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Modes of Occurrence and Abundance of Trace Elements in Pennsylvanian Coals from the Pingshuo Mine, Ningwu Coalfield, Shanxi Province, China

Ning Yang, Shuheng Tang *, Songhang Zhang and Yunyun Chen

School of Energy Resources, China University of Geosciences, Beijing 100083, China; yangning@cugb.edu.cn (N.Y.); zhangsh@cugb.edu.cn (S.Z.); chenyy@cugb.edu.cn (Y.C.)

* Correspondence: tangsh@cugb.edu.cn; Tel.: +86-10-8232-2005

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Abstract: The Pingshuo Mine is an important coal mine of the Ningwu coalfield in northern Shanxi Province, China. To investigate the mineralogy and geochemistry of Pingshuo coals, core samples from the mineable No. 4 coals were collected. The minerals, major element oxides, and trace elements were analyzed by scanning electron microscopy (SEM), LTA-XRD in combination with Siroquant software, X-ray fluorescence (XRF), inductively coupled plasma mass spectrometry (ICP-MS) and ICP-CCT-MS (As and Se). The minerals in the Pennsylvanian coals from the Pingshuo Mine dominantly consist of kaolinite and boehmite, with minor amounts of siderite, anatase, goyazite, calcite, apatite and florencite. Major-element oxides including SiO_2 (9.54 wt %), Al_2O_3 (9.68 wt %), and TiO_2 (0.63 wt %), as well as trace elements including Hg (449.63 ng/g), Zr (285.95 μg/g), Cu (36.72 μg/g), Ga (18.47 μg/g), Se (5.99 μg/g), Cd (0.43 μg/g), Hf (7.14 μg/g), and Pb (40.63 μg/g) are enriched in the coal. Lithium and Hg present strong positive correlations with ash yield and SiO_2, indicating an inorganic affinity. Elements Sr, Ba, Be, As and Ga have strong positive correlations with CaO and P_2O_5, indicating that most of these elements may be either associated with phosphates and carbonates or have an inorganic–organic affinity. Some of the Zr and Hf may occur in anatase due to their strong positive correlations with TiO_2.

Keywords: Pennsylvanian coals; minerals; trace elements; Ningwu Coalfield

1. Introduction

Coal is the most abundant fossil fuel in China, and it is a reliable long-term fuel source for China and other countries, including Turkey and South Africa. With the increasing use of coal in China, a large amount of pollutants is produced, not only in the form of gas emissions but also as ash residues. However, many valuable elements in the coal and coal ash are not yet extracted and used, with the exception of Ge [1–3]. Studies on the geochemistry of elements in coals serve as the basis for the environmental impacts of coals and the efficient use of valuable elements. Many previous investigations have studied the geochemistry and mineralogy of coal deposits around the world, such as the Guanbanwusu and Haerwusu Surface Mines in the Junger Coalfield, northern China [4,5], the Donglin Coal Mine and Xinde Mine, southwestern of China [6,7], Yili Basin, northwestern China [8], the Mariza-east lignite deposit, Bulgaria [9], the Çan coals, Çanakkale, Turkey [10] and Gray Hawk Coal, Eastern Kentucky, USA [11].

As the province with the highest coal production in China, Shanxi has produced up to 505 Mt of raw coal in 2014, which accounts for more than a quarter of China's total coal production. To further understand hazardous elements in coals and in coal combustion products, many coal production areas have been investigated. In this paper, we reported the data on the mineralogy and elemental geochemistry of the No. 4 Coal in the Pingshuo mine, Ningwu Coalfield, Shanxi Province, China.

2. Geological Setting

The Pingshuo mine district covers an area of 396 km^2. The total coal reserves are estimated at 13 billion tons. The mine district is part of the Ningwu Coalfield, which has an area of 2761 km^2 and is located in the northern region of Shanxi Province, north China, in the Ningwu Syncline Basin (Figure 1). To the west of the basin are the Lvliang Mountains and to the east is Wutai Mountain. The base rocks of the basin are metamorphic rocks of the Archean Group [12].

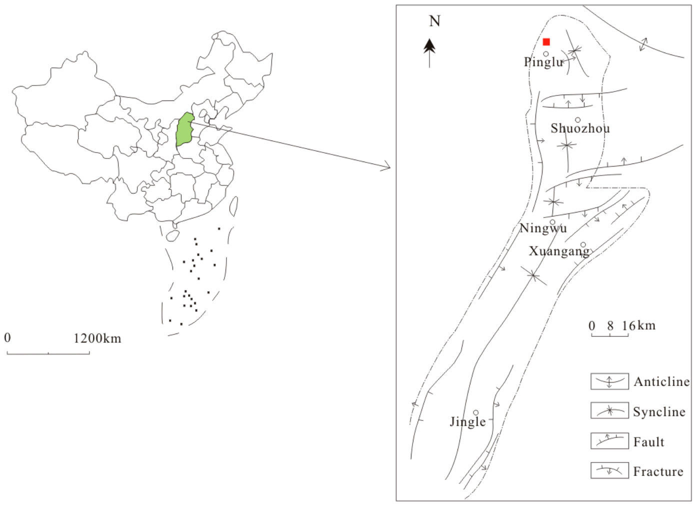


Figure 1. Location of the Pingshuo Mine in the Ningwu Coalfield, Shanxi Province, China.

Coal-bearing strata in the study area occur in the Taiyuan Formation of Upper Carboniferous age and the Shanxi Formation of Lower Permian age [13]. In ascending stratigraphic order, the coal-bearing strata in the area are No. 11, 10, 9, 5 and 4 of the Taiyuan Formation and No. 3 of the Shanxi Formation (Figure 2a).

