Enrichment Characteristics of Macerals during Triboelectrostatic Separation in the View of Surface Microstructure, Pore distribution, and Typical Electrical Parameters

Xin He, Hao Sun, Mengya Ma, Xinxi Zhang, and Wenfeng Wang

ABSTRACT: Vitrinite and inertinite, respectively, are the reactive and inert macerals for coal liquefaction, which could be effectively enriched in triboelectrostatic separation specialized in particle processing. Inertinite has a higher specific surface area and more pores than vitrinite and a more balanced mesopores distribution, while the mesopores in vitrinite are mainly focused in the 4 nm × 7 nm range. As for electrical properties, inertinite has a higher relative dielectric constant than vitrinite in all granularities, while its resistivity is only higher than vitrinite in the <74 μm fraction, which means inertinite and vitrinite tend to have negative and positive charges, respectively, in their mutual friction, but inertinite (<74 μm) has a stronger ability to maintain surface charge. During triboelectrostatic separation, the 105 μm × 150 μm fraction of clean coal has the highest vitrinite content, whereas inertinite tends to concentrate at tailings <74 μm under the co-effect of separation granularity limit and electrical characteristics of macerals; this conclusion has a certain guiding significance to maceral separation.

1. INTRODUCTION

Coal will remain a primary energy source in China for a long period because it is the richest domestic energy reserve. However, the increasingly serious environmental pollution and growing demand for petroleum resources are pressing the coal industry to turn to coal-to-chemicals projects, especially coal liquefaction technologies. The first coal direct liquefaction demonstration plant developed by the Shenhua Group completed its successful trial run at the end of 2008 and has operated steadily up to now. Low-rank coal characterized by a high reactivity and low ash content makes it a suitable feed for coal liquefaction. Many studies have pointed out that the maceral composition of the feed has a great impact on the oil yield rate and conversion rate of the liquefaction process. It is widely believed that vitrinite and liptinite are reactive macerals, leading to a high oil yield and reaction efficiency, while inertinite is considered as an inert maceral, reducing the conversion rate. Thus, the separation and enrichment of macerals in low-rank coal is the key point in developing and popularizing coal direct liquefaction technology.

Sink-and-float method and density gradient centrifugation (DGC) are the early approaches to separating coal macerals. They are both based on the density difference between macerals and centrifuging is taken as the main tool in DGC. DGC was first conducted and improved by Dyrkacz, who found that higher purity of macerals could be achieved in modified processes by avoiding mineral interference. Flotation also has been applied to extracting macerals. The contact angle of liptinite, vitrinite, and inertinite was measured by Arnold and Aplan as 90 to 130, 60 to 70, and 25 to 40°, respectively. Shu et al. and Barraza et al. performed maceral-enrichment experiments by adopting a flotation column, and the vitrinite content in the overflow (clean coal) was increased to some extent after yield reduction and other parameter controls. Effect analyses of size distribution of macerals on flotation were studied by Jorjani et al., and the results reported that vitrinite was mainly concentrated in two size ranges as 40 μm × 75 μm and <25 μm. Reflux classification as a recent hydraulic technique was employed to separate macerals by Xie and Tran with different emphases. The former was underlining effect improvement and 15% increment in vitrinite enrichment, while the latter gave a detailed introduction on principles of reflux classification. Triboelectrostatic separation, as an effective beneficiation method on fine particles, was also proposed for concentrating macerals for its advantages in...
conserving water, avoiding chemical contaminations, sparing complicated filtration procedure, and so forth. Zhou et al. carried out electrical separation on macerals after surface treatment with aromatic agents, which stimulated the recovery rate of vitrinite. Hower et al. found that the product collected at the negative electrode contains more vitrinite, while inertinite tended to gather at the opposite electrode in the triboelectrostatic separation of bituminous coal.

