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Mineralogical components of some thermally decomposed lignite and lignite ash from the Ptolemais basin, Greece

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Abstract

Two samples of Pliocene lignites from the Ptolemais basin of Greece, one from the upper and one from the lower lignite seams, were heated and dried in air at 50°C intervals from 50 to 1200°C. The two lignite samples initially contained the same minerals, namely calcite, dolomite, quartz, kaolinite, illite, pyrite and gypsum, but in different proportions. The lignite sample from the upper lignite seam is rich in Fe_2O_3, CaO and SO_3, while that from the lower lignite seam is rich in SiO_2 and Al_2O_3.

Hematite, periclase, melilites, merwinite, calcium ferrite and brownmillerite are constituents of the 1200°C lignite ash from both samples. The heating conditions and the chemistry of the samples lowered the formation temperatures of brownmillerite, which appeared in both samples at 950°C. In the Fe_2O_3, CaO- and SO_3-rich sample, magnesioferrite is present from 850 to 1100°C and hematite appears at 300°C. In the SiO_2- and Al_2O_3-rich sample, magnesioferrite was not detected at any temperature and hematite appeared at 600°C.

Anhydrite, which normally decomposes in air at 1638°C, is the main constituent at 1150°C, on heating the lignite sample that was rich in Fe_2O_3, CaO and SO_3. Anhydrite diminishes at 1200°C. In the SiO_2- and Al_2O_3-rich lignite sample, anhydrite is main constituent at 1100°C, but diminishes considerably at 1150°C and decomposes at 1200°C.

1. Introduction

Numerous studies have shown a diversity of physical and chemical changes in coal mineral matter during combustion (Mitchell and Gluskoter, 1976; Harris et al., 1979;

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Pearson and Kwong, 1979; Unsworth et al., 1991). Knowledge of changes in a coal's mineral composition during combustion has application in industry as well as in the assessment of any potential environmental consequences of coal combustion (Ward, 1984; Gay and Davis, 1987; Filippidis and Georgakopoulos, 1992; Georgakopoulos et al., 1992; Guthrie, 1992; Thomas, 1992; Kassoli-Fournaraki et al., 1993).

Recently, we conducted heating experiments (100–1000°C at 100°C intervals) on the mineralogical components of a lignite sample from the Ptolemais basin of Greece (Filippidis et al., 1992; Kassoli-Fournaraki et al., 1992). Chemical analysis of the inorganic matter, contained in the sample at 600°C, showed mainly (in weight %) $CaO = 41.7$, $Fe_2O_3 = 16.1$, $MgO = 6.9$, $SiO_2 = 5.2$ and $Al_2O_3 = 2.5$.

Our aim in this study was to compare the mineralogical changes, on heating, in two lignite samples from the Ptolemais basin, from 450 to 1200°C at 50°C intervals. The two lignite samples displayed differences in their original mineral proportions, which resulted in differences in their chemical composition during and after combustion.

2. Geologic setting

The Florina–Vegoritis–Ptolemais graben forms a large basin, filled with Neogene–Quaternary sediments, and extends from the Greek–Former Yugoslavian Republic of Macedonia borders in a south by southeast direction to the Kozani area (southeast of Ptolemais, Fig. 1). The sediments of the Ptolemais basin unconformably overlay both Paleozoic metamorphic rocks and Mesozoic crystalline limestones (Pavlides, 1985; Pavlides and Mountrakis, 1987).

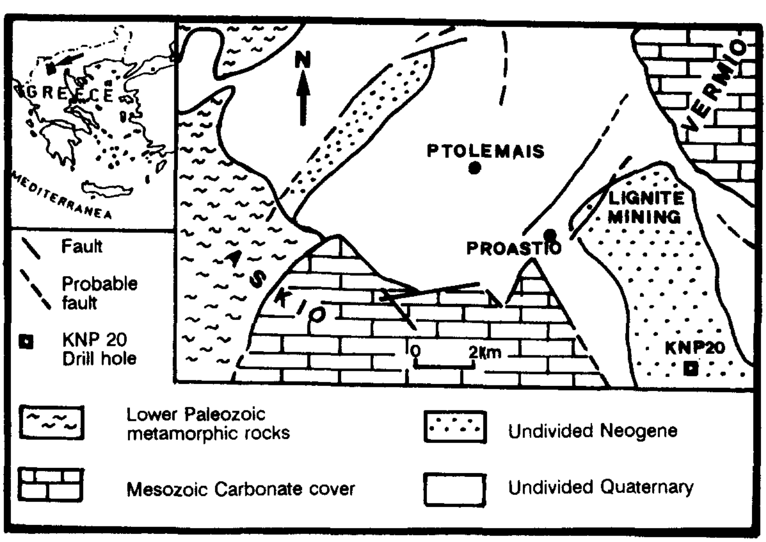

Fig. 1. Geological map of Ptolemais basin area (after Pavlides, 1985; Pavlides and Mountrakis, 1987) and location of drill hole KNP20.