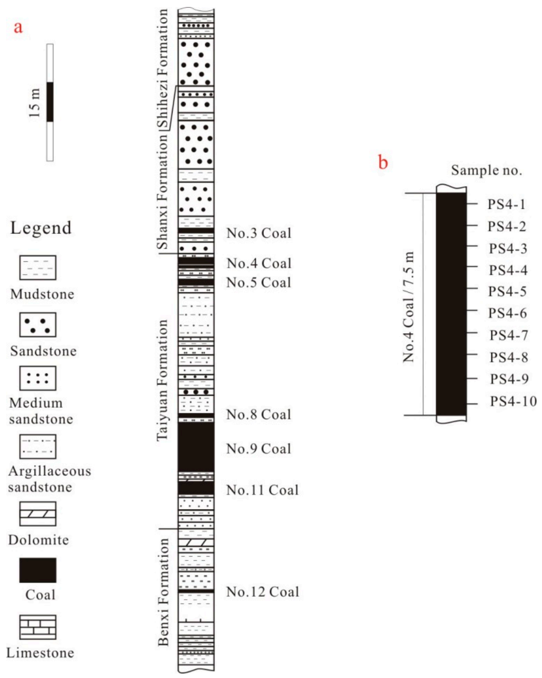


Figure 2. Generalized stratigraphic column: (**a**) of the Late Pennsylvanian-Permian coal measures in the Ningwu coalfield and sampling profiles; and (**b**) of the No. 4 Coal in the Pingshuo Coal Mine.

3. Samples and Analytical Procedures

To examine the mineralogical and geochemical composition of the Ningwu coals, a total of ten samples were collected from the No. 4 Coal in the Pingshuo Coal Mine, Ningwu Coalfield. The cumulative thickness of the No. 4 Coal is approximately 7.5 m. From top to bottom, the ten samples are labeled PS4-1 to PS4-10 (Figure 2b). All samples were air-dried, sealed in polyethylene bags to prevent oxidation, and splits were ground to pass 200-mesh and were stored in brown glass bottles for chemical analyses.

Proximate analyses (A_{ad}, M_{ad}, and V_{daf},) of the coal samples were conducted in accordance with ASTM standards (ASTM D3173-11 [14], ASTM D3175-11 [15], and ASTM D3174-11 [16], respectively). Total sulfur was determined according to the ASTM D3177-02 standard [17].

A scanning electron microscope (SEM) was used to study the surface characteristics and the distribution of minerals in the coal. The accelerating voltage was 20 KV, and the beam current was 10^{-10} A.

LTAs (low-temperature ashes) of the powdered coal samples were produced by an EMITECH K1050X plasma asher (Quorum, Lewes, UK) prior to XRD analysis. XRD analysis of the LTAs was performed on a D/max-2500/PC powder diffractometer (Rigaku, Tokyo, Japan) with Ni-filtered Cu-K radiation and a scintillation detector. Each XRD pattern was recorded over a 2θ interval of 2.6°–70°, with a step size of 0.01°. X-ray diffractograms of the LTAs and non-coal samples were subjected to quantitative mineralogical analysis using the Siroquant™ interpretation software system (Sietronics, Mitchell, Australia). More analytical details are given by Dai *et al.* [18,19] and Wang *et al.* [20].

X-ray fluorescence (XRF) spectrometry (ARL ADVANT′XP+) was performed to determine the major-element oxides (*i.e.*, SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, MnO, Na_2O, K_2O, and P_2O_5) of the high-temperature coal ash samples.

Trace elements, except for As, Se, Hg and F, were determined by inductively coupled plasma mass spectrometry (ICP-MS). For ICP-MS analysis, coal samples were digested using an UltraClave Microwave High Pressure Reactor (Milestone, Sorisole, Italy). The basic load for the digestion tank was composed of 330-mL distilled H_2O, 30-mL 30% H_2O_2, and 2-mL 98% H_2SO_4. Initial nitrogen pressure was set at 50 bars and the highest temperature was set at 240 °C for 75 min. The reagents for 50-mg sample digestion were 2 mL 40% HF, 5 mL 65% HNO_3 and 1 mL 30% H_2O_2. Multi-element standards were used for calibration of trace element concentrations. More details are given in Dai *et al.* [21]. Arsenic and Se were analyzed by a collision/reaction cell technology of inductively coupled plasma mass spectrometry (ICP-CCT-MS), as described by Li *et al.* [22]. Fluorine was determined by the ion-selective electrode (ISE) method. Mercury was determined using a Milestone DMA-80 Hg Analyzer (Milestone).

The quantitative analysis of minerals and determinations of elements were completed at the State Key Laboratory of Coal Resources and Safe Mining of China University of Mining and Technology (Beijing, China).

4. Results and Discussion

4.1. Coal Chemistry

The results of the proximate analysis and the total sulfur of the Late Pennsylvanian coal samples are presented in Table 1. Ash yields of the Pingshuo No. 4 coal range from 12.96% to 32.14%, with an average of 21.42%, indicating a medium ash coal according to Chinese National Standards (GB/T 15224.1-2004, which shows 10.01%–16.00% ash yield is for low-ash coal, 16.01%–29.00% ash yield for medium-ash coal, and >29.00% ash yield for high-ash coal) [23]. The ash yields range irregularly from bottom to top through the coal-seam section (Figure 3).

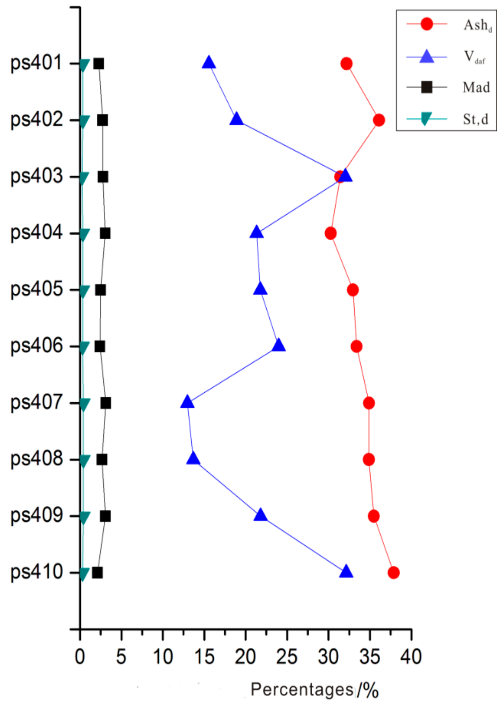


Figure 3. Variation of total sulfur and proximate analysis through the Pingshuo coal section.

Table 1. Proximate analysis and total sulfur in the Pingshuo coals (%).

