0.9	2.3	28.0	7.8	5.3	1.1	1.5
13	22.2	66.2	12.1	62.0	5.6	0.6	3.5	28.2	8.5	5.3	1.5	1.7
14	23.5	64.0	13.1	63.2	5.6	0.6	3.0	27.6	10.3	5.1	2.3	2.9
15	36.4	–	12.2	60.0	5.5	0.9	2.9	30.7	8.3	3.5	2.8	2.1
16	53.0	–	9.3	60.6	5.0	0.9	3.0	30.5	6.2	1.8	3.2	1.2

daf – dry and ash-free, d – dry, a – as received basis

Results

The results of the proximate and ultimate analyses, and the proportions of the various sulphur forms, are presented in Table 1.

The sampled coal seam is comprised of coal and shale coal, interbeded with layers of fossiliferous clay and coaly clay with thicknesses up to 0.30 m. These conditions suggest dynamic tectonic conditions during organic matter deposition.

The main lithotype of the investigated coal is humoclarain (up to 80%), with xylain, humovitrain, and fusain being present in smaller amounts.

Analysis of sulphur forms

The Maritza West coals are high in sulphur, the proportion of which is commonly above 7wt% and ranges from 4wt% to more than 11wt%. The average total sulphur content is 7.5wt%, and ranges from 4.1wt% to 13.5wt% (Table 1). Organic sulphur is the major part of total sulphur in the sampled low-ash coals: it averages 4.2wt% and ranges from 1.8wt% to 6.0wt%. Pyritic sulphur is commonly less abundant than organic sulphur, though it is the predominant type in some samples with high ash contents. The average pyritic sulphur content is 1.6wt%, and ranges from 0.6wt% to 3.2wt%. The studied coals have unusually large proportions of sulphate sulphur, which ranges from 0.3wt% to 2.9wt% (average 1.7wt%).

Mode of occurrence and morphology of major sulphur-bearing minerals

Pyrite and its decomposition products (gypsum and jarosite) account for most of the inorganic sulphur content in the studied coals. They are the main sulphur-bearing minerals and vary widely in mode of occurrence.

The modes of pyrite occurrence suggest that its generation occurred in two main stages: a syngenetic stage during peat formation, and a subsequent epigenetic stage. Syngenetic pyrite was observed as separate framboids, framboid clusters, and individual euhedral crystals or clusters therefore along bandings, mainly in humodetrinite and humotelinite (Fig. 4 – b, d, e; Fig. 5 – a, b; Fig. 7 – a). The pyrite framboids are comprised of well shaped crystals and are often observed in the investigated coals (Fig. 7 – b, c, d). The morphology of the crystals is normally cubic or octahedral, and the size of separate globules ranges from 1 to 50–60 μm (Fig. 4 – b, f; Fig. 5 – a; Fig. 7 – b, c, d). The sizes of euhedral pyrite crystals vary greatly, reaching up to 1–2 μm.

Epigenetic pyrite is present as massive irregular grains with sizes ranging from 20 to 300 μm (Fig. 6 – d, f), spheroidal aggregates (Fig. 6 – d), and clusters of framboids and euhedral pyrite crystals in coal fractures (Fig. 4 – f; Fig. 6 – a, b). Pyrite-filled cell cavity structures of textoulminite, fusinite, and funginite were found also (Fig. 4 – e, f). A close association between syngenetic and epigenetic pyrite, clay minerals, and gypsum in all samples has been established (Fig. 6 – a, b, c, f; Fig. 7 – e, g, h).

A remarkably high quantity of gypsum was found in the Maritza West coal. This mineral accounts for much of the sulphate sulphur, the data for which are presented in Table 1. Gypsum was found mainly as lenses, fine crusts, or as infilling in cross bedding marks and cracks in the coal. A close association between gypsum, clay minerals, and pyrite was more commonly observed (Fig. 7 – e, f, h). The gypsum occurs as well shaped, short or long prismatic crystals (Fig. 7 – e, f) and twins. The sizes of the gypsum crystals are 5–10 × 30–90 μm for long prismatic and 2–3 ×