Our group has conducted a series of work on triboelectrostatic separation of macerals (vitrinite and inertinite) in low-rank coal used as liquefaction feed, including the relationship between infrared analyses and enrichment effect of macerals, charging mechanism study based on their electrical property differences, and tribocharging behaviors of macerals with typical minerals and various other materials. Previous results showed that vitrinite and inertinite could be separated and enriched at negative and positive plates, respectively, by triboelectrostatic separation based on differences of macerals in various aspects. However, there are still some issues that have not been discussed in detail such as the relevance between electrical characteristics and separation results of macerals. Besides, only a few investigations have been made in the related area; for instance, the effect of dielectric properties on coal char was studied by Liu et al. and Li et al. Filipé et al. have pointed that electrical resistivity and porosity of black shales are positively correlated. To systematically investigate the relationships among pore structure, electrical characteristics, and separating behaviors of macerals in coal, the objectives of this research herein has been to study: (a) surface microstructure characterization and pore distribution of macerals; (b) electrical parameters changing patterns of macerals under different environmental conditions and granularity; and (c) enrichment characteristics of macerals in low-rank coal during triboelectrostatic separation based on the above studies.

2. MATERIALS AND METHODS

2.1. Sample Preparation and Characterization. As the only designated feed in the Shenhua liquefaction project, a Jurassic-age subbituminous coal was sampled from the Dalita Coal Mine and used in this study. According to the test requirement of this study, high-purity fractions of vitrinite and inertinite were extracted from raw coal. Liptinite was excluded in this research for its extremely low content (<3%) and negligible impact on coal liquefaction. The extraction of inertinite and vitrinite was a full physical process to avoid any possible contaminations caused by contacting chemicals: first, preliminary concentrates of vitrinite and inertinite was gained by manually picking vitrain and fusain in raw coal, respectively. Then the preliminary concentrates of vitrinite and inertinite could be enriched by triboelectrostatic separation based on differences of macerals in various aspects. However, there are still some issues that have not been discussed in detail such as the relevance between electrical characteristics and separation results of macerals. Besides, only a few investigations have been made in the related area; for instance, the effect of dielectric properties on coal char was studied by Liu et al. and Li et al. Filipé et al. have pointed that electrical resistivity and porosity of black shales are positively correlated. To systematically investigate the relationships among pore structure, electrical characteristics, and separating behaviors of macerals in coal, the objectives of this research herein has been to study: (a) surface microstructure characterization and pore distribution of macerals; (b) electrical parameters changing patterns of macerals under different environmental conditions and granularity; and (c) enrichment characteristics of macerals in low-rank coal during triboelectrostatic separation based on the above studies.

2.2. Electrical Parameter Measurement of Macerals. Relative dielectric constant (RDC) \( \varepsilon_r \) and resistivity \( \rho \), which represent the polarization ability and resistance characteristics, respectively, are the two most significant electrical parameters of materials. Measuring schematics of RDC and resistivity on macerals are separately shown in Figure 1. In this research, the test sample was ground into a powder and positioned in the sample pool constituted by two parallel plates for RDC and resistivity measurements. In the case of RDC, the two plates were directly connected to the testing electrodes of a capacitance meter (TH2618B, Tonghui, China) and an additional grounding electrode was also needed. RDC \( \varepsilon_r \) of the samples could be determined by the eq 1 according to the capacitance comparison method.

\[
\varepsilon_r = \frac{C}{C_s}
\]

where \( C \) (F) and \( C_s \) (F) represent the capacitance of empty capacitor and capacitor filled with the tested sample, respectively.

In terms of resistivity test, as long as a high-voltage insulation measuring apparatus (AT682SE, Applent Instruments, China) accessed two parallel plates, the \( \rho \) of the sample could be calculated according to eq 2.

\[
\rho = \frac{R S}{d}
\]

where \( R \) is the measuring resistance value of the sample, and \( S \) (m²) and \( d \) (m) represent the effective area and distance of two plates, respectively.

The impact of environment and particle granularity changes on two electrical parameters of both macerals were also monitored; the measurement set points of temperature, relative humidity (RH), and granularity are independently arranged as follows: 10, 20, 30, 40, and 50 °C; 25, 40, 55, 70, and 80%; and <74 \( \mu \)m, >74 \( \mu \)m \( \times \) 105 \( \mu \)m, 105 \( \mu \)m \( \times \) 150 \( \mu \)m, 150 \( \mu \)m \( \times \) 250 \( \mu \)m, and >250 \( \mu \)m, respectively. During the tests, three procedures were taken for minimizing error: (1) a shield electrode and grounding device were applied to RDC and resistivity measurements separately; (2) the measuring system was placed at a constant temperature and humidity for maintaining continuously stable surrounding; and (3) each value was taken as an average of three repeated tests.