Sample	Proximate Analysis			$S_{t,d}$
	M_{ad}	V_{daf}	A_d	
PS4-1	2.28	32.19	15.56	0.33
PS4-2	2.74	36.11	18.91	0.36
PS4-3	2.78	31.43	32.02	0.25
PS4-4	3.05	30.27	21.33	0.33
PS4-5	2.49	32.95	21.78	0.34
PS4-6	2.41	33.40	24.00	0.32
PS4-7	3.12	34.89	12.96	0.43
PS4-8	2.66	34.88	13.69	0.41
PS4-9	3.07	35.47	21.80	0.43
PS4-10	2.12	37.88	32.14	0.31
Average	2.67	33.95	21.42	0.30

M, moisture; V, volatile matter; A, ash yield; St, total sulfur; ad, air-dry basis; d, dry basis; daf, dry and ash-free basis.

The volatile matter yields of the No. 4 coal vary from 30.27% to 37.88% through the coal-seam section, with a mean of 33.95%, suggesting that the Late Pennsylvanian coals in the Pingshuo coal mine are medium-high volatile bituminous coals based on Chinese Standard MT/T 849-2000 (28.01% to 37.00% for medium-high volatile coal, 37.01% to 50.00% for high volatile coal and >29.00% for super high volatile coal) [24].

The coals from the Pingshuo coal mine have a moisture content of 2.12% to 3.12%, with an average of 2.67%, indicating a low-medium rank coal, in accordance with MT/T 850-2000 (⩽5% for low moisture coal, 5% to 15% for medium moisture coal, and >15% for high moisture coal) [25].

The total sulfur of the No. 4 coals ranges from 0.25% to 0.43%, averaging 0.30%, which corresponds to a super-low sulfur coal according to Chinese National Standards (GB/T 15224.2-2004) (<0.5% for super-low sulfur coal, 0.51%–0.9% for low-sulfur coal and 0.9%–1.50% for medium-sulfur coal) [26].

4.2. Minerals in Coal

The mineral phase percentages were calculated on a coal ash basis from the XRD results obtained for the low temperature ashes and are reported in Table 2. The results show that minerals in the Pingshuo coals mainly consist of kaolinite, followed by boehmite (averaging 5.09%), siderite (1.35%), anatase (0.29%), goyazite (0.8%), calcite (0.07%), apatite (0.01%) and florencite (0.33%) (Figure 4).

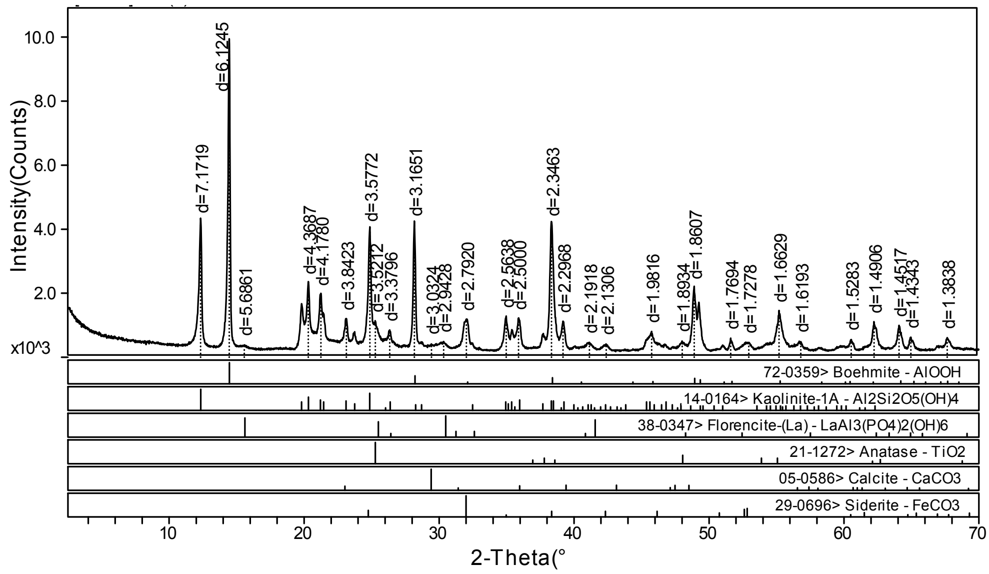


Figure 4. XRD patterns of coal samples (PS4-9).

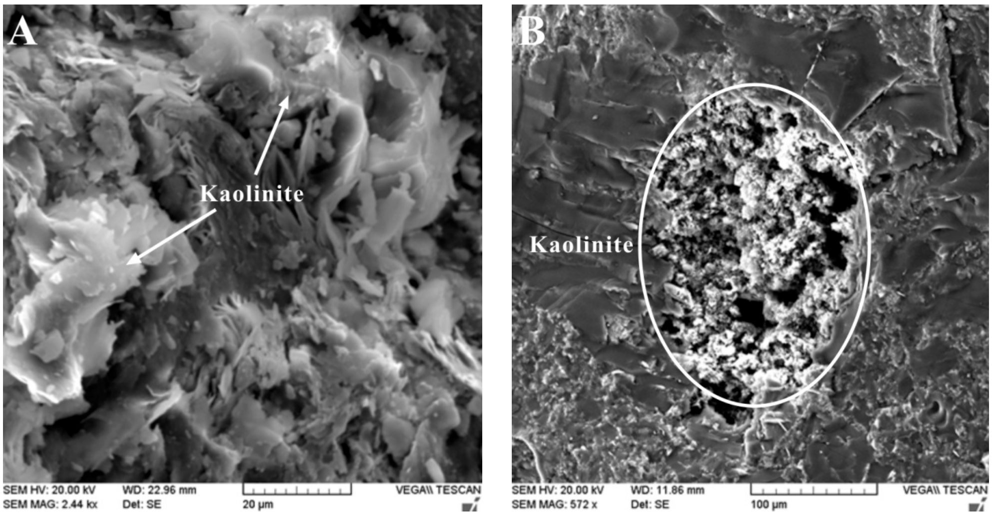
Table 2. Mineral contents in coal samples from the Pingshuo Mine measured by LTA-XRD (%).