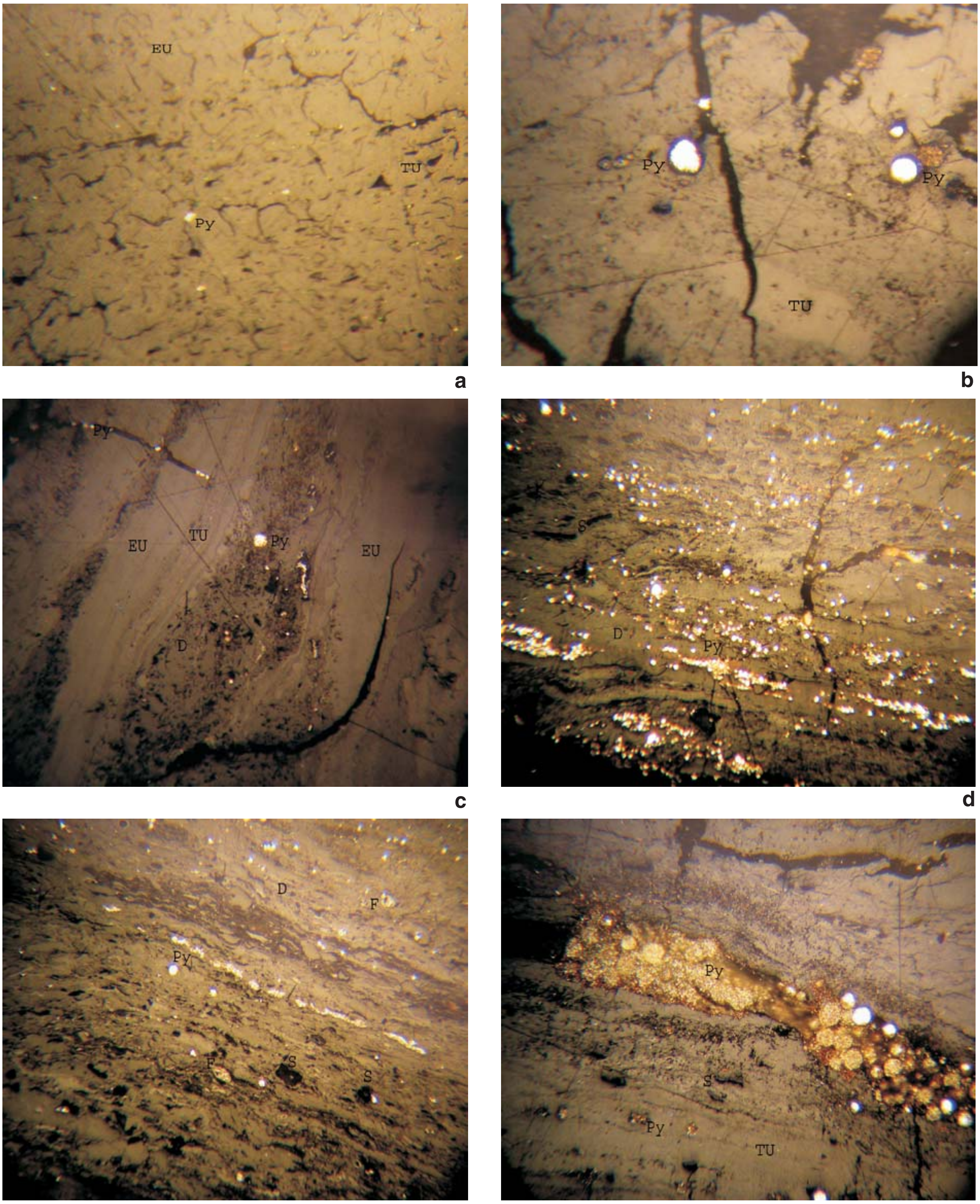


Figure 4. Photomicrographs of various macerals and pyrite within the coal matter from the Maritza West Basin. All photos were made with reflected white light under oil immersion. a – transition of texto-ulminite (TU) to eu-ulminite (EU), with intermixed fine-grained pyrite (Py), magnification 500×. b – framboidal pyrite with various huminite macerals and texto-ulminite (TU), 500×. c – transition of texto-ulminite (TU) to eu-ulminite (EU), along with densinite (D), inertodetrinite (Id), and framboidal pyrite (Py), 200×. d – fine-grained framboidal pyrite (Py) intermixed with humodetrinite, densinite (D), 500×. e – pyrite (Py) within cell structures of funginite (F), with attrinite (At), densinite (D), and sporinite (S), 500×. f – framboidal and euhedral pyrite (Py) within micro-cleat and fine-grained pyrite dispersed along humotelinite and humodetrinite, texto-ulminite (TU), sporinite (S), 500×.

