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Mild oxidation of Jincheng No. 15 anthracite

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Abstract：Jincheng No. 15 anthracite （J15A） was subjected to ruthenium ion-catalyzed oxidation to characterize its structural feature. The results show that J15A is abundant in *peri*-condensed aromatic structure. The NaOCl oxidation of J15A promoted by pretreatment with H_2O_2 was conducted under mild conditions to produce benzene polycarboxylic acids （BPCAs）. The pretreatment with H_2O_2 is proved to enhance the yields of BPCAs. It is potential to obtain BPCAs in high yield and selectivity by NaOCl oxidation of pretreated J15A with H_2O_2.

Keywords：anthracite；benzene polycarboxylic acid；mild oxidation

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Coal oxidation is effective to obtain organic acids and investigate coal structure at molecular level[1-4]. The high rank coals present poor oxidation reactivity but they can produce a large amount of benzene polycarboxylic acids （BPCAs） by oxidation[5]. BPCAs are important chemicals. They can be used in polymer field （e. g. , terephthalic and pyromellitic acid） and pharmaceutical field （e. g. , trimesic and mellitic acid）[6,7]. Qin et al[8] optimized the alkali-oxygen oxidation of Yangquan anthracite to effectively get BPCAs. Wang et al[9] investigated the high-temperature alkali-oxygen oxidation of a bituminous coal to produce BPCAs，which found that high-temperature drastically decreased the alkali consumption and improved the production efficiency of BPCAs. However，high temperature and high pressure would limit the practical application of alkali-oxygen oxidation of coals.

As an effective and mild method for selective elimination of aromatic rings，ruthenium ion-catalyzed oxidation （RICO） has been successfully used to characterize alkylene bridges，alkyl side chains and condensed aromatic structures in coals[3,10~14] and other fossil resources[15~18]. However，the industrial application of RICO is difficult due to its high cost. Sodium hypochlorite （NaOCl） is potential to be used as an oxidant for coal oxidation to generate organic acids because of its easy availability，high reactivity，environmental friendliness，and electrolytic renewability[19]. Mayo et al[4] conducted oxidations of Illinois No. 6 coal and its pyridine-inextractable portion in aqueous NaOCl. The resulting products could be classified into black acids （up to 1000 Da） and nearly colorless water-soluble acids （300 ~ 400 Da）. According to our previous work[20], BPCAs with 4-6 -COOH are main products from NaOCl oxidation of several Beisu coal chars with highly condensed aromatic structures.

H_2O_2 is usually used as a pretreatment agent to promote hydrogenolysis[21]，extraction[22]，desulfurization[23]，and flash pyrolysis[24] of coals. Our group[25] investigated the effect of the pretreatment with H_2O_2 on NaOCl oxidation of Shengli lignite. It was found that the pretreatment with H_2O_2 improved the yields of organic acids，and suppressed the formation of chloro-substituted alkanoic acids.

Jincheng is rich in anthracites and is one of major anthracite bases in China. The reserves of Jincheng No. 15 anthracite （J15A） account for ca. 33% of the total anthracite reserves in the region[26]. J15A is considered to be an inferior coal because of its high ash yield and high sulfur content. In this study，we first investigated the structural features of J15A using RICO and then focused on the BPCA distributions and yields from NaOCl oxidation promoted by pretreatment with H_2O_2.

1　Experimental

1.1　Coal sample

J15A was collected in Jincheng，Shanxi province，China. It was pulverized to pass through a

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200-mesh sieve (particle size of < 75 μm) followed by desiccation in a vacuum oven at 80 ℃ for 24 h prior to use. Table 1 shows the proximate and ultimate analyses of J15A. CH_2Cl_2, CS_2, (CH_3CH_2)$_2O$, HCl aqueous solution (36%), H_2O_2 aqueous solution (30%), and NaOCl aqueous solution (6% available chlorine) used in the experiments are analytical reagents. All the organic solvents were distilled before use.