Samples	PS4-1	PS4-2	PS4-3	PS4-4	PS4-5	PS4-6	PS4-7	PS4-8	PS4-9	PS4-10
LTA yield	15.04	15.25	30.58	20.62	20.81	23.11	13.15	21.03	28.89	13.91
Kaolinite	13.72	11.16	27.82	12.47	12.53	11.49	7.72	13.36	14.30	12.79
Boehmite	0.92	0.05	1.87	5.77	7.89	11.44	4.47	6.04	11.61	0.83
Arsenopyrite	0.33									
Rutile	0.08			0.06						
Siderite		3.54	0.31			0.16	0.88	1.54	1.70	
Apatite		0.27			0.27					
Hexahydrite		0.12								
Goyazite		0.08		2.25	0.08					
Anatase		0.03	0.58			0.02		0.11	0.69	
Florencite					0.02				0.49	
Calcite							0.08		0.06	
Quartz										0.29

Kaolinite is a very common clay mineral in coal [27,28]. As presented in Table 2, kaolinite is the most abundant mineral in the Pingshuo coals, with abundance varying from 7.72% to 27.82% (13.74% on average). Kaolinite occurs as infillings of cells or fractures (Figure 5a) and as thin-layered or flocculent forms (Figure 5b) in the No. 4 Coal.

The percentage of boehmite in Pingshuo coals varies from 0.05% to 11.61%, with an average of 5.09%. As for the variation of content in the coal profile, boehmite is lower in the top and bottom portions than in the middle portion. Some trace elements (Ga, F) occur in boehmite according to a previous study [5].

Other minerals are detected in only a few samples, and there are no obvious vertical trends of their distribution.



(**a**) (**b**)

Figure 5. Minerals in the Pingshuo coals (SEM, secondary electron images): (**a**) kaolinite in thin-layered forms; and (**b**) flocculent kaolinite.

4.3. Abundance of Elements in Pennsylvanian Coals

The percentages of major-element oxides and concentrations of trace elements in the Pennsylvanian coal samples from the Pingshuo Mine, in comparison with the average values of Chinese coals [29] or world hard coals [30], are listed in Table 3.

Table 3. Contents of major-element oxides and trace elements in Pennsylvanian coals from the Pingshuo Mine (LOI, oxides in %, elements in μg/g) (whole-coal basis).

Elemental Contents	Samples											
	PS4-1	**PS4-2**	**PS4-3**	**PS4-4**	**PS4-5**	**PS4-6**	**PS4-7**	**PS4-8**	**PS4-9**	**PS4-10**	**Average**	**Coal** [b]
LOI	84.9	84.8	69.4	79.4	79.2	76.9	86.9	79.0	86.1	71.1	79.8	-
SiO_2	8.17	7.15	16.3	9.84	9.47	9.08	5.18	5.86	7.61	16.7	9.54	8.47 [a]
TiO_2	0.42	0.57	0.81	0.90	0.69	0.79	0.43	0.36	0.93	0.41	0.63	0.33 [a]
Al_2O_3	6.63	6.61	14.0	8.10	10.4	13.2	6.15	6.44	11.2	14.1	9.68	5.98 [a]
Fe_2O_3	0.06	3.33	0.36	0.53	0.58	0.42	0.67	0.59	1.13	0.33	0.80	4.85 [a]
Na_2O	0.01	0.01	0.03	0.02	0.02	0.02	0.01	0.01	0.02	0.03	0.02	0.16 [a]
K_2O	0.01	0.02	0.18	0.09	0.17	0.10	0.05	0.06	0.11	0.06	0.08	0.19 [a]
CaO	0.09	0.25	0.10	0.28	0.11	0.10	0.12	0.10	0.17	0.11	0.14	1.23 [a]
MgO	0.02	0.15	0.06	0.06	0.08	0.06	0.07	0.08	0.10	0.05	0.07	0.22 [a]
P_2O_5	0.02	0.21	0.02	0.86	0.02	0.03	0.04	0.02	0.19	0.14	0.15	0.09 [a]
MnO	-	0.06	-	-	-	-	0.01	-	0.01	-	0.03	0.02 [a]
Li	4.82	12.2	27.4	10.6	3.95	11.5	12.6	10.2	3.12	42.4	13.9	14.0
Be	2.66	1.88	2.49	4.16	2.48	1.94	1.70	1.55	1.60	0.87	2.13	2.00
F	81.5	105	123	384	194	203	116	11	241	117	167	140
Sc	2.08	13.3	2.55	4.84	1.75	1.59	2.74	1.62	2.03	1.56	3.40	3.00
V	18.7	24.7	46.6	42.3	42.4	35.8	39.2	26.4	40.3	21.9	33.8	21.0
Cr	5.09	6.99	12.4	12.9	9.21	21.9	8.75	10.9	16.0	16.3	12.1	12.0
Co	2.83	1.52	0.68	0.66	1.11	0.88	1.29	1.29	1.04	1.08	1.24	7.00
Ni	4.31	4.54	3.99	1.57	2.28	4.02	1.94	2.31	3.13	5.46	3.36	14.0
Cu	85.6	54.2	39.4	39.3	31.1	33.8	24.2	23.0	24.3	12.4	36.7	13.0
Zn	65.3	26.8	19.6	16.6	14.7	13.5	18.8	12.3	23.5	12.5	22.4	35.0
Ga	11.4	21.5	19.8	32.4	25.0	16.1	19.4	18.8	10.9	9.62	18.5	9.00
Ge	1.16	1.23	0.41	1.93	0.80	0.48	0.73	0.76	0.52	0.38	0.84	2.78
As	1.71	1.87	0.94	2.54	0.83	0.77	0.92	0.71	0.75	0.79	1.18	5.00
Se	3.56	4.85	5.68	6.60	5.75	9.73	5.77	4.08	6.46	7.45	5.99	2.00
Hg	0.18	0.13	0.08	0.09	0.34	0.09	0.07	0.09	0.37	0.31	0.18	0.15
Rb	0.28	0.19	3.77	1.70	3.88	1.82	0.81	1.27	1.96	1.55	1.72	8.00