2.3. Triboelectrostatic Separation of Macerals. Grind-
including different components will enter the system by a feeder and move with the airflow created by the centrifugal fan. There are two ways for the sample to get charged in the tribo-charger; collisions between different components and frictions between components themselves and the inner wall of the tribo-charger. Then, components with different charges (positive or negative) will be separated and gathered at the opposite plates to form clean coal and tailings, respectively. Ash content and maceral identification were applied to all size fractions of collected clean coal and tailings.

3. RESULTS AND DISCUSSION

3.1. Characterization Analyses of Macerals. Results of maceral identification, ultimate analysis, and proximate analysis of raw coal (RC), vitrinite (MV), and inertinite (MI) are shown in Tables 1 and 2. Maceral identification shows that purities of MV and MI are both over 90% without any detectable minerals, and the MV is mainly composed of colliotelinite and collodetrinite, while semifusinite is the only major maceral in MI. On the basis of the ultimate analysis, the H/C ratio of MV and MI could be calculated as 0.78 and 0.66, respectively, which aligns with the stronger liquefaction reactivity of vitrinite for its high hydrogen content benefits a lot in the liquefaction process. This also well conforms with the higher vitrinite reflectance of vitrinite for it represents better maturity of organic matter. The above analyses also conformed with the higher volatile and lower ash content of MV from the proximate analysis.

SEM–EDS characterizations of MV and MI are shown in Figure 3. Both macerals were observed under magnification of $2 \times 10^2$, $2 \times 10^3$, and $5 \times 10^3$. Minerals marked in yellow as area 1 and area 2 could be found in vitrinite (Figure 3a) and inertinite (Figure 3b); they have been identified as clays (Figure 3c) and pyrite (Figure 3d), separately. In addition, the tubular structures circled with blue in inertinite (Figure 3b) are the deformed cell cavities of fusinite, which could also be observed clearly on a single particle (Figure 3f) and formed the abundant pore structures of inertinite. Surface pores of two macerals could be clearly observed at magnifications $2 \times 10^3$ and $5 \times 10^3$, and the subtle visible folds circled with green on vitrinite (Figure 3e) should be caused by the brittle fracturing of the vitrinite. In all images, it is obvious that the flatness of surface and evenness on vitrinite is much better than that on inertinite.

Pore distribution including cumulative pore volume (CPV) and PV of two macerals are shown in Figure 4. SSA of vitrinite and inertinite are 5.348 and 12.100 m$^2$/g, respectively. Obviously, inertinite has more pores than vitrinite based on the data of SSA and CPV, which is consistent with SEM images. The PV shows that mesopores is the overwhelming majority in both macerals, while micropore and macropore barely presented, but it should be noted that mesopores in vitrinite are mainly concentrated in the 4 nm $\times$ 7 nm range, whereas inertinite has a much more balanced distribution in the overall scope with a range of 5 nm $\times$ 8 nm. The above pore analyses of macerals in this designated liquefaction low-rank coal deviate from previous literature which indicated that micropores and macropores are the majority of vitrinite and inertinite, respectively. However, it is certain that pores in vitrinite are smaller than those in inertinite.

3.2. Electrical Parameter Analysis of Macerals. 3.2.1. Relative Dielectric Constant Analyses of Macerals. RDC represents the dielectric and polarization properties of materials and could also indicate the electrostatic storage capacity of materials in the electric field. Usually, when triboelectrification happens between two materials, the sample with higher RDC is inclined to get a negative charge and the other one tends to be positively charged. The RDC of vitrinite and inertinite under different granularity, temperature, and RH.