Table 1　Proximate and ultimate analysis of J15A

Proximate analysis w /%			Ultimate analysis w_{daf}/%				
M_{ad}	A_d	V_{daf}	C	H	N	S	O^a
2.31	38.82	4.04	52.04	1.147	0.541	4.19	0.95

a by difference

1. 2　Procedure for RICO and subsequent treatments

According to a reported procedure[14,27], ca. 0.4 g J15A, 40 mg $RuCl_3$, 40 mL CH_3CN, 40 mL CCl_4, and 60 mL distilled water were added to a 250 mL spherical flask. Then 8 g $NaIO_4$ was added to the mixture followed by magnetical agitation at 35 ℃ for 72 h. The reaction mixture was separated by filtration and phase separation to afford the organic phase (OP) and aqueous phase (AP). Both the filter cake and AP were extracted with CH_2Cl_2. The extraction solution was incorporated with the above OP and then dried over anhydrous $MgSO_4$. Then solvents in OP and AP were removed by rotary evaporation followed by esterification with CH_2N_2 in (CH_3CH_2)$_2O$ to afford corresponding methyl esterified OP (MEOP) and methyl esterified AP (MEAP).

1. 3　Pretreatment of J15A with H_2O_2 and subsequent NaOCl oxidation

As described in our previous work[25], J15A (ca. 10 g) was mixed with 50 mL of H_2O_2 aqueous solution in a 250 mL beaker with magnetical stir at room temperature for 12 h. The reaction mixture was filtrated. The filtrate was evaporated under reduced pressure followed by esterification with CH_2N_2. The residue, i. e., pretreated J15A (PTJ15A), was dried and transferred into a desiccator for subsequent oxidation in NaOCl aqueous solution.

As shown in Figure 1, ca. 9 g PTJ15A and 135 mL NaOCl aqueous solution were added to a 250 mL spherical flask and fully mixed by magnetical agitation at 35 ℃ for 72 h. Then 1 g Na_2SO_3 was added into the spherical flask to decompose excess NaOCl. Then the reaction mixture was separated to filter cake 1 (FC_1) and filtrate 1 (F_1) by filtration

through a membrane filter with 0.45 μm of pore size. FC_1 was dried in a vacuum at 80 ℃ for 24 h and then weighed. F_1 was acidized with HCl until pH < 2 to convert -COONa to -COOH in the reaction mixtures, and filtrated to afford filter cake 2 (FC_2) and filtrate 2 (F_2). F_2 was sequentially extracted with CS_2 to afford extraction solution 1 (ES_1) and inextractable solution 1 (IES_1). IES_1 was then extracted with (CH_3CH_2)$_2O$ to afford extraction solution 2 (ES_2) and inextractable solution 2 (IES_2). ES_1 and ES_2 were dried over anhydrous $MgSO_4$ and filtrated to remove $MgSO_4 \cdot nH_2O$. Then the solvents in ES_1, ES_2 and IES_2 were removed to afford the extract 1 (E_1), extract 2 (E_2) and inextractable fraction (IEF), correspondingly. E_1, E_2 and IEF were esterified with CH_2N_2 to afford methyl esterified E_1 (MEE_1), methyl esterified E_2 (MEE_2) and methyl esterified IEF (MEIEF), respectively.
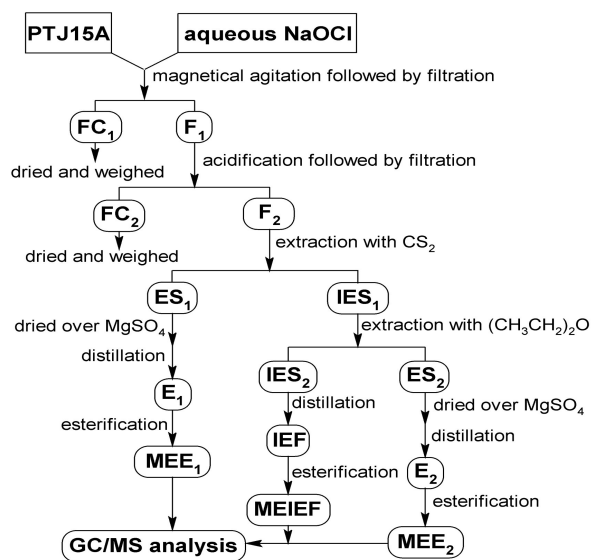