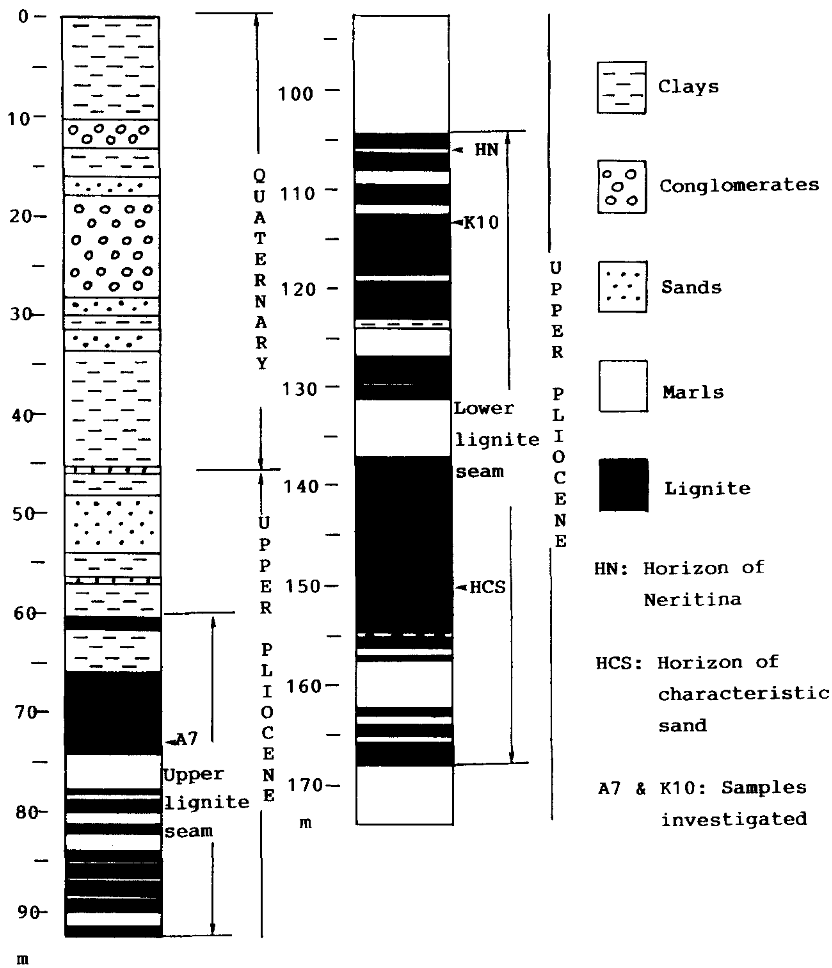

Fig. 2. Stratigraphic section of the investigated drill hole KNP2O.

The Neogene–Quaternary sediments of the basin are divided into three lithostratigraphic formations. The lower formation (Upper Miocene to Lower Pliocene) consists mainly of conglomerates, marls, sands and clays. The middle formation (Pliocene) contains the lignite beds (Fig. 2). The upper formation (Quaternary) consists of terrestrial and fluvioterrestrial conglomerates, lateral fans and alluvial deposits. The middle formation contains two lignite seams (upper and lower) which have been exploited by opencast mining since 1953. The lignite beds alternate with marls, clays and sands (Gross, 1966; Koufos and Pavlides, 1988; Kaouras, 1989).

In the opencast mine of the Southern lignite field, the average thickness of the overburden (based on data from nine bore holes) is 80 m and the average thickness of the lignite is 58 m. The marl band, which separates the upper and lower lignite seams, is the thickest sterile band (Fig. 2). The average thickness of the intermediate sterile bands is 31 m.

3. Materials and methods

The investigated lignite samples were obtained from a drill hole core (KNP20) in the Southern lignite field of the Ptolemais basin. The 10-cm-thick sample A7 (73.40–73.50 m depth) is from the upper lignite seam, while sample K10, of the same thickness (113.40–113.50 m depth) is from the lower lignite seam. Each lignite sample was air dried and crushed to pass a 250 μm sieve. They were further divided into 24 samples each.