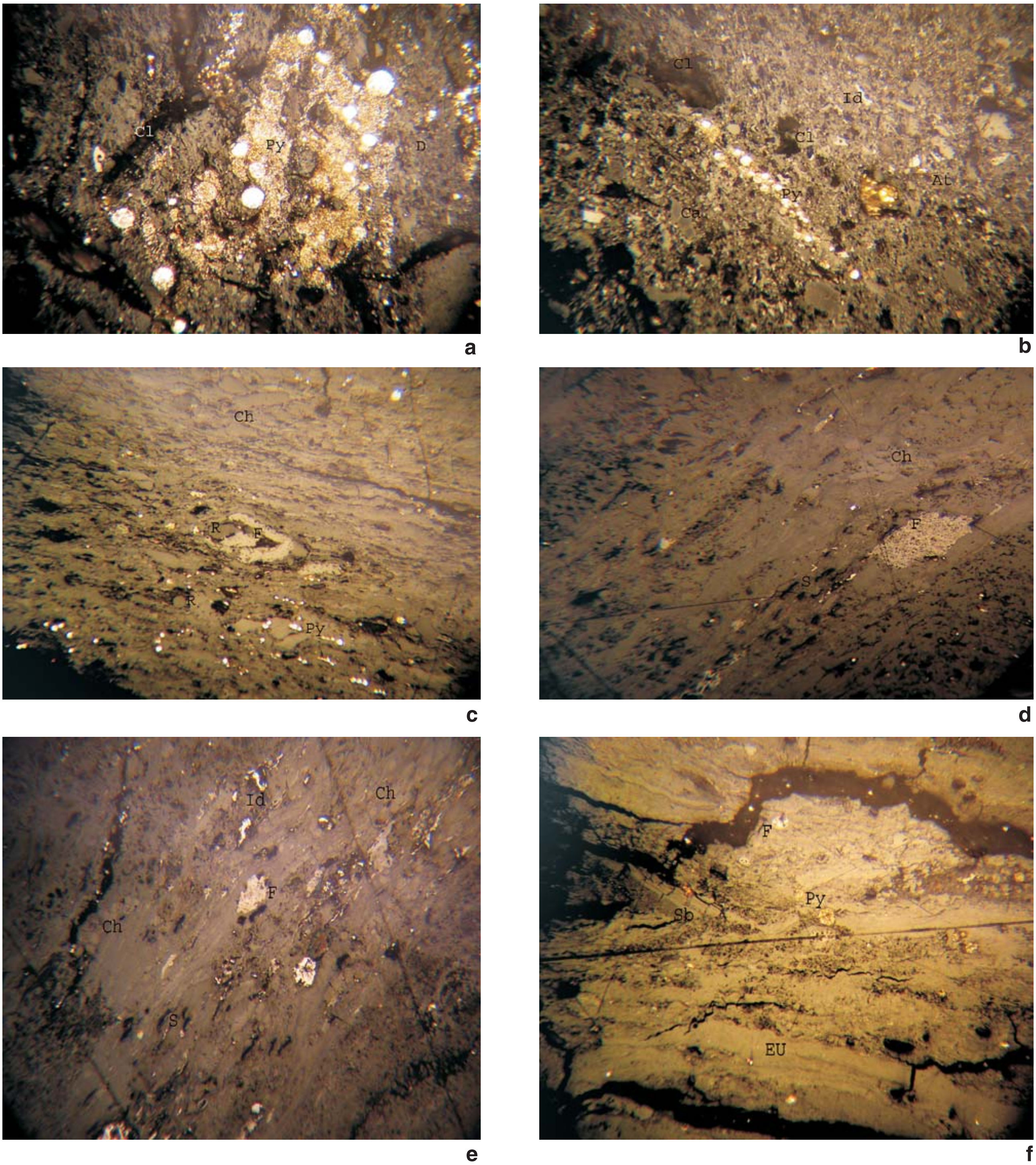


Figure 5. Photomicrographs of various macerals and minerals in coal from the Maritza West Basin. All photos were made with reflected white light under oil immersion. a – framboidal, euhedral pyrite (Py), and cleat-filling clay minerals (Cl) mixed with densinite (D), 500×. b – pyrite (Py), calcite of organic origin (Ca), and clay minerals (Cl) intermixed with attrinite (At) and inertodetrinite (Id), 500×. c – fine-grained framboidal pyrite (Py), clay minerals, resinite particles (R), and funginite (F) with corpohuminite (Ch) and humodetrinite, 500×. d – funginite (F) and inertodetrinite mixed with corpohuminite (Ch) and humodetrinite, 500×. e – corpohuminite (Ch), sporinite (S) and inertodetrinite (Id), 200×. f – fine-grained framboidal pyrite (Py) dispersed with huminite macerals, funginite (F) filled with pyrite; suberinite (Sb), eu-ulminite (EU), 500×.