Figure 1　Procedure for NaOCl oxidation of PTJ15A

1.4　Analytical methods

J15A, PTJ15A and the residue from NaOCl oxidation of PTJ15A (i. e., FC_1) were dehydrated and were analyzed by a Nicolet Magna IR-560 Fourier transform infrared (FT-IR) spectrometer by collecting 50 scans at a resolution of 8 cm^{-1} in reflectance mode with measuring regions of 4 000 ~ 400 cm^{-1}. All the FT-IR spectra were processed on automatic baseline correction and smooth with Omnic software.

All the methyl esterified samples including MEOP, MEAP, MEE_1, MEE_2 and MEIEF were analyzed with a Hewlett-Packard 6890-5973 gas chromatography/mass spectrometer (GC/MS), which is equipped with a capillary column coated with HP－5MS (cross－link 5% PH ME siloxane, 60 m length, 0.25 mm inner diameter, 0.25 μm film

thickness）and a quadrupole analyzer with a *m/z* range from 33 to 500 and operated in electron impact （70 eV）mode. The capillary column was heated at 5 ℃/min from 60 to 300 ℃, and held at 300 ℃ for 5 min. Data acquired were processed using Chemstation software. Compounds were identified by comparing mass spectra with NIST05a library data. Quantitative analysis was conducted using dimethyl phthalate as the external standard.

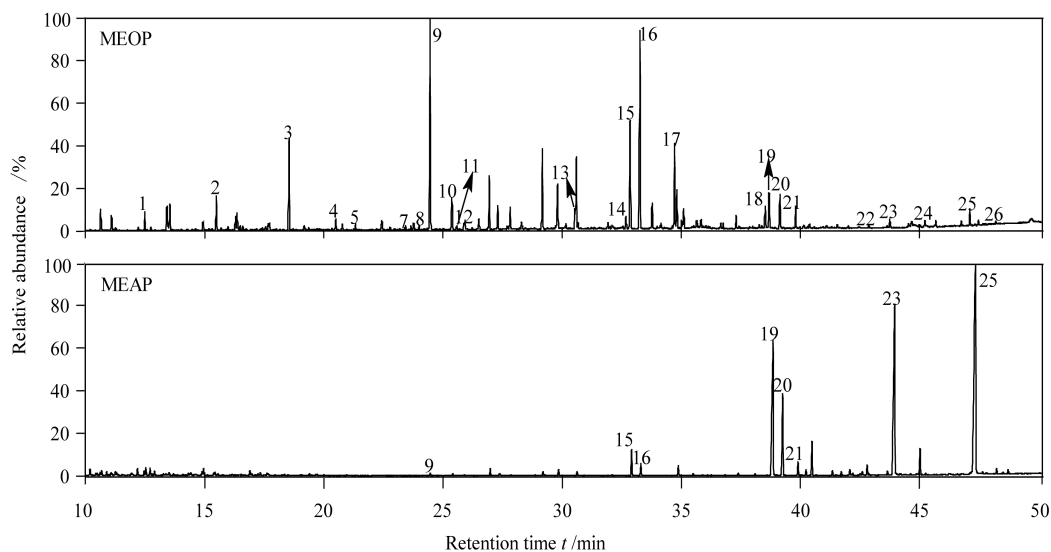## 2　Results and discussion

2.1　Structural features of J15A through

RICO

RICO was effective to convert arylalkanes, α, ω-diarylalkanes, and condensed aromatics to alkanoic acids（AAs）, alkanedioic acids（ADAs）, and benzene polycarboxylic acids（BPCAs）. According to GC/MS analysis, a total of 26 organic acids were detected in the products from RICO of J15A, as summarized in Figure 2 and Table 2. They could be classified into 10 AAs, 5 ADAs, 1 alkanetricarboxylic acid（ATCA）and 10 BPCAs. Their yields decreased as following：BPCAs ≫ AAs> ADAs>ATCA.