Five polished sections of each original air-dried lignite sample were studied under a reflected light microscope and by scanning electron microprobe JSM 840S with a LINK AN 10000 EDS microanalyzer.

Approximately 0.5 g of the air-dried lignite samples was placed in Pt boats and dried/heated in air at 50°C intervals from 50 to 1200°C. Each sample was heated for a minimum of 2 hours. The temperature of the furnace was controlled within ±2°C. After air quenching and cooling, the samples were examined under a stereomicroscope to determine if the sample had undergone complete combustion.

Table 1
Ash content of the lignite samples, heated at different temperatures for two hours

Weight before heating (g)		Temperature of heating (°C)	Weight after heating (g)		Ash [a] (%)	
A7	K10		A7	K10	A7	K10
0.578	0.511	50	0.541	0.488	93.6 [a]	95.5 [a]
0.585	0.570	100	0.514	0.507	87.9 [a]	88.9 [a]
0.579	0.569	150	0.494	0.495	85.3 [a]	87.0 [a]
0.595	0.551	200	0.489	0.451	82.2 [a]	81.9 [a]
0.579	0.568	250	0.357	0.387	61.7 [a]	68.1 [a]
0.583	0.560	300	0.160	0.235	27.4 [a]	42.0 [a]
0.579	0.581	350	0.112	0.098	19.3 [a]	16.9 [a]
0.584	0.518	400	0.077	0.059	13.2 [a]	11.4 [a]
0.584	0.442	450	0.061	0.044	10.4	10.0
0.592	0.447	500	0.060	0.039	10.1	8.7
0.597	0.448	550	0.059	0.039	9.9	8.7
0.582	0.449	600	0.056	0.037	9.6	8.2
0.577	0.443	650	0.055	0.036	9.5	8.1
0.594	0.457	700	0.056	0.037	9.4	8.1
0.603	0.444	750	0.056	0.034	9.3	7.7
0.590	0.449	800	0.055	0.034	9.3	7.6
0.579	0.447	850	0.053	0.034	9.2	7.6
0.588	0.448	900	0.053	0.034	9.0	7.6
0.578	0.463	950	0.052	0.035	9.0	7.6
0.594	0.444	1000	0.053	0.033	8.9	7.4
0.583	0.457	1050	0.052	0.034	8.9	7.4
0.590	0.457	1100	0.049	0.031	8.3	6.8
0.639	0.446	1150	0.052	0.030	8.1	6.7
0.597	0.463	1200	0.044	0.030	7.4	6.5

[a] Includes uncombusted lignite.

Table 2
Mineralogical composition of lignite ash obtained at different temperature intervals and determined by X-ray diffraction analysis (XRD)

Minerals	Sample No.	Temperature (°C)												
		RT [a]	50	100	150	200	250	300	350	400	450	500	550	600
Calcite	A7	(+) [b]	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+ [c]	+	+	+	+
	K10	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	+	+	+
Dolomite	A7	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	+	+	+
	K10	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	+	+	+
Quartz	A7	X [d]	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	+	+	+
	K10	X	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	+	+	+
Kaolinite	A7	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	–	–	–
	K10	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	–	–	–
Meta-kaolinite	A7	– [e]	–	–	–	–	–	–	–	–	–	+	+	+
	K10	–	–	–	–	–	–	–	–	–	–	+	+	+
Illite	A7	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	–	–	–
	K10	(+)	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	+	–	–	–
Pyrite	A7	0 [f]	(+)	(+)	(+)	(+)	(+)	(+)	(+)	–	–	–	–	–
	K10	0	(+)	(+)	(+)	(+)	(+)	(+)	(+)	+	–	–	–	–
Hematite	A7	–	–	–	–	–	–	(+)	(+)	+	+	+	+	+
	K10	–	–	–	–	–	–	–	–	–	–	–	–	+
Gypsum	A7	(+)	(+)	(+)	–	–	–	–	–	–	–	–	–	–
	K10	(+)	(+)	(+)	–	–	–	–	–	–	–	–	–	–
Bassanite	A7	–	–	–	(+)	(+)	(+)	–	–	–	–	–	–	–
	K10	–	–	–	(+)	(+)	(+)	–	–	–	–	–	–	–
Anhydrite	A7	–	–	–	–	–	–	(+)	(+)	+	+	+	+	+
	K10	–	–	–	–	–	–	–	–	–	+	+	+	+