Table 3. *Cont.*

Elemental Contents	Samples											
	PS4-1	PS4-2	PS4-3	PS4-4	PS4-5	PS4-6	PS4-7	PS4-8	PS4-9	PS4-10	Average	Coal [b]
Sr	74.5	710	35.6	2908	57.7	52.1	166	70.8	557	264	229	423
Zr	107	250	691	276	365	184	282	144	405	157	286	52.0
Mo	2.10	1.51	0.77	1.34	2.01	1.82	1.80	2.07	1.63	1.04	1.61	4.00
Cd	0.33	0.43	0.91	0.39	0.50	0.25	0.39	0.25	0.57	0.24	0.43	0.20
Sn	3.60	3.12	4.79	3.23	5.52	2.73	1.52	1.90	3.52	2.13	3.21	2.00
Sb	0.29	0.38	0.27	0.27	0.53	0.19	0.41	0.32	0.46	0.14	0.33	2.00
Cs	0.02	0.02	0.13	0.07	0.19	0.08	0.03	0.06	0.09	0.13	0.08	1.00
Ba	16.2	64.9	25.0	213	26.7	20.4	30.0	20.8	54.6	28.5	50.0	56.0
Hf	2.74	6.02	15.6	6.75	9.59	4.84	7.51	3.87	10.4	4.06	7.14	2.40
Ta	0.47	1.12	1.41	1.36	3.33	1.02	0.77	0.62	1.27	0.88	1.22	0.70
W	5.02	1.20	1.40	1.86	1.66	1.53	0.55	0.84	1.14	0.84	1.60	2.00
Tl	0.05	0.06	0.04	0.05	0.10	0.05	0.09	0.09	0.13	0.09	0.07	0.40
Pb	31.4	38.2	39.5	40.6	97.3	40.0	32.1	24.6	49.2	13.3	40.6	13.0
Bi	0.21	0.41	0.54	0.56	0.61	0.55	0.33	0.35	0.54	0.32	0.44	0.80
Th	5.52	13.1	4.85	20.6	12.0	2.76	14.3	10.0	13.8	1.02	9.78	6.00
U	1.87	3.33	4.56	5.40	5.86	3.85	4.12	2.84	4.40	2.08	3.83	3.00

LOI, loss on ignition; [a] Chinese average coals value by Dai *et al.* [29]; [b] world hard coals by Ketris and Yudovich [30].

The major elements in the Pingshuo coals are dominated by SiO_2 and Al_2O_3. The main carriers of these elements are quartz and clay minerals [31,32]. Average values of major-element oxides in high-temperature ashes of Pingshuo coal samples are as follows: SiO_2 (9.54 wt %), Al_2O_3 (9.68 wt %), Fe_2O_3 (0.80 wt %), TiO_2 (0.63 wt %), CaO (0.14 wt %), K_2O (0.08 wt %), MgO (0.07 wt %), Na_2O (0.02 wt %), P_2O_5 (0.15 wt %) and MnO (0.27 wt %). Compared with the average values of Chinese coals [29], the Pennsylvanian coals from the Pingshuo Mine contain higher proportions of SiO_2, Al_2O_3, TiO_2, and P_2O_5 and lower proportions of Fe_2O_3, CaO, K_2O, MgO and Na_2O.

The SiO_2/Al_2O_3 ratios range from 0.68 to 1.23, with an average of 0.99 for the Pennsylvanian coals. This range is lower than those of other Chinese coals (1.42) [25] and lower than the theoretical SiO_2/Al_2O_3 ratio of kaolinite (1.18), in accordance with the occurrence of boehmite and the lack of quartz in the coal.

In comparison with Chinese coals [29] or world hard coals [30], Zr (CC = 5.50) is substantially higher in the Pennsylvanian coals from the Pingshuo Mine. Copper (CC = 2.82), Ga (CC = 2.05), Se (CC = 2.99), Cd (CC = 2.14), Hf (CC = 2.97) and Pb (CC = 3.13) are slightly higher than the average values of Chinese coals [29], whereas Li (CC = 0.99), Be (CC = 1.07), F (CC = 1.20), Sc (CC = 1.13), V (CC = 1.61), Cr (CC = 1.00), Zn (CC = 0.64), Hg (CC = 1.20), Sr (CC = 1.16), Sn (CC = 1.60), Ba (CC = 0.89), Ta (CC = 1.75), W (CC = 0.80), Bi (CC = 0.55), Th (CC = 1.63) and U (CC = 1.27) are very close to those of Chinese coals, and the remaining trace elements are lower than the averages of Chinese and world hard coals (CC < 0.5) (CC, concentration coefficient, the ratio of trace element concentration in coal samples investigated *vs.* averages for Chinese or world hard coals [33]) (Figure 6).

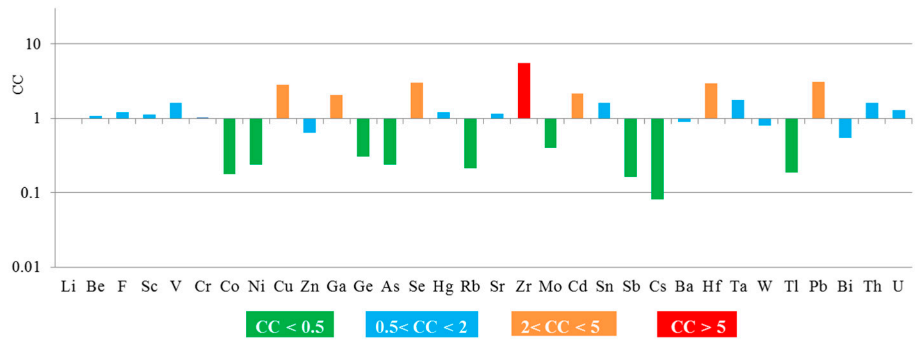


Figure 6. Concentration coefficients (CC) of trace elements in the Pingshuo coals.

4.4. Geochemical Associations in the Coal Samples

The modes of occurrence of trace elements in the Pingshuo coals were preliminarily investigated using cluster analysis and correlation coefficients, which are effective indirect methods for coal geochemistry [34]. Pearson correlation coefficients were used in hierarchical clustering. Elements that are the most strongly correlated are linked first, followed by elements or element groups with decreasing correlations, until a complete dendrogram is achieved, as reported by Zhao *et al.* [35].

Associations of trace elements in the Pingshuo coals are indicated by the hierarchical clustering dendrogram (Figure 7). Elements in the Pingshuo coals are divided into six groups. Apart from the inter-correlation among elements in the same group, each group contains elements of different sub-groups that have different correlations with ash yield and some oxides in the coals. The modes of occurrence of trace elements could be deduced based on the cluster analysis results and correlation coefficients.