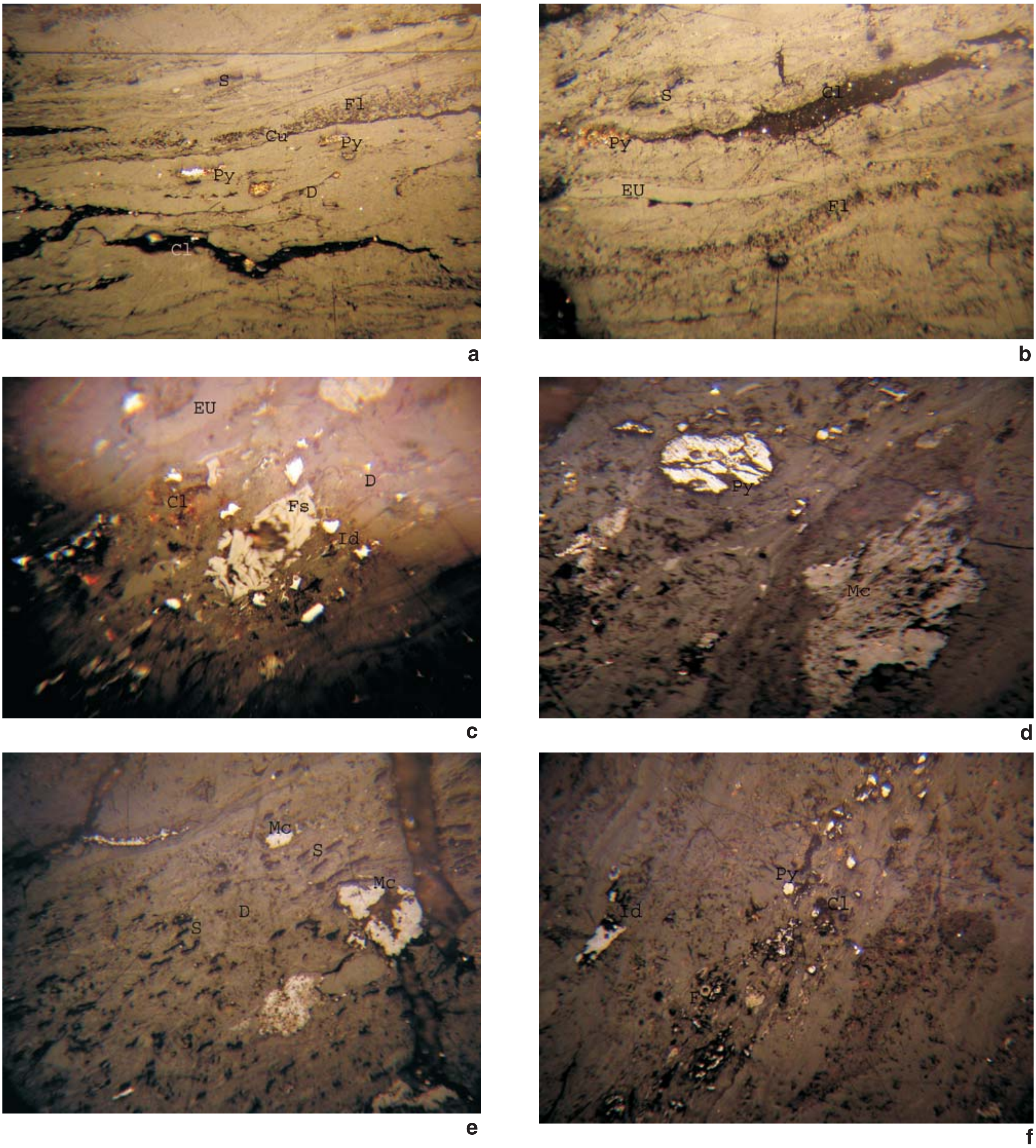


Figure 6. Photomicrographs of macerals and minerals in coal from the Maritza West Basin. All photos were made with reflected white light under oil immersion. a – clay minerals (Cl), and pyrite within micro-cleats of coal; cutinite (Cu), fluorinite (Fl), sporinite (S), and fine-grained pyrite intermixed with densinite (D), 500×. b – association between pyrite (Py) and clay minerals (Cl) filling coal micro-cleat; sporinite (S) and fluorinate (Fl) mixed with humotelinite and humodetrinite, 200×. c – fusinite (Fs), inertodetrinite (Id) and clay minerals (Cl) intermixed with eu-ulminite (EU) and densinite (D), 500×. d – massive pyrite (Py), inertodetrinite (Id), and macrinite (Mc) with humodetrinite, 500×. e – macrinite (Mc) particles, fine-grained pyrite, clays, and sporinite (S) intermixed with densinite (D), 500×. f – framboidal and massive pyrite (Py), clay minerals (Cl), funginite (F), and inertodetrinite (Id) mixed with humodetrinite and humocollinite, 200×.