Table 1. Maceral Characterization of Samples

| Characterization parameters | RC | MV | MI |
|-----------------------------|----|----|----|
| Ultimate analysis (wt %)    |    |    |    |
| O, oxygen; C, carbon; H, hydrogen; N, nitrogen; St, total sulfur; M, moisture; A, ash yield; V, volatile matter; FC, fixed carbon; ad, air-dry basis; d, dry basis; daf, dry and ash-free basis. |
| proximate analysis (wt %)   |    |    |    |
| O, oxygen; C, carbon; H, hydrogen; N, nitrogen; St, total sulfur; M, moisture; A, ash yield; V, volatile matter; FC, fixed carbon; ad, air-dry basis; d, dry basis; daf, dry and ash-free basis. |
were measured and are displayed in Figures S1 and S2, respectively. It was observed clearly that vitrinite and inertinite samples with different granularity all share the same change pattern under temperature and RH variations: the RDC of macerals decrease with rising temperature and increase with RH. As concluded in our previous work,25 water molecules
with high polarity have a strong impact on the electrical properties of macerals. The increasing RH would result in an increase in the moisture content of the sample and an increase in RDC, whereas rising temperature has the opposite effect. From Figures S1 and S2, the RDCs of the two macerals all decline as the granularity decreases, but the drop in inertinite was sharper than that in vitrinite. This illustrates that granularity has an obvious impact on the RDC of macerals, and inertinite is more sensitive to particle variation. The sensitivity of inertinite must be caused by its poorer uniformity of surface roughness compared with vitrinite as the SEM images presented; the dense and homogeneous structure of vitrinite makes it influenced less by size change after crushing.

Although the RDC of inertinite is higher than that of vitrinite in all granularities, their RDC difference values are also different in each granularity. These differentials (inertinite minus vitrinite) are as follows: 1.27 (>250 \(\mu m\)), 1.26 (150 \(\mu m\) × 250 \(\mu m\)), 0.81 (105 \(\mu m\) × 150 \(\mu m\)), 0.74 (74 \(\mu m\) × 105 \(\mu m\)), and 0.42 (<74 \(\mu m\)). Generally, greater the RDC difference between two macerals, the larger chance that they

Figure 4. Pore distribution of macerals.

Figure 5. RDC and resistivity average change rate of macerals with RH and temperature under size distribution: (a) RDC average change rate with RH, (b) RDC average change rate with \(T\), (c) \(R\) average change rate with RH, and (d) \(R\) average change rate with \(T\). (RH = relative humidity, \(T\) = temperature, \(R\) = resistivity).
get opposite charge; thus, based on this analysis, the best separation effect should happen in samples with sizes >250 μm. For further investigation into the impact of surroundings on RDCs of macerals, the RDC average change rates of macerals with temperature and RH in all size distribution are shown in Figure 5a,b, respectively. It is evident that all change rates increased at smaller particle sizes, which is because more pores appeared in low granularity, leading to a stronger impact brought by moisture change. It seemed a little confusing that the RDC average change rates of vitrinite with temperature and RH are higher than those of inertinite in most granularities, considering inertinite has higher SSA and more pores. In view of this situation, the authors proposed a possible reason here: the increased moisture tends to gather on the smoother surface of vitrinite, while inertinite absorbs them in its pores, combined with the fact that RDC measurements were mainly affected by the surface change of samples, all of which resulted in the above phenomenon.

3.2.2. Resistivity Analyses of Macerals. Resistivity values of vitrinite and inertinite under different granularities, temperature, and RH are shown in Figures S3 and S4, respectively. Resistivity indicates the ability of materials to maintain surface charge; usually, higher resistivity means a stronger holding capacity. As demonstrated in these figures, the resistivity of both macerals in all granularities has the same change pattern: decreasing with the rising RH and temperature. The resistivity change pattern of macerals with RH could be explained as follows: the rising RH easily increased the moisture content in the sample, which will enhance conductivity and weaken the resistivity of the sample since water is a common conductor. As for the change pattern of macerals with temperature, there is a common rule that rising temperature leads to the lower

| condition | T (°C) | RH (%) | sorting voltage (kV) | transport volume (m³/h) | feeding rate (kg/h) | positive plate yield (%) | ash content (%) | negative plate yield (%) | ash content (%) |
|-----------|--------|--------|----------------------|-------------------------|---------------------|-------------------------|----------------|-------------------------|----------------|
| C1        | 22.5   | 41     | 40                   | 2800                    | 22.96               | 55.56                   | 10.04          | 44.44                   | 4.33           |
| C2        | 14.3   | 68     | 40                   | 2800                    | 22.96               | 58.18                   | 8.92           | 41.82                   | 5.53           |