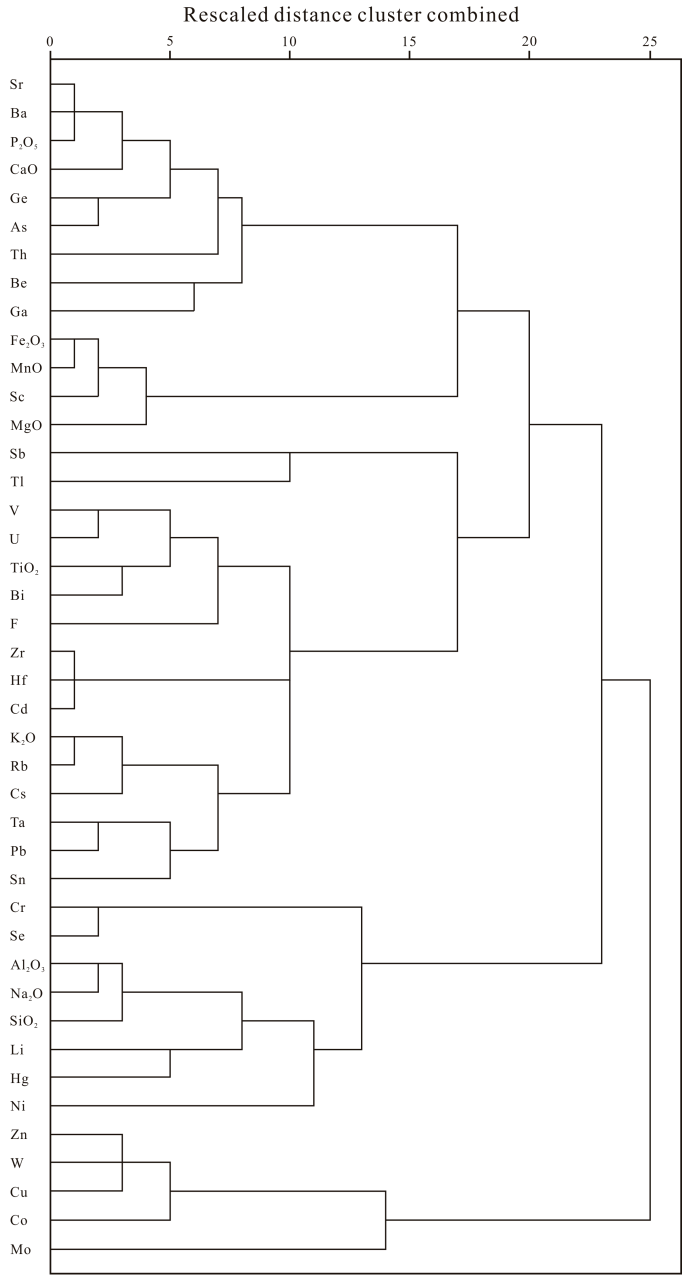


Figure 7. Cluster analysis of the geochemical data of the Pennsylvanian Pingshuo coal samples.

Group 1 includes Al_2O_3, SiO_2, Na_2O, Li, Hg, Ni, Se, and Cr (Figure 7). Elements in Group 1 are all strongly correlated with Al_2O_3 or SiO_2, with high correlation coefficients ($r > 0.60$, ranging from 0.62 to 0.81). In addition, these elements are slightly correlated with ash yields (rLi-ash = 0.72, rHg-ash = 0.56, rNi-ash = 0.54, rSe-ash = 0.53, and rCr-ash = 0.55). Most of the elements in this group probably have the same modes of occurrence.

Group 2 includes P_2O_5, CaO, Ba, Sr, As, Th, Be, and Ga (Figure 7). Their correlation coefficients with P_2O_5 are rBe = 0.68, rGa = 0.61, rAs = 0.78, rSr = 0.99, rBa = 0.99, and rTh = 0.66, indicating that P_2O_5 and these elements are probably associated with phosphate minerals (goyazite, apatite, and florencite in coal samples), which were detected by previous investigations [35–37]. Ba, Sr, As, and Th also have strong correlations with CaO, and they have a carbonate affinity.

Group 3 includes Fe_2O_3, MgO, MnO and Sc (Figure 7). Elements in this group have a strong correlation with each other ($r > 0.92$). Fe_2O_3 has strong correlations with total sulfur in the coal samples (Figure 8), indicating most of elements in Group 3 probably occur in sulfides in the Pingshuo coals.

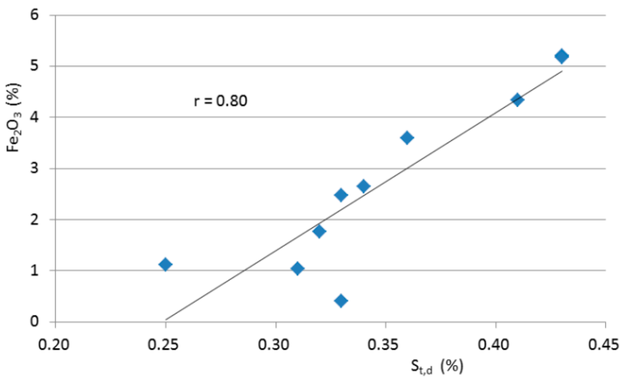


Figure 8. Correlations between Fe_2O_3 and total sulfur in the Pingshuo coals.

Group 4 includes three subgroups. Subgroup 1 includes TiO_2, Bi, V, U, F, Zr, Hf and Cd. The correlation coefficients of trace elements in this subgroup with TiO_2 are larger than 0.72 (rBi = 0.87, rV = 0.74, and rU = 0.72), indicating that TiO_2 and these elements probably occur in anatase. Subgroup 2 includes K_2O, Rb and Cs. Elements in this group have strong correlations with K_2O (rRb = 0.99 and rCs = 0.86). Subgroup 3 includes Ta, Pb and Sn. These elements are weakly correlated with ash yield (r varies from −0.19 to 0.36), SiO_2 (r varies from −0.30 to 0.32) or Al_2O_3 (r varies from −0.01 to 0.32).

Group 5 includes Zn, W, Cu, Co and Mo (Figure 7). These metal elements present negative correlations with ash (r ranging from −0.81 to −0.29), SiO_2 (r is from −0.81 to −0.18) and Al_2O_3 (r ranging from −0.60 to −0.39).

Group 6 includes Sb and Tl (Figure 7). Their correlation coefficient is 0.55, and there are no obvious correlations with ash, SiO_2, Al_2O_3 or other elements.

4.5. Modes of Occurrence of Some Trace Elements in Coals

Based on the cluster analysis, many elements are associated with SiO_2, Al_2O_3 and other elements in the coals with different degrees of affinity. Apart from these correlations, ash yield and other factors may also affect the abundance and modes of occurrence of trace elements in coals.