5–10 μm for short prismatic crystals. It has been considered that this gypsum is a weathering product of both sulphide and carbonate minerals. It has also been deposited with epigenetic sulphides from circulating hydrothermal solutions. According to Vassilev and Vassileva (1996) some finely dispersed gypsum crystals and gypsum-filled coal cavities may have formed by the crystallisation of calcium and sulphate ions dissolved in pore water during coal storage.

Petrographic analysis

The petrographic composition of the Maritza West coal is shown in Table 2, and is based on group macerals distribution. The coals show comparatively low variations in maceral group composition. They are characterized by a relatively high content of huminite macerals. Huminite content ranges from 77–90 vol%, and liptinite from 2–13 vol%. Inertinite macerals range from 0–14 vol%.

The huminite maceral group consists predominantly of highly dispersed macerals from the humodetrinite subgroup – densinite and attrinite. Commonly they are closely associated with pyrite and clay minerals, and occasionally occur as separate lenses or small bands (Fig. 4 – c, d, e, f; Fig 5 – a, b, c, d, f; Fig. 6 – a, b, c, e, f). The macerals ulminite (texto-ulminite and eu-ulminite), gelinite, and corpohuminite are present in smaller amounts. They occur as isolated bands and lenticular bodies (Fig. 4 – a, b, c, f; Fig. 5 – c, d, e; Fig. 6 – b, c, f). The cell lumens of texto-ulminite and eu-ulminite are occasionally filled with pyrite, clay minerals, or resinite.

The liptinite group macerals are dominated by sporinite, cutinite, resinite, and liptodetrinite. Suberinite and fluorinate are present in separate samples. Microsporinite is the dominant sporinite variety (Fig. 4 – e; Fig. 5 – e). Cutinite is usually not well preserved, variously sized particles of which were found dispersed among the humodetrinite (Fig. 6 – a). Liptodetrinite is present in almost all samples in proportions reaching up to 3%. Resinite is associated mainly with humotelinite and humodetrinite (Fig. 5 – c). Suberinite and fluorinite were observed as small lenses between humodetrinite and the other liptinite macerals (Fig. 5 – f; Fig. 6 – a, b).

There are relatively high contents of inertinite macerals in the Maritza West coal (up to 14%). They are dominated by fusinite (mainly degradofusinite), semifusinite, and inertodetrinite. Funginite and macrinite are present only in some samples (Fig. 4 – e; Fig. 5 – c, f; Fig. 6 – d, e, f). The cell lumens of fusinite, semifusinite, and funginite are often filled with clay minerals and occasionally with pyrite (Fig. 4 – e; Fig. 5 – f; Fig. 6 – c). All inertinite macerals are closely associated with clay, pyrite, and humodetrinite (Fig. 5 – b, e; Fig. 6 – c, d, f).

A number of minerals have been identified, including pyrite, hematite, limonite, quartz, opal, montmorilonite, illite, chlorite, kaolinite, K-feldspar, gypsum, jarosite, calcite, and aragonite. The major minerals in the Maritza West coal are pyrite, gypsum, kaolinite, and illite. Quartz, montmorillonite, chlorite, K-feldspar, and calcite are present in smaller amounts. Other inorganic phases (hematite, limonite, aragonite, jarosite, and opal) are present only as accessory minerals (under 1%).

Vertical distribution of macerals, sulphur forms, and ash content

The vertical distribution of maceral groups, different sulphur forms (total, pyritic, organic, and sulphate) in volume

Table 2. Maceral composition of coal from Maritza West Basin, Bulgaria

Samples No.	Maceral composition per total matter (vol. %)				Maceral composition per organic matter (vol. %)		
	Huminite H	Liptinite L	Inertinite I	Mineral matter Mm	Huminite H	Liptinite L	Inertinite I
1	–	–	–	–	–	–	–
2	62	6	1	31	90	9	1
3	73	6	5	16	87	7	6
4	55	6	–	39	90	10	–
5	61	6	1	32	90	9	1
6	59	10	8	23	77	13	10
7	61	7	6	27	83	10	7
8	62	5	3	30	89	7	4
9	67	8	8	17	80	10	10
10	73	2	6	13	84	2	14
11	74	2	10	14	86	2	12
12	72	3	8	17	87	3	10
13	69	4	7	20	86	5	9
14	64	10	8	18	78	12	10
15	54	7	–	39	89	11	–
16	–	–	–	–	–	–	–