Minerals	Sample No.	Temperature (°C)											
		650	700	750	800	850	900	950	1000	1050	1100	1150	1200
Calcite	A7	−	−	−	−	−	−	−	−	−	−	−	−
	K10	+	−	−	−	−	−	−	−	−	−	−	−
Dolomite	A7	+	+	−	−	−	−	−	−	−	−	−	−
	K10	−	−	−	−	−	−	−	−	−	−	−	−
Quartz	A7	+	+	+	−	−	−	−	−	−	−	−	−
	K10	+	+	+	+	+	−	−	−	−	−	−	−
Hematite	A7	+	+	+	+	+	+	+	+	+	+	+	+
	K10	+	+	+	+	+	+	+	+	+	+	+	+
Anhydrite	A7	+	+	+	+	+	+	+	+	+	+	+	+
	K10	+	+	+	+	+	+	+	+	+	+	+	−
Lime	A7	+	+	+	+	+	−	−	−	−	−	−	−
	K10	+	+	+	+	+	−	−	−	−	−	−	−
Periclase	A7	−	−	+	+	+	+	+	+	+	+	+	+
	K10	+	+	+	+	+	+	+	+	+	+	+	+
Melilites [g]	A7	+	+	+	+	+	+	+	+	+	+	+	+
	K10	+	+	+	+	+	+	+	+	+	+	+	+
Merwinite	A7	−	+	+	+	+	+	+	+	+	+	+	+
	K10	−	+	+	+	+	+	+	+	+	+	+	+
Calcium ferrite	A7	−	−	+	+	+	+	+	+	+	+	+	+
	K10	−	−	+	+	+	+	+	+	+	+	+	+
Magnesioferrite	A7	−	−	−	−	+	+	+	+	+	+	−	−
	K10	−	−	−	−	−	−	−	−	−	−	−	−
Brownmillerite	A7	−	−	−	−	−	−	+	+	+	+	+	+
	K10	−	−	−	−	−	−	+	+	+	+	+	+

Samples heated to 400°C and higher, were ground to a fine powder and investigated by XRD methods. The 50–350°C samples, as well as the two original lignites, were also ground to fine powder and disaggregated. After centrifugation, parallel-oriented X-ray samples were prepared. This method was used because of the high content of unburnt lignite contained in these samples (see Table 1).

The diffractograms for random- and parallel-oriented samples were obtained by a Philips diffractometer with Ni-filtered Cu-$K\alpha$ radiation. Silicon powder was used as an external standard. The scanning speed was 1° per minute over the interval 3–63 of 2θ and 0.25° per minute over the interval 20–36 of 2θ. The 1000°C lignite ash samples were analysed by atomic absorption spectroscopy.

4. Results and discussion

The original lignite samples (A7 and K10) were studied under reflected light and by SEM–EDS, and showed that both samples contained framboidal pyrite and quartz. In each sample, individual framboids and colonies were recognized. Most framboids were well preserved with concentric arrangement of the microcrysts, but massed spherules were found as well.

The original lignite samples (A7 and K10) both contained the same minerals (Table 2). Sample A7 contained more framboids than K10, thereby accounting for the differences in the Fe_2O_3 and the SO_3 contents of their 1000°C ash samples (Table 3). The SO_3 content (Table 3) could also indicate that the A7 sample contained more gypsum than K10. The SiO_2 content could be assigned to quartz, kaolinite and illite, the Al_2O_3 content to kaolinite and illite, the Fe_2O_3 content to pyrite, the CaO content to calcite, dolomite and gypsum, the MgO content to dolomite, the K_2O content mainly to illite, and the SO_3 content to pyrite and gypsum. It is obvious that the two lignite samples contain the same minerals in different proportions, and this is reflected in the chemistry of their 1000°C lignite ash. In later discussions, the chemical composition is used when comparing the two samples throughout the temperature range.

Fig. 3 shows the variation in ash content ± uncombusted lignite vs. temperature. The two lignite samples behave similarly at the different temperatures. Above 200–400°C the lignite is consumed, leaving an ash content of around 7% at 1200°C (Table 1; Fig. 3).