Figure 2　Total ion chromatograms of MEOP and MEAP from RICO of J15A

Table 2　Yields（mmol·mol^{-1}, daf）of different types of organic acids from RICO of J15A

Peak	Parent compounds	Yield	Peak	Parent compounds	Yield
	AAs			ADAs	
4	decanoic acid	0.04	8	octanedioic acid	0.02
7	undecanoic acid	0.03		ATCA	
12	dodecanoic acid	0.03	6	propane-1,2,3-tricarboxylic acid	0.01
13	tetradecanoic acid	0.05		BPCAs	
14	pentadecanoic acid	0.10	9	phthalic acid	1.80
17	palmitic acid	0.60	10	isophthalic acid	0.02
18	stearic acid	0.17	11	terephthalic acid	0.01
22	icosanoic acid	0.02	15	hemimellitic acid	2.22
24	docosanoic acid	0.05	16	trimellitic acid	2.01
26	tetracosanoic acid	0.02	19	benzene-1,2,3,4-tetracarboxylic acid	12.37
	ADAs		20	pyromellitic acid	4.99
1	succinic acid	0.18	21	benzene-1,2,3,5-tetracarboxylic acid	0.81
2	glutaric acid	0.19	23	benzene pentacarboxylic acid	15.44
3	adipic acid	0.56	25	benzene hexacarboxylic acid	22.21
5	heptanedioic acid	0.03			

The range of carbon number of the detected AAs was more than C_9, suggesting that J15A contained a certain amount of long-chain alkyl groups. The

detected ADAs ranged from succinic acid（C_6, peak 1）to octanedioic acid（C_{10}, peak 8）. The most ADAs were succinic, glutaric and adipic acids（peaks

1, 2 and 3). The three ADAs might result from their corresponding α, ω-diarylalkanes. Alternatively, they were generated from 9, 10-dihydrophenanthrene (or acenaphthene), indane and tetralin, respectively[12,28]. The detected minimal amount of ATCA (peak 6) indicated that only a small amount of 1, 2, 3-triarylpropane existed in J15A.

The dominant BPCAs consisted of 3 benzene dicarboxylic acids (peaks 9, 10 and 11), 2 benzene tricarboxylic acids (peaks 15 and 16), 3 benzene tetracarboxylic acids (peaks 19, 20 and 21), a benzene pentacarboxylic acid (peak 23) and a benzene hexacarboxylic acid (peak 25). BPCAs were produced by selective oxidation of condensed aromatic structures. As listed in Table 2, the richest benzene hexacarboxylic acid implied that J15A was abundant in highly condensed aromatic structure. Although the aromatic system was destroyed, the detailed original structural feature was still deduced based on the BPCA types. The original condensed aromatic structure could be classified into linable type (*cata*-condensed type), biphenyl type and most compact aromatic structures (*peri*-condensed type)[18].

The isophthalic acid, terephthalic acid, trimellitic acid and benzene-1,2,3,5-tetracarboxylic acid are resulted from the oxidation of condensed aromatic structures with biphenyl types. As Table 2 shows, the yields of these BPCAs were quite low, indicating only a minor amount of biphenyl condensed aromatic structures existed in J15A.
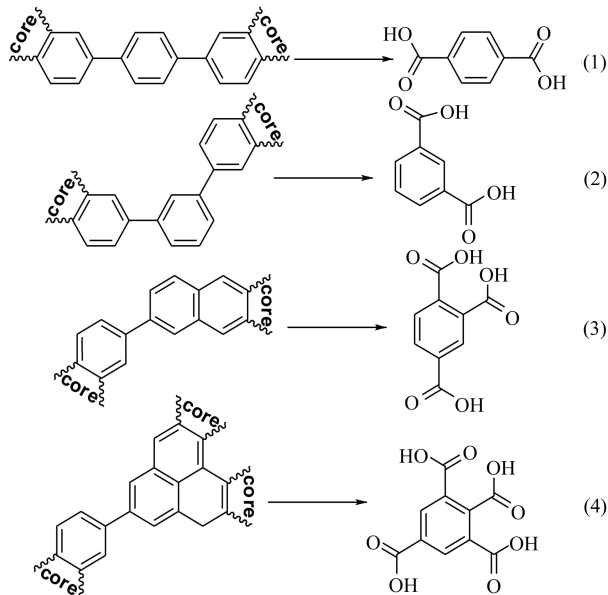
The phthalic acid and pyromellitic acid with high yields were generated from degradation of condensed aromatic structures with *cata*-condensed type, which suggested *cata*-condensed aromatic structures accounted for quite a certain percentages in J15A.