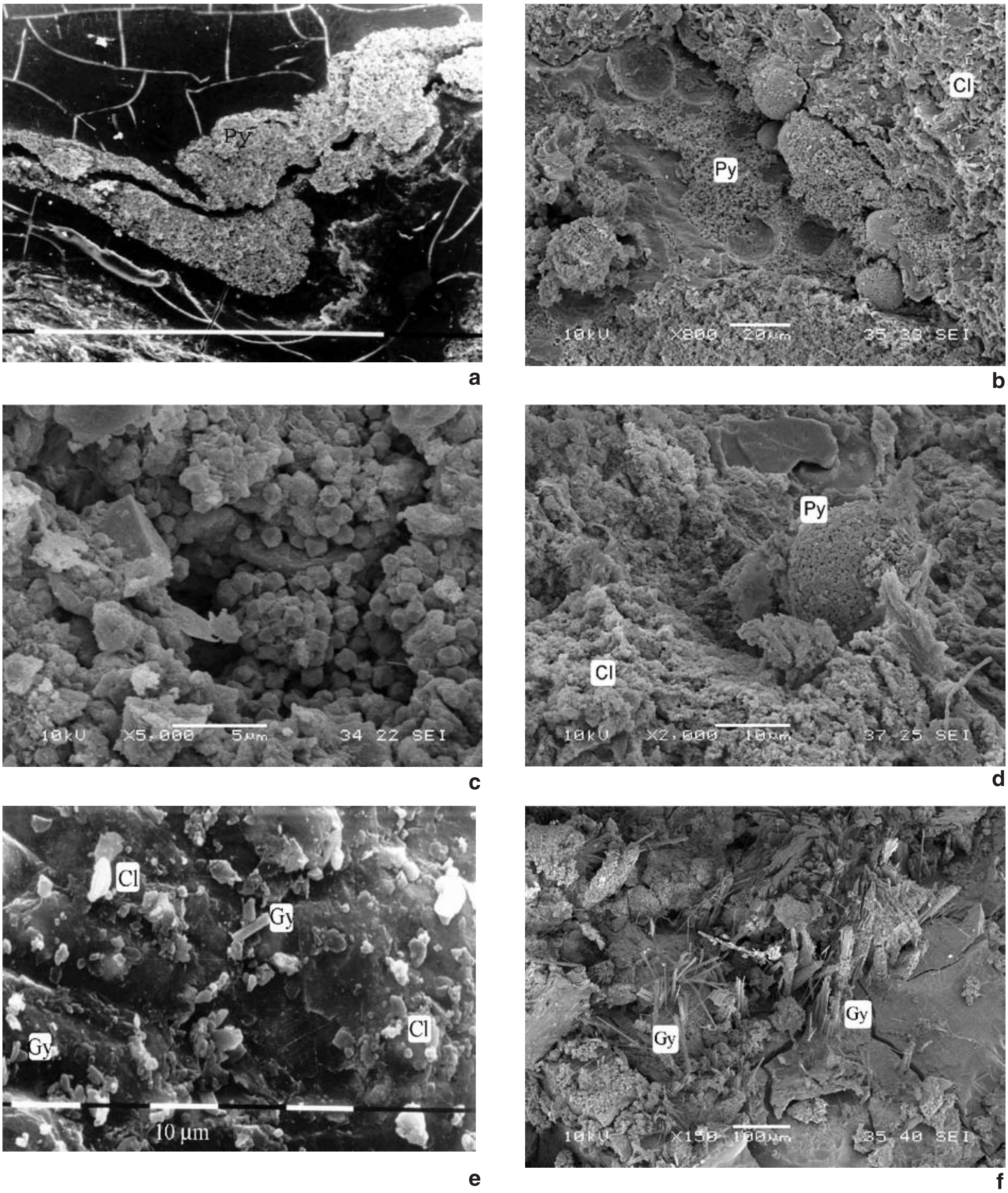
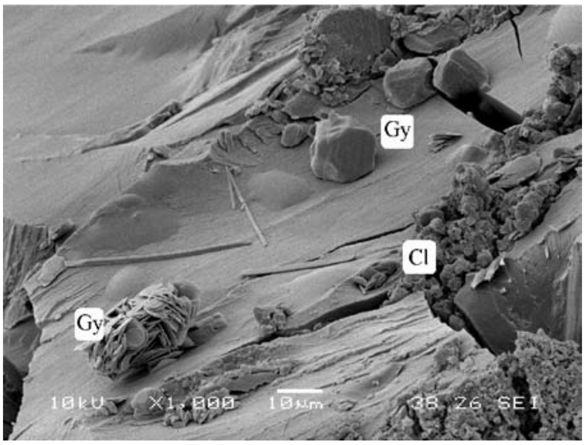