4.5.1. Li and Hg

Lithium (Li) is a very important element used for batteries and in other industries [38]. Many authors have investigated Li enrichment [38,39]. The average Li content in coals is 14 μg/g globally and 31.8 μg/g in China [29]. The Li contents of the Pingshuo coals range from 3.12 to 42.40 μg/g, averaging 13.87 μg/g (Table 3), close to the world hard coal content (CC = 0.99). Modes of occurrence of Li in coal have not been fully addressed in other studies because of its low atomic number and its reduced level of toxicity to the environment compared to many other elements [4]. Limited previous studies show that Li in coals is generally associated with aluminosilicate minerals [4,38,39] and organic matter. Lithium in the Pingshuo coals presents strong positive correlations with ash yield, SiO_2 and Al_2O_3 (Figure 9), indicating that Li may be associated with aluminosilicate minerals in the Pingshuo coals.

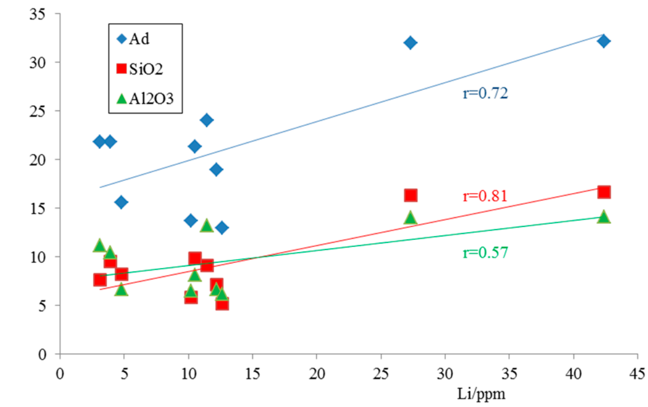


Figure 9. Correlations between Li and ash yield (Ad), SiO_2 and Al_2O_3.

The coal samples from Pingshuo are enriched in mercury (Hg) (CC = 1.20). The Hg concentrations vary between 0.07 and 0.37 µg/g, with an average value of 0.18 µg/g. There have been many reports on the modes of occurrence of Hg in coals [40,41], and most of the results agree that mercury in coals has a sulfide affinity, usually in pyrite [42–44], getchellite [45], or organic matter [46]. However, none of these associations are indicated in the Pingshuo coals. There is no obvious correlation between Hg and total sulfur contents in the coal samples (Figure 10), and the correlation coefficients between Hg and ash yield, SiO_2 and Li are 0.56, 0.62 and 0.78, respectively, indicating that Hg is associated with aluminosilicate minerals other than sulfides.

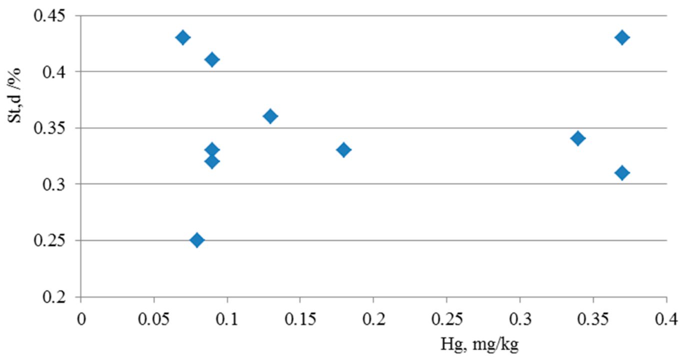


Figure 10. Correlations between Hg and total sulfur.

4.5.2. Sr, Ba, Be, As and Ga

The concentrations of Sr and Ba in coal samples are very close to the averages for Chinese or world hard coals (CC = 1.16 and 0.89), with average contents of 489.50 and 50.02 µg/g, respectively. The concentration of P_2O_5 shows positive correlations with Sr and Ba in the coal samples (Figure 11), with correlation coefficients of 0.99, indicating that most of the Sr and Ba in Pingshuo coals may occur in aluminophosphates (goyazite and gorceixite), as previously reported [35,47]. Sr and Ba also have positive correlations with CaO in the samples (Figure 11), indicating that the Sr and Ba in the coals exhibit some carbonate affinity.

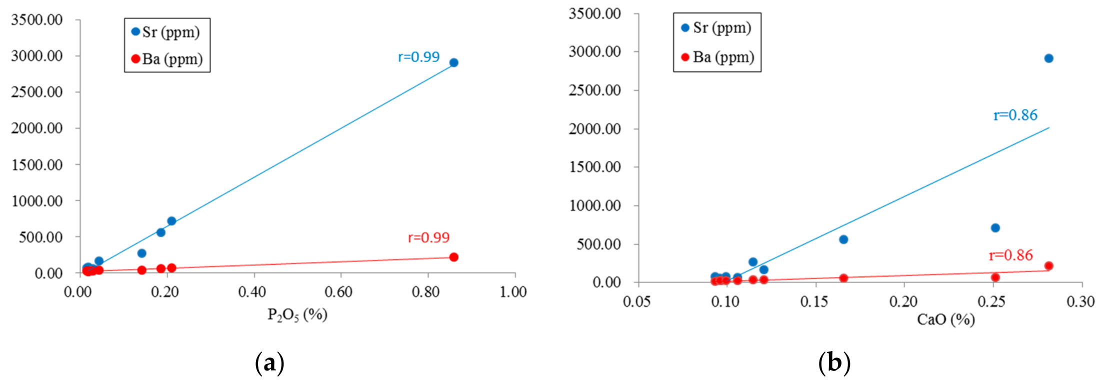


Figure 11. Correlations of Sr and Ba with: P_2O_5 (**a**); and CaO (**b**).

The concentration of beryllium (Be) in the Pingshuo coals is close to the average content of Chinese coals (CC = 1.07). Modes of occurrence of Be in the coal have been addressed in previous studies [9,41], and Be has a mixed inorganic–organic affinity in the Yimin Ge-rich coals [48]. The correlation coefficients for Be-P_2O_5 (r = 0.68), Be-Sr (r = 0.71) and Be-Ba (r = 0.73) in the present study indicate that the Be has a mixed inorganic–organic affinity.