The hemimellitic acid, benzene-1, 2, 3, 4-tetracarboxylic acid, benzene pentacarboxylic acid and benzene hexacarboxylic acid were produced from *peri*-condensed aromatic structure. Their yields were significantly more than other ones. It indicated that a noticeable amount of aromatic species presented as *peri*-condensed type.
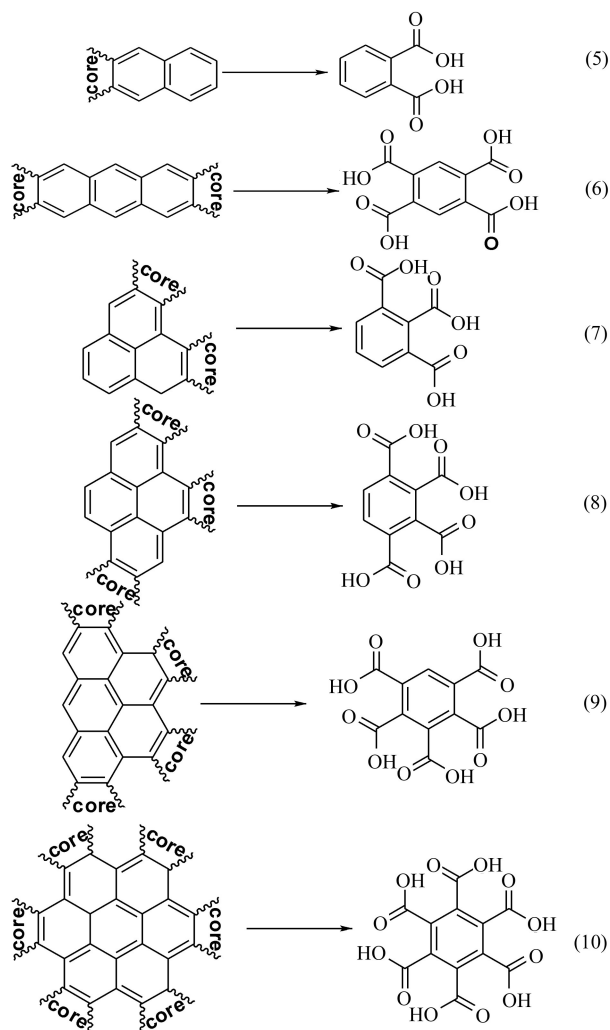
2.2 NaOCl oxidation of PTJ15A

According to our previous study[29], the yields of BPCAs were 20.68 mg/g in NaOCl oxidation of J15A, but they increased up to 42.28 mg/g in NaOCl oxidation of PTJ15A. The result proved that the pretreatment with H_2O_2 enhanced J15A oxidation with NaOCl. In spite of this, the yields of FC_1 remained high. The improved method was still necessary.

Mae et al[22,24] supposed that the pretreatment with H_2O_2 introduced -COOH and -OH into coals, and hence facilitated coal extraction and flash pyrolysis. We also found that the absorbances of bands corresponding to -COOH and -OH were weaker in FT-IR spectrum of Shengli lignite than in the one of Shengli lignite pretreated with H_2O_2. As exhibited