Filippidis et al. (1992) and Kassoli-Fournaraki et al. (1992) heated a lignite sample from the same borehole, but at 0.65 m beneath sample A7. Although they were heating in the range of 100–1000°C at 100°C intervals, they reported similar observations up to

Notes to Table 2:
[a] Room temperature.
[b] (+) identified using three to six weak to very weak XRD reflections.
[c] Present.
[d] Identified using Scanning Electron Microprobe with EDS microanalyser (SEM–EDS) and XRD.
[e] Not detected.
[f] Identified using reflected light microscope; SEM–EDS and XRD.
[g] Detected as gehlenite and åkermanite.

Table 3
Chemical composition (wt%) of the 1000°C lignite ash, determined by atomic absorption spectometry

Sample No.	A7	K10
SiO_2	3.38	24.43
TiO_2	0.14	0.51
Al_2O_3	2.63	14.77
Fe_2O_3	17.91	7.22
MnO	0.05	0.03
MgO	7.97	8.93
CaO	38.25	32.60
Na_2O	0.23	0.52
K_2O	0.15	1.14
P_2O_5	0.46	0.36
SO_3	28.81	9.10
Total	99.98	99.61

1000°C with the exception of hematite. Hematite was determined to be present (locally introduced through fractures) in the original lignite sample of their previous studies (Filippidis et al., 1992; Kassoli-Fournaraki et al., 1992); it was not detected in the original lignite samples of the present study. Further small differences between the results of the two studies in the temperatures (± 150°C) concerning the appearance or disappearance of the various mineral phases, could be attributed to the different heating intervals, the different chemical composition, or the detection limit of the X-ray diffraction method.

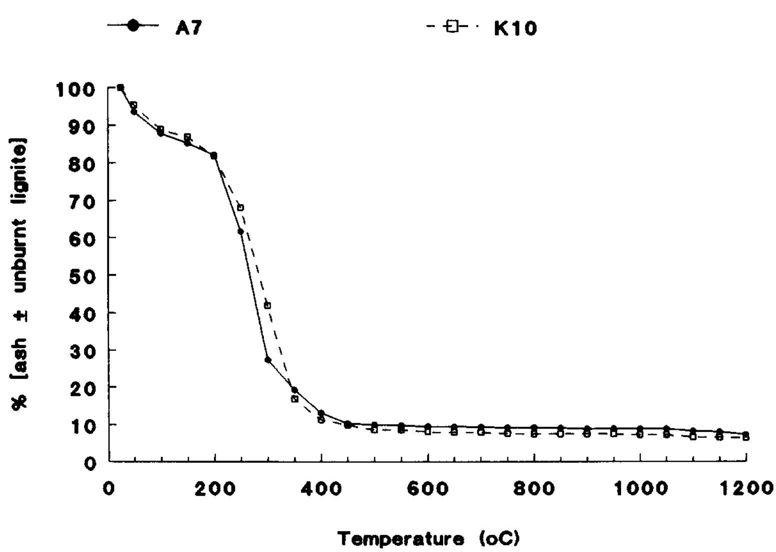

Fig. 3. Plot of variation in ash content ± uncombusted lignite vs. temperature. The samples above 400°C are free of any uncombusted lignite.

Because each lignite ash sample is a mineralogically complicated system, it is not possible to fully explain all the reactions involved in each system. Nevertheless, comments are possible in regard to the formation of some of the more important minerals detected above 900°C. For lower temperatures, explanations can be found in previous papers (Filippidis and Georgakopoulos, 1992; Filippidis et al., 1992; Kassoli-Fournaraki et al., 1992).

Anhydrite forms by dehydration of bassanite, but anhydrite and hematite can be formed by a pyrite-calcite reaction (Mitchell and Gluskoter, 1976). Periclase is formed from the decomposition of dolomite through magnesite. The formation of melilites, merwinite and brownmillerite may involve chain reactions between carbonates, silicates and oxides (MacKenzie and Alasti, 1978; Deer et al., 1986). Periclase-hematite reactions are responsible for the formation of magnesioferrite, while calcium ferrite is formed through a lime-hematite reaction or even by a magnesioferrite-calcite (from dolomite) reaction.