Figure 7. a – SEM photomicrograph of cleat-filling pyrite (Py), secondary electrons, the size of the scale on the micrograph is 100 μm. b – SEM photomicrograph of clusters of pyrite framboids and fine-grained euhedral pyrite crystals (Py) and clay (Cl), secondary electrons. c – clusters of octahedral and cubic pyrite crystals, SEM, secondary electrons. d – SEM photomicrograph of a single pyrite framboid (Py) and clay minerals (Cl), secondary electrons. e – SEM photomicrograph of short prismatic gypsum crystals (Gy) and clay minerals (Cl), secondary electrons. f – SEM photomicrograph of well shaped, long, prismatic gypsum crystals (Gy), secondary electrons. g – association between clay (Cl) and gypsum (short prismatic crystals and “gypsum rose”) (Gy), SEM, secondary electrons. h – association between well shaped gypsum crystals (Gy) and clay minerals (Cl) – kaolinite, SEM, secondary electrons.



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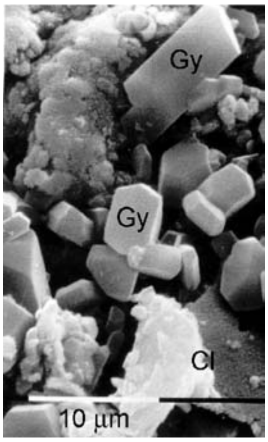

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Figure 7, continued.

percents, and the ash contents along a complete sampled section of the Kipra coal bed are shown in Fig. 8.

The amount of total sulphur generally decreases from the base to the top of the sampled coal bed. The highest contents of total, sulphate, and pyritic sulphur were recorded near the clay coal layers. The highest organic sulphur contents were found in the middle part of the coal bed. This is perhaps related to acidic water conditions that were favourable for tissue preservation in the paleoswamp (Padgett et al. 1999). The total and organic sulphur quantities decrease near the top and bottom of the coal seam. There is a strong relation between the high amounts of sulphate sulphur and the clay layers within the coal seam.

Discussion

According to the chemical classification system for high-temperature compositions (HTA), the Maritza West HTA belongs to the ferricalsialic chemical type. According to the genetic mineral classification system for mineral classes in crystalline matter, the coal has low detrital and high authigenic mineral abundances, with sulphide-sulphate authigenic mineral tendency. The coal and coal ash reveal the highest contents of S, Fe_2O_3, MnO, P_2O_5, and residual CO_2, and the lowest values of FC, C, TiO_2, K_2O, and K_2O/Na_2O (Vassilev and Vassileva, 1998). Vassilev and Vassileva (1998) have pointed out that the high-ash Maritza West coal is enriched in Fe-sulphides, gypsum, and Fe sulphates along with a number of other mineral phases. Those authors have also shown that the characteristic genetic features of these coals are as follows: intensive infiltration and crystallization of syngenetic carbonate, sulphide, and sulphate mineralization; limited detrital supply; abundance of unstable weathering phases and elements; and probably low contents of organically bound, and high concentrations of ion-exchanged inorganic elements.

The present investigation reveals that the Maritza West coals have very high sulphur contents (above 7wt%), and that pyrite, gypsum, and Fe sulphates are their main sulphur-bearing minerals.

Extremely high gypsum contents near the coal partings, and comparatively high contents in the upper part of coal seams, were recorded. There is a strong connection between the high gypsum contents and the specific paleoenvironment during peat formation. The coal-bearing sediments formed in a typical regressive continental facies near a marine basin. The sedimentation occurred in flat littoral areas. The presence of gypsum in the coal layers, and its comparatively high content in the upper part of coal beds, indicates intensive hypergene coal changes. According to Siskov et al. (1982), this caused the peat bog to dry up quickly after it became covered by sediments.

The presence of high concentrations of organic and inorganic sulphur in the Maritza West coal basin can be explained with reference to the specific paleogeographic environment during coal generation. High groundwater levels with low vertical circulation, and underlying marine-influenced sediments and coal layers created conditions favourable to the formation of organic sulphur (Kostova 1999).

Conclusions

– The studied coals are characterised by high sulphur contents, commonly above 7wt%, which range from 4wt% to more than 11wt%. The relative proportions of the various sulphur forms are as follows: average total sulphur of 7.5wt%, ranging from 4.1wt% to 13.5wt%; average pyritic sulphur of 1.6wt%, ranging from 0.6wt% to 3.2wt%; average organic sulphur of 4.2wt%, ranging from 1.8wt% to 6.0wt%; and average sulphate sulphur of 1.7wt%, ranging from 0.3wt% to 2.9wt%.
– Pyrite and gypsum are the main sulphur-bearing minerals. The syngenetic pyrite occurs mainly as isolated or clustered framboidal bodies and well-shaped euhedral crystals along stratification bands, amongst the humodetrinite and humotelinite. Epigenetic massive pyrite and pyrite-filled cell lumens were also observed. Gypsum occurs as lenses, fine crusts, or as infilling in cross bedding marks and cracks in the coal; it is present as well shaped, short and long prismatic crystals.
– The total sulphur contents slightly decrease from the base toward the top of the coal bed. The highest contents of total, sulphate, and pyritic sulphur occur near the clay coal layers. The highest contents of organic sulphur were found in the middle part of the coal bed.
– The high contents of organic sulphur are perhaps due to the presence of high amounts of sulphate ions in the groundwater, high groundwater levels, and the pre-