in Figure 3, the band around 3 408 cm^{-1} attributed to -OH was remarkable in FT-IR spectrum of PTJ15A, whereas the band was hardly detectable in that of J15A. It proved that -OH was introduced to J15A after pretreatment with H_2O_2. The introduced -OH improved the oxidation reactivity and the hydrophilicity of J15A. And the increased hydrophilicity also contributed to interaction between J15A particles and NaOCl aqueous solution, and thereby promoted NaOCl oxidation of J15A. The band around 1 618 cm^{-1} corresponded to vibration of the condensed aromatic ring. Its absorbance was significantly stronger in FT-IR spectrum of PTJ15A than that of J15A. It suggested that, compared with J15A, more organic matters were exposed on the surface of PTJ15A. This was possibly attributed to that H_2O_2 pretreatment removed minerals on the surface. In addition, the absorbance of the bands corresponding to vibrations of condensed aromatic ring and -OH were similar in FTIR spectra of PTJ15A and FC$_1$. Taking consideration of the high yields of FC$_1$, FC$_1$ was supposed to be subject to next NaOCl

oxidation, which would produce BPCAs in high yield and selectivity. In other words, the sequential NaOCl oxidation of J15A by pretreatment with H_2O_2 should be adopted to convert most organic matters to soluble organic molecules.

BPCAs were main products of oxidation of J15A and were paid much attention in the investigation. As shown in Figure 4 and Table 3, a total of 10 BPCAs were detected in ES$_2$ and IES$_2$.
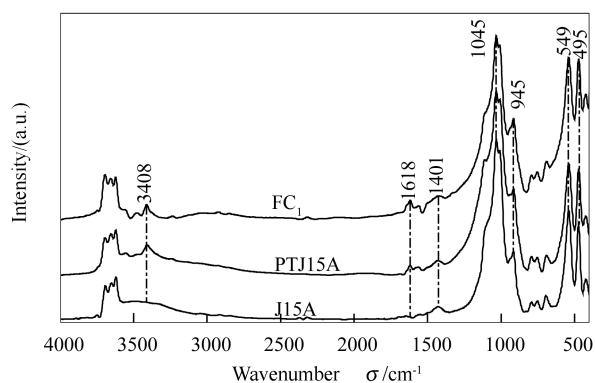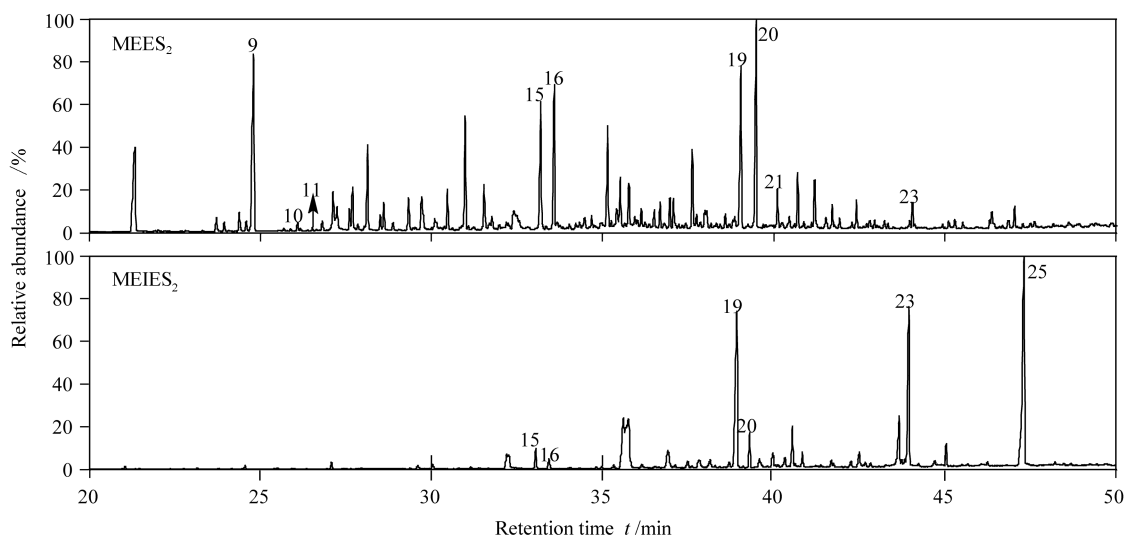

Figure 3　FT-IR spectra of J15A, PTJ15A and FC$_1$

Figure 4　Total ion chromatograms of MEE$_2$ and MEIEF from NaOCl oxidation of PTJ15A