Figure 6. Maceral composition of products in triboelectrostatic separation: (a) clean coal of C1, (b) tailings of C1, (c) clean coal of C2, and (d) tailings of C2.
resistivity of nonconductors (coal in this case) since resistivity is an inherent property of materials and is only affected by a few factors. In addition, inertinite still has a wider variation range than vitrinite in resistivity changes, which is caused by the same reason described in the RDC case (Section 3.2.1): vitrinite has a greater degree of dense and even surface structure, thus it is less susceptible to the grain-size variation.

The resistivity differences (inertinite minus vitrinite) of macerals in all granularities are presented as follows: \(-2.80 \, (\text{>250 \(\mu\)m}), -2.20 \, (150 \, \text{\(\mu\)m} \times 250 \, \text{\(\mu\)m}), -0.75 \, (105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m}), -0.06 \, (74 \, \text{\(\mu\)m} \times 105 \, \text{\(\mu\)m}), \) and 0.11 \, (<74 \, \text{\(\mu\)m}). Based on the above data, inertinite should have the better enrichment result in the <74 \, \text{\(\mu\)m} size fraction, while vitrinite would behave better in all other granularities, especially in the >250 \, \text{\(\mu\)m} fraction. The resistivity average change rates of the two macerals with temperature and RH in all size distributions are also studied and demonstrated in Figure 5c, d, respectively. Rates of both macerals increased with decreasing granularity, and it is easy to understand that smaller particle size means more surface area affected by the environment which is the same as the RDC case. Another point needing attention is that vitrinite always has a higher resistivity average change rate with RH than inertinite, while they have almost even change rates with temperature. It is easy to understand the former phenomenon in Figure 5c based on the proposed speculation in RDC analyses (Section 3.2.1): resistivity average change rate of vitrinite with RH is more obvious as increased moisture tends to gather on its smoother surface. The latter could be interpreted as resistivity being the natural characteristic of the two macerals and temperature is the only common impact factor of resistivity, and these change rates with temperature are more stable in a relatively narrow temperature range in this study.

3.3. Triboelectrostatic Separation of Macerals. Triboelectrostatic separation experiments have been applied to vitrinite and inertinite in low-rank coal. In our previous study, the increasing temperature (\(T\)) and decreasing RH have been confirmed to have a positive effect on separation; so the impact of the environment will not be scrutinized in this work. Condition C1 was set as high \(T\) and low RH, while C2 was set with low \(T\) and high RH in Table 3, and system factors in both conditions have the same values. The yield and ash content of products collected from the two opposite electrodes are also exhibited. Clean coal and tailings are distributed in the negative plate and the positive plate, respectively. Higher yield and lower ash content of clean coal are obtained in condition C1, which agrees with the previous study.