A wide variety of As-bearing phases has been observed in the Guizhou high-As coals, including pyrite, Fe-As oxides, and As-bearing clays [49,50]. Arsenic in the Chongqing coals is correlated with Fe_2O_3, suggesting a pyrite affinity [6,44]. The correlation coefficient between As and ash yield is 0.20, which indicates As in the Pingshuo coals has an inorganic–organic affinity. Arsenic has a positive correlation with CaO and P_2O_5 (rCaO = 0.79, rP_2O_5 = 0.78), which suggests that some As may be affiliated with phosphates and carbonates in the Pingshuo coals.

According to previous research, Gallium (Ga) is generally related to clay minerals in coal [39,51]. The modes of Ga occurrence in the Guanbanwusu, Haerwusu and Heidaigou coals are slightly different. Ga in the Haerwusu coals occurs mainly in boehmite and organic matter [27]. The correlation coefficient between Ga and ash yield is −0.15, suggesting that Ga has mixed affinities in coal samples. The strong positive correlation between Ga and P_2O_5 (r = 0.61) suggests that Ga mainly occurs in inorganic associations.

4.5.3. Zr and Hf

The concentration of Zirconium (Zr) in Pingshuo coals is considerably higher than the average content of Chinese coals (CC = 5.50), whereas that of hafnium (Hf) is slightly higher (CC = 2.97). Zr and Hf show similar distributions in the coal samples (Figure 12), and their correlation coefficient is 0.99, indicating that they have nearly the same modes of occurrence in the Pingshuo coals.

Zircon is the most common zirconium mineral; therefore, Zr is believed to be at least partly due to the probable presence of this heavy mineral in these samples [5]. The fine Zr-phases (<0.5 μm), probably zircon, were detected in anatase [39,51]. Zr and Hf have strong positive correlations with TiO_2 and K_2O (Figure 13), indicating that some of Zr and Hf may occur in zircons included in the anatase.

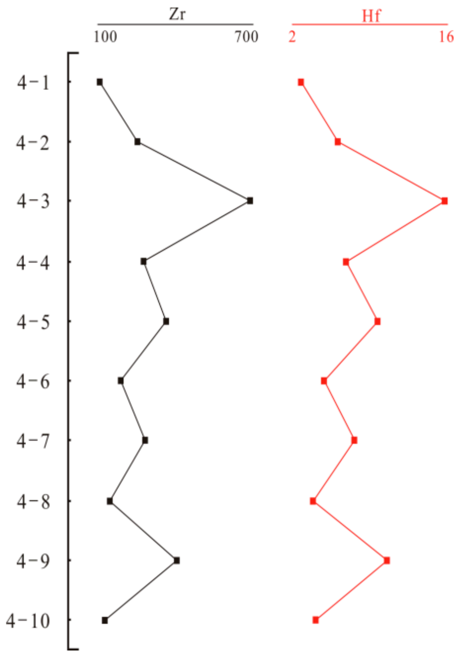
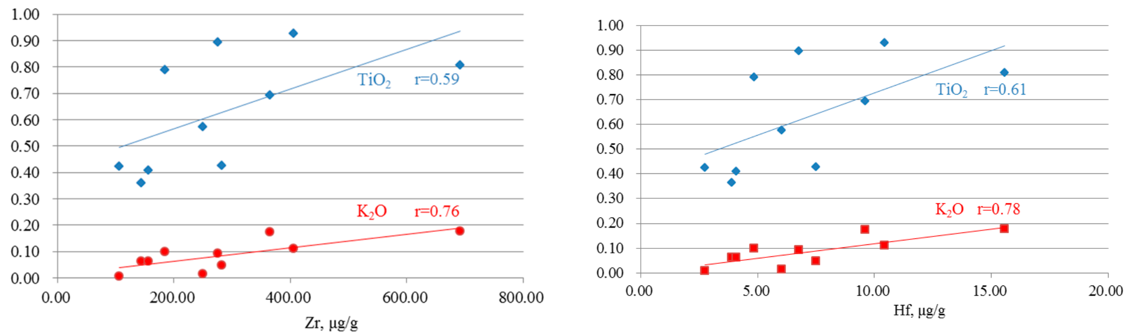


Figure 12. Vertical variation of Zr and Hf in the profile of the Pingshuo coals.



(**a**) (**b**)

Figure 13. Correlations of Zr and Hf with: TiO_2 (**a**); and K_2O (**b**).

5. Conclusions

The Pingshuo coals of the Ningwu Coalfield have a medium-ash yield (averaging 21.42%) and an ultra-low-sulfur content (0.30%), with mean volatile matter and moisture contents of 33.95% and 2.67%, respectively.

The minerals in the Pingshuo coals mainly consist of kaolinite, followed by boehmite (averaging 5.09%), siderite (1.35%), anatase (0.29%), goyazite (0.8%), calcite (0.07%), apatite (0.01%) and florencite (0.33%).

The Pennsylvanian coals from the Pingshuo Mine contain higher proportions of SiO_2, Al_2O_3, TiO_2, and P_2O_5 and lower proportions of Fe_2O_3, CaO, K_2O, MgO and Na_2O compared with the average values for common Chinese coals [26].

Based on the CC values, Hg and Zr concentrations are considerably higher in the Pennsylvanian coals from the Pingshuo Mine. Concentrations of elements Cu, Ga, Se, Cd, Hf and Pb are slightly higher, and the those of Li, Be, F, Sc, V, Cr, Zn, Sr, Sn, Ba, Ta, W, Bi, Th and U are very close to common Chinese and world hard coals. The remaining trace elements are much lower than the averages for common Chinese and world hard coals.

The modes of occurrence of trace elements in the Pingshuo coals were preliminarily investigated by using cluster analysis and correlation analysis. Elements Li and Hg show strong positive correlations with ash yield and SiO_2, indicating that Li and Hg occur in mineral phases. Elements Sr, Ba, Be, As and Ga have strong positive correlations with CaO and P_2O_5, indicating that most of these elements may be affiliated with phosphates and carbonates or have an inorganic–organic affinity in the Pingshuo coals. Elements Zr and Hf have strong positive correlations with TiO_2 and K_2O, indicating part of them occur in anatase.

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Author Contributions: Ning Yang conceived the overall experimental strategy and analyzed testing data of coal samples. Shuheng Tang guided all experiments. Songhang Zhang and Yunyun Chen processed pictures. All authors participated in writing the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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