Table 3　Yields（mg·g^{-1}, daf）of BPCAs in ES$_2$ and IES$_2$ from NaOCl oxidation of PTJ15A

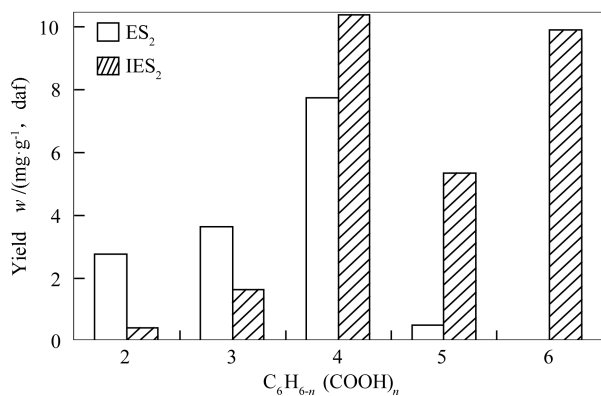| Peak	Parent Compounds	ES$_2$	IES$_2$
9	phthalic acid	1.74	0.08
10	isophthalic acid	<0.01	
11	terephthalic acid	<0.01	
15	hemimellitic acid	1.83	0.53
16	trimellitic acid	0.76	0.33
19	benzene-1,2,3,4-tetracarboxylic acid	3.63	6.87
20	pyromellitic acid	2.22	0.86
21	benzene-1,2,3,5-tetracarboxylic acid	0.38	
23	benzene pentacarboxylic acid	0.40	5.58
25	benzene hexacarboxylic acid		10.19

Figure 5   Distributions of BPCA in ES$_2$
and IES$_2$ from NaOCl oxidation of PTJ15A

The yields of different BPCAs in ES$_2$ decreased in the following order as exhibited in Figure 5: benzene tetracarboxylic acids > benzene tricarboxylic acids > benzene dicarboxylic acids > benzene pentacarboxylic acid. No benzene hexacarboxylic acid was detected. The yield of benzene pentacarboxylic acid was the lowest, while the yield of tetramethyl benzene-1, 2, 3, 4-tetracarboxylate (peak 19) was the most. The result suggested that the solvent extraction could enrich BPCAs with less than 5 -COOH. The yields of different BPCAs in IES$_2$ decreased as follows: benzene tetracarboxylic acids > benzene hexacarboxylic acid > benzene pentacarboxylic acid > benzene tricarboxylic acids > benzene dicarboxylic acids.

The yields of benzene tricarboxylic and benzene dicarboxylic acids were quite small. The yield of benzene hexacarboxylic acid (peak 25) was the highest as 10.19 mg/g with a single BPCA. It was feasible to obtain this compound in high yields.

3   Conclusions

The organic structure of J15A was characterized by RICO. It could be concluded that J15A is rich in condensed aromatic structures. Specifically, the *peri*-condensed aromatic structure is the dominant type. The *cata*-condensed one accounts for a certain amount. A minor part of condensed aromatic structures exist as biphenyl type in J15A. The pretreatment with H$_2$O$_2$ improved J15A oxidation with NaOCl leading to the increase of BPCAs yields. The tetramethyl benzene-1, 2, 3, 4-tetracarboxylate was the most in ES$_2$ and benzene hexacarboxylic acid dominated in IES$_2$. It was feasible to enrich the two BPCAs in high yield and selectivity. The NaOCl oxidation of J15A promoted by pretreatment with H$_2$O$_2$ was proved to be an effective method to get BPCAs.

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晋城 15 号无烟煤的温和氧化

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摘　要：用钌离子催化氧化法探究了晋城 15 号无烟煤（J15A）的结构特征，结果表明，J15A 富含紧密的芳香性结构。在温和条件下，先用 H_2O_2 对 J15A 进行预处理，然后用 NaOCl 对其氧化从而生成苯多酸。结果表明，H_2O_2 预处理可提高 NaOCl 氧化 J15A 生成苯多酸的产率，而且该方法可以高收率、高选择性地获取苯多酸化学品。

关键词：无烟煤；苯多酸；温和氧化

中图分类号：TQ531　　**文献标识码**：A