The maceral composition of all products collected in C1 and C2 are shown in Figure 6. Compared with raw coal (ash content 7.55%, vitrinite 57.55%, and inertinite 42.45% on a mineral-free basis), it could be found in both conditions that clean coal has higher vitrinite content, lower ash content, and inertinite content, whereas tailings have a reverse situation. Hence, vitrinite is enriched in clean coal collected from the negative plate, while inertinite is mainly gathered in tailings at the positive plate, which accords with RDC analyses of macerals that inertinite has a higher RDC and tends to get negative charge and vitrinite with a smaller RDC is easily positively charged in its triboelectrification. Maceral composition of clean coal and tailings under different size distributions were also studied in this graph. The granularities are set as >150 \, \text{\(\mu\)m}, 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m}, 74 \, \text{\(\mu\)m} \times 105 \, \text{\(\mu\)m}, \) and <74 \, \text{\(\mu\)m}; the >250 \, \text{\(\mu\)m} and 250 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m} \text{\(\mu\)m} fractions were removed because triboelectrostatic separation is only effective with fine particles. From Figure 6, it is easy to find that the highest vitrinite content always appeared in the sample at 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m}, while most inertinite enriched in the <74 \, \text{\(\mu\)m} sample. According to the electrical parameters research, the largest RDC difference of the two macerals occurs in the >150 \, \text{\(\mu\)m} fraction, and vitrinite also has a much higher resistivity than inertinite in the same range. It can be inferred that the best vitrinite separation effect should happen at the >150 \, \text{\(\mu\)m} fraction in clean coal. However, the actual best effect belongs to the 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m} fraction, although the separating results of the >150 \, \text{\(\mu\)m} fraction was also good (it should be noted that the >150 \, \text{\(\mu\)m} fraction makes up less than 3% of the total). This situation should be attributed to the effective particle size limit of the triboelectrostatic separation method. Previous literature has recommended <74 \, \text{\(\mu\)m as the effective size for particles applied in triboelectrostatic separation because clean coal has lower ash content under this condition. In this study, the 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m} fraction belongs to the effective size of this method, and vitrinite has a greater charge maintenance ability than inertinite in this size range, which all lead to the best enrichment of vitrinite. Observed from the aspect of inertinite, it has higher resistivity at <74 \, \text{\(\mu\)m fraction, but the largest RDC difference between the two macerals was presented in the >150 \, \text{\(\mu\)m} fraction, and the real best gathering effect of inertinite happened at the <74 \, \text{\(\mu\)m fraction in the tailings of C1 and C2. Based on the above analysis, it could be deduced that the RDC difference between vitrinite and inertinite mainly determines the gathering plate of macerals in separation, whereas resistivity relatively has the stronger impact on their enrichment effect. In the effective sorting size range (<150 \, \text{\(\mu\)m}) of triboelectrostatic separation, the best gathering effect of vitrinite happens in the 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m} fraction where vitrinite has the highest resistivity difference with inertinite, and the inertinite mainly enriches at <74 \, \text{\(\mu\)m fraction where it has the higher resistivity. Combining the above analyses, during triboelectrostatic separation, particle size of the sample should be controlled at 105 \, \text{\(\mu\)m} \times 150 \, \text{\(\mu\)m if vitrinite is the target enrichment maceral, and <74 \, \text{\(\mu\)m is the better granularity choice if it aims at gathering inertinite.

4. SUMMARY
Surface microstructure, pore distribution, and electrical parameters (relative dielectric constant and resistivity) of two maceral groups (vitrinite and inertinite) with high purity extracted from low-rank coal were observed by SEM–EDS, surface area analyzer, and electrical parameters measurement systems in this paper. Triboelectrostatic separation experiments were also conducted with raw coal, and the enrichment characteristics of vitrinite and inertinite were analyzed combined with the above characterization. The main conclusions of this research are as follows:

1. Inertinite has more pores, larger SSA, and greater surface roughness than vitrinite. Mesopores carry out the major role of pore composition in both macerals, but most of the mesopores in vitrinite are mainly gathered in the 4 nm × 7 nm range, while inertinite has more balanced distribution in all ranges with relative concentration at 5 nm × 8 nm.

2. Relative dielectric constants of inertinite are constantly higher than that of vitrinite in all granularities, which indicates that inertinite tends to charge negatively, while
vitrinite is easily positively charged in its triboelectrostatic separation, and their largest RDC difference happens in the >250 μm fraction; Resistivity values of vitrinite are higher than those of inertinite in most size ranges but the <74 μm fraction, which means that inertinite with the smaller particle size has stronger ability in maintaining surface charge.

3. Vitrinite enriches in the clean coal on the negative plate, whereas inertinite gathers in tailings at the positive plate during triboelectrostatic separation. Meanwhile, the 105 μm × 150 μm fraction of clean coal has the highest vitrinite content, while the <74 μm fraction of tailings obtains the most inertinite, which indicates that the enrichment of vitrinite (active maceral in coal liquefaction) and effect of subsequent liquefaction could both be further improved based on size control.

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**ASSOCIATED CONTENT**

 Supporting Information The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c02791.

Relative dielectric constant measurement data of vitrinite and inertinite and resistivity measurement data of vitrinite and inertinite (PDF)

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Notes

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