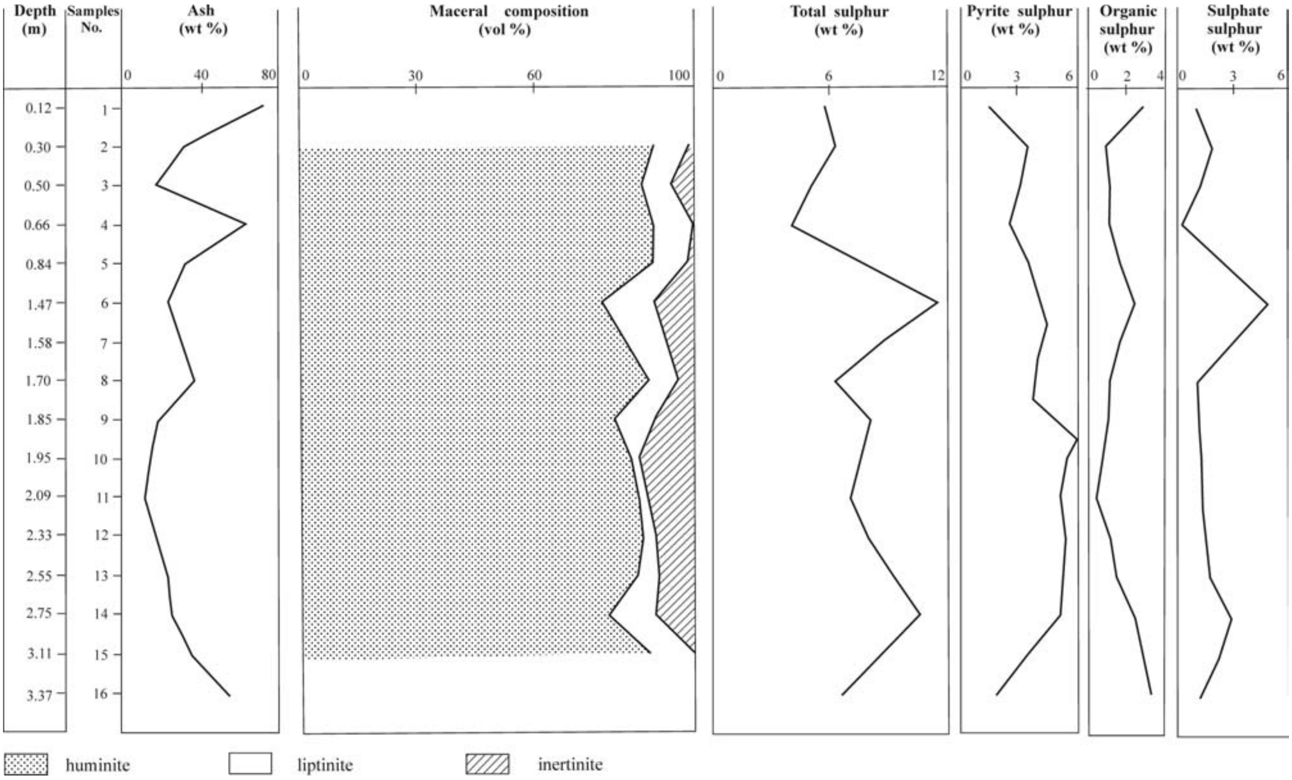

Figure 8. Vertical distribution of the maceral groups and sulphur forms (total, pyrite, organic and sulphate) in volume percents, and the ash content along a complete sampled section from the Kipra coal bed from Maritza West Basin, Bulgaria.

sence of a favourable depositional environment during peat formation.

– There is a strong connection between the coal's unusually high gypsum content and the specific paleogeographic environment during peat formation. These coal-bearing sediments were formed in a typical regressive continental facies near a marine basin. The presence of gypsum in the coal layers, and its comparatively high content in the upper part of the coal beds, indicates intensive hypergene coal changes that caused the peat bog to dry quickly after becoming covered with sediments.

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