The small differences in temperature (±150°C) for the appearance or disappearance of the various mineral phases, observed between the two investigated lignite ash

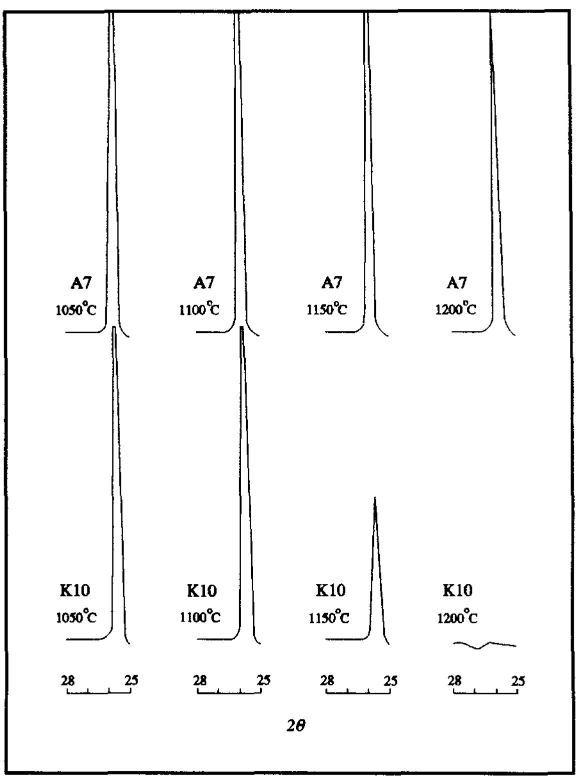

Fig. 4. Plot of the (002) XRD reflection of anhydrite during heating of the lignite samples (*A7* and *K10*) from 1050 to 1200°C.

samples, are attributed to the differences in their chemical composition or to the detection limit of the X-ray diffraction techniques.

Magnesioferrite was not detected at any temperature in the heating treatment of sample K10 which contains less Fe_2O_3, CaO and SO_3; it was detected in sample A7 from 850 to 1100°C. Hematite appears at 300°C in the Fe-rich lignite sample A7, whereas it first appears at 600°C in the Fe-poor K10 lignite sample. Brownmillerite in the system $CaO–Al_2O_3–Fe_2O_3$ is formed at 1150°C and higher (MacKenzie and Alasti, 1978; Drabik and Petrovic, 1985). Filippidis et al. (1992) did not observe brownmillerite in the 1000°C lignite ash sample. The heating condition of the present experiments and the chemistry of the samples could have lowered the formation temperature of brownmillerite. In the heating experiments of both lignite samples, A7 and K10, brown millerite appears from 950°C (Table 2).

Anhydrite appears at 300°C for sample A7, which is richer in Fe_2O_3, CaO and SO_3, and it is present up to 1200°C, whereas anhydrite appears at 450°C for sample K10, but it does not remain at 1200°C (Table 2). On heating, anhydrite inverts to a high-temperature form at 1193°C, and decomposes at 1638°C (Deer et al., 1962; Hanic et al., 1985). In the heating treatment of sample A7, anhydrite is a main constituent up to 1150°C, but begins to diminish at 1200°C. In sample K10, it is present at 1100°C, diminishes considerably at 1150°C and disappears at 1200°C (Fig. 4). The chemistry of the lignite samples apparently affects the stability of anhydrite, which is completely decomposed at 1200°C on heating lignite sample K10.

5. Conclusions

The two investigated lignite samples, A7 from the upper and K10 from the lower lignite seams of the Ptolemais basin, contained the same minerals originally (calcite, dolomite, quartz, kaolinite, illite, pyrite and gypsum) in different proportions. This difference is reflected in the chemical composition of their 1000°C lignite ash. Sample A7 is richer in Fe_2O_3, CaO and SO_3, while sample K10 is richer in SiO_2 and Al_2O_3.

During the heating/drying treatment in air, from 50 to 1200°C at 50°C intervals, the two samples behaved more or less the same in regard to their weight loss. At low temperatures, the weight loss is attributed to inherent water. Between 200 and 400°C the lignite is consumed, leaving an ash content of around 7% at 1200°C.

During heating, new minerals are formed and different chemical reactions occur. The minerals present in all heating experiments at 1200°C are: hematite, periclase, melilites, merwinite, calcium ferrite and brownmillerite. In mineralogical changes through all temperature intervals, the two lignite samples behaved more or less similarly, except in the cases of hematite, magnesioferrite and anhydrite. Hematite appeared at 300°C on heating Fe-rich lignite sample A7; hematite appears at 600°C on heating the Fe-poor sample K10. In the lignite sample A7, which is richer in Fe_2O_3, CaO and SO_3, magnesioferrite is detected from 850 to 1100°. In the lignite sample K10 it is not detected at any temperature. Anhydrite is present from 300 to 1200°C during heating of sample A7, while it is detected from 450 to 1150°C in the heating experiments of sample K10; it is completely decomposed at 1200°C in sample K